United States Patent Office 2,973,018 Patented Feb. 28, 1961

2,973,018

CELLULOSE TRIACETATE PILE FABRIC AND METHOD OF MAKING SAME

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No Drawing. Filed Feb. 23, 1956, Ser. No. 567,079

Claims priority, application Great Britain Mar. 9, 1955

13 Claims. (Cl. 139—391)

This invention relates to pile fabrics and their manufacture.

The invention comprises pile fabrics in which the pile consists of or contains short lengths of twisted yarn made up at least in part of artificial fibres in which the twist has been set by increasing the degree of crystallinity of the material of the artificial fibres in the twisted yarn. The invention also comprises the manufacture of the novel pile fabrics, as by the method hereinafter described.

The artificial fibres in the pile may with advantage be made from cellulose triacetate (i.e. a cellulose acetate having an acetyl value above 50% and preferably above 60% reckoned as acetic acid) or from a synthetic high linear polymer, e.g. a polyamide such as polyhexamethylene adipamide (6,6-nylon), the product obtained by polymerising aminocaproic acid or caprolactam (6-nylon), or an aromatic polyester such as polyethylene terephthalate. However fibres of other synthetic polymers which can have their degree of crystallinity increased, for instance by a heat treatment or by steaming, may be used.

The term "degree of crystallinity," as used in this specification, denotes a property which shows itself in the sharpness of the X-ray diagram given by the fibre, the sharper the diagram the higher being the degree of crystallinity. Thus the degree of crystallinity of the material of a fibre is to be regarded as having been increased by a given treatment when the X-ray diagram given by the fibre is sharper after the treatment than before, without reference to whether the increased sharpness is due to an increase in the ratio between the proportion by weight of the material which is more or less crystalline to the proportion which for practical purposes is amorphous, or to an increase in the size of the crystallites (accompanied or not by an increase in the crystalline:amorphous weight ratio), or to an increased local concentration of the crystalline fraction (e.g. in the skin of the fibre) or to any two or all of these factors or to any other factors as yet unknown, since it is very difficult, if not impossible, to determine how far an increase in the sharpness of the X-ray diagram in any particular case is due to any one factor.

The degree of twist of the yarn in the pile may be as low as 1–2 turns per inch, or it may be much higher, e.g. up to 40–50 turns per inch. The twist may be inserted as a separate operation, or partly or wholly in the course of another operation, for example folding. The yarn from which the pile is made may be a singles yarn or a doubled or other folded yarn in which successive twists may be either in the same direction or in opposite directions. As a general rule the final twist, i.e. the twist in a singles yarn, or the twist last applied in a folded yarn, expressed as the number of turns per inch, should be less than

$$\frac{600}{\sqrt{d}}$$

where d is the denier of the yarn, but not so low as to give less than one complete turn in the pile length.

The increase in the degree of crystallinity of the material of the artificial fibres, one effect of which is to set the twist in the yarn, may be effected at any stage in the manufacture of the finished pile fabric. (It will be convenient to use the term "yarn" to include both the filamentary material to which the twist is actually applied, and also the same material after it has been cut into short lengths before, in the course of, or after its incorporation in the fabric to form the pile.) For example the twist may be set in a continuous filament or staple fibre yarn immediately after, and advantageously continuously with, its insertion, or at a later stage, for instance while the yarn is in hank or package form, or while it is travelling from one package to another. On the other hand the setting of the twist may be deferred until after the yarn has been incorporated in the pile fabric.

Since the twist is to be set in the yarn by increasing the degree of crystallinity of the material of the artificial fibres, this must, when the twist is inserted, naturally be below the maximum possible. This is the case with fibres of high linear polymers as normally obtained by melt spinning methods, and with cellulose triacetate fibres obtained by wet, dry, or melt spinning, or by the complete acetylation of partly acetylated or unacetylated cellulose fibres in which the cellulose molecules are not already highly orientated. Thus any of these methods can be used to produce triacetate fibres for use in accordance with the invention. Methods of making melt spun cellulose triacetate yarns which are suitable for employment in accordance with the invention are described in U.S. applications Ser. Nos. 243,994, filed August 28, 1951, and now abandoned, 292,772, filed June 10, 1952, now U.S. Patent No. 2,888,711, granted June 2, 1959, 304,441, filed August 14, 1952, and now abandoned, 338,834, filed February 25, 1953, now U.S. Patent No. 2,831,748, granted Apr. 22, 1958, and 423,743, filed April 16, 1954, and now abandoned; wet spun cellulose triacetate yarns may be made for example by the process of U.S. Patent No. 2,657,973.

Various methods are available for increasing the degree of crystallinity of the materials of the various artificial fibres and thus for setting the twist in yarns containing them. For example polyamide and polyester fibres may be heated in the absence or presence of steam as in the known processes for rendering articles made from such fibres dimensionally stable. Processes for increasing the degree of crystallinity of cellulose triacetate fibres (or "annealing" them) are described in U.S. applications Ser. Nos. 400,798, filed December 28, 1953, and now U.S. Patent No. 2,862,785, granted Dec. 2, 1958, 467,223, filed November 5, 1954, 509,327, filed May 18, 1955, and now abandoned.

For example a twisted cellulose triacetate yarn may be subjected to dry heat at a temperature of about 170°–225° or 230° C., e.g. by means of radiant heat or a liquid or gaseous heat transfer medium or by passing it over a plate or roller or other solid member heated to the desired temperature. The twist may be set in yarn which already forms the pile in a fabric by passing hot air through or over the fabric or by passing the fabric over a heated plate or roller, preferably in such a way that the pile, but not the backing, comes into contact with the hot surface. If a substance having some softening or swelling action on cellulose triacetate is brought into contact with the yarn the twist may be set in the yarn at a lower temperature. For example wet steam and hot water are effective at temperatures of 120°–160° C.; normally liquid aromatic hydrocarbons, and halo-

genated aliphatic hydrocarbons boiling at temperatures above 50° C. such as propyl bromide, are effective between about 60° and 90° C.; mixtures of acetone and water or of acetone and an aliphatic alcohol containing up to 4 carbon atoms in the molecule, in which the proportion of acetone is between 30 and 70% by weight, are effective at temperatures between about 15° and 40° C.; and methylene chloride and chloroform vapours are effective at temperatures between about 15° and 30° C. Ethylene glycol, glycerol, and other liquid di- or polyhydroxy compounds can also be used, and at temperatures between about 130° and 200° C. raise the degree of crystallinity very rapidly. Naturally the higher the temperature the shorter is the time needed to set the twist in the yarn, other things being equal. The time required for a given increase in the degree of crystallinity in the material of a continuous yarn can also be shortened by simultaneously stretching the yarn as described in U.S. application S.N. 509,327.

When the twist is set in the yarn by treatment with a liquid swelling agent for the material of the yarn, e.g. with aqueous acetone when the yarn consists of cellulose triacetate, the setting treatment may be combined with a simultaneous solvent dyeing operation by dissolving a suitable dyestuff in the aqueous acetone or other swelling agent. Suitable dyestuffs include acid wood dyes such as Croceine Scarlet 3BS (Colour Index No. 277), Solway Green G. S. (Schultz, Farbstofftabbelen (7th edition) No. 1201), Wool Fast Blue BL (Ibid No. 974), Amide Yellow E (Colour Index No. 11) and 1-amino-4-phenylaminoanthraquinone-2-sulphonic acid (blue); and disperse dyes of the type commonly used for dyeing cellulose acetate textiles, e.g. 1-amino-4-hydroxyanthraquinone (red) and 4-nitro-2'-chloro-4'-(di-beta-hydroxyethyl)-amino-azobenzene (scarlet).

The handle of the pile fabrics is to some extent affected by the method used to set the twist in the yarn. Thus when the twist is set in a cellulose triacetate yarn by means of dry heat a somewhat stiffer pile results than when the twist is set by steaming or by treatment with hot water, while the softest effects of all are usually obtained when the twist is set by means of an organic liquid or vapour which is a swelling agent for the cellulose triacetate. If in any particular case the pile is stiffer or harsher than is desired, it can be rendered less stiff by subsequent ironing, by passing the fabric between fluted rollers, or by brushing the pile.

When the twist is set in the yarn before it is incorporated in a fabric the yarn may with advantage, after the setting operation, be subjected to a partial untwisting or other opening operation, both to increase its voluminosity, and also, especially when the twist has been set by a dry heating operation, to soften the yarn to some extent. Such a partial untwisting should not be sufficient to impart to the yarn any important degree of curliness or crimp and may amount to between about 2 and 25% and especially about 5–15% of the twist, or of the final twist in a folded yarn. If it is desired to open the yarn to some extent without actually untwisting it, this may be done by running it under tension round a friction device, e.g. a smooth cylindrical bar, in such a way as to cause a fairly sharp change in the direction of travel of the yarn.

The invention is applicable not only to relatively short pile fabrics, such as carpets and velvets, but also to long pile fabrics, such as artificial furs. Yarns for use in forming the pile in carpets may for example be of high denier and low twist, e.g. of denier between 2,500 and 10,000 and total twist between about 2 and 7 or 8 turns per inch, or they may be of lower denier and higher twist, e.g. of denier between about 800 and 1,500 with a total twist for example of about 10–15 turns per inch (subject always to the limitation that the twist must be below

$$\frac{600}{\sqrt{d}}$$

as already set out), so that a finer or pin-point effect is produced. Yarns for use in making a dress velvet may be 2-, 3- or 4-fold, with for example a total denier of about 50–100 or 200 and a total twist of about 2–40 turns per inch, and especially about 10–40 turns per inch. It will of course be understood that all these figures are given by way of example only.

The invention is further illustrated by the following examples.

Example I

Melt-spun cellulose acetate yarns of filament deniers 10, 15 and 25 respectively were made by the methods described in U.S. applications S.Nos. 243,994 and 423,743, and were cut into staple lengths and spun by the woolen system into a yarn of denier 3,200 with a twist of 2.5 turns per inch; two ends of this yarn were then doubled, with a twist of 4 turns per inch in the opposite direction, to give a carpet yarn of denier 6,400. In each instance part of this yarn was used to form the pile of a Wilton-quality carpet, which was subsequently heated to 212° C. for 3 minutes by contact of the pile with a heated metal plate. Another part was heated to the same temperature and for the same time before being used to form the pile in a similar carpet. In both cases the pile was characterised by a high degree of springiness and resilience, and a somewhat harsh handle; the handle could however be softened by brushing the pile or, when the yarn was given the heat treatment before being formed into the pile, by passing it under tension through an angle of 160° round a smooth steel bar of diameter ¼ inch.

Example II

Of the yarns described in Example I, that made by doubling the 3,200 denier 15 filament denier yarn made by the method of U.S. application S.N. 243,994, was treated in hank form with a 60% by volume aqueous acetone at 23° C. for half an hour, after which it was washed with water and dried. The resulting yarn was springy and resilient but at the same time relatively soft in handle, and when used in the production of Wilton-quality carpetting, gave a pile having these properties.

When the aqueous acetone contained in solution 10 grams per litre of Alcovar Red BEG, the yarn was dyed to a brilliant depth of a wine shade having good fastness to light and acid fading. Other colours could be obtained by using other dyestuffs, for instance a good scarlet with 4-nitro-2'-chloro-4'-(di-beta-hydroxyethyl)-amino-azobenzene.

Example III

Another yarn which gave a resilient pile was obtained from a dry-spun continuous filament triacetate yarn of total denier 4,000 by giving it a twist of 8 turns per inch and then heating it at constant length to 230° C. for 30 seconds. In this case also the handle of the pile could be rendered softer either by brushing the pile or by opening the yarn, before using it to form the pile, by passing it under tension round a steel bar as described in Example I, or by giving it a reverse twist of about 0.75 turn per inch.

Example IV

A number of polyaminocaproic acid (6-nylon) yarns of denier 30, filament denier 6, and twist 7 turns per inch were folded into a 9,000 denier yarn with a twist of 3 turns per inch in the opposite direction. This yarn was then heated to 160°–170° C. for 5 minutes, and used to form the pile in Wilton-quality carpetting.

Having described our invention, what we desire to secure by Letters Patent is:

1. Pile fabrics in which the pile contains short lengths of twisted yarn made up at least in part of heat set cellulose triacetate fibres having an acetyl value above 60% reckoned as acetic acid, the twist ranging between a minimum of one turn per pile length to a maximum of

$$\frac{600}{\sqrt{d}}$$

turns per inch where d is the denier of the yarn.

2. Process for the manufacture of pile fabrics, which comprises the steps of forming into a pile a yarn comprising fibres selected from the group consisting of dry-spun, wet-spun and melt-spun cellulose triacetate artificial fibres, the said yarn having a twist of less than

$$\frac{600}{\sqrt{d}}$$

turns per inch where d is the denier of the yarn, there being at least one complete turn of twist in the pile length, and setting the twist in the yarn at any stage in the process by giving the yarn a treatment selected from the class consising of dry heat at 170–230° C., wet steam at 120–160° C. and water at 120–160° C. for a time sufficient to convert the cellulose triacetate into a form which gives a sharp X-ray diagram.

3. Process according to claim 2, wherein said treatment comprises heating dry at 170–230° C.

4. Process according to claim 2, wherein said treatment comprises heating in wet steam at 120–160° C.

5. Process according to claim 2, wherein said treatment comprises heating in water at 120–160° C.

6. Process according to claim 2, wherein the twist is set in the yarn before the formation of the pile.

7. Process according to claim 2, wherein the twist is set in the yarn after the formation of the pile.

8. Process according to claim 2, wherein after the twist has been set the twisted yarn is subjected to an opening operation in which it loses 2–25% of its final twist.

9. Process for the manufacture of pile fabrics, which comprises the steps of forming into a pile a yarn comprising fibres selected from the group consisting of dry-spun, wet-spun, and melt-spun cellulose triacetate artificial fibres, the said yarn having a twist of less than

$$\frac{600}{\sqrt{d}}$$

turns per inch where d is the denier of the yarn, there being at least one complete turn of twist in the pile length, and setting the twist in the yarn by subjecting it to the action of a mixture of 30–70% by weight of acetone with 70–30% by weight of a liquid of formula $C_nH_{2n+1}OH$, where n is 0–4, at a temperature between 15° and 40° C. for a time sufficient to convert the cellulose triacetate into a form which gives a sharp X-ray diagram.

10. Process according to claim 9, wherein the liquid mixture contains in solution a dye for the cellulose triacetate.

11. Process for the manufacture of pile fabrics, which comprises the steps of forming into a pile cellulose triacetate yarn in which the cellulose triacetate is in a metastable form in which the proportion in the crystalline state is too small to give a sharp X-ray diffraction diagram, the said yarn having a twist of less than

$$\frac{600}{\sqrt{d}}$$

turns per inch, where d is the denier of the yarn, and setting the twist in the yarn at any stage in the process by heating it to a temperature at which the cellulose triacetate changes to the stable form in which the proportion thereof which is crystalline is sufficient to give a sharp X-ray diffraction diagram.

12. Process according to claim 11, wherein the twist is set by heating the yarn in the presence of steam.

13. Process according to claim 11, wherein the twist is set by heating the yarn in the absence of steam.

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