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SYNTHETIC FIBERS

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This invention relates to synthetic fibers, and more particularly to an improved method for setting filaments, fibers, films, fabrics, and the like derived therefrom.

5 This invention is concerned with improve-
ments in the new class of fibers obtained from
synthetic linear condensation polymers described
in Patents 2,071,250, 2,071,252 and 2,071,253. The
10 fibers are formed from the mentioned polymers
which have the unusual property of being capa-
ble of being spun by extruding, or otherwise, into
filaments which can be cold drawn, that is by
being drawn in the solid state into useful tex-
15 tile fibers which like natural fibers exhibit upon
X-ray examination molecular orientation along
the fiber axis. Of these polymers, the poly-
amides are especially useful in the preparation
of fibers, bristles, films, fabrics, and the like. It
20 is with these polyamides that the present inven-
tion is particularly applicable. The polyamides
are of two types, those derived from polymeriz-
able monoaminomonocarboxylic acids and their
amide-forming derivatives, and those derived
25 from the reaction of suitable diamines with suit-
able dicarboxylic acids or amide-forming deriva-
tives of dibasic carboxylic acids.

Although the synthetic fiber-forming poly-
amides as a class are characterized by high melt-
ing points and low hygroscopicity, it has previ-
30 ously been found that water, and particularly
steam, has a remarkable effect on the properties
of filaments and the like prepared therefrom.
Thus, steam treatment improves the crimp re-
tentivity of crimped polyamide filaments, i. e.
35 sets the crimp. The setting effect of steam is
not limited to crimped filaments, but is also use-
ful in setting filaments in a straight position.
Hot water has a similar effect and is particu-
larly useful in setting polyamide bristles. Poly-
40 amide bristles which have been treated with hot
liquid water or steam are much superior in recov-
ery from deformation and in wearing qualities
than untreated bristles. Furthermore, these
processes are not limited to single filaments but
45 are applicable to yarns, fabrics, and the like.
For example, when these setting treatments are
applied to fabrics held in the desired position,
the fabrics become much less susceptible to fu-
ture wrinkling. One drawback, however, of the
50 mentioned setting methods is that the time re-
quired to secure the desired setting is frequently
quite long.

55 An object of this invention is to improve the
properties of filaments, fibers, bristles, films, fab-
rics, and the like derived from synthetic linear

condensation polyamides, and particularly to im-
prove the ability of such articles to recover from
deformation. A further object is to accelerate
the setting effect of setting agents, particularly
hot water (including steam), on polyamide fila-
ments. Other objects will appear hereinafter.

5 These and other objects, which will become
apparent as the description proceeds, are accom-
plished by subjecting articles, such as filaments,
fibers, bristles, films, fabrics, and the like, derived
10 from synthetic linear condensation polyamides to
a setting treatment, which comprises subjecting
the articles in the form or shape it is desired to
impart thereto to the action of a hot mild swell-
ing agent, preferably hot water, in the presence
15 of a water-soluble sulfite.

The term "setting" as used herein denotes im-
parting to the article treated a more or less per-
manent shape or form to which the material
tends to return after being subjected to defor-
20 mation. The term "hot water" is used to include
steam. "Sulfite" is used in its broad sense to
include bisulfites.

I have now found that the setting effect of
hot water on polyamide filaments and the like
25 can be greatly accelerated by carrying out the
process in the presence of a water-soluble sul-
fite. This fact is demonstrated by the following
specific example in which approximately 20 min-
utes' treatment of a sample of crimped poly-
30 amide filaments with saturated steam was re-
quired to produce a product of high crimp re-
tentivity, whereas crimped filaments taken from
the same original sample which were first moist-
ened with an aqueous solution of sodium sulfite,
35 gave a product of equally good crimp retentivity
in 10 seconds' treatment with steam under com-
parable conditions. This pronounced accelerat-
ing effect of sulfite is obviously of great eco-
nomic importance. For example, it makes it pos-
40 sible to conduct such operations as crimp setting
continuously with a great saving of time. With-
out sulfite treatment, it is generally necessary to
resort to a batch process to secure the desired
45 improvement in crimp retentivity.

Although wetting agents are not necessary in
the process of the present invention, their use
is sometimes beneficial since the use of the sul-
fite in conjunction with a wetting agent tends to
50 further accelerate the action of the steam. Par-
ticularly useful wetting agents are sodium ole-
ate, C-hexadecylbetaine, gelatin, sodium silicate,
dodecyl sodium sulfate, Turkey red oil, alkyl
naphthalene sodium sulfonates, alkyl sodium
55

sulfates, fatty acid esters of ethanol sulfuric acid, and sulfonated oils.

Of the water-soluble sulfites, the alkali metal sulfites are preferred. Sodium sulfite is a particularly effective agent. Water-soluble bisulfites can also be used. As typical examples of water-soluble sulfites may be mentioned ammonium sulfite, magnesium sulfite, sodium bisulfite, sodium sulfite, potassium sulfite, and potassium bisulfite. Mixtures of water-soluble sulfites can also be used in the process of this invention.

Since the polyamide articles treated according to the process of this invention become "set" in the position or shape in which they are treated, it is essential that the articles be treated in the position it is desired that they should assume. To obtain crimped polyamide filaments of lasting crimp, for example, it is necessary that the crimped filaments be treated with hot water while in the crimped position, i. e., under low tension. Similarly, if large polyamide filaments are to be used as straight bristles, the filaments should be treated in a straight position. Likewise, in order to obtain a fabric which is free from wrinkles and a tendency to wrinkle, the fabric should be treated in a position free from wrinkles. Thus, to secure a stocking of good appearance and good resistance to wrinkling, the stockings should be treated while on a suitable form under sufficient tension to avoid wrinkles. This treatment of a fabric on a form will be referred to as "boarding".

The following examples illustrate the invention in greater details. Examples 1 and 2 illustrate the setting of crimped filaments by means of steam, whereas Example 3 illustrates the setting of a polyamide fabric. In Examples 1 and 2 the effect of setting is expressed quantitatively in terms of the "crimp recovery from stretch" of the filaments. "Crimp recovery from stretch" is determined empirically as follows: The crimped filaments are stretched until the crimps are just straightened out and then an additional 10% stretch is applied based on the straight length of the filaments. The filaments so stretched are immersed in boiling water for 30 seconds, removed, and allowed to dry in the relaxed condition for two minutes. The "crimp recovery from stretch" is obtained in per cent by multiplying by 100 the quotient obtained by dividing the difference between initial straightened length and recovered crimped length by the difference between initial straightened length and initial crimped length. This test is of value in comparing the crimp retentivity, that is the degree to which different samples have been "set" in the crimped form.

Example I

A sample of 80-denier, 10-filament yarn spun from poly-hexamethylene adipamide was soaked in water and passed at a rate of 100 feet per minute through a mineral oil bath heated to 150° C., employing a bath travel of four inches. This heat treatment will be referred to as "conversion", since it renders the yarn susceptible to spontaneous crimping. The oil treated yarn was washed twice with toluene, twice with acetone, and then soaked in water. The wet yarn was cold drawn 175%, i. e., until its length was 2.75 times its original length. Part of the yarn was crimped by hanging it loosely on a bar and permitting it to dry. Spontaneous crimping occurred during drying. A second part of the yarn was impregnated with a 2% aqueous solution of

sodium sulfite for 24 hours, removed from the sulphite solution, and allowed to crimp by drying it in the relaxed condition. The crimp quality of both samples was excellent. A third sample was impregnated with 5% sodium sulfite solution for 24 hours and then dried in a relaxed condition. The crimp which developed in this yarn was somewhat inferior to that of the other samples.

The three samples of crimped yarn were then "set" by exposure to saturated steam at 100° C. for 10 seconds. The sample which had not been impregnated with sodium sulfite had a "crimp recovery from stretch" of $62 \pm 20\%$, where 20% represents the standard deviation calculated from ten determinations which averaged 62%. The samples which had been impregnated with 2% and 5% of sodium sulfite, respectively, had "crimp recoveries from stretch" of $91 \pm 5\%$ and $89 \pm 7\%$, respectively. The accelerating effect of the sodium sulfite is quite apparent from these tests. It is further evident that the sodium sulfite impregnated yarns had more uniform retentivity, since various portions of the same sample showed less deviation from the mean.

Example II

Four 85-denier, 10-filament polyhexamethylene adipamide yarns were soaked in water and "converted" at a rate of 100 feet per minute by passing the wet yarns separately through a 4-inch mineral oil bath heated to 150° C. The four yarns were then combined into a single yarn which was washed free of oil by passing it around a series of rollers located in a warm washing bath containing soap. The yarn was then rinsed with warm water, cold drawn 175% while still wet, and wound on a bobbin. The bobbin containing the yarn was stored in water.

One sample of the converted and drawn yarn was crimped and set as follows: The wet yarn was unwound from the bobbin and passed through squeeze rolls to remove excess water. From the squeeze rolls the yarn was taken by an air aspirator which injected the yarn at a rate of 82 feet per minute into the top of a vertical column, nine feet long and three inches in diameter. A current of air just insufficient to support the weight of the yarn was passed up the column. Under these conditions the yarn dried in the column and crimped spontaneously. On reaching the bottom of the column the yarn was laid down on a moving endless belt traveling at a rate of 7.5 feet per minute. The belt carried the yarn through a horizontal chamber filled with steam at 117° C. The length of this steam setting chamber was such that the yarn was in contact with steam for approximately 24 seconds. On leaving the setting chamber the yarn was wound on a bobbin. The "crimp recovery from stretch" of the resultant yarn was $54 \pm 6\%$ (5 determinations).

A second sample of the converted and drawn yarn was impregnated with an aqueous solution containing 1% sodium sulfite and 0.25% isopropyl naphthalene sodium sulfonate and was then crimped and set as in the case of the first sample. The "crimp recovery from stretch" of this yarn was $106 \pm 7\%$ (5 determinations).

Example III

A stocking knitted from cold drawn polyhexamethylene adipamide yarn containing about 7 turns per inch twist was immersed in an aqueous solution containing 1% of sodium sul-

fitte and 0.25% isopropyl naphthalene sodium sulfonate for 15 minutes. The stocking was then placed in a suitable rigid form approximating the shape of a leg, the form being of sufficient size to hold the fabric under tension to avoid wrinkles. While on this form the stocking was placed in a saturated steam chamber for about five minutes. The stocking so boarded was free from wrinkles and showed substantially no tendency to wrinkle when subsequently dyed in a hot dyeing solution. Any superficial wrinkles which may develop are easily removed by a short after-boarding operation. On the other hand, if an unboarded stocking is similarly dyed permanent wrinkles develop. Wrinkles developed in this way are not easily removed since the hot dyeing solution has a setting effect on the fabric. Although sodium sulfite is not essential in the boarding operation, its presence accelerates the setting operation.

The foregoing examples illustrate the application of this invention to the setting of polyamides in the form of yarns and fabrics. It will be apparent that other polyamide articles, e. g., bristles, films, and sheets, can be similarly set.

The invention is not limited to the particular polyamide cited in the foregoing examples but is applicable broadly to synthetic linear condensation polyamides. As examples of other polyamides of the diamine-dibasic acid type may be mentioned polytetramethylene sebacamide, polypentamethylene adipamide, polypentamethylene sebacamide, polyhexamethylene sebacamide, polyoctamethylene adipamide, polydecamethylene adipamide, and poly-p-xylylene sebacamide. Polyamides of the amino acid type, e. g., polymerized 6-aminocaproic acid, polymerized 9-aminononanoic acid, and polymerized 11-aminoundecanoic acid may also be used. Interpolyamides, i. e., polyamides derived from a mixture of polyamide-forming reactants capable of yielding more than one polyamide if reacted in suitable combinations, can also be set by the method of this invention. As an example of such an interpolyamide might be mentioned the polymer derived from equimolecular amounts of hexamethylenediamine, decamethylenediamine, adipic acid, and sebacic acid. By the term "linear condensation polyamide" I mean to designate polyamide compounds of the types herein disclosed and their equivalents, whether or not such compounds are produced by chemical condensation or by some other process.

It is also within the scope of the invention to apply the setting treatment to polyamide articles containing modifying agents, e. g., plasticizers, pigments, dyes, luster modifying agents, antioxidants, oils, resins, etc. The invention can also be applied to mixed yarns and fabrics, i. e., yarns and fabrics containing other types of filaments in addition to the polyamide filaments.

As already indicated, the setting treatment can be effected by means of hot liquid water or steam. For many purposes, e. g., in setting the crimp in wool-like polyamide filaments, steam is most satisfactory, the best results being obtained with saturated steam at 100-150° C. In applying this invention to the setting of bristles, however, hot water, particularly water at 85-100° C., is preferred. The time required to secure the necessary setting will, of course, vary with the nature of the polyamide article, particularly its thickness or diameter. In general it will vary from a few seconds to a few minutes. As indi-

cated in Example I, ten seconds' treatment in the case of crimped filaments of the size used in making fabrics is sufficient to secure good crimp setting.

Although water is the preferred setting agent, other mild swelling agents for the polyamides can be used as setting agents in the process of this invention either in the form of hot liquids or vapors. For example, hydroxylated non-solvents for the polyamides, particularly the lower boiling aliphatic alcohols such as methanol and ethanol, can be used for this purpose.

Only a small amount of sulfite is required in the process of this invention. The best results are obtained by impregnating the polyamide article to be set by immersing it for a short time in a 0.1 to 5.0% aqueous solution of the sulfite. On removal from the bath the article can be treated directly with the setting agent or it may first be dried. If hot water is to be used as the setting agent, no preliminary impregnation is necessary since the sulfite can be incorporated directly in the hot water.

It has been emphasized that during the setting treatment the polyamide articles must be held in the position it is desired to impart thereto. It follows, therefore, that if a fabric free from wrinkles is desired, the fabric should be set while held in a position free from wrinkles. On the other hand, if a fabric with creases or pleats is desired, the fabric should be treated in a creased or pleated position. By application of this invention it is possible to prepare "fashioned" wearing apparel, i. e., apparel having the shape it is to assume in use. A typical example of this type of wearing apparel is a hat.

This invention furnishes a convenient and economical process for improving the properties of polyamide articles, particularly filaments and fabrics. It greatly improves the ability of these articles to recover from deformation. This improvement in recovery is manifested in the case of crimped filaments by improved crimp retentivity, in the case of bristles by resistance to curling, and in the case of fabrics by resistance to wrinkling. Although the same effects can be obtained with hot water or steam in the absence of sulfite, the use of sulfite according to the process of this invention reduces the time required for this purpose.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A method for improving the properties of synthetic linear condensation polyamides in the form of filaments, fibers, bristles, films, fabrics and the like which comprises treating said polyamides in such form with a setting agent in the presence of a water-soluble sulfite.

2. A method for improving the properties of synthetic linear condensation polyamides in the form of filaments, fibers, bristles, films, fabrics and the like which comprises treating said polyamides in such form with a hot mild swelling agent for the polyamides in the presence of a water-soluble sulfite.

3. The process set forth in claim 2 in which said agent is water at a temperature of 85-100° C.

4. The process set forth in claim 2 in which said agent is steam at a temperature of 100-150° C.

5. The process set forth in claim 2 in which said sulfite is sodium sulfite.

6. The process set forth in claim 2 in which said polyamides are in the form of stockings.

5 7. A process which comprises treating a synthetic linear condensation polyamide in the form of filaments, fibers, bristles, films, fabrics and the like with an aqueous solution of water-soluble sulfite and then subjecting the treated
10 polyamide to a setting treatment by means of

heated fluid exerting a mild swelling action on the polyamide.

8. A process which comprises treating synthetic linear condensation polyamides in the form of yarn with an aqueous solution of water-soluble sulfite, crimping the yarn, and then subjecting the crimped yarn to a setting treatment by means of heated fluid exerting a mild swelling action on the polyamides.

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