United States Patent Office 3,564,834 Patented Feb. 23, 1971

3,564,834

TWIST SET POLYAMIDE YARN AND PROCESS FOR ITS MANUFACTURE

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No Drawing. Continuation-in-part of application Ser. No. 723,871, Apr. 24, 1968, which is a continuation-in-part of application Ser. No. 671,600, Sept. 29, 1967. This application July 9, 1969, Ser. No. 840,526

Int. Cl. D02g 1/02, 3/30

U.S. Cl. 57—140 8 Claims

ABSTRACT OF THE DISCLOSURE

Crepe fabrics having the lively and supple qualities associated with silk crepes are prepared from certain twisted, heat-treated polycarbonamide yarns. These fabrics are washable and resistant to water spotting.

CROSS REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of U.S. patent application Ser. No. 723,871 filed Apr. 24, 1968, now abandoned which is a continuation-in-part of U.S. patent application Ser. No. 671,600, filed Sept. 29, 1967 and now abandoned.

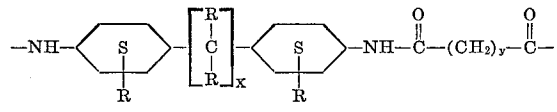
BACKGROUND OF THE INVENTION

This invention concerns twisted and heat-treated (twist-set) yarns of polyamides which are readily woven to produce crepe fabrics similar to silk crepes. The yarns of the invention develop a high amount of torque and low modulus of rigidity while subjected to swelling agents.

Crepe fabrics are commonly prepared from water-sensitive yarns such as rayon or silk. When fabrics prepared from highly twisted rayon or silk yarns are subjected to relaxed treatment in hot water, the filaments swell. As a result of the swelling or growth of the filaments, the highly twisted yarn tends to unwind. However, due to restraints imposed by the fabric construction, the yarns cannot untwist appreciably. Instead, the torsional energy is dissipated by formation of kinks or partial loops. This distortion of the yarn in the fabric creates a desirable texture in the fabric. This texture is identified by the term "crepe figure."

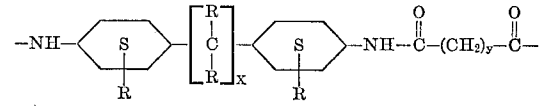
SUMMARY OF THE INVENTION

This invention provides a highly twisted polyamide yarn suitable for preparation of crepe fabrics, by fiber swelling techniques. The yarn of this invention does not have excessive "twist liveliness," which would interfere with weaving but is capable of generating torque and lower modulus when treated with swelling agents in an aqueous medium at the boil. This invention is a twist-set yarn comprised of continuous filaments of polyamides wherein at least 50 mol percent of the repeating units have the formula



wherein X represents 1 or 2, y represents an integer in the range of 7 to 14, R represents the same or different members of the class consisting of hydrogen and methyl and at least 40% of the diamino constituent in said repeating units being of the trans-trans stereoisomer characterized in that said yarn has a twist multiplier above 1.5, an orientation index of at least 0.7 and prefebaly 1.0, and a liveliness multiplier less than 0.4. Preferably the yarn

has a buckling index of 300 and most preferably 1000. Preferably the polyamide has a structure wherein X is 1, R is H and y is 7 or 10. In one embodiment, two types of filaments are present such as above described, one being a homopolymer and the other a copolymer. The yarn in the form of a crepeable fabric is also within the scope of this invention. This invention also comprises a process for preparing the yarn of claim 1 comprising extruding a plurality of filaments of a polyamide wherein at least 50 mol percent of the repeating units have the formula:



wherein X represents 1 or 2, y represents an integer in the range of 7 to 14, R represents the same or different members of the class consisting of hydrogen and methyl and at least 40% of the diamino consituent in said repeating units being of the trans-trans stereoisomer, characterized by obtaining an orientation index above 0.7 and preferably above 1.0 by drawing or annealing, twisting the filaments to a twist multiplier above 1.5 and heat treating the twisted yarn at a temperature of between 55° C. and the second order transistion temperature T_g of the polymer to a liveliness multiplier of less than 0.4.

The term "PACM" means bis(4-aminocyclohexyl) methane and the term "G–PACM" means the generic family of diamines described in the diamine portion of the above-mentioned structural formula. In addition, the polymer class is indicated by "G–PACM polymer." The yarn class is indicated by G–PACM yarn, it being understood that these latter terms are applied only when a major proportion of the polymer or yarn is derived from G–PACM diamines. Twist multiplier orientation index, liveliness multiplier, buckling index are defined by tests described hereinafter.

Polymers and yarns within the G–PACM class have been previously described in British patent specification No. 1,091,007, U.S. Pat. 3,249,591, and in Bolton and Kirk U.S. Pat. 2,512,606. Other G–PACM polymers and G–PACM yarns will be described in subsequent paragraphs.

Typical polymers for use in the yarn of the invention are the polyamides prepared from bis(4-aminocyclohexyl) methane and sebacic, azelaic, or dodecanedioic acid. Liveliness is the tendency of a fabric to revert quickly to a planar arrangement after being distorted.

The yarns of the invention may contain filaments of different composition in mixtures with the PACM filaments. In any case however at least 50% by weight of the filaments in the yarn are composed of PACM polyamides prepared from diamines having the prescribed PACM structure and from dibasic linear aliphatic acids having 9 to 16 carbon atoms.

Mixed yarns wherein filaments of two different PACM polyamide species are included in a single yarn bundle are used with special advantage in the present invention. The twisted heat-treated yarns prepared from these mixed yarns according to the present invention provide even greater suppleness and liveliness in fabrics than yarns with a single PACM fiber species.

A particularly useful yarn is made by a co-spinning process in which half the filaments are comprised of polymer prepared from PACM of 70% tt content and dodecanedioic acid, and the other half are filaments comprised of a copolymer prepared from PACM of 70% tt content and a mixture of dodecanedioic acid and isophthalic acid. The isophthalic acid is preferably present in an amount sufficient to provide 10% by weight of the isophthalic copolymer component in the final polymer.

It has been found that only those PACM polyamide

yarns which have a buckling index of at least 300 are adequate for preparing creped fabrics. Especially preferred are yarns having a buckling index above 1000. Buckling index is a measure of the buckling capacity of twist-set yarn when held between two fixed points and subjected to a swelling agent. This measurement permits one to predict whether an adequate crepe figure can be developed.

In order to obtain G–PACM yarns with satisfactory orientation index, adequate care is needed in melt spinning and quenching. Quenching promotes development of molecular orientation without excessive crystallization. Excessive crystallization in G–PACM yarns tends to promote low tensile strength in the yarn. The amount of crystallization is preferably controlled by use of a superheated steam atmosphere in the chamber surrounding the spinneret (200 to 350° C. and atmospheric pressure). Reduced crystallization of the polymer is obtained by increased steam flow or increased temperature.

After spinning and quenching the yarn is drawn and annealed to obtain an orientation index above 0.7 and to promote higher tesile strength. The draw ratio required is quite low usually between about 1.2× and 2.0×. Drawing may be done at room temperature by conventional mechanical techniques in one or two stages. Annealing of the yarn may be a separate operation or may be done simultaneously with the second stage of the drawing operation by passing the yarn many turns around the final set of rolls enclosed in a heated air chamber. The annealing chamber air temperature is preferably 120 to 180° C.

After drawing and annealing the filaments are twisted on standard twisting equipment such as a down-twister or up-twister, to develop true twist, to give a twist multiplier (T.M.) above 1.5 and preferably above 3.2.

Subsequently, the yarn is heat treated (twist-set).

Twist-setting is usually carried out by exposing the twisted yarn to a heated atmosphere, preferably by exposure on a yarn package to saturated steam at 221° F. (105° C.). Exposure for a time of 1½ hours is usually satisfactory. High humidity air or saturated steam may be used at temperatures from 55° C. to the transition temperature T_g of the polymer. The twist-set yarn has an orientation index above 0.7 and preferably above 1.0. The twist liveliness multiplier is less than about 0.4.

The twist-set yarn may be woven or knit by conventional methods. The resulting fabric is potentially crepeable when fabric construction is substantially equivalent to that used for silk or rayon of the same denier. Crepe figure is developed by treatment of the fabric in an aqueous bath of a swelling agent at a temperature near the boiling point preferably at 90–100° C. The bath contains a swelling agent of sufficient concentration to produce a diametrical swelling of the filaments of at least 5%. The following swelling agents are particularly suitable: butyl benzoate, pelargonic acid, phenol, octyl alcohol, chlorobenzene, biphenyl. The swelling agents may be removed by boiling the fabric in water or in a slightly alkaline solution (a solution containing 1.5 g./l. sodium carbonate). Some of the swelling agents may be removed simply by heating the fabric. The product may be finally rinsed by tumbling in cold water. A fabric with crepe figure is obtained.

Crepe fabrics which contain 50% or more than 50% PACM–12 or PACM–9 polymers are, after heat setting, softer and more lively crepe fabrics than fabrics based primarily on PACM–10 fibers. The PACM–9 and PACM–12 crepes are therefore preferred.

In order to obtain lively, supple fabrics, the yarns must be carefully processed in the twist-setting operation, to avoid excessive crystallization in twist-setting.

Quite unexpectedly, the filaments used in the yarns of this invention can be swelled to a high degree without a high degree of shrinkage. It is believed that this feature is partly responsible for the exceptional liveliness and suppleness of the treated fabrics. This high degree of liveliness and suppleness is typical of that obtained from silk fabric of the same construction. At the same time fabrics prepared from the twist-set yarns of the invention resist water-spotting and are washable.

Yarn test methods

The buckling index is defined by the formula

$$B=\frac{T_{rw}\times L}{E_w\times I_B}$$

wherein:

T_{rw} is the torque in dyne-cm. developed by the wet yarn at 95° C. in an aqueous solution containing 5 g./l. pelargonic acid as swelling agent;

L is the length (cm.) of the yarn in the test device. In characterizing the products of this invention this value is always 10 cm.

E_w is the wet modulus of the multifilament yarn in dynes/cm.^{2} at 95° C. in an aqueous solution containing 5 g./liter pelargonic acid;

I_B is the section modulus in bending or moment of inertia (cm.^{4}) of the multifilament yarn about an axis through its centroid.

Torque development T_{rw} under hot-wet swelling conditions is beneficial for promoting high buckling index. An equally important factor is the wet modulus in the hot swelling bath. Low hot-wet modulus promotes high buckling index. The yarn can only buckle if the torque to modulus ratio is sufficiently high.

The I_B factor is related to the filament shape and diameter in the unswelled condition and to the number of filaments. Filaments of smaller diameter will in general bend more easily than filaments of greater diameter, although cross-sectional shape should be considered. A sufficiently high buckling index is only obtainable in filaments which have not been over-crystallized in twist-setting, in yarn drawing, or in other processing. At the same time, sufficient orientation must be provided at the start and retained through the swelling treatment to give the resilient properties of silk.

The calculation of buckling index requires determination of wet torque development T_{rw} and wet modulus E_w of the yarn. These values are each determined in an aqueous bath at 95° C. containing 5 g./l. pelargonic acid as swelling agent. It should be understood that this is not necessarily the concentration which should be used in the treatment of fabrics. In the actual dyeing or finishing of fabrics with pelargonic acid or other swelling agents, the amounts of swelling agent to be used should be related to the amount of fiber in the dye bath.

Wet torque development T_{rw} is determined as follows: A weight of 0.02 g.p.d. is attached to one end of a twist-set yarn sample. The other end of the yarn sample is attached to a clamp axially mounted to a moving coil of a galvanometer. The clamp and weight are attached to the yarn at points which are 10 cm. apart. The yarn sample and attached weight are suspended from the clamp. A fork device is moved into place around the weight to prevent the weight from rotating. The fork does not restrain vertical movement which may occur during shrinkage.

The total length of the above-mentioned yarn sample with weight suspended and fork device in place is immersed in an aqueous dispersion of pelargonic acid containing 5 g./l. of pelargonic acid, maintained at 95° C. The yarn sample is immersed for 15 minutes. The swelling agent releases latent torque in the yarn sample which displaces the galvanometer needle. After fifteen minutes, the operator counterbalances the torque by increasing the current to the galvanometer provided by the power supply. Finally, by carefully adjusting the current, the galvanometer pointer is brought to rest at the zero point, the current as measured by an ammeter,

calibrated to read T_{rw} in dyne-cm. which is the wet torque development value.

The calibration of the torque tester may be achieved by the use of two torsional hairsprings which are placed in parallel and whose total torque in dyne cm./revolution is known (either specified by the manufacturer or determined by comparison with a standard torque cell). These springs are then given various amounts of twist (0 to 90°) and the torque generated by them is applied directly to the moving coil of the galvanometer. A current is applied through the moving coil of the galvanometer thereby maintaining the null position of the pointer at various externally applied known torques. Hence, by this technique the torque generated by a given current in the galvanometer's moving coil is equated to the torque applied by the hairsprings and the relationship between the galvanometer current and the externally applied torque is determined. The springs used in this calibration had a total spring constant of 200 dyne cms./revolution. The galvanometer had a range of ±50 dyne cms. of torque. The calibration was performed by twisting the springs through angles up to ±90° and noting the current.

The wet modulus E_W is the modulus of a yarn at 95° C. in an aqueous dispersion containing 5 g./l. pelargonic acid. The test simulates the buckling process for yarns in a crepe fabric. A fixed length of twist-set yarn (10 cm.) with residual twist liveliness released is placed between two sets of clamps in an Instron tensile tester with just sufficient tension to straighten the yarn. An aqueous bath containing 5 g./l. pelargonic acid is brought up over the yarn specimen in the clamps and maintained there for 15 minutes during which time the maximum shrinkage tension (T_{max}) develops. At the end of 15 minutes the two clamps of the tester are brought together at constant speed with the yarn still immersed in the aqueous hot dispersion. The tension on the yarn is reduced from T_{max} to a tension of 0.033 g./denier which is expressed by the symbol T_{033}. A plot of tension in g./denier versus actual change in length may be prepared automatically by the instrument. The wet modulus E_W of twist-set yarns of the invention is determined by dividing the change in load ΔT (expressed in dynes/cm.^{2}) by the fractional change in length in going from the maximum tension down to 0.033 g./denier. The change in tension ΔT must be converted from g./denier to dynes/cm.^{2} by use of yarn denier and polymer density. For this purpose one assumes that the multi-filament yarn is round in cross-section and is free of air space. From this it follows that the basic formula

$$E_W = \frac{\Delta T}{\Delta L/L}$$

expressed in g./denier may be converted to

$$E_W = \frac{(T_{max} - T_{033}) \times 8.83 \times 10^8 \times \text{polymer density}}{\Delta L/L}$$

expressed in dynes/cm.^{2}, wherein

T_{max} and T_{033} are tensile tester loads in g./denier,
polymer density is in g./cm.^{3},
denier is the weight of multifilament yarn in g. for 900,000 cm.,
ΔL is the change in length during unloading from T_{max} to T_{033},
L is the original length in cm., and
E_W is the wet modulus expressed in dynes/cm.^{2}.

The section modulus in bending (moment of inertia) I_B of the yarn around its centroid is defined by the formula:

$$I_B = \frac{n\pi D^4}{64}$$

wherein n is the number of filaments in the yarn bundle, and D is a calculated diameter of the filament (cm.) assuming that the filament is round (regardless of actual shape). I_B is expressed in cm.^{4} units. The diameter D is determined from denier and polymer density in g./cm.^{3} by the formula:

$$D = 1.19 \times 10^{-3} \times \sqrt{\frac{\text{denier per filament}}{\text{density}}}$$

Denier per filament is the weight in g. of 900,000 cm. of filament. The average density of the polymer in the preferred yarn prepared from 50% by weight PACM-12 filaments and 50% PACM-12/PACM-1 (90/10) filaments is 1.04 g./cm.^{3}.

Twist multiplier is determined by fastening a 10 in. (25.4 cm.) length of yarn between the clamps of a twist counter at a tension of 0.1 g./denier (including wgt. of holder) and unwinding until the twist angle is zero. Twist level is reported in turns/cm. Twist multiplier T.M. is calculated from

$$T.M. = \frac{\text{turns/cm.} \times \sqrt{\text{denier}}}{28.8}$$

For purposes of the present invention all yarns are assumed to be made of round filaments. The error due to differences in diameter of odd cross-section fibers and round fibers is believed to be compensated by improved packing of fibers. The denier used for determining twist multiplier is the denier of untwisted yarn with crimp extended at a tension of 0.1 g./denier. The singles and plied yarns within the invention have a twist multiplier above 1.5 and a liveliness multiplier below 0.4. In determining twist multiplier for a singles component of plied yarns, the net turns/cm. for the singles yarn is considered. In plying operations, if ply twist is in the same direction as the singles twist, the turns/cm. for the plied yarn is added to the turns/cm. for the original twisted singles yarn. If ply twist is in the opposite direction, the turns/cm. in plying must be subtracted from the turns/cm. of singles yarn. The resulting net twist count in turns/cm. for the singles yarn in the plied condition is used in the twist multiplier formula. However, it should be noted that the denier used in the formula for determining twist multiplier of a singles component is the total denier including all of the components in the plied filament bundle.

The liveliness multiplier (L.M.) is a factor which relates twist liveliness to denier:

$$L.M. = \frac{T.L. \times \sqrt{\text{denier}}}{28.8}$$

where T.L. is twist liveliness in turns/cm. of the singles component or the plied yarn. Yarns of equal liveliness multiplier have approximately an equal ability to kink during weaving.

Twist liveliness is determined at 21° C. and 75% relative humidity. Samples are removed tangentially from the package by rolling the package so that no twist is added or removed. A weight of 1 g. is fastened to one end of a suspended strand which is of known length (about 10 cm.) The yarn is allowed to untwist as a result of the action of the weight, until it reaches equilibrium. The net number of turns is counted during unwinding. For example, if a yarn originally has "Z" twist and turns 100 turns "S" direction and then back 25 turns "Z" direction during unwinding and stops at equilibrium, the net number of turns would be 75. The twist liveliness is finally calculated by dividing the net turns to equilibrium by the original length of the test sample in cm. before unwinding. The answer is expressed in turns/cm. In 60 denier yarns of the invention, the twist liveliness is below 0.6 turns/cm.

Orientation index, as used herein, is a measure of crystalline orientation in the fiber. The orientation of crystallites in PACM polyamide fibers appears to change rapidly when heat is applied in any way. The amount and direction of change may be greatly affected by such factors as torsion, tension, compression, or slack in the fiber

during heating. The orientation index is obtained from an X-ray diffraction pattern. The measurement as described herein appears to be more responsive to crystallite changes than is the traditional arc measurement for X-ray diffraction.

The orientation index is determined from a flat-plate wide-angle X-ray diffraction pattern. A flat-plate vacuum camera is used with nickel filtered copper X-rays at 40,000 volts potential and 20 milliamp current, e.g. with a General Electric Company XRD–5 X-ray unit with CA–8 X-ray tube. The beam is collimated with an outside pinhole diameter of about 0.625 mm. and an inside pinhole diameter of about 0.50 mm. with a separation of 7.0 cm. Careful technique must be employed to assure measurement of the maximum density in both regions which is then corrected by subtraction of the proper background density before the ratio calculation. Analysis of both regions must be made on the same X-ray negative. In making the exposure, care must be taken to assure that the yarn has no twist or crimp when supported in the camera and that all of the filaments in the yarn are aligned in a parallel manner. A yarn sample thickness of no more than about 0.50 mm. is used with a sample to film distance of 7.5 cm. The pattern for the subject fibers shows a well-defined meridional reflection at a Bragg spacing of about 10.5 A. and an equatorial diffraction, or amorphous halo, at a Bragg spacing of about 4.7 A. The orientation index is calculated as the ratio of the maximum intensity of this meridional spot divided by the maximum intensity of this equatorial halo. The intensities are measured on the X-ray negatives using conventional microphotometry techniques. The negatives are prepared in a conventional manner using No Screen X-ray film manufactured by the Eastman Kodak Company, Rochester, N.Y. (or X-ray film providing equivalent contrast). The film is developed for 3 minutes at 20° C. with Kodak X-ray developer at the concentration recommended by the manufacturer. Exposure times for the X-ray negatives should be such that the microphotometer registers optical densities for both regions of less than 1.0 but not less than 0.10.

Some G-PACM yarns which include a high shrinkage group of glaments and a low shrinkage group of filaments which are preferred for preparation of crepeable yarns of the invention are further characterized by a difference in filament length which develops during boil-off or relaxation of greater than 1%. Untwisted yarns which develop a further difference in filament length (for example, at least 0.5%) on further heat setting (such as at 177° C. for 2 minutes) are also suitable in this invention.

The second group (i.e., low shrinkage group) may have different shrinkage properties because it contains 5–50% of a copolymeric component of the same diamine and a different acid or a different diamine and same acid as the polymer of the first group (i.e., high shinkage group); or may differ from the polyamide of the filaments of the first group in trans-trans steric configuration by more than 10% by weight; or may differ from the polyamide of the filaments of the first group by having a relative viscosity at least 9 units less than that of the polyamide of the first group; or may have a chain length of the diacid constituent of the polyamide less than the chain length of the diacid constituent of the polyamide of the first group; or may have an orientation index that is at least 1.0 greater than that of the first group of filaments.

The shrinkage of the yarns is determined on skeins of measured length as follows: A skein of about 1500 denier is prepared by winding an appropriate number of turns of about 110 cm. length per turn, on a reel. For 60 denier yarn, 25 turns are used. The skein is advantageously tied at two diametrically opposed points before removal from the reel. The length of the collapsed skein (approximately 55 cm.) is measured accurately under a load of 4 mg.d. (milligrams per denier) based on the denier of the collapsed skein, i.e., about 3,000, namely a total load of about 12 grams. A water bath is heated to boiling over a ½ hour period. When the temperature reaches 50° C. the skeins, with a suspended weight at one of the tie points amounting to 4 mg.d. are placed in the bath and remain there for 10 minutes after the boiling point is reached. They are then removed and air dried with the weights attached. The length is then determined again and the percent shrinkage calculated from the equation

$$\text{Percent shrinkage, } S=\frac{100(L_0-L_1)}{L_0}$$

where L_0 equals length of skein before boiling, L_1 equals length of skein after boiling.

The percent difference in filament length (DFL) between the high and low shrinkage filaments is determined by cutting a 40 cm. strand from the yarn package after shrinking, doubling the strand, and tying the free ends together to form a loop about 19 cm. long. The face ends above the knot are taped to a vertical meter rule and the lengths of isolated individual filament loops are measured by hanging a 0.4 milligram per denier weight in a loop (such a weight, for example, for a 60 denier yarn before shrinkage can be provided by a 0.6 g. paper clip in each individual filament loop). At least 5 low shrinkage and 5 high shrinkage filaments are measured in this manner. Percent DFL is calculated from the following equation:

$$\text{Percent } DFL=\frac{100(L_2-L_1)}{L_1}$$

where L_2 is the average length of the longer filaments and L_1 is the average length of the shorter filaments.

The DFL after exposure to heat-setting temperature is measured by hanging a similar boiled-off, air dried skein in an oven at 177° C. for 2 minutes under the same total load as was used in boil-off and then determining the DFL as described above for the boiled-off skein.

For measurement of DFL on the shrunk twisted yarns a different procedure was used. A 20 inch length of yarn was doubled and the free ends tied and taped to a vertical meter rule. A 0.6 gram paper clip was employed as a weight to straighten the yarn. The yarn length (L_1) was then measured. The yarn was then extended by pulling on the paper clip until the point where all evidence of loopiness disappeared and the yarn length (L_2) was measured. These values are then inserted into the percent DFL equation.

Fabrics from twist-set yarns

For the twist-set yarns of the invention, torque is generated by treatment with swelling agents. The fabrics may be either woven or knitted. The yarns of the invention may be alternated in the fabric with other yarns such as silk, rayon, acetate, 6-nylon, 6-6 nylon, polyester, or acrylics. The yarn of the invention may be used either in the filling or in the warp of woven fabrics, however to obtain woven crepe fabrics with satisfactory crepe figure the PACM polyamide yarn of the invention must always be present in the filling direction to the extent of at least 50%. Preferably the fabric has also at least 50% by weight PACM polyamide in the warp direction of the fabric.

The fabric construction may consist of simple plain weave construction, or various other constructions may be used. For example, the fabric may be a crepe de chine, a crepe satin, a double tafetta crepe, a double twill crepe, a double faced crepe, a georgette, a chiffon, or any type of fabric wherein the construction is loose enough to permit movement of fibers during swelling.

In the preparation of fabrics, several ends of the same twist-set yarn may be combined and additional twist may be applied. The twist-set yarns may be used as single yarns or they may be plied. It should be understood that the plied yarns after twisting in the plied state should be twist-set by heat treatment if the product yarns are to be considered within the scope of this invention.

A variety of processes are available for treating the woven fabrics with swelling agents. The swelling agent may be applied as a separate step from an aqueous bath at the boil or it may be applied in combination with dye steps. In this latter case, the swelling agent acts as a carrier for improved dyeing. The hot-wet treatment may for example be applied in a paddle dyer, in a beck, in a tumble washer.

The concentration of swelling agent will depend upon the particular agent chosen. Preferably, the carrier weight is determined on the basis of fiber weight.

Because of the limited solubility of pelargonic acid, solutions are advantageously prepared by first converting the acid to the sodium salt which is quite soluble. The sodium salt solution may readily be converted to a dispersion of pelargonic acid in water by addition of acetic acid or other acids.

Fabric properties

The suppleness of the crepe fabric of this invention may be measured by its flexural rigidity. Flexural rigidity is derived from the bending length and the weight per unit area of the fabric, and is defined as the moment of couple required to bend a strip of fabric of unit width in a circle having a unit radius. Bending length is measured by means of a strip of fabric suspended in a heart-shaped loop (a double cantilever). The heart loop test is used as described in ASTM Standards 1958, part 10, published by the American Society for Testing Materials, Philadelphia, Pa., pages 557–562 (ASTM test designation D1388–552, Method B). Fabric samples 1x6½ inches (2.54x16.5 cm.) are used and cut to measure bending length in both fabric directions. The samples are mounted such that measured specimen length is 6 inches (15.24 cm.). Flexural rigidity is the product of the bending length (cm.) and weight per unit area (mg./cm.^{2}) of the fabric. Particularly suitable crepe de chine fabrics of this invention will have a flexural rigidity of 6 to 28.5 mg./cm.

In the following examples, polymer A and polymer B refer to the following:

(A) Polymer is prepared from bis(4-aminocyclohexyl) methane and dodecanedioic acid in a manner similar to that described in Belgian Pat. 668,705. The diamine consists of 70% of the tt isomer. The polymer has a molecular weight of about 14,000 as determined by the end group analysis. The polymer has a second-order transition temperature (T_g) of 172° C. which is the temperature at which crystallization begins as measured by X-ray diffraction.

(B) In a similar fashion a copolymer is prepared from bis(4-aminocyclohexyl)methane and a mixture of dodecanedioic and isophthalic acids. The mixed acids contain 8.7% by weight of isophthalic acid which is sufficient to provide 10% by weight of the isophthalic copolymer component in the final polymer. The diamine contains 70% of the tt isomer. The copolymer has a second-order transition temperature (T_g) of 180° C. as defined above.

EXAMPLE I

Filaments of polymer A are melt spun through a spinneret containing 34 orifices, and drawn 1.9× by use of 120° C. draw roll and annealed on a 165° C. roll to obtain a yarn of 60 denier. The yarn is then twisted 19.7 turns/cm., wound on an aluminum spool and then heated for 90 minutes at 82° C. and 75% relative humidity. The yarns so produced have a twist multiplier of 5.4 and a twist liveliness of 0.2 turns/cm. (liveliness multiplier=0.054). The T_{rw} is 5.4 dyne-cm.; E_W is 3.0×10^9 dyne/cm.^{2}; I_B is 0.00971×10^{-9} cm.^{4}, and the buckling index (B) is 1850.

EXAMPLE II

Filament of polymer A and copolymer B are melt cospun through a spinneret containing 34 orifices, polymer A passing through 17 orifices and copolymer B passing through 17 orifices and drawn in a single bundle 1.9× by use of 120° C. draw roll and annealed on a 165° C. roll to obtain a yarn of 60 denier. This yarn is then twisted 19.7 turns/cm. and then heated in this condition for 90 minutes at 82° C. and relative humidity of 75%. The yarns so produced have a twist multiplier of 5.4 and a twist liveliness of 0.2 turn/cm. (liveliness multiplier=0.054). The T_{rw} is 6.3 dyne/cm., E_W is 2.0×10^9 dyne/cm.^{2}; I_B is $.00971\times10^{-9}$ cm.^{4}; and the buckling index (B) is 2730.

EXAMPLE III

A plain weave fabric (crepe de Chine) is woven using the twist-set yarn of Example I as the filling at 86 picks/inch. The warp has 101 ends/inch and is comprised of the same yarn as Example I, but has a twist of only 2 turns/cm. and is not heat treated (not twist-set). This griege fabric is then treated for 15 minutes with a swelling agent consisting of 1.25 gm./liter solution of pelargonic acid in a 170 liter paddle dyer (3.33/1 swelling agent/fabric weight ratio). The temperature of the solution is 95° C. (Under these conditions of swelling agent composition, concentration, and temperature the filament swells 16.5% relaxed on a hot stage microscope. Shrinkage of a skein under 4 milligram per denier load in a bath of the same concentration and temperature is 3.8%.) The resulting crepe fabric has flexural rigidity of 27.5 mg./cm. and is lively.

EXAMPLE IV

A plain woven fabric is woven using the twist-set yarn of Example II as the filling at 86 picks/inch. The warp has 101 ends/inch is comprised of the same yarn as Example II, but has a twist of only 2 turns/cm. and is not heat treated (not twist-set). This greige fabric is then treated for 15 minutes with a swelling agent consisting of 0.62 gm./liter solution of pelarognic acid in a 170 liter paddle dyer (2/1 swelling agent/fabric weight ratio). The temperature of the solution is 95° C. (Under these conditions of swelling agent composition, concentration, and temperature the filament diameter swells 6.5% relaxed on a hot stage microscope and skein shrinkage under 4 mg.p.d. is 3.0%.) The resulting crepe fabric has flexural rigidity of 28.0 mg./cm. and has a silk-like liveliness.

EXAMPLE V

A fabric is woven (plain weave) using the twist set yarn of Example II as the filling at 86 picks/inch. The warp has 101 ends/inch and is comprised of the same yarn as Example II, but has a twist of only 2 turns/cm. and is not twist-set. This greige fabric is then treated for 15 minutes with a swelling agent consisting of 1.5 cm./liter solution of pelargonic acid in a 170 liter paddle dyer (4/1 swelling agent/fabric weight ratio). The temperature of the solution is 95° C. (Under these conditions of swelling agent composition. concentration, and temperature the filament diameter swells 18.0% relaxed on a hot stage microscope and skein shrinkage under 4 m.g.p.d. is 6.7%.) The resulting crepe fabric has a flexural rigidity of 25.5 mg./cm. and is lively.

EXAMPLES VI, VII, VIII

Crepeable yarns were obtained from 60 denier cospun yarns containing filaments of two different compositions as shown in Table I. In each yarn 18 filaments were of one composition and 18 filaments of another composition. In the table the symbol P has been used under composition to indicate PACM. All of the compositions have a trans-trans stereoisomer content above 40% in the diamine constituent. The two compositions in each case were spun through a separate set of orifices in the same spinneret all of the orifices being of Y-shaped cross-section. The melt temperature is indicated in Table I along with the sipnneret temperature. The yarn was spun in quenching chamber which was filled with steam, except in the yarn of Example VII which was spun into ambient

atmosphere (air). The rate of steam flow and steam temperature in the chamber are shown in Table I.

The yarn then was passed around a pair of rolls. It passed continuously from the first pair of rolls to a pair of first-stage draw rolls running at somewhat faster speed as indicated in Table I, then the yarn proceeded to a pair of second-stage draw rolls and was annealed by passing several times around the pair of rolls. This second-stage pair of draw rolls was enclosed in a heated chamber. From this point the yarn passed continuously to a roll operating at somewhat lower speed and finally to a wind-up roll as indicated in the table.

The properties of the three mixed filament PACM yarns are shown in Table II. The filaments were trilobal in cross-section and the modification ratio of the trilobal filaments is indicated in the table. The modification ratio is the ratio of the radius of a circumscribing circle touching the three tips of the trilobal cross-sectional configuration divided by the radius of an inscribing circle touching the three innermost portions of the cross-section. It will be noted that each of the yarns has an orientation index greater than 0.7 and a tenacity greater than about three grams per denier.

The 60 denier G-PACM yarns were separatey woven into crepe de Chine fabrics. In each case warps yarns were 5 "Z" turns/inch. Filling yarns were 60 turns/inch, half being twisted in "S" direction, half in "Z" direction. The filling yarns were twist set prior to weaving at 220° F./100% relative humidity for 90 minutes (twist liveliness multiplied less than 0.4; twist multiplier 6.4).

Loom construction was 90 ends per inch, 84 picks per inch, in a plain weave construction. Filling yarns were woven 2 picks "S" twist, 2 picks "Z" twist.

The woven fabrics were scoured in skein form by dropping the fabric into an aqueous bath at 210° F. containing 2.0 g./l. "Duponol" RA surface active agent and 2.0 g./l. tetrasodium pyrophosphate. The fabric skein was submerged for 15 minutes in the hot aqueous bath while maintained in a loose, open condition. It was then briefly drained and then submerged for 15 minutes in a bath of cold water. The fabric was then left to drain.

The fabric was next tacked (without leader) while wet and scoured in a 18-inch paddle beck with butyl benzoate carrier solution. Tacking means sewing the fabric into the form of a tube and closing the ends by sewing. The combined action of the butyl benzoate in swelling the high twist yarns and the working of the fabric in the beck dye bath resulted in the development of crepe. The guide bars were adjusted and the minimum reel speed used to give at least one fabric skein revolution per minute. Air was injected into the evenly tacked fabric through the fabric to initiate (and as necessary to maintain) a good balloon.

The beck bath (275 liters) was prepared at 120° F. with surface active agents, mild alkaline scour agents, and 10.0 g./l. of DAC–888 (80% butyl benzoate and 20% emulsifying agent). The bath was adjusted to pH 6.0 with acetic acid, raised to 200° F. at 3° F./minute and run for 2.5 hours. It was then cooled to 170° F. and allowed to overflow. The fabric was rinsed well in warm water. The bath was set at 120° F. with 1.0 g./l. "Duponol" RA (condensation product of ethylene oxidide and a fatty alcohol) and 1.0 g./l. tetrasodium pyrophosphate. The temperature was raised to 180° F. at 3°/minute and kept at that temperature for 30 minutes, cooled to 170° F., overflowed to cool. At 140° F. the bath was dropped. The fabric was rinsed well in warm water. The untacked wet fabric was vacuum extracted avoiding wriankles and creases. The fabric was dried, fully relaxed on leader, at 200° F. in a Famatex heating frame.

The three crepe de chine fabrics, after butyl benzoate scour in the 18 inch paddle beck, all gave a distinctive crepe appearance. The scoured crepe fabrics were heat-set, fully relaxed on leader cloth, in a Famatex heating frame at 375° F., 30 seconds. After heat-setting the P–10//P–10/P-I fabric was stiffer than the P–12 (45 tt)//P–12 (80 tt) fabric or the P–9//P–12 fabric. It was less soft and less lively than the other two fabrics.

EXAMPLES IX, X, XI

Thirty denier yarns were prepared from PACM–9, PACM–10 and PACM–12 polymer. Each yarn consisted of a single homopolymer as indicated in Table I. The processing conditions were similar to those described in Examples VI, VII and VIII. The conditions are recorded in Table I. These 30 denier G–PACM yarns were separately woven into georgette fabrics. In each case both warp and fill yarns were 60 turns/inch, half in "S" direction, half in "Z" direction. The yarns were twist set at 220° F./100% relative humidity for 90 minutes (twist liveliness 0.6 t.p.i. untwist; twist multiplier 4.5.

Loom construction was 110 ends per inch, 84 picks per inch in a plain weave construction. Warps yarns were in the order 1 end "S" twist, 1 end "Z" twist. Filling yarns were woven 2 picks "S" twist, 2 picks "Z" twist.

The georgette fabrics were finished exactly as described for the crepe de Chine fabrics in Examples VI, VII and VIII. All three georgette fabrics gave good crepe texture, when scoured with 10 g./l. of DAC–888 (containing 80% butyl benzoate and 20% emulsifying agents). After heat-setting at 375° F. for 30 seconds the PACM–9 and PACM–12 fabrics were soft and lively, while the PACM–10 fabric tended to be papery and not soft or lively. It should be understood that the construction of the fabric and finishing procedures can be varied to make a softer fabric from the PACM–10 yarn, but in any case the PACM–9 and PACM–12 yarns are preferred over all other PACM yarns.

EXAMPLE XII

A 30 denier yarn containing 9 filaments of PACM–12 and 9 filaments of PACM–12/PACM-I (90/10) may be converted to a warp knit fabric with crepe figure under the following procedure. Each filament in the yarn is about 3.3 denier. One half of the mixed filament yarn is twisted 45 turns per inch in the "S" direction and the other half is twisted 45 turns per inch in the "Z" direction. The yarns are then twist set as for woven fabrics (220° F., 100% RH, 90 min.). A tricot warp is then prepared from these yarns in which the twist directions of ends are alternated across the width of the fabric (to neutralize the effects of any residual torque). The fabric is then knit using a top bar motion of 1–0, 0–1, 2–3, 2–1 and a bottom bar motion of 1–0, 2–3.

The fabric is knit on a Mayer machine, 32 gauge, 42 inches. The knit quality is 9 inch/480 courses and the knit ratio is 0.72. The knit fabric construction is 42 wales by 64 courses. The gray fabric is finished in a manner similar to woven crepes including open width scour, beck scour with carrier (swelling agent), dry, heat-set, beck dye and dry. The final fabric weight is 2.4 oz./yd.^{2}; thickness 0.014 inch; bulk 4.4 cm.^{3}/gm. The resulting fabric has a dry handle and textured surface similar to woven crepes. The stitch configuration also imparts stability and draping qualities generally associated with woven fabrics. This stitch configuration also confers highly desirable properties when low or zero twist yarn is used.

The fabric is finished by scouring with successive passes to the bath at 140°, 160°, 180° and 212° F. Crepe figure is developed by scouring at 212° F. with 5 g./l. of DAC–888 (a commercial material containing 80% by weight butyl benzoate and 20% by weight of emulsifying agent). The butyl benzoate is an effective swelling agent for G–PACM yarns. After crepe development, the fabric may be dried and heat-set in the conventional manner.

EXAMPLE XIII

An interlock circular knit fabric having crepe-like aesthetics is prepared using the 30 denier mixed filament yarn of Example XII. Half of the desired quantity of 30–18 yarn is twisted 45 "S" turns per inch and twist set

at 190° F. for 90 min. at 100% RH; the second half of the yarn is twisted 40 "Z" turns per inch and twist set in the same manner. The two halves are then plied 4 "Z" turns per inch and twist set at 220° F. for 90 minutes at 100% RH, in the manner employed with yarns for woven fabrics. The plied yarn can be handled satisfactorily without undesirable twisting and tangling. The yarn is placed on 3° cones with a coning oil and knit on a 28-cut interlock circular knitting machine such as manufactured by Scott and Williams Co. The knitted tube is finished by the following sequence:

(1) Batcher tank scour:

2 passes at room temperature with 0.5 g./l. of a surface active agent Triton X–100
2 passes at 212° F.
1 pass at room temperature

(2) Beck scour at 110° F., with detergent, raising temperature 3°/min. to 180° F. and run for 30 minutes and the beck is cleared. This is followed by a scour with 5 g./l. of DAC–888 (a commercial material containing 80% by weight benzoate and 20% by weight emulsifying agent) for 30 minutes at the boil. The fabric is rinsed, centrifuged dry and dried on a pin tenter at 250° F., with the pins set 1 inch under wet fabric width. The fabric is heat set in a pin tenter with 10% overfeed (30 seconds in each box) at 300° F. (Box 1), 370° F. (Box 2), 250° F. (Box 3).

The fabric can now be printed or beck dyed similar to the warp knit fabric described.

TABLE I.—PREPARATION OF G-PACM YARNS

Example Number	VI	VII	VIII	IX	X	XI
Composition:						
(1)	P-10	P-12	P-9	P-9	P-10	P-12
(2)	P-10/P-I	P-12	P-12			
Percent t-t	55//55	45//80	55//70	55	55	70
RV or $\overline{Mn}$	42//38	17,000//11,000	41//60	41	42	43
Component percent by weight	50//50	50//50	50//50	100	100	100
Melt temperature,° C	320	305	309	309	320	330
Spinneret temperature,° C	350	305	335	335	350	
Quench chamber:						
Steam flow, lbs./hr	0.4		0.4	0.4	0.4	0.4
Temperature,° C	300		300	300	300	330
First roll speed, yds./min	2,100	910	2,000	2,000	2,100	1,800
First-stage draw roll, yds./min	3,000	1,800	3,000	3,000	3,000	3,000
Second-stage draw roll and anneal:						
Yds./mi	2,847	1,650	2,846	2,846	2,847	2,918
Temperature,° C	195	125	160	160	195	120
Let-down roll, yds./min	3,000	1,650	2,866	2,866	3,000	2,932
Wind-up, yds./min	2,954	1,640	2,800	2,806	2,954	2,853

TABLE II.—PROPERTIES OF G-PACM YARNS BEFORE TWISTING

Example Number	VI	VII	VIII	IX	X	XI
Composition:						
(1)	P-10	P-12	P-9	P-9	P-10	P-12
(2)	P-10/P-I	P-12	P-12			
Yarn denier	60	60	60	30	30	30
Number of filaments	18/18	18/18	18/18	18	18	18
Modification ratio	1.65//1.66	2.3/1.9	1.35//1.60	1.35	1.65	1.31
Tenacity, g./denier	3.7	2.8	3.8	3.2	3.9	3.7
Elongation, percent	33	28	31	25	34	37
Orientation index	3.1//1.4	1.4//2.1	0.9//1.7	0.9	3.1	0.9
Skein shrinkage in—						
Water at 100° C						
With 4 mg./denier load	7.3	6.6	12.6	10.5	[1] 6.6	[1] 8.1
DFL, percent	1.0	5.2	8.7			

[1] No restraining load.

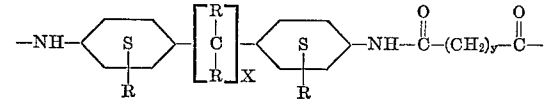
TABLE III.—PROPERTIES OF G-PACM TWIST-SET YARN

Example No	VI	VII	VIII	IX	X	XI
Composition:						
(1)	P-10	P-12	P-9	P-9	P-10	P-12
(2)	P-10/P-I	P-12	P-12			
Turns/inch	60	60	60	60	60	60
Twist set temp., ° F	220	220	220	220	220	220
Wet torque dev., T_{rw}, dyne-cms.	8.6	8.5	11.1	1.15	0.9	2.75
Wet modulus, E_w, dynes/cm.^{2}	0.9×10^9	2.0×10^9	1.2×10^9	0.32×10^9	2.20×10^9	2.8×10^9
Bending modulus, I_b	12.33×10^{-12}	16.39×10^{-12}	13.69×10^{-12}	5.77×10^{-12}	6.12×10^{-12}	6.19×10^{-12}
Buckling index, B	7,747	2,599	6,757	6,224	668	1.586
Liveliness multiplier, L.M	[1]	[1]	[1]	[1]	[1]	[1]

[1] Below 0.4.

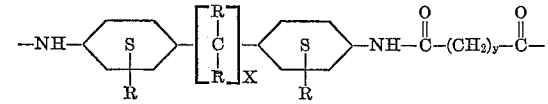
I claim:

1. A crepe fabric comprised of filaments of polyamides wherein at least 50 mol percent of the repeating units have the formula:



wherein X represents 1 or 2, y represents an integer in the range of 7 to 14, R represents the same or different members of the class consisting of hydrogen and methyl and at least 40% of the diamino constituent in said repeating units being of trans-trans stereoisomer.

2. A twist-set yarn comprised of continuous filaments of polyamides wherein at least 50 mol percent of the repeating units have the formula:



wherein X represents 1 or 2, y represents an integer in the range of 7 to 14, R represents the same or different members of the class consisting of hydrogen and methyl and at least 40% of the diamino constituent in said repeating units being of the trans-trans stereoisomer, said yarn having a twist multiplier above 1.5, a liveliness multiplier less than 0.4, and a buckling index of at least 300.

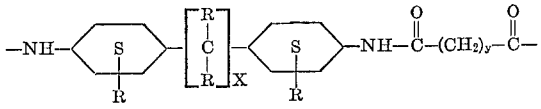
3. The yarn of claim 2 characterized by a twist multiplier of about 3.2.

4. The yarn of claim 2 characterized by a buckling index of at least 1,000.

5. The yarn of claim 2 wherein X is 1, R is H and y is 10.

6. The yarn of claim 2 wherein about 50% of the filaments are homopolymer and about 50% are copolymer.

7. A process comprising extruding a plurality of filaments of a polyamide wherein at least 50 mol percent of the repeating units have the formula:



wherein X represents 1 or 2, y represents an integer in the range of 7 to 14, R represents the same or different members of the class consisting of hydrogen and methyl and at least 40% of the diamino constituent in said repeating units being of the trans-trans stereoisomer, drawing the filaments, twisting the filaments to a twist multiplier above 1.5 and heat treating the twisted yarn at a temperature of between 55% and the second order transition temperature T_g of the polymer to a liveliness multiplier of less than 0.4.

8. The process of claim 7 wherein the draw ratio in the drawing step is between about 1.2 to 2.0×.

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JOHN PETRAKES, Primary Examiner

U.S. Cl. X.R.

57—157; 260—78

PO-1050
(5/69)

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,564,834 Dated February 23, 1971

Inventor(s) Raymond J. Elia

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 15, line 9, Claim 3, "about" should read -- above

Column 16, line 8, Claim 7, "55%" should read -- 55°C. -

Signed and sealed this 15th day of June 1971.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents