

[54] COMPOSITE FILAMENT
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[56] **References Cited**

UNITED STATES PATENTS			
3,399,108	8/1968	Olson.....	161/175
3,551,277	12/1970	Tamura.....	161/175
3,607,610	9/1971	Fukuma.....	161/175
3,620,904	11/1971	Tamura.....	161/175
3,779,853	12/1973	Olson.....	161/175

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[30] **Foreign Application Priority Data**
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 [58] Field of Search 161/172, 173, 175, 177; 260/78 R, 78 S, 857 R; 28/72 HR

[57] **ABSTRACT**
 A crimpable composite filament wherein the one component is homogeneous polyamide and the other random copolyamide, is provided. The obtained filament is superior in crimp developing ability and the knit from the filament is especially suitable for, among others, stretch hosiery.

9 Claims, 7 Drawing Figures

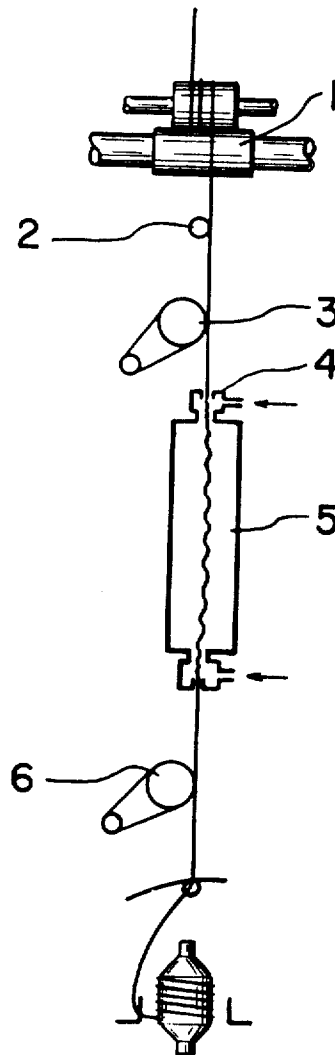


Fig. 1

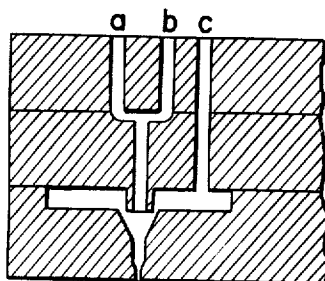


Fig. 2

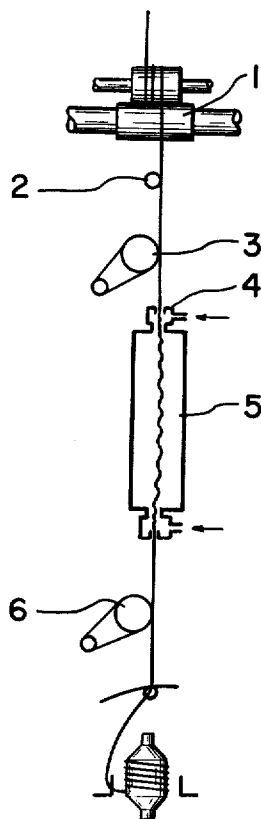


Fig. 3

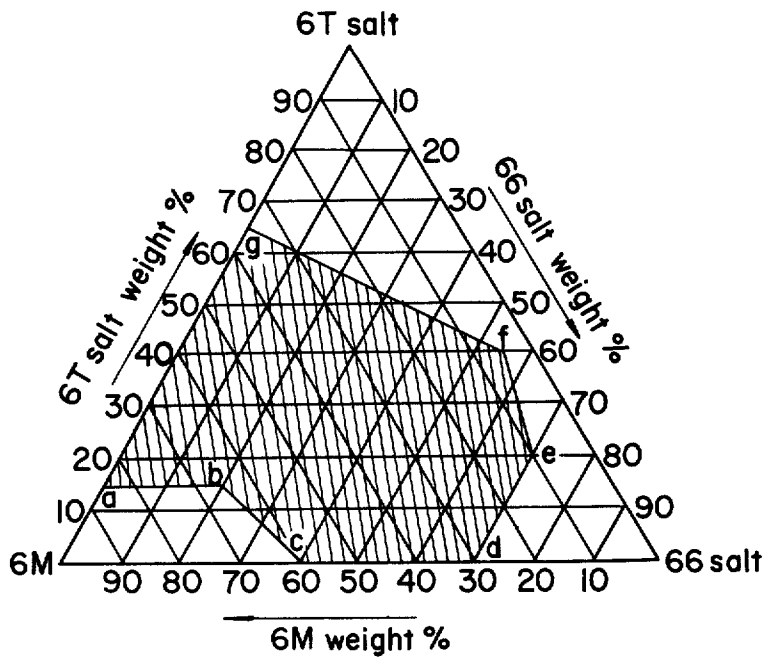


Fig. 4

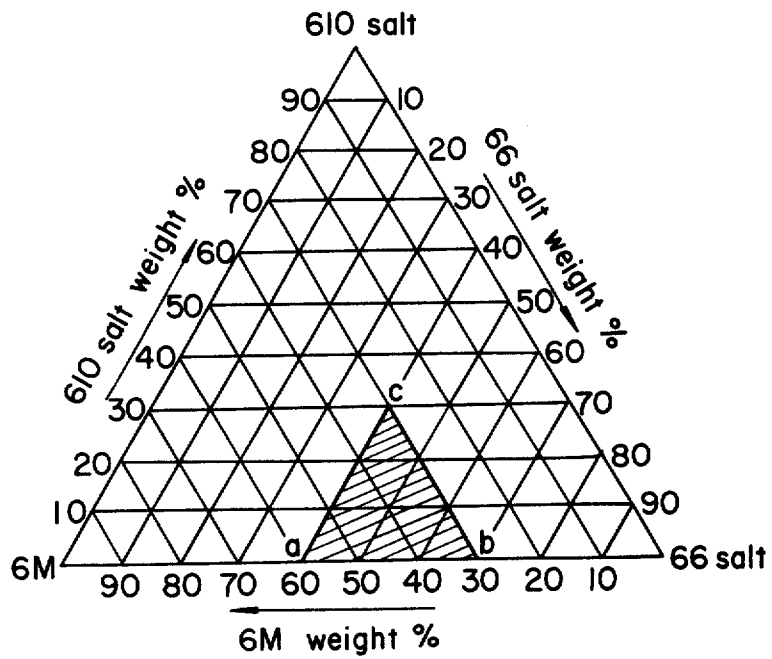


Fig. 5

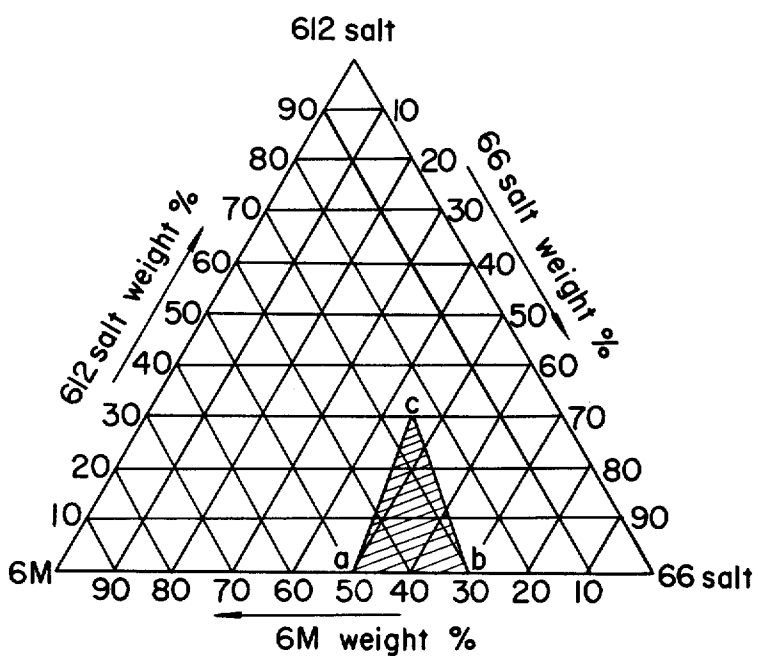


Fig. 6

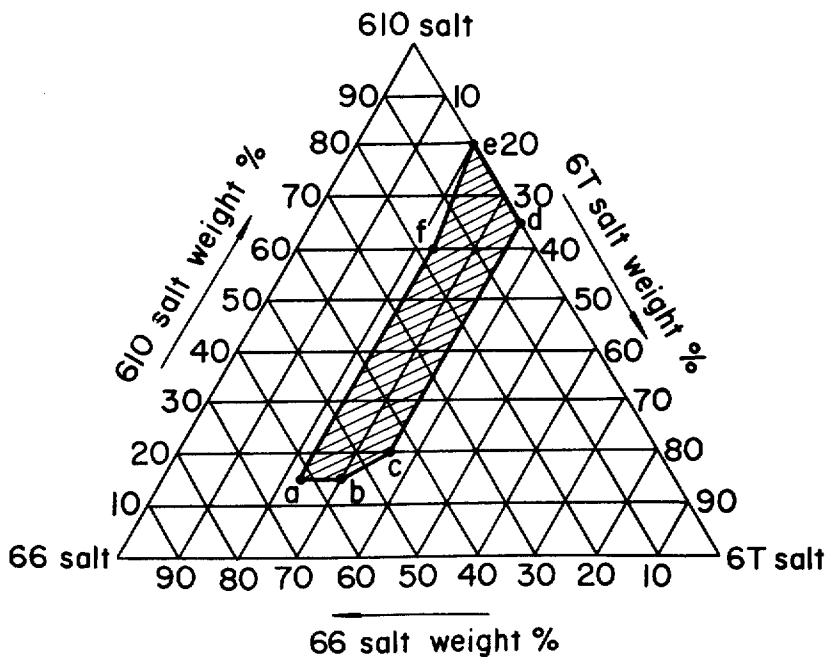
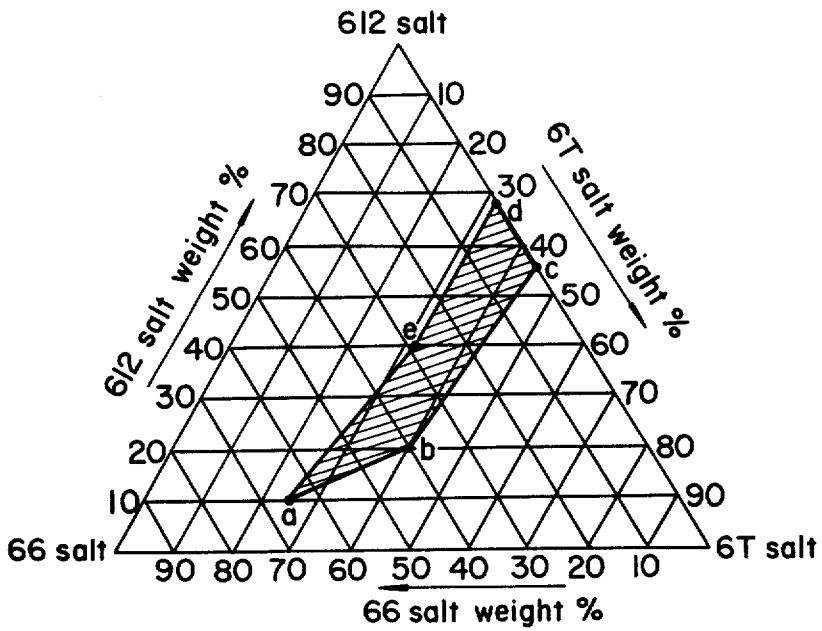


Fig. 7



COMPOSITE FILAMENT

BACKGROUND

Heretofore, composite filament arranged in side-by-side or kidney shape in cross sectional view of the fiber was proposed for the crimpable composite fiber.

However, these known crimpable composite fibers show only insufficient crimp and cannot retain satisfactory bulk and crimp, due to restrained force when knitted.

Further, shrinkage in boiling water of these known fiber was low. Preferred shrinkage properties were especially required for panty stocking yarn, which is one of the predominant uses for this kind of fibers. Filaments with shrinkage in boiling water more than 15 percent necessitate a longer knitted fabric with coarse structure, due to taking into consideration heat shrinkage in boiling water and crimping shrinkage when knitted product is heat relaxed. Accordingly, for these known fibers, the conventional knitting machine can not be used and alteration of the machine such as modification of fabric tube is required. In addition, large knit structure causes disadvantage in later processes such as crimp producing process and tends easily to induce defect in knitted fabric.

To meet the above mentioned requirements, various improvements in making composite filament having less shrinkage and large crimping force were intended by selecting certain types of copolymers or employing improved after-treatment method, etc. These improvements satisfy requirements to some extent, but few of these improvements could satisfy above mentioned two requirements.

For one of these improvements, Japanese Patent Publication No. 25507/1967 was proposed. The gist of this Japanese Patent Publication was:

1. using the composition as the one component substantially comprising crystallizable homogeneous polyamide, and the composition substantially comprising random non-isomorphic copolyamide containing at least 20 percent of each two of contained polymer unit (preferably, one of the polymer units being same with homogeneous polyamide),
2. spinning simultaneously both components to form eccentrically arranged composite filament,
3. drawing thus obtained filament in a condition wherein only homogeneous polymer components can be crystallized,
4. crystallizing copolymerization composition to produce crimp by heat-treating the filament in relax condition and, then
5. stretching the filament to remove crimp.

This process enables to obtain crimpable composite fiber with latent crimp which satisfy the afore-said requirements. However, in this process, since stretching step (5) is necessitated, enhancement of the productivity is immensely difficult. Similarly, for the step (4), except specific copolymer, treatment under severe condition is required for crystallization, and accordingly, physical properties are remarkably lowered.

SUMMARY

This invention relates to crimpable composite filament obtained by

- i. eccentrically forming into fiber, homogeneous polyamide and random copolyamide,
- ii. drawing thus obtained fiber,

iii. heat-relaxing the resultant filament, so as said random copolyamide component not to exhibit crystalline structure by X-ray diffraction and to develop a part of crimp of latent crimpability.

Preferable shrinking percentage in boiling water of thus produced filament is less than 15 percent, most preferably around 10 percent. And number of crimps of thus produced crimpable filament is less than 25, preferably less than 20/25 mm.

Knitted fabric from thus obtained composite fiber develops crimp upon sufficient heat relax treatment by boiling water or steam and crystallization of copolymer occurs. Thus, the obtained fiber is superior product with high crimp developing ability.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 shows the spinneret assembly for composite spinning used for the present invention.

FIG. 2 shows sheet of processing the yarn according to the invention.

FIG. 3 shows triangle co-ordinate diagram for copolyamide 6/66/6T.

FIG. 4 shows triangle coordinate diagram for copolyamide 6/66/610.

FIG. 5 shows triangle coordinate diagram for copolyamide 6/66/612.

FIG. 6 shows triangle coordinate diagram for copolyamide 66/6T/610.

FIG. 7 shows triangle coordinate diagram for copolyamide 66/6T/612.

DETAILED DESCRIPTION

Homogeneous polyamide used for the present invention includes polycapramide (nylon 6), polyhexamethylene diamide (nylon 66), polyundecamide (nylon 11), polydodecamide (nylon 12), polyhexamethylene decamide (nylon 610) and polyhexamethylene dodecamide (nylon 612).

Random copolyamide used for the present invention includes:

1. bicomponent copolymer of 6/66 prepared from ϵ -caprolactam (hereinafter referred to as 6M) and hexamethylene diammonium adipate (hereinafter referred to as 66 salt) with the amount of 6M in the range of 30-60 weight percent;
2. tricomponent copolymer of 6/66/6T prepared from 6M, 66 salt and hexamethylene diammonium terephthalate (hereinafter referred to as 6T salt), wherein its composition is represented by the area covered by *a, b, c, d, e, f*, and *g* in FIG. 3, with the amount of 6M, 66 salt and 6T at each point of *a, b, c, d, e, f*, and *g* being respectively represented by weight as 85:0:15, 65:20:15, 60:40:0, 30:70:0, 10:70:20, 5:55:40 and 35:0:65;
3. tricomponent copolymer of 6/66/610 prepared from 6M, 66 salt and hexamethylene diammonium sebacate (610 salt), wherein its composition is represented by the area covered by *a, b* and *c* in FIG. 4, with the amount of 6M, 66 salt and 610 salt at each point of *a, b* and *c* being respectively represented by weight as 60:40:0, 30:70:0 and 30:40:30.
4. tricomponent copolymer of 6M, 66 salt and the salt of hexamethylene diamine and dodecan dicarboxylic acid (612 salt), wherein its composition is represented by the area covered by *a, b* and *c* in FIG. 5, with the amount of 6M, 66 salt and 612 salt at each point of *a, b* and *c* respectively represented by weight as 50:50:0, 30:70:0 and 25:45:30.

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5. tricomponent copolymer of 66/6T/610 prepared from 66 salt, 6T salt and 610 salt, wherein its composition is represented by the area covered by *a, b, c, d, e*, and *f* in FIG. 6 with the amount of 66 salt, 6T salt and 610 salt at each point of *a, b, c, d, e*, and *f* being respectively represented by weight as 62.5:22.5:15, 55:30:15, 45:35:20, 0:35:65, 0:20:80 and 17.5:22.5:60;

6. tricomponent copolymer of 66/6T/612 prepared from 66 salt, 6T salt and 612 salt, wherein its composition is represented by the area covered by *a, b, c, d* and *e* in FIG. 7, with the amount of 66 salt, 6T salt and 612 salt at each point of *a, b, c, d* and *e* being respectively represented by weight as 65:25:10, 40:40:20, 0:45:55, 0:32.5:67.5 and 27.5:32.5:5.40.

The components outside above specified composition cannot satisfy the afore-said requirement for copolyamide, and therefore are excluded from the present invention.

For example, tricomponent copolymer of 6/66/6T having copolymerization ratio by weight of 5/70/25 or 5/60/35, tricomponent copolymer of 66/6T/610 having copolymerization ratio by weight of 60/30/10, 40/40/20, 20/40/40 and 10/40/50, tricomponent copolymer of 66/6T/612 having copolymerization ratio by weight of 70/25/5, 60/30/10, 35/45/20, 20/50/30 or 10/50/40; etc can satisfy the crystallization condition for the present invention, but cannot produce enough crimp for crimp yarn, due to small shrinkage in boiling water which eventually causes only less than 10 percent of difference of shrinkage in boiling water between that of homogeneous polyamide.

On the other hand, tricomponent copolymer of 6/66/6T having copolymerization ratio by weight of 25/72.5/2.5 or 80/10/10; tricomponent copolymer of 6/66/610 with copolymerization ratio by weight of 20/70/10, 20/60/20, 20/50/30 or 15/45/40; tricomponent copolymer of 6/66/612 with copolymerization ratio by weight of 20/50/30, 20/60/20 or 20/70/10; tricomponent copolymer of 66/6T/610 with copolymerization ratio by weight of 20/20/60, 30/20/50, 40/20/40, 50/20/30, 70/10/20, 50/10/40, 30/10/60 or 20/10/70; tricomponent copolymer of 66/6T/612 with copolymerization ratio by weight of 20/30/50, 35/30/35, 65/20/15, 40/20/40, 10/20/70 or 30/10/60; etc can satisfy the requirement of shrinkage in boiling water as the copolymer of the invention, but cannot satisfy the crystallization condition specified in the present invention.

Besides, tricomponent copolymer of 6/66/6T with copolymerization ratio by weight of 10/85/5, 10/80/10, 5/85/10, 5/90/5, 90/5/5, 85/10/5, or 85/5/10 can not satisfy both of crystallization condition and shrinkage in boiling water for copolymer specified according to the present invention.

Further, tricomponent copolymer of 6/66/6T with copolymerization ratio of 20/20/60 or 25/20/65 which satisfy the condition of crystallization condition and shrinkage in boiling water, extremely lowers spinning ability and drawing ability, and therefore excluded from the present invention.

Random copolymer used for the present invention should be the copolymer, fiber from which shows more than 10 percent of the difference in thermal shrinkage between the fiber from homopolyamide and cannot be turned into crystalline structure as described in Japanese Patent Publication 25507/1967 upon exposure of

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steam at 100° - 200°C for 10⁻⁴ - 0.5 second in relaxed status, does not show by densitomer X-ray reflection characteristics along equatorial direction in the plane of (100), and (010, 110) and accordingly shows only one peak instead showing inherently distinct two peaks and, upon relax treatment by boiling water or steam forms crystalline or pseudo-crystalline structure.

Drawing of random copolymer filament selected for the present invention by ordinary thermal drawing condition of polyamide fiber does not give rise to crystallization of polymer.

Heretofore, for the typical copolymer component for crimpable composite fiber, copolyamide of 66/610 with copolymerization ratio being 30-70 percent, copolyamide of 66/61(hexamethylene diammonium isophthalate) with copolymerization ratio of 60-97 percent, copolyamide of 66/6 with copolymerization ratio of 5-20 percent were proposed and these copolyamides possess satisfactory thermal shrinkage.

However, in case of these copolyamides crystallization proceeds by heat relax treatment after drawing, and in order to utilize thermal shrinkability for the effective production of crimp, it was necessary to produce enough crimp at this stage. As a result, removal of crimp by stretching after heat relax treatment was necessary. Besides, reduction of crimp production at the heat relax treatment step with the aim of eliminating stretching step of (5), remarkable crystallization of copolymer in that step proceeds and therefore thermal shrinkage of copolymer cannot be utilized for effective crimping. And thus sufficient crimp cannot be ensured by the treatment of crimp production for knit.

In other words, for these known typical copolyamides, in order to develop as much crimp as possible at the heat relax treatment step, it was most effective to utilize for crimp production thermal shrinkage of copolymer and consequently, removal of crimp at stretching step of (5) was indispensable.

On the contrary, according to the present invention no crystallization proceeds during heat relax treatment step by partially developing crimp and at this step residual crimpability can be retained. Thus in the present invention, no difference in crimp which is induced by total production of crimp at this step is observed and there is no need to develop excessive crimp, thereby simultaneously satisfying crimp developing ability, shrinkage in boiling water and suitability for knit fabric or hosiery goods.

In the heat relax treatment, it is most effective to develop 5-30 percent of crimp from the view points of crimp developing ability, shrinking percentage in boiling water, productivity of yarn and suitability for knit.

Composite filament having these properties can be obtained by introducing the yarn to heat-relax treatment zone under steam or heated gaseous fluid, which zone is provided after draw step. Drawn filament was passed through chamber or tube under steam or fluid heated at 100°-200°C, for less than 0.5 second. At this step, filament is fed to the chamber to obtain relax percentage of the filament being more than 15 percent, preferably 20-25 percent. By employing this heat-relax condition mentioned above, shrinking percentage in boiling water of the filament can be lowered to around 10 percent, while substantially avoiding crystallization of random copolymer component of the fiber. Thus, commercially feasible high production speed of more than 800 m/min, preferably 800-1000 m/min. can be attained. FIG. 2 shows preferable example of heat relax

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treatment zone. Undrawn filaments are fed from drawing zone (including delivery roll 3) to heat treatment chamber 5. This chamber is equipped with an inlet for filament. Steam or other heated fluid is blown from a nozzle part in an arrow direction. Heat-relax condition of the filament can be kept excellent by blowing simultaneously from both of the inlet and the outlet. Filaments passed through this heat treatment zone are wound on a bobbin by a spindle.

As mentioned above, in the present invention copolymerization component is not crystallized during heat relax treatment step and there is no need to completely develop crimp at this step. Accordingly, stretching step to remove crimp is not necessitated and treating speed is remarkably enhanced up to 1200 m/min, due to mild heat relax treatment.

The present invention is further illustrated by the following examples.

In the example, shrinking percentage is calculated according to the following equation:

$$\text{Shrinking percentage in boiling water (\%)} = \frac{L - L_1}{L} \times 100(\%)$$

wherein,

L is the length of the skein prepared by winding 50 loops and then suspending with a weight of 0.1 g/d, and

L₁ is the length of skein by removing the weight suspended as described above for 5 minutes, allowing to dry in air 24 hours and suspending with a weight of 0.1 g/d.

Crimp developing ability is calculated according to the following equation:

$$\text{Crimping developing ability} = \frac{m - m_1}{m} \times 100(\%)$$

wherein

m₁ is the length of the skein prepared by winding the composite filaments yarn in 50 loops, applying by steam atmosphere at normal pressure for one minute under suspension with a weight of 0.0012 g/d, allowing to dry in air for 60 minutes under this weight,

and

m is the length of the skein by removing the weight of 0.0012 g/d, allowing to stand for 1 minute and then suspending a weight of 0.33 g/d for one minute.

The number of crimps means number of crimps in 25mm wherein the crimp filament is suspended with a weight of 2 mg/d, by counting from the one peak to the other peak as one crimp or from the one trough to the other trough as one crimp.

Relax percentage is calculated according to the following equation: n

$$\text{Relax (\%)} = \frac{V - V_1}{V} \times 100(\%)$$

wherein,

V is the speed of the drawing roll, and V₁ is the speed of the delivery roll.

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Relative viscosity ($\eta_{rel.}$) of the polymer is viscosity of the solution whereby 1g of polymer is dissolved into 100ml. of 95.5 percent of sulfuric acid.

In the following examples, measurement of stretch-back was conducted as in Example 1.

EXAMPLE 1

To 120 kg of 50 percent aqueous solution of hexamethylene diammonium adipate (hereinafter referred to as 66 salt) in an 400-l autoclave was added and dissolved 40 kg of ϵ -caprolactam (hereinafter, referred to as 6M) to obtain the weight ratio of 66 salt to 6M being 60/40. Then the inside of the autoclave was purged with nitrogen for the polymerization. The autoclave was heated at 230°C for 1 hour, while gradually removing steam and keeping the inside pressure at 17.5 kg/cm². Then the inside pressure was brought from 17.5 Kg/cm² to an atmospheric pressure over a period of 1 hour and heating was continued for 1 hour while flowing nitrogen at an atmospheric pressure. From thus obtained random copolyamide 66/6 (copolyamide of polyhexamethylene adipamide and polycapramide) having relative viscosity of 2.43 as the one component and homopolyamide 66 (polyhexamethylene adipamide hereinafter referred to as Nylon 66) having relative viscosity of 2.47 as the other component, composite spinning in the same volume ratio of the components was conducted through a kidney-type composite spinneret having 5 orifices, each with a diameter of 0.3 mm ϕ .

In FIG. 1, the random copolyamide 66/6 component is supplied from groove (2), and the homopolyamide from grooves (b) and (c). As shown in FIG. 2, thus obtained non-spun filaments are spun to 3.5 times through feed roll 1 and draw roll 3 and then heat relaxed by introducing the filament yarn to 30 cm long-heating chamber 5 under steam kept at 155°C, wherein steam was introduced from ejector 4 at the top of heating chamber 5 and the supply speed of the yarn was 800 m/min. and the yarn was heat-relaxed at a rate of 21 percent of relax in the heating chamber 5 by adjusting the speeds of draw roll 3 and delivery roll 6 to give 21d/5f composite filament developing 20 percent of latent crimp (number of crimps being 11/25mm).

For comparison, undrawn yarn of the composite filaments of (II) prepared in the similar manner as described above, was drawn to 3.5 times to obtain 20d/5f. Crimp developing ability and shrinking percentage in boiling water of these filaments (I) and (II) were measured. Also, evaluation was made by the following way. The filament was knitted by a circular knitting machine with a diameter of 3 $\frac{3}{4}$ inch and having 400 needles into plain stitch fabric under knitting tension of 1 g and with the course of 62/inch and wale of 32/inch. The fabric was subjected to heat relax treatment for 5 minutes in boiling water at 100°C, followed by the steps of allowing to dry in air elevating the heating temperature from normal temperature to 100°C over 40 minutes, continuing heating treatment at 100°C for 30 minutes, reducing the temperature to 60°C over 15 minutes, and then dried in air for 24 hours. Stretch-back was measured in term of tension (g), when stretched to 50 percent.

The results were shown in Table 1.

Table 1

sample	Properties of yarn			Evaluation of knit		
	Crimp	Developping Ability	Shrinkage percentage in Boiling Water	Crimpability	Crimp Irregularity	stretch back
I	23%		9%	good	none	97
II	7%		18%	poor	many streak irregularities	54

As is clearly seen from the result, the treatment according to the invention remarkably enhances crimp producing ability, restrains shrinking percentage in boiling water and gives extremely excellent properties for knitting. On the other hand the single component filament prepared only from copolyamide component of the composite fiber (I), was in the same condition as described above drawn and subjected to heat-relax treatment, and then X-ray measurement according to Japanese Patent Publication No. 25507/1967 was conducted. Each of the testing results after drawing filament subjecting to the heat-relaxed treatment and treating by boiling water were summarized in Table 2. The result shows crystallization of said copolyamide in the process of heat-relaxed treatment.

under steam atmosphere at 160°C, wherein the yarn was heat-relaxed in the heating chamber 5 with a speed of 800m/min. And with 22 percent of heat-relax percentage to give 22d/5f composite filament (III) developing 20 percent of latent crimp (number of crimp being 11/25mm).

On the other hand, the same procedure was followed except that using copolyamide with a copolymerization of hexamethylene diammonium adipate to ϵ -caprolactam being 30/70 by weight and having relative viscosity of 2.41. And there was obtained a composite fiber (IV) of 22d/5f with number of crimps being 12/25mm. Crimp developing ability and the evaluation of the fibers (III) and (IV) after being knitted and treated by boiling water were shown in Table 3.

Table 2

After drawing	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter trace. (one peak)
After heat-relax treatment	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter (one peak)
After boiling-water treatment	Crystalline structure was observed. Separation of two peaks at 100 and 010, 110 was clearly observed by densit-o-meter.

EXAMPLE 2

From the copolyamide prepared as in Example 1 with a copolymerization ratio of hexamethylene diammo-

In addition, single-component filaments of copolyamide ingredients of the fibers (III) and (IV) of the fibers (III) and (IV) were subjected to X-ray measurement. The result was summarized in Table 4.

Table 3

sample	Properties of yarn		Crimpability	Evaluation of knit		
	Crimp	Developping Ability		Shrinking Percentage	Crimp irregularity	stretch back
III	24%		9%	good	none	101
IV	15%		9%	poor	some streak irregularities	53

Table 4

Sample	III	IV
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)
After heat relax treatment	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace.	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace

nium adipate to ϵ -caprolactam being 70/30 by weight and having a relative viscosity of 2.43 as the one component and nylon 66 as the other component, composite spinning as in Example 1 in the same volume ratio of the components was conducted through a kidney-type composite spinneret as shown in FIG. 1 having 5 orifices, each with a diameter of 0.3 m ϕ . Thus obtained undrawn filaments were spun to 3.5 times and heat-relaxed by introducing the drawn yarn at a relax percentage of 21 percent to 30cm long-heating chamber

For comparison, the yarn having the same composition of (IV) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication 25507/1967 to obtain composite fiber of 22d/5f. The knitted fabric from thus obtained composite fiber was treated as above mentioned. Crimpability and stretching back ability were good. Crimp developing ability and shrinking percentage in boiling water were 22 percent and 8 percent respectively.

From the above result, in view of crimp developing ability, stretch back of knit and crimping status yarns to be crystallized in heat relax step do not show good result by the treatment of the present invention and show good result by heat-relaxed-stretching treatment as described in Japanese Patent Application No. 25507/1967. Thus, the copolymer of the present invention gives good result only by satisfying the crystallization condition described in the present specification.

EXAMPLE 3

The same procedure for preparing composite yarn as in Example 1 was followed except that the copolyamide of nylon 66/6 with a copolymerization ratio of 40/60 and having a relative viscosity of 2.48 was employed. From thus obtained non-spun filament composite filaments yarn of 21d/5f was obtained by following the same procedure for sample (I) of Example 1, except by employing heat-relax treatment in an atmosphere of hot air at 175°C. Testing result was 22 percent for crimp developping ability and 11 percent for shrinking percentage in boiling water. Evaluation of knitted fabric and X-ray measurement gave substantially same result as that for sample (I) of Example 1.

EXAMPLE 4

To 135 Kg of 40 percent aqueous solution of hexamethylene diammonium adipate (66 salt) in a 400-l autoclave, was added and dissolved 16 Kg of hexamethylene diammonium terephthetele (6T salt) and subsequently was added and dissolved 30 Kg of ϵ -caprolactam (6M), to obtain the weight ratio of 6M : 66 salt : 6T salt = 30 : 54 : 16. Then the inside of the autoclave was purged with nitrogen for polymerization. The autoclave was heated at 230°C for 1 hour, while gradually removing steam and keeping the inside pressure at 17.5 Kg/cm². Then the inside pressure was brought from 17.5 Kg/cm² to an atmospheric pressure over a period of 1 hour and heating was continued for 1 hour while flowing nitrogen at an atmospheric pressure for about 1 hour. From thus obtained tricomponent copolyamide of 6/66/6T having relative viscosity of 2.51 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning in the same volume ratio of the components was conducted through a kidney-type composite spinneret (in FIG. 1) having 5 orifices, each with a diameter of 0.3 mm ϕ .

In FIG. 1, the copolyamide component is supplied from groove (a), and nylon 66 from grooves (b) and (c). As shown in FIG. 2, thus obtained undrawn filaments are spun to 3.3 times through feed roll 1 and draw roll 3 and then heat relaxed by introducing the drawn yarn to 30 cm long-heating chamber 5 under steam at 160°C supplied from ejector 4 at the top of heating chamber 5 wherein the yarn was supplied at a speed of 700 m/min and was heat-relaxed at a rate of 23 percent in the heating chamber 5 by adjusting the

speeds of draw roll 3 and delivery roll 6 to give 22d/5f composite filament developing 25% of latent crimp (number of crimps being 17/25mm).

Crimp developing ability and shrinking percentage in boiling water of the fiber were 25 percent and 8 percent, respectively. Evaluation of crimpability and stretch-back ability for knitted fabric of the yarn were good. The result of X-ray measurement was summarized in Table 5.

Table 5

After Drawing	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter trace. (one peak)
After heat-relaxed Treatment	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter (one peak)
After boiling-water Treatment	Crystalline structure was observed. Separation of two peaks at 100 and 010, 110 was observed by densit-o-meter

EXAMPLE 5

From the tricomponent random copolyamide 6/66/6T prepared as in Example 4 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium terephthalate being 20/50/30 by weight and having relative viscosity of 2.47 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 4 was conducted. Thus obtained undrawn filaments were drawn to 3.4 times on a drawing pin heated at 110°C (2 in FIG. 2) and then by introducing the drawn yarn at a relax percentage of 21 percent to 30 cm long-heating chamber 5 under steam at 173°C wherein steam was supplied from heat ejector at the top of heating chamber 5. The yarn was supplied to the heating chamber 5 under steam at 173°C with a speed of 1100 m/min. to obtain 21d/5f composite filament with 15% of latent crimp (number of crimps being 9/25mm). Crimp developing ability and shrinking percentage in boiling water of the filaments yarn were 24 and 11 percent, respectively. Evaluation of knitted fabric and X-ray measurement gave substantially same result as that of Example 4.

EXAMPLE 6

From the tricomponent random copolyamide 6/66/6T prepared as in Example 4 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium terephthalate being 30/65/5 by weight and having relative viscosity of 2.48 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite filament (III) of 22d/5f with number of crimps being 15/25mm was obtained in the manner as described in Example 4.

On the other hand, using random copolyamide with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium terephthalate being 80/10/10 and having relative viscosity of 2.46, the composite filament of 22d/5f (IV) with number of crimps being 14/25mm was obtained by treating in the same manner as above-mentioned.

Crimp developing ability of filaments and evaluation of knitted prepared and treated by boiling water, as in Example 1, of the filament (III) and (IV) were shown in Table 6. In addition, X-ray measurement was made, as

in Example 1, for the fibers (III) and (IV) and the result was shown in Table 7.

EXAMPLE 8

Table 6

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp irregularity	Stretch back
	Crimp Developing Ability	Shrinking Percentage			
III	24%	8%	good	none	113
IV	17%	8%	poor and rough crimp	some streak irregularities	56

Table 7

sample	III	IV
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)
After heat relax treatment	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace	Possesses crystalline structure separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace

For comparison, the yarn having the same composition of (IV) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication 25507/1967 to obtain composite fiber of 22d/5f. The knitted fabric from thus obtained composite filaments yarn was treated as mentioned above. Crimpability and stretching back ability of knitted fabric were good. Crimp developing ability and shrinking percentage in boiling water of the yarn were 23 and 8 percent, respectively.

From the above results, in view of crimp developing ability, stretch back of knitted fabric and crimping status, yarns composed of crystallizable copolymer in the heat relax step do not show good result by the heat-relax treatment of the present invention but show good result by heat-relaxed-stretching treatment as described in Japanese Patent Application 25507/1967. Thus the material of the present invention gives good result only by using specific random copolymer having the crystallization condition, as described in the present specification.

EXAMPLE 7

The same procedure for preparing composite yarn as in Example 4 was followed except that the copolyamide with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium terephthalate being 40/10/50 by weight and having relative viscosity of 2.50 was employed. Thus obtained undrawn composite filament was drawn to 3.3 times by a drawing pin (2 in FIG. 2) heated at 110°C, introduced to a 30 cm long-heating chamber 5 under steam at 160°C wherein steam was supplied from objector 4 at the top of heating chamber 5, and then heat-relaxed, wherein the yarn was supplied to the heating chamber 5 with a speed of 800 m/min. and heat relaxed at 21 percent of relax, to obtain 23d/5f composite filament developing 20 percent of crimp (number of crimps being 14/25 mm). Crimp developing ability and shrinking percentage in boiling water of the filaments yarn were 25 and 10 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 4.

The composite fiber of 21d/5f was prepared in the same manner as in Example 4 except that undrawn filaments yarn obtained in Example 4 was heat-relaxed in an hot-air atmosphere at 180°C. Crimp developing ability and shrinking percentage in boiling water of the filament were 23 and 8 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 4.

EXAMPLE 9

Undrawn composite fibers were prepared following the same manner as in Example 4, except that nylon 6 was used as homogeneous polyamide, and heat-relaxed in the similar manner to obtain composite filaments of 22 d/5f with number of crimps being 15/25mm.

Crimp developing ability and shrinking percentage in boiling water of the fiber were 23 and 9 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 4.

EXAMPLE 10

To 150 Kg of 40 percent aqueous solution of hexamethylene diammonium adipate (66 salt) in an 400-l autoclave, was added and dissolved 5 Kg of hexamethylene diammonium sebacate and subsequently was added dissolved 35 Kg of ϵ -caprolactam (6M) to obtain the weight ratio of 6M : 66 salt : 610 salt = 35 : 60 : 5. Then the inside of the autoclave was purged with nitrogen for polymerization. The autoclave was heated at 230°C for 1 hour, while gradually removing steam and keeping the inside pressure at 17.5 Kg/cm². Then, the inside pressure was brought from 17.5 Kg/cm² to an atmospheric pressure over a period of 1 hour and heating was further continued for 1 hour while flowing nitrogen at an atmospheric pressure. From thus obtained tri-component random copolyamide of 6/66/610 having relative viscosity of 2.50 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning in the same volume ratio of the components was conducted through a kidney-type composite spinneret (in FIG. 1) having 5 orifices, each with a diameter of 0.3 mm ϕ .

In FIG 1, the copolyamide component is supplied from groove (a), and nylon 66 from grooves (b) and (c). As shown in FIG. 2, thus obtained undrawn filaments are drawn to 3.5 times through feed roll 1 and

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draw roll 3 and then heat relaxed by introducing to 25 cm long-heating chamber 5 under steam kept at 155°C wherein steam was supplied from ejector 4 at the top of heating chamber 5 and whereby the yarn was supplied at a speed of 800 m/min. and the yarn was heat-relaxed at a rate of 22 percent in the heating chamber 4 by adjusting the speeds of draw roll 3 and delivery roll 6 to give composite fiber (V) of 22d/5f with number of crimps being 15/25 mm.

For comparison, undrawn yarn of the composite filaments of (VI) prepared in the similar manner as described above, was drawn to 3.5 times to obtain 20d/5f. Table 8 shows crimp developing ability and shrinking percentage in boiling water of these filaments (V) and (VI), and evaluation of knitted fabric of respective filaments yarn with the same condition as in Example 1.

Table 8

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp irregularity	Stretch back
	Crimp Developing Ability	Shrinking Percentage			
V	23%	11%	good	none	102
VI	7%	18%	poor	many streak irregularities	57

As is clearly seen from the result, the treatment according to the invention remarkably enhances crimp developing ability, restrains shrinking percentage in boiling water and gives extremely excellent properties for knitting.

On the other hand, the single-component filament prepared only from random copolyamide components of the composite fiber (V) was, in the same condition as described above, drawn and subjected to heat-relax treatment, and then X-ray measurement according to Japanese Patent Publication No. 25507/1967 was conducted. Each of the testing results after drawing filament, subjecting to the heat-relax treatment and treating by boiling water were summarized in Table 9. The result shows no crystallization of said copolyamide during heat-relax treatment.

Table 9

After drawing	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter trace. (one peak)
After heat-relaxed treatment	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter (one peak)
After boiling water treatment	Crystalline structure was observed. Separation of two peaks at 100 and 010, 110 was clearly observed by densit-o-meter

EXAMPLE 11

From the random tricomponent copolyamide 6/66/610 prepared as in Example 10 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium sebacate being 50/45/5 by weight and having relative viscosity of 2.53 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 10 was conducted.

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Thus obtained undrawn filaments were drawn to 3.3 times through feed roll 1 and draw roll 3 as shown in FIG. 2 and then introduced to 30 cm long-heating chamber 5 under steam at 165°C supplied from heat ejector 4 at the top of heating chamber 5, wherein the yarn was supplied to the heating chamber 5 with a speed of 1000 m/min. and heat relaxed at 20 percent of relax by adjusting the speeds of draw roll 3 and delivery roll 6, to obtain 21d/5f composite filament producing 10 percent of crimp (number of crimps being 7/25 mm.)

Crimp developing ability and shrinking percentage in boiling water of filaments were 23 and 11 percent, respectively. Evaluation of knitted fabric and X-ray measurement gave substantially same result as that of Example 10.

EXAMPLE 12

From the random tricomponent copolyamide 6/66/610 prepared as in Example 10 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium sebacate being 35/50/15 by weight and having relative viscosity of 2.45 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite filament (VII) of 22d/5f with number of crimps being 14/25mm was obtained in the manner as described for sample (V) in Example 10.

On the other hand, using random copolyamide with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium sebacate being 20/50/30 and having relative vis-

cosity of 2.47, the composite filament of 22d/5f (VIII) with number of crimps being 14/25 mm was obtained by treating in the same manner as above-mentioned.

Crimp developing ability of the filaments and evaluation of knitted fabric, prepared and treated by boiling water as in Example 1, of (VII) and (VIII) were shown in Table 10. In addition, X-ray measurement was made for the fibers (VII) and (VIII), and the result was shown in Table II.

Table 10

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp irregularity	stretch back
	Crimp Developing Ability	Shrinking Percentage			
VII	25%	10%	good	none	96
VIII	16%	9%	insufficient, rough crimp	some streak irregularities	55

Table 11

sample	VII	VIII
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)
After heat relax treatment	Incomplete in crystalline structure. No peak-separation was observed by densit-o-meter (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace.	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace.

For comparison, the yarn having the same composition of (VIII) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication No. 25507/1967 to obtain composite fiber of 22d/5f. The knitted fabric from thus obtained composite fibers was treated as above mentioned. Crimpability and stretching back ability of knitted fabric were good. Crimp developing ability and shrinking percentage in boiling water were 25 and 8 percent, respectively.

From the above results, in view of crimp developing ability, stretch back of knit and crimping status, yarns crystallizable in heat relax step do not show good result by the treatment of the present invention, and show good result by heat-relaxed-stretching treatment as described in Japanese Patent Publication No. 25507/1967. Good result can be obtained only by using the material of the present invention satisfying the condition described in the present specification.

EXAMPLE 13

The same procedure for preparing composite yarn as in Example 10 was followed except that random tri-component copolyamide 6/66/610 with a copolymerization ratio of 30/50/20 by weight and having relative viscosity of 2.49 was used and that nylon 6 having relative viscosity of 2.45 was used as homogeneous polyamide, to obtain undrawn filament. From thus obtained undrawn filament, the composite filaments of 22d/5f with number of crimps 14/25 mm was prepared by following the same manner for sample (V) as described in Example 10, except that the non-spun yarn was drawn and subjected to heat relax treatment in hot air atmosphere at 175°C. Crimp developing ability and shrinking percentage in boiling water of the fiber were 23 and 12 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of the sample (V) of the Example 10.

EXAMPLE 14

To 150 Kg of 40 percent aqueous solution of hexamethylene diammonium adipate (66 salt) in an 400-l autoclave, was added and dissolved 10 Kg of the salt of hexamethylene diamine and dodecane dicarboxylic acid (612 salt) and subsequently was added and dissolved 30 Kg of ϵ -caprolactam (6M), to obtain the weight ratio of 6M : 66 salt : 612 salt = 30 : 60 : 10. Then the inside of the autoclave was purged with nitrogen for polymerization. The autoclave was heated at 230°C for 1 hour, while gradually removing steam and keeping the inside pressure at 17.5 Kg/cm². Then the inside pressure was brought from 17.5 Kg/cm² to an atmospheric pressure over a period of 1 hour and heating was continued for 1 hour while flowing nitrogen at an atmospheric pressure for about 1 hour. From thus obtained random tri-component copolyamide having relative viscosity of 2.45 as the one component and nylon

66 having relative viscosity of 2.47 as the other component, composite spinning in the same volume ratio of the components was conducted through a kidney-type composite spinneret (in FIG. 1) having 5 orifices, each with a diameter of 0.3 mm ϕ .

In FIG. 1, the copolyamide component is supplied from groove (a), and nylon 66 from grooves (b) and (c). As shown in FIG. 2, thus obtained non-spun filaments were spun to 3.5 times through feed roll 1 and draw roll 3 and then heat relaxed by introducing to 30 cm long-heating chamber 5 under steam at 160°C wherein steam was supplied from ejector 4 at the top of heating chamber 5 and the yarn was supplied at a speed of 800 m/min., and the yarn was heat-relaxed at a rate of 23 percent in the heating chamber 5 by adjusting the speeds of draw roll 3 and delivery roll 6 to give 22d/5f composite filament developing 25 percent of crimp with number of crimps being 17/25mm.

Crimp developing ability and shrinking percentage in boiling water of the fiber were 25 and 9 percent, respectively. Evaluation of knitted fabric, crimpability and stretch-back ability were good. The X-ray measurement gave substantially same result as that of the sample (V) of the Example 10.

EXAMPLE 15

From the random tri-component copolyamide prepared as in Example 14 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and salt of hexamethylene diamine and dodecane dicarboxylic acid being 35/60/5 by weight and having relative viscosity of 2.46 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 14 was conducted. Thus obtained undrawn filaments were drawn to 3.5 times and then introduced to 30 cm long-heating chamber 5 under steam atmosphere at 168°C, wherein the yarn was supplied with a speed of 1100 m/min. and heat relaxed at 21 percent of relax to obtain 21d/5f composite filament developing 10 percent of crimp with number of crimps being 7/25 mm.

Crimp developing ability and shrinking percentage in boiling water of the fiber were 24 and 11 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 14.

EXAMPLE 16

From the random tri-component copolyamide 6/66/612 prepared as in Example 14 with a copolymerization ratio of ϵ -caprolactam, hexamethylene diammonium adipate and the salt of hexamethylene diamine and dodecane dicarboxylic acid being 30/55/15 by weight and having relative viscosity of 2.48 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite filaments (IX)

of 22d/5f with number of crimps being 17/25mm was obtained in the manner as described in Example 14.

On the other hand, using random tricomponent copolyamide with a copolymerization ratio of 20/50/30 by weight and having relative viscosity of 2.46 the composite filament (X) of 22d/5f with number of crimps being 16/25mm was obtained by treating in the same manner as above-mentioned. Crimp developping ability and evaluation of knit prepared and treated by boiling water as in Example 1 of (IX) and (X) were shown in Table 12. In addition, X-ray measurement was made for the single-component filament of copolyamide ingredients of the fiber (IX) and (X) and the result was shown in Table 13.

Table 12

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp irregularity	stretch back
	Crimp Developping Ability	Shrinking Percentage			
IX	26%	11%	good	none	103
X	17%	10%	poor	some streak irregularities	51

Table 13

	IX	X
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)
After heat relax treatment	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace.	Possesses crystalline structure. Separation of two peaks in 100 plane and 010,110 plane was clearly observed by densit-o-meter trace.

For comparison, the filaments yarn having the same composition of (X) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication No. 25507/1967 to obtain composite fiber of 22d/5f. The knitted fabric from thus obtained composite fibers was treated as above mentioned. Crimpability and stretching back ability of the knitted fabric were good. Crimp developing ability and shrinking percentage in boiling water of the filaments were 24 and 7 percent, respectively.

From the above results, in view of crimp developping ability and stretch back of knit and crimping status, yarns to be crystallized in heat relax step do not show good result by the treatment of the present invention but, show good result by heat-relaxed-stretching treatment as described in Japanese Patent Publication No. 25507/1967. Good result can be obtained only by using the material of the present invention satisfying the condition described in the present specification.

EXAMPLE 17

The same procedure for preparing composite yarn as in Example 14 was followed except that random tricomponent copolyamide 6/66/612 with a copolymerization ratio of 40/55/5 by weight and having relative viscosity of 2.43. From thus obtained undrawn filament, the composite fiber of 22d/5f with number of crimps being 18/25mm was prepared by following the same manner as described in Example 14, except that undrawn yarn was subjected to heat-relax treatment in hot air atmosphere at 175°C. Crimp developing ability and shrinking percentage in boiling water of the yarn were 24 and 10 percent, respectively. Evaluation of

knitted fabric and X-ray measurement gave substantially same result as that of Example 14.

EXAMPLE 18

To 240 Kg of 25 percent aqueous solution of hexamethylene diammonium sebacate (610 salt) in an autoclave, was added and dissolved 30 Kg of hexamethylene diammonium terephthalate (6T salt) and subsequently was added and dissolved 25 Kg of 40 percent aqueous solution of hexamethylene diammonium adipate (66 salt), to obtain the weight ratio of 66 salt: 6T salt: 610 salt = 10 : 30 : 60. Then the inside of the autoclave was purged with nitrogen for the polymerization. The autoclave was heated at 230°C for 1

hour, while gradually removing steam and keeping the inside pressure at 17.5 Kg/cm². Then the inside pressure was brought from 17.5 Kg/cm² to an atmospheric pressure over a period of 1 hour and heating was continued for 1 hour while flowing nitrogen at an atmospheric pressure. From thus obtained random tricomponent copolyamide of 66/6T/610 having relative viscosity of 2.45 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 1 was conducted. Thus obtained undrawn filaments were drawn to 3.3 times on a drawing pin heated at 110°C (2 in FIG. 2) and then by introducing to 30cm long-heating chamber 5 under steam at 165°C supplied from heat ejector at the top of heating chamber 5, wherein the yarn was supplied to the heating chamber 5 with a speed of 800m/min. and heat-relaxed at 22 percent of relax to obtain 21d/5f composite filament developing 15 percent of crimp (number of crimps being 12/25mm). Crimp developing ability and shrinking percentage in boiling water of the filaments yarn were 23 and 11 percent, respectively. Evaluation of knitted fabric and X-ray measurement gave substantially same result as that for sample (I) of Example 1.

EXAMPLE 19

From the random tricomponent copolyamide 66/6T/610 prepared as in Example 18 with a copolymerization ratio of hexamethylene diammonium adipate, hexamethylene diammonium terephthalate and hexamethylene diammonium sebacate being 15/25/60 by weight and having relative viscosity of 2.43 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite fiber of

22d/5f with number of crimps being 14/25mm (IX) was obtained as in Example 18.

On the other hand, using random copolyamide 66/6T/610 with a copolymerization ratio of 20/20/60 by weight and having relative viscosity of 2.45, the composite filament of 22d/5f (XII) with number of crimps being 14/25mm was obtained by treating in the same manner as above-mentioned.

Crimp developing ability of the filaments yarn and evaluation of knitted fabric prepared and treated by boiling water as in Example 1 of (XI) and (XII) were shown in Table 14. In addition, X-ray measurement was made for the fibers (XI) and (XII) and the result was shown in Table 15.

Table 14

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp Irregularity	stretch back
	Crimp Developing Ability	Shrinking Percentage			
XI	25%	9%	good	none	111
XII	16%	8%	poor	some streak irregularities	60

Table 15

Sample	XI	XII
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one-peak)
After heat relax treatment	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010.110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure. Separation of two peaks in 100 plane and 010.110 plane was clearly observed by densit-o-meter trace.	Possesses crystalline structure. Separation of two peaks in 100 plane and 010.110 plane was clearly observed by densit-o-meter trace.

For comparison, the yarn having the same composition of (XII) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication No. 25507/1967 to obtain composite fiber of 22d/5f. The knitted fabric from thus obtained composite yarn was treated as above mentioned. Crimpability and stretching back ability of the knitted fabric were good. Crimp developing ability and shrinking percentage in boiling water were 24 and 9 percent, respectively.

From the above results, in view of crimp developing ability and stretch back of knit and crimping status, yarns to be crystallized in heat relax step do not show good result by the treatment of the present invention and shown good result by heat-related-stretching treatment as described in Japanese Patent Publication No. 25507/1967. Good result can be obtained only by using the material of the present invention satisfying the condition described in the present specification.

EXAMPLE 20

The same procedure for preparing composite yarn as in Example 18 was followed except that bicomponent copolyamide 6T/610 with a copolymerization ratio of 20/80 by weight and having relative viscosity of 2.51. From thus obtained undrawn composite filament, the composite fiber of 20d/5f with number of crimps being 13/25mm was prepared by following the same manner as described in Example 18, except that undrawn yarn was drawn and subjected heat-relax treatment in hot air atmosphere at 180°C. Crimp developing ability and shrinking percentage in boiling water of filament yarn were 25 and 10 percent, respectively. Evaluation of

knit and X-ray measurement gave substantially same result as that for sample (XI) of Example 19.

EXAMPLE 21

To 220 Kg of 25 percent aqueous solution of the salt of hexamethylene diamine and dodecane dicarboxylic acid (612 salt) in an 400-1 autoclave, was added and dissolved 35 Kg of hexamethylene diammonium terephthalate (6T salt) and subsequently was added and dissolved 25 Kg or 40 percent aqueous solution of hexamethylene diammonium adipate (66 salt), to obtain the weight ratio of 66salt : 6T salt : 612 salt = 10 : 35 : 55. From thus obtained tricomponent copolyamide of 66/T/612 prepared as in Example 18 having

relative viscosity of 2.43 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 1 was conducted. Thus obtained undrawn filaments were drawn 3.3 times on a drawing pin heated at 110°C (2 in FIG. 2) and then heat relaxed as in Example 18 to obtain 22d/5f of composite fiber with number of crimps being 13/25mm. Crimp developing ability and shrinking percentage in boiling water of the fiber were 25 and 9 percent, respectively. Crimpability and stretching back ability of the knitted fabric therefrom were good. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 1.

EXAMPLE 22

From the random tricomponent copolyamide 66/6T/612 prepared as in Example 21 with a copolymerization ratio of 25/35/40 by weight having relative viscosity of 2.46 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite filament (XIII) of 20d/5f with number of crimps being 13/25mm was obtained in the manner as described in Example 18. On the other hand, using copolyamide 66/6T/612 with a copolymerization ratio being 35/25/40 by weight and having relative viscosity of 2.47, the composite filament (XIV) of 20d/5f with number of crimps being 14/25mm was obtained by treating in the same manner as above mentioned.

Crimp developing ability and evaluation of knit prepared and treated by boiling water as in Example 1 of (XIII) and (XIV) were shown in Table 16. In addition, X-ray measurement was made for the fibers (XIII) and (XIV), and the result was shown in Table 17.

Table 16

sample	Properties of yarn		Crimpability	Evaluation of knit Crimp irregularity	stretch back
	Crimp	Developping Ability			
III	23%		good	none	110
XIV	17%		insufficient	some streak irregularities	61

Table 17

Sample	XIII	XIV
After drawing	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter trace (one peak)
After heat relax treatment	Incomplete in crystalline structure. No peak separation was observed by densit-o-meter (one peak)	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace
After treatment in boiling water	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace.	Possesses crystalline structure. Separation of two peaks in 100 plane and 010, 110 plane was clearly observed by densit-o-meter trace.

For comparison, the yarn having the same composition of (XIV) was heat-relaxed-stretched as in the manner of Example 1 of Japanese Patent Publication No. 25507/1967 to obtain composite fiber of 20d/5f. The knitted fabric from thus obtained composite fibers was treated as above mentioned. Crimpability and stretching back ability were good. Crimp developing ability and shrinking percentage in boiling water were 24 and 8 percent, respectively.

From the above results, in view of crimp developping ability, stretch back of knit and crimping status, yarns to be crystallized in heat relax step do not show good result by the treatment of the present invention but show good result by heat-relaxed-stretching treatment as described in Japanese Patent Publication No. 25507/1967. Good result can be obtained only by using the material of the present invention satisfying the condition described in the present specification.

EXAMPLE 23

The same procedure for preparing composite yarn as in Example 1 was followed except that copolyamide tri-component copolyamide 66/6T/612 with a copolymerization ratio of 5/35/60 by weight and having relative viscosity of 2.48. From thus obtained non-spun filament, the composite fiber of 22d/5f with number of crimps being 13/25mm was prepared by following the same manner as described in Example 18, except that undrawn yarn was drawn and subjected to heat-relax treatment in hot air atmosphere at 180°C.

Crimp developping ability and shrinking percentage in boiling water of the filaments yarn were 24 and 11 percent, respectively. Crimpability and stretching back ability of the knitted fabric were good. Evaluation of knit and X-ray measurement gave substantially same result as that for sample (1) of Example 1.

EXAMPLE 24

From the tricomponent random copolyamide 6/66/6T as in Example 4 with a copolymerization ratio of 30/30/40 by weight and having relative viscosity of 2.53 as the one component and nylon 6 having relative viscosity 2.45 as the other component, composite spinning as in Example 4 was conducted. Thus obtained undrawn filaments were drawn to 3.3 time on a drawing pin heated at 110°C (2 in FIG. 2) and then by introducing to 30cm long-heating chamber under steam at 173°C, wherein the steam was supplied from heat ejection

tor at the top of heating chamber 5 and the yarn was supplied with a speed of 800m/min., and heat-relaxed at 24 percent of relax, to obtain 23d/5f composite filament developing 30 percent of crimp with number of crimp being 21/25mm. Crimp developping ability and shrinking percentage in boiling water of the filaments yarn were 27 and 10 percent, respectively. Evaluation of knitted fabric in terms of crimpability and stretch-back ability were good. X-ray measurement gave substantially same result as that of Example 4.

EXAMPLE 25

The same procedure as in Example 4 was followed except that random tricomponent copolyamide 6/66/6T with a copolymerization ratio of 50/4/45 by weight and having relative viscosity of 2.45 was used as the one component, and the composite fiber of 21d/5f with number of crimps being 17/25mm was obtained. Crimp developping ability and shrinking percentage in boiling water of the yarn were 27 and 10 percent, respectively. Evaluation of knitted fabric in terms of crimpability and stretch-back ability were good. X-ray measurement gave substantially same result as that of Example 4.

EXAMPLE 26

From the tricomponent random copolyamide of 6/66/6T prepared as in Example 4 with a copolymerization ratio of 15/65/20 by weight and having relative viscosity of 2.53 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning was conducted through a kidney-type composite spinneret as shown in FIG. 1 having 15 orifices, each with a diameter of 0.5mmφ.

In FIG. 1, the copolyamide component is supplied from groove (a), and nylon 66 from grooves (b) and (c). As shown in FIG. 2, thus obtained undrawn filaments were spun to 3.3 times through feed roll 1 and draw roll 3 and then heat relaxed by introducing to 30cm long-heating chamber 5 under steam at 165°C wherein the steam was supplied from ejector 4 at the top of heating chamber 5 and the yarn was supplied at a speed of 900m/min and the yarn was heat-relaxed at a rate of 19 percent of relax to give 1600d/60f composite filament with number of crimps being 5/25mm.

Crimp developping ability and shrinking percentage in boiling water of the fiber were 15 and 8 percent, respectively.

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Thus obtained filament yarn was twisted 40T/m. and tufted carpet with sufficient bulky touch was prepared under the condition with pile length being 8mm and the weight being 750 g/m². It was observed that thus obtained carpet had extremely superior tufting properties. The number of crimps after dyeing was 17/25mm.

EXAMPLE 27

The same procedure as in Example 26 was followed except that tricomponent random copolyamide 6/66/6T with a copolymerization ratio of 10/50/40 by weight and having relative viscosity of 2.51 was used as the one component to obtain undrawn filament. After drawing 3.3 times on a drawing pin heated at 120°C (2 in FIG. 2), the yarn was then heat relaxed to obtain composite fiber of 1600d/60f with number of crimps being 3/25mm. Crimp developing ability and shrinking percentage in boiling water of the fiber were 13 and 7 percent, respectively. The carpet prepared as in Example 26 from thus obtained composite yarns was rich in bulkiness with superior quality. It was confirmed that the yarn was extremely excellent in tufting properties.

EXAMPLE 28

From the random tricomponent copolyamide 66/6T/610 as in Example 18 with copolymerization ratio of 50/30/20 by weight and having relative viscosity of 2.54 as the one component and nylon 66 having relative viscosity of 2.47 as the other component, composite spinning as in Example 26 was conducted. Thus obtained undrawn filament were drawn to 3.3 times on a drawing pin heated at 120°C (2 in FIG. 2) and then by introducing to 30cm long-heating chamber under steam at 170°C, wherein the steam was supplied from ejector 4 at the top of heating chamber 5 and the yarn was supplied with a speed of 900m/min., and heat-relaxed at 18 percent of relax, to obtain composite fiber of 1600d/60f with number of crimps being 3/25mm. Crimp developing ability and shrinking percentage in boiling water of the filaments yarn were 14 and 8 percent, respectively. The carpet prepared as in Example 26 from thus obtained composite yarns was rich in bulkiness with superior quality. It was confirmed that the yarn was also extremely excellent in tufting properties.

EXAMPLE 29

The same procedure as in Example 28 was followed except that random tricomponent copolyamide 66/6T/610 with a copolymerization ratio of 40/30/30 by weight and having relative viscosity of 2.45 was used as the one component, and the composite filaments yarn of 1600d/60f with number of crimps being 4/25mm was obtained. Crimp developing ability and shrinking percentage in boiling water of the fiber were 14 and 9 percent, respectively. Carpet prepared as in Example 26 from thus obtained yarns was rich in bulkiness and highly resilient with superior quality. The yarn was also extremely excellent in tufting properties.

EXAMPLE 30

The same procedure as in Example 28 was followed except that random tricomponent copolyamide 66/6T/610 with a copolymerization ratio of 25/30/45 by weight and having relative viscosity of 2.51 was used as the one component, and the composite filaments of 1600d/60f with number of crimps being 6/25mm was obtained. Crimp developing ability and shrinking per-

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centage in boiling water of the yarn were 15 and 7 percent, respectively. Carpet prepared as in Example 26 from thus obtained yarns was rich in bulkiness and highly resilient with superior quality. The yarn was also extremely excellent in tuft properties.

EXAMPLE 31

The same procedure as in Example 26 was followed except that random tricomponent copolyamide 66/6T/612 with a copolymerization ratio of 50/30/20 by weight and having relative viscosity of 2.42 was used as the one component, to obtain undrawn composite yarn. Then, the yarn was drawn and subjected to heat-relax treatment as in Example 28, to obtain composite filaments yarn of 1600d/60f with number of crimps being 5/25mm. Crimp developing ability and shrinking percentage in boiling water of the yarn were 14 and 8 percent, respectively. Carpet prepared as in Example 26 from thus obtained yarns was rich in bulkiness and highly resilient with superior quality. The yarn was also extremely excellent in tufting properties.

EXAMPLE 32

The same procedure as in Example 31 was followed except that tricomponent copolyamide 66/6T/612 with a copolymerization ratio of 35/35/30 by weight and having relative viscosity of 2.52 was used as the one component, and the composite fiber of 1600d/60f with number of crimps being 8/25mm was obtained. Crimp developing ability and shrinking percentage in boiling water of the fiber were 15 and 9 percent, respectively. Carpet prepared as in Example 26 from thus obtained yarns was rich in bulkiness and highly resilient with superior quality. It was confirmed that the yarn was also extremely excellent in tufting properties.

EXAMPLE 33

Undrawn yarn was prepared as in Example 4 from random tricomponent copolyamide of 6/66/6T with a copolymerization ratio by weight of 20/30/50 and having relative viscosity of 2.55 as the one component and nylon 66 having relative viscosity of 2.47 as the other component. Thus obtained undrawn composite filament was drawn to 3.3 times by a drawing pin (2 in FIG. 2) heated at 110°C, introduced to a 30cm long-heating chamber 5 under steam atmosphere at 165°C, supplied from ejector 4 at the top of heating chamber 5 and then heat-relaxed, wherein the yarn was supplied to the heating chamber 5 with a speed of 800m/min. and heat relaxed at 23 percent of relax, to obtain 21d/5f composite filament developing 20 percent of latent crimp with number of crimps being 13/25mm. Crimp developing ability and shrinking percentage in boiling water of the fiber were 25 and 11 percent, respectively. Evaluation of knit and X-ray measurement gave substantially same result as that of Example 4.

What is claimed is:

1. A crimpable partially heat relaxed composite nylon filament comprising eccentrically arranged homopolyamide and random copolyamide components, the homopolyamide component being selected from the group consisting of nylon 6, nylon 11, nylon 12, nylon 66, nylon 610 and nylon 612, the random copolyamide component being non-crystalline and being made up of monomeric units of

a. nylon 6 and nylon 66 containing 30 to 60 weight percent of nylon 6.

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- b. nylon 6, nylon 66 and nylon 6T of a composition falling within the shaded area of FIG. 3,
- c. nylon 6, nylon 66 and nylon 610 of a composition falling within the shaded area of FIG. 4,
- d. nylon 6, nylon 66 and nylon 612 of a composition falling within the shaded area of FIG. 5,
- e. nylon 66, nylon 6T and nylon 610 of a composition falling within the shaded area of FIG. 6, or
- f. nylon 66, nylon 6T and nylon 612 of a composition falling within the shaded area of FIG. 7.

2. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 6 and nylon 66 containing 30 to 60 weight percent of nylon 6.

3. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 6, nylon 66 and nylon 6T of a composition falling within the shaded area of FIG. 3.

4. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 6, nylon 66 and nylon 610 of a composition falling within the shaded area of FIG. 4.

5. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 6, nylon 66 and nylon 612 of a composition falling within the shaded area of FIG. 5.

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6. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 66, nylon 6T and nylon 610 of a composition falling within the shaded area of FIG. 6.

7. A filament according to claim 1, wherein the random copolyamide component comprises monomeric units of nylon 66, nylon 6T and nylon 612 of a composition falling within the shaded area of FIG. 7.

8. A filament according to claim 1, wherein the filament has 5 to 30 percent of crimp developing ability, less than 15 percent shrinkage in boiling water and a difference of at least 10 percent in the boiling water shrinkages of the homopolyamide and copolyamide components.

9. The process for producing a composite filament according to claim 8, comprising extruding melts of the homopolyamide and copolyamide components to form an eccentrically arranged filament, drawing the filament, and heat relaxing the drawn filament about 15 to 25 percent under conditions which do not crystallize the random copolyamide component, whereby the filament develops 5 to 30 percent of crimp developing ability, less than 15 percent shrinkage in boiling water and a difference of at least 10 percent in the boiling water shrinkages of the homopolyamide and copolyamide components.

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