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SIDE-BY-SIDE SELF-CRIMPING CONJUGATE FILAMENTS

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ABSTRACT OF THE DISCLOSURE

A side-by-side conjugate filament is disclosed in which one of the components is a fibre-forming polyamide or polyester and the other component is a fibre-forming polyamide/polyester mixture. The mixture is such that one polymer forms a continuous phase with the other polymer dispersed therein in the form of fine fibrils.

The present invention is concerned with improvements in or relating to conjugate filaments and is especially concerned with conjugate filaments in which one component is a polyester of a polyamide and the other component is a polyamide/polyester mixture.

By the term conjugate filament is meant a unitary filament consisting of at least two polymeric fibre-forming components extending continuously along the length thereof in an adhering sheath/core or side-by-side relationship.

It has been proposed in British patent specification No. 805,033 to produce conjugate filaments in which one component is a polyamide and the other component a polyester, such filaments having a high potential crimp and good crimp retention. However such filaments formed from these components in a side-by-side arrangement tend to fibrillate, that is separate into two unitary filaments, during drawing and hence they have only limited commercial application. This problem can be alleviated by forming the conjugate filament with the components in a sheath and core arrangement thus effectively preventing fibrillation, however, the production of this type of conjugate filament is technically less attractive than the side-by-side type.

We have now found that a conjugate filament, in which the components exist in a side-by-side relationship, having high crimp and good crimp permanence and substantially free from fibrillation can be obtained using a polyamide and a polyamide/polyester mixture as the two components. To obtain maximum adhesion it is preferable the polyamide/polyester mixture should consist of a matrix of the polyamide having the polyester dispersed therein in the form of long fine fibrils to obtain maximum adhesion between the two components. It has been found that the polyester fibrils exert a disproportionately large effect on the physical characteristics of the mixture in filament form, thus the initial modulus is increased by an amount which is greater than would be expected from the percentage of polyester present and the filaments exhibit a spontaneous crimp when the two polyamide components are the same. Surprisingly good adhesion between the components is obtained when the polyamide is the dispersed phase, it being thought that this may be the result of the polyamide fibrils diffusing into the wholly polyamide component and thereby forming anchor points which prevent fibrillation. Conjugate filaments of this type normally have a higher crimp than those in which the polyamide is the continuous phase.

We have also found non-fibrillated conjugate filaments are obtained when one component is a polyester and the other component is a poly/polyamide mixture in which the polyester is the disperse phase.

The present invention does not include those conjugate filaments in which a polyester forms one component and a polyester forms the continuous phase of the other component, since the dispersed polyamide fibrils add nothing the ability of the filament to crimp or to the adhesion between the components.

The conjugate filaments of this invention crimp spontaneously when held in a relaxed condition after drawing. Subsequent heat relaxation treatments further increase the crimp and, surprisingly, it has been found that improved crimp is obtained when the filaments are fatigued, that is repeatedly stretched and relaxed under ambient conditions.

Accordingly therefore from one aspect the present invention provides a drawn molecularly oriented spontaneously conjugate filament containing two components one of which components is a fibre-forming polyamide or polyester and the other of which componets is a fibre-forming polyamide/polyester mixture. Preferably the components exist in a side-by-side relationship.

It is preferred that the percentage of polymer constituting the disperse phase in the mixture be not less than 20% and more preferably not less than 25% of the combined weight of the polymers in the mixture.

It is also preferred that in the polyamide/polyester mixture the polyamide is the continuous phase and the polyester is dispersed therein in the form of fibrils having a length of not less than 80 microns.

The polyamide/polyester component should preferably have an initial modulus of at least 50 gm. per denier per 100% of extension, measured at 2% extension when in the form of a unitary filament, and a glass/rubber transition temperature measured in the dry state of not less than 100° C.

In order to avoid fibrillation it is preferred that the heterofilaments are drawn immediately after spinning, or are stored at a relative humidity of at least 65% and a temperature of 71–73° F. before drawing if there is a time lag between spinning and drawing.

From another aspect therefore the present invention provides a process for the manufacture of a heterofilament as hereinbefore defined comprising simultaneously extruding the components through the same extrusion orifices in a side-by-side relationship, solidifying and winding up the filament onto a package and storing the package at a relative humidity of at least 65% and a temperature of 71–73° F. before drawing or drawing immediately after completion of winding the filament onto the package, drawing the filament by at least three times its original length and relaxing to allow the crimp to develop.

Drawing may be carried out by passing the filament between a first and second set of rotating rolls, the second set rotating at a greater peripheral speed than the first, there being, preferably, a snubbing pin positioned between the sets of rolls to locate the point of draw of the filament. For best results the filaments should be heated during drawing by passage over a heated plate or by use of a heated snubbing pin, the temperature of the plate or pin preferably being above 100° *C.*

Care must be taken in the preparation and spinning of polyamide/polyesters polymers to avoid hydrolytic degradation of the polyester by the action of water on the polyester linkages and to avoid interaction between the polyamide and polyester components. Suitable polyester/polyamide mixture may be prepared and spun as one component of the heterofilament by the process described in our copending patent application No. 46,796/65.

The polyamides, when used as one component, may be homopolymers or copolymers. Suitable polyamides are polyhexamethylene adipamide and polyepsilon caprolactam and copolymers thereof, especially the 80/20 polyhexamethylene adipamide/polyepsilon caprolactam copolymer.

Polyethylene terephthalate is a suitable polyester to use as one component.

The polyamide/polyester mixture may consist of polyhexamethylene adipamide or polyepsilon caprolactam admixed with polyethylene terephthalate or the polyester formed from ethylene glycol and 4/4′ dicarboxy phenoxy ethane. Either the polyamide or the polyester may constitute the continuous phase, the disperse phase being distributed therethrough in the form of fine fibrils. For the purposes of the present invention polyesters include copolyesters.

The following examples illustrate but do not limit the invention.

Example 1

A conjugate filament was obtained by the extrusion of polyhexamethylene adipamide (PHA) and a mixture of polyhexamethylene adipamide and polyethylene terephthalate (PET) (75:25 parts by weight) in a side-by-side arrangement at a temperature of 285° C. The solidified filament was wound up and stored at a temperature of about 72° F. and a relative humidity of about 67% and subsequently drawn at a feeding speed of 50 ft./min. and a draw ratio of 5.5 over a hot plate at 150° C. without fibrillation. The filament had a denier of 13.1 a tenacity of 7.2 g./d. and an extension to break of 18.5%. A fine helical crimp having good crimp permanence was obtained when the drawn filament was relaxed at ambient atmospheric conditions and there was complete absence of fibrillation.

After relaxing in water at near boiling point for one minute and then further relaxing for one minute out of water the conjugate filament had the following crimp characteristics:

Crimp ratio (CR): 33%
Crimp frequency (CF): 3 (crimps/cm.)
Helix diameter (d): 2.0 mm.
Crimp ratio was calculated from the expression:

$$C.R.=\frac{Lo-a}{Lo}\times 100$$

Crimp frequency from the expression

$$CF=n/a \text{ (crimps/cm.)}$$

where

Lo=length of uncrimped filament
a=length of crimped filament under zero tension
n=number of loops in crimped length a of the filament.

The spontaneous crimp developed by the filaments obtained in this example were characterised in the following experiment. A 10 cm. length of undrawn filaments obtained above was inserted between the jaws of an instron tensile tester, extended at a rate of 100 cm./min. to a draw ratio of 4.1 and the load released at the same speed: the distance apart of the jaws when the load became zero was used as a means of filament length with the crimp removed (L_0). The test was carried out at normal temperature and at a relative humidity of >65%. The crimp characteristics were as shown in Table 1.

TABLE 1

	Minimum	Maximum	Mean
Denier	19.7	23.0	21.3
CR percent *	23.0	30.8	27.0
CF (per cm.)*	1.5	1.9	1.7
RF (per cm.)*	0.08	0.18	0.14
CF +	0.13	0.63	0.39
d (mm.)	1.8	3.9	2.8

*Mean of 10 tests.
+Mean of 6 tests.

Reversal Frequency (RF) i.e. the number of crimp reversals per cm. is calculated from the expression:

$$RF=r/a$$

where r=number of crimp reversals per crimped length a.

Crimp Permanence (CP) is calculated from the expression:

$$CP=\frac{L_o-b}{L_o-a}$$

where L_0 and a have the same significance as above, and b=length of crimped filament after placing the crimped filament under a load of 0.3 g./d. for one minute and then relaxing in the untensioned state for one minute.

Examples 2–5

A series of drawn unfibrillated conjugate filament yarns containing six filaments were prepared in the manner described in Example 1. Details of the polymer components, spinning and drawing temperatures and yarn properties are given in Table 2 below.

TABLE 2

Example No.	Polymer Components		Spinning temp., ° C.	Drawing Conditions		Yarn physical properties		
	Mixture	Homo-polymer		Hotplate temp., ° C.	Draw ratio	Denier	Tenacity g./d.	Ext. to break, percent
2	PET/PHA (75/25)	PHA	292	140	5.0	13.7	5.8	14.5
3	PET/PHA (75/25)	PHA	292	20	4.5	9.8	4.6	36.9
4	PET/PHA (25/75)	PET	290	140	5.0	13.3	5.1	16.7

The spun filaments in the above examples were cooled and wound up and drawn immediately or held at a temperature of about 72° F. and a relatively humidity of about 67% for several hours before drawing. Under these conditions separation of the components was not encountered during drawing. A fine helical crimp having good crimp permanence was obtained when the drawn yarn was relaxed at ambient atmospheric conditions.

After relaxing in water at near boiling point for one minute and then further relaxing for one minute out of water, the filaments had the following characteristics:

	CR, percent	CF/cm.	d mm.
Example No.:			
2	50	6	1.0
3	29	2	1.0
4	58	4	2.0

The spontaneous crimp developed by the filaments of Examples 2 and 4 was characterised in the manner used in Example 1 at a draw ratio of 4.5, the crimp characteristics are shown in Table 3.

TABLE 3

		Denier*	CR*, percent	CF* .per cm	RF*, per cm.	CP+	d*, mm.
Ex. No:							
2	Minimum	10.2	27	2	0.25	0.33	1.1
	Maximum	18.0	34	4	0.40	0.52	2.3
	Mean	15.1	31	3	0.33	0.41	1.6
4	Minimum	9.2	27	4	0.26	0.44	0.9
	Maximum	13.7	36	5	0.45	0.89	1.4
	Mean	11.4	32	4	0.31	0.68	1.2

*Mean of ten tests.
+Mean of six tests.

A series of other combinations of polymers was rod spun as monofilaments at a rate of extrusion of 1.3 g./min. and a wind up speed of 600 ft./min. The undrawn filaments were drawn over a heated plate immediately after extrusion and subsequently relaxed in water at near boiling point for one minute and then further relaxed for one minute out of the water and the crimp characteristics determined. Details of spinning and drawing and crimp characteristics are given in Table 4.

TABLE 4

	Components		Spinning temp., °C.	Draw ratio	Plate temp., °C.	CR percent	CF, per cm.	d. mm
	Polyamide	Mixture						
Ex.:								
6	PHA/PEC* (80/20)	PHA/PET (20/80)	288	5	140	74	22	0.8
7	PHA	PET/PHA (40/60)	275	4.5	135	61	3.5	1.6
8	PEC	PEC/PET (25/75)	270	4.5	135	37	2.0	1.8
9	PHA	PEC/PET (25/75)	275	4.0	135	61	4.7	1.6
10	PHA	CPE.2G [1]/PHA (50/50)	296	4.0	150	63	7.1	1.0

*PEC=polyepsilon caprolactam.
+CPE.2G=polyester prepared from ethylene glycol and 4,4′ dicarboxyphenoxyethane.

There was no evidence of fibrillation during drawing in any of the above experiments.

What we claim is:

1. A drawn molecularly oriented spontaneously crimping conjugate filament containing at least two components existing in a side-by-side relationship, one of which components is a fibre-forming polyamide or polyester and the other of which components is a fibre-forming polyamide/polyester mixture.

2. A filament according to claim **1** wherein the polyamide/polyester mixture contains not less than 20% by weight of the disperse phase.

3. A filament according to claim **2** wherein the polyamide/polyester mixture contains not less than 25% by weight of the disperse phase.

4. A filament according to claim **2** wherein one component is a polyamide and the other component is a polyamide/polyester mixture in which the polyamide is the continuous phase and the polyester is dispersed therein in the form of fibrils having a length of not less than 80 microns.

5. A filament according to claim **4** wherein the polyamide/polyester component has an initial modulus of at least 50 g. per denier per 100% extension, measured at 2% extension, and a glass-rubber transition temperature of not less than 100° C. when measured in the dry state.

6. A filament according to claim **5** wherein the polyamide is polyhexamethylene adipamide and the polyamide/polyester mixture is polyhexamethylene adipamide/polyethylene terephthalate.

7. A filament according to claim **2** wherein the polyamide is polyhexamethylene adipamide and the polyamide/polyester component is a mixture of polyhexamethylene adipamide/and a polyester formed from ethylene glycol and 4,4′ carboxy phenoxy ethane in equal proportions by weight.

8. A filament according to claim **2** wherein one component is a polyamide and the other component is a polyamide/polyester mixture in which the polyester is the continuous phase and the polyamide is dispersed therein in the form of fine fibrils.

9. A filament according to claim **8** wherein the polyamide is polyhexamethylene adipamide and the polyamide/polyester component is polyepsilon caprolactam/polyethylene terephthalate in which the polyepsilon caprolactam is the disperse phase and constitutes 25% by weight of the mixture.

10. A filament according to claim **8** wherein the polyamide is polyepsilon caprolactam and the polyamide/polyester component is polyepsilon caprolactam/polyethylene terephthalate in which the polyepsilon caprolactam is the disperse phase and constitutes 25% by weight of the mixture.

11. A filament according to claim **8** wherein the polyamide is polyhexamethylene adipamide and the polyamide/polyester mixture is polyhexamethylene adipamide/polyethylene terephthalate in which the polyhexamethylene adipamide is the disperse phase and constitutes 25% by weight of the mixture.

12. A filament according to claim **2** wherein one component is a copolymer polyhexamethylene adipamide and polyepsilon caprolactam containing 20% by weight of the polyepsilon caprolactam and the other component is polyhexamethylene adipamide/polyethylene terephthalate containing 20% by weight of polyhexamethylene adipamide.

13. A filament according to claim **2** wherein one component is a polyester namely polyethylene terephthalate and the polyamide/polyester component is polyhexamethylene adipamide/polyethylene terephthalate containing 75% by weight of the polyamide as the continuous phase and the polyester dispersed therein in the form of fibrils having a length of not less than 80 microns.

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