(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 115 041**
A2

(12) # EUROPEAN PATENT APPLICATION

(21) Application number: **83112917.6**

(22) Date of filing: **21.12.83**

(51) Int. Cl.³: **D 01 F 6/80**
D 01 D 5/08, D 01 D 5/16

(30) Priority: **31.12.82 IT 2507782**

(43) Date of publication of application:
08.08.84 Bulletin 84/32

(84) Designated Contracting States:
AT BE CH DE FR GB LI NL

(71) Applicant: **SNIA FIBRE S.p.A.**
Via Friuli 55
I-20031 Cesano Maderno(IT)

(72) Inventor: **Casati, Alvaro**
Via Poliani 7
I-20030 Palazzolo Milanese(IT)

(72) Inventor: **Zuntini, Carlo**
Via De Santis 15/A
I-20030 Palazzolo Milanese(IT)

(72) Inventor: **D'Andolfo, Francesco**
Villaggio Snia
I-20031 Cesano Maderno(IT)

(72) Inventor: **Fugiglando, Paolo**
Viale Ca' Granda 16/B
I-20162 Milano(IT)

(72) Inventor: **Leoncini, Novello**
Via Cavour 17
I-20051 Limbiate(IT)

(74) Representative: **Garrone, Fernando**
Internazionale Brevetti s.r.l. Via Brentano 2
I-20121 Milano(IT)

(54) **A method of preparing a polyamidic fiber suitable for staple spinning, and fiber obtained thereby.**

(57) The invention is concerned with a method of preparing a suitable fiber for staple processing and having good moisture absorption characteristics. The method consists of preparing a copolymer of Nylon 6 and poly(4, 7-dioxydecamethylene) adipamide containing 10 to 40% by weight of the latter component, spinning said copolymer by melting at a temperature in the 246°C to 300°C range and extruding through a die having capillaries with larger diameter dimensions than those used in the standard polyamide staple spinning, cooling the filaments by blowing air, hot drawing the filaments in air at a draft ratio in the 2.2 to 3.6 range, effecting a heat setting operation under tension, at least the final step of the heat setting operation being effected at a temperature close to the softening temperature of said filaments, and subjecting the resulting filaments to a mainly antistatic type of finish. The invention also relates to the polyamidic fiber thus obtained.

EP 0 115 041 A2

"A METHOD OF PREPARING A POLYAMIDIC FIBER SUITABLE FOR STAPLE SPINNING, AND FIBER OBTAINED THEREBY"

This invention relates to a polyamidic short fiber or staple suitable for staple spinning, and to a method for the preparation thereof.

Known in the art are polyamidic staples which may be processed with the methods of staple processing, i.e. carding, yarn forming from short fibers, and subsequent textile processing such as weaving, etc., but they have only enjoyed, heretofore, limited applications because of their poor moisture absorption, which results in discomfort for the wearer of the textile articles manufactured from them.

Known in the art is also a polyamidic block co-polymer, as described and claimed in US Patent No. 4,130,602, which is formed by co-melting a polyamide suitable for melt spinning, such as Nylon 6, with a polydioxyamide, and in particular with poly(4,7-dioxydecamethylene) adipamide. Said block co-polymer will be referred to hereinafter as polymer S, for brevity. Said block co-polymer exhibits the advantageous property of absorbing significant amounts of moisture, thereby it should afford, when processed into textile articles, a comparable comfort for the wearer to that afforded by articles made of cotton and other natural fibers.

Attempts have been made at spinning that block co-polymer so as to obtain suitable fibers to be processed, using the methods and equipment which are typical of staple spinning, but it has been found that that spinning method cannot be applied to it for

a number of reasons. First of all, the process cannot be carried out materially on industrial scale with regularity because the fiber lacks the required features for the treatment on the card, on the spinners and on other related equipment. It is not always possible to make certain of why it should be so, because the properties required of a fiber for spinning are multivarious and not always clearly understood. Of proven import is the fiber strength at the initial stage of its deformation curve, as expressed by its initial elastic modulus, its elongation, dimensional stability, ability to promote mutual adhesion of the fibers as the various fibers are twisted to form a yarn, and so forth.

Experiments carried out by the Applicant have shown, moreover, that polymer S fibers, as obtained with conventional polyamide spinning techniques, cannot be processed on an industrial scale with the staple spinning method, and that even where such difficulties are not taken into account, i.e. even within the experimental limits within which a spun product of sort can be obtained, this has commercially unacceptable characteristics due to its low strength and lack of an adequate dimensional stability.

Thus, the need was felt for a way to change the properties of the fibers formed from polymer S so as to make them suitable for conversion with staple spinning techniques and provide suitable products for the desired textile applications. The problem was

further aggravated by the fact that, as mentioned, not all the properties the fiber should possess are perfectly understood, nor are the reasons why fibers formed from polymer S with standard polyamidic spinning methods did not possess such properties.

The Applicant has now unexpectedly found that the problem is solved, and that a polymer S fiber can be obtained which is suitable for staple spinning, while quite satisfactory articles can be achieved thereby, through a modified and improved spinning method which comprises essentially the following steps:

1. One starts from a copolymer of Nylon 6 and poly(4,7-dioxydecamethylene) adipamide, which contains 10 to 40% by weight, preferably 15 to 30% by weight, of the latter component.
2. The copolymer is melt-spun at a temperature in the 246 °C to 300°C range, by extrusion through a die the capillaries whereof have a diameter comprised between 0.15 and 0.45 mm.
3. Upon exiting the die, the extruded filaments are cooled by air blowing .
4. The filaments are hot drawn in air at a draft ratio in the 2.2 to 3.6 range.
5. Thereafter, a heat setting operation is performed under tension on hot calenders, at least the final phase of said heat setting operation being effected at a temperature comprised between 180 to 210°C.

As mentioned hereinabove, the method of this invention is useful in the continuous preparation of fibers suitable for staple processing from a block copolymer of Nylon 6 and poly(4,7-dioxydecamethylene) adipamide having the property of absorbing moisture, but being difficult to spin with the prior methods.

The starting copolymer of Nylon 6 and poly(4,7-dioxydecamethylene) adipamide, containing 10 to 40%, preferably 15 to 30%, and even more preferably 15 to 25%, by weight of the second component, is obtained by co-melting the components as described in the cited US Patent No. 4,130,602.

The copolymer is then spun, melting the dried chips in extruder such that the temperature of the molten polymer is in the range from 246 to 300°C, preferably in the range from 260 and 280°C, and then extruding the molten polymer through a die the capillaries whereof have a diameter comprised between 0.15 and 0.45 mm.

It has been found that optimum results are to be obtained with a die having capillaries with a diameter in the 0.20 to 0.30 mm range.

On exiting the die, the extruded filaments are cooled by blowing, which operation is well known in in the art of polyamide spinning, but the following prescriptions are observed.

The flow rate of the cooling air should range from 100 to 300 m^3/hour, because higher flow rates would result in filament breakage and lower flow rates than indicated would not cool adequately the bun-

dle of filaments leaving the die.

Further, the cooling air should be conditioned to a relative humidity in the 50 to 70 % range at an approximately ambient temperature, preferably at a temperature in the 10°C to 22°C range.

The cooled filaments are collected into bins, and optionally conditioned or maintained in a conditioned environment prior to the drawing step, at a temperature of 15-30°C and relative humidity in the 50% to 70% range. Advantageously, conditioning, where required, may be effected using the same air which is utilized to cool the filaments as they are extruded.

Drawing is carried out in air with heat application, preferably in two successive steps The first drawing phase takes place at a temperature in the 40°C to 80°C range, and the second step is carried out at a higher temperature of about 110°C to 150°C.

In actual practice, two sets of draft rollers are provided wherebetween a means, such as a furnace, is interposed which is operative to keep the yarn temperature relatively constant during the second drawing step which takes place within the furnace.

The temperature of the draft rollers is set at such a level as to heat the yarn progressively from the temperature of the first drawing step (in the 40°C to 80°C range) to the final temperature of the second drawing step varying, as mentioned, from 110°c to 150°C. The overall draft ratio is in the 2.2 to 3.6 range, preferably in the 2.7 to 3.2 range.

The drawn filaments are then subjected to a heat setting treatment under tension on hot calenders, such that at least during the final phase of the treatment the temperature is at a level in the 180°C to 210°C range; in other words, temperature during this phase should be the highest that the yarn can stand before it starts to melt. In practice, heat setting would be carried out, of preference, in two stages. During the initial stage, or so-called "preparatory" stage, temperature is raised to a value in the 100°C to 150°C range and the yarn maintained at this temperature for about 4 to 8 seconds.

During the final heat setting stage, under tension, the temperature is the highest acceptable for the yarn and is in the 180°C to 210°C range.

The yarn is maintained at this temperature for about 14 to 28 seconds. It has been found that at lower heat setting temperatures than indicated the desired yarn characteristics cannot be achieved.

Finishing is then applied at a temperature of about 15 to 55°C.

If required, further drying may follow prior to the subsequent utilization in the staple processing.

The fiber obtained with the method of this invention has the following characteristics:

Filament count	: 1.5 to 10 den/filament
Ultimate strength	: 5 to 7 g/den
Ultimate elongation	: 55% to 70%
Initial modulus	: 6 to 8 g/den
Shrinkage, measured as explained hereinafter	: 1.5 % to 5%.

Shrinkage is measured in boiling water in conformity with the ASTM 2259-77 Method as adapted for the fibers in question.

The fibers thus obtained are suitable for a staple processing using conventional methods and equipment, a particular example whereof will be described hereinafter.

The fiber obtained from the polymer S may be successfully utilized both in the production of fibers made of 100% copolymer S, and of mixed yarn with other synthetic or natural fibers. With the yarn thus produced, both shuttle fabrics (dry goods and shirts) and knitwear may be made.

Both in the production of fabrics and subsequent steps (dyeing, setting), conventional processing cycles are employed with excellent results, as may be seen from the examples which follow.

The fabrics thus produced have shown to develop clearly valuable characteristics from the standpoint of mechanical strength (wear, tear, etc.), wrinkle-proofness, dimensional stability with maintenance treatments (washing in water and dry cleaning). Among the characteristics of the finished product obtained from polymer S fiber according to this invention, the anti-pilling properties and specially soft and pliable hand (comparable to cashmere) of knitwear.

The examples which follow illustrate the method of this invention and fibers and yarns to be obtained

with said method, but should not be construed as limitative of the invention scope.

EXAMPLE I

Preparation of polymer S fiber.

200 g/min of polymer S comprising 80% Nylon 6 and 20% poly(4,7-dioxydecamethylene) adipamide (also referred to as polymer S 80/20 hereinafter), having a 0.05% residual humidity, are melted at 280°C and extruded through a conventional circular die of steel having 475 holes, size 0.30/0.25, i.e. a diameter of 0.3 mm and height of 0.25 mm. The spinning rate is 700 m/min.

On leaving the die, the filaments are cooled by air blowing at a flow rate Q of 200 m^3/hour, temperature of 21°C, and relative humidity (U.R.) of 50%.

The cooled filaments are then collected into bins and maintained in this condition (T = 21°C and U.R. = 50%) until drawing.

Drawing is carried out in two steps , the first at 60°C and the second at 100°C. The initial velocity (of the first draft rollers) is 33 m/minute and the velocity of the final draft rollers is 103 m/min, thereby the overall draft ratio is 3.12.

The drawn fiber is then subjected to heat setting under tension, first at 120°C for 6 seconds, and then at 195 °C for 20 seconds. Thereafter, the fiber is passed through a finishing bath at 52°C, the bath containing 30.0 g/l finish. The finish concentration on the fiber shows to be, by analysis, of 0.9 %, the concentration being measured by extraction.

The fiber is then dried on a hot drum (at 120°C), crimped to impart it with 6 loops/cm, and cut with the short cotton mill method to 40 mm.

The resulting fiber (fiber S 80/20) has the following characteristics:

Filament count	: 1.65 den/filament
Ultimate strength	: 6.5 g/den
Ultimate elongation	: 62%
Initial modulus	: 6.5 g/den
Free shrinkage in boiling H_2O	: 2.7%

EXAMPLE II

Example I is gone through again, except that the following conditions are used in order to obtain a wool-type staple.

1. Starting polymer

200 g/min of copolymer having 80% Nylon 6 and 20% poly(4,7-dioxydecamethylene) adipamide, residual humidity 0.07 % .

2. Spinning

Melting temperature	= 270°C
Die	= 300 holes, 0.30/0.25 mm
Spinning rate	= 700 m/min

3. Cooling by blowing

Air flow rate	= 200 m^3/hour
Temperature	= 21°C
U.R.	= 60 %

4. Conditioning in the bins

Temperature	= 21°C
U.R.	= 60 %

Hot drawing in air

first stage temperature	= 60°C

second stage temperature = 100 °C

first draft roller speed = 32 m/min

final draft roller speed = 103 m/min

overall draft ratio = 3.22

5. Heat setting

Initial stage = 6 seconds at 120°C

Final stage = 20 seconds at 19›°C

6. Finishing

Bath temperature = 55°C

Bath contents = 40 g/l

Finish concentration on the fiber = 1.2 %

Drying

drum temperature = 120°C

Crimp = 4.5 loops/cm

Cutting = wollen cut 80 to 100 mm .

The resulting fiber has the following characteristics:

Filament count = 3.02 den/filament

Ultimate strength = 6.1 g/den

Ultimate elongation = 66%

Initial modulus = 6.1 g/den

Shrinkage = 3.2%

EXAMPLE III

This Example compares the water retention and water absorption and de-absoprtion characteristics of polymer S fibers according to this invention to prior synthetic fibers and raw cotton.

On measuring water retention in accordance with the ASTM D 2402/69 Method, the following values are obtained:

Polymer S fiber according to Example I : 24%

Nylon 6 : 15%

Raw cotton : 38%

Water absorption and de-absorption have been measured as follows. The fiber sample whose water absorption properties are to be measured is first thoroughly dried, and then maintained over a time period in a conditioned environment at ambient temperature and controlled relative humidity. Next, the amount of moisture (expressed as percentage by weight) which the fiber takes back is measured by weighing at each assigned value of relative humidity.

To determine the de-absorption characteristics a similar procedure is followed, except that one starts with a wet fiber (saturated with moisture) and that the amount of moisture present in the fiber is measured for decreasing values of relative humidity of the conditioned environment.

The results are shown in the following Table.

Test	U.R. %	Moisture % by weight - Nylon 6	Moisture % by weight - Fiber S-80/20
Absorption	33	2.73	4.00 to 5.00
	65	4.59	6.5 to 9
	93	7.98	16 to 20
Deabsorption	93	8.95	17 to 20
	65	4.40	6.5 to 9.0
	33	2.52	3.5 to 5.0

It will be apparent that the fiber of polymer S has considerably better absorption, de-absorption, and retention of water characteristics than the synthetic fiber of Nylon 6.

EXAMPLES IV and V

Cotton mill processing.

These Examples concern cotton process of the fibers of polymer S 80/20 obtained in accordance with Example I .

In particular, yarns are produced with a cotton count Ne 24 in 100% fiber S having a denier rating of 1.65 den and 40 mm cut, obtained with the procedure of Example I .

The fiber S staples, 1.65 den is processed without any additional antistatic finishing.

The staples are "opened" on an opener having a single beating reel, at a number of reel beats per minute equal to 40 , and at 800 reel rpm.

Then, a carding operation is effected on a flat card having a staple direct feed system.

The carded webs are then processed with two passes through a roller drawing frame at the speed of 240 m/min.

Subsequent conversion on the roving frame is effected on a frame having high pressure swinging arms and twin belt drawing facilities adjusted as

follows:

Input card sliver count Ne	: 0.14
Output roving count Ne	: 1.02
Roving twists/inch	: 0.45
Spindle rpm	: 600
Pre-drawing	: 1.19
Roll setting for 1.65 den fiber in mm	: 58-48

Yarn conversion is carried out on a ring having suitable high pressure swinging arms for synthetic fibers, using the following settings:

Input roving count Ne	: 1.02
Output yarn count Ne	: 24
Twists per inch of 1.65 den fiber	: 15.7
Spindle rpm	: 9,500

The resulting yarn has the textile characteristics set forth in the following Table.

	Yarn from 40 mm, 1.65 den Fiber S staple
Count (Ne).....	24.1
Average breaking load (g).....	637
Breaking length (km)....	26.0
Breaking elongation (%).	25.2
Load coefficient of variation (%).....	15.5
USTER-U evenness (%) ...	13.2

CLAIMS

1. A method of preparing a suitable synthetic fiber for staple processing having good moisture absorption characteristics, the method comprising the steps of :

1) preparing a copolymer of Nylon 6 and poly(4.7-dioxydecamethylene) adipamide containing 10 to 40 % by weight of the second mentioned component;

2) spinning said copolymer by melting at a temperature in the 246 to 300°C range, and extruding through a die the capillaries whereof have a diameter comprised between 0.15 and 0.45 mm;

3) cooling the filaments exiting said die by air blowing;

4) hot drawing the filaments in air at a draft ratio in the 2.2 to 3.6 range;

5) effetting a heat setting operation under tension on hot calenders, at least the final stage of said operation being carried out at a temperature comprised between 180°C to 210°C.

2. A method according to Claim 1, wherein said copolymer comprises 15% to 25% by weight of poly'4.7-dioxydecamethylene)adipamide.

3. A method according to Claim 1, characterized in that said copolymer is melt-spun at a temperature comprised between 260 and 280°C.

4. A method according to Claim 1, characterized in that said draft ratio is comprised between 2.7 and 3.2 .

5. A method according to claim 1, characterized

in that said copolymer of Nylon 6 and poly(4.7-dioxydecamethylene) adipamide contains 15% to 30 % of the second component.

6. A method according to Claim 1, characterized in that said extrusion step (2) is carried out using a die provided with capillaries having a diameter dimension in the 0.20 mm to 0.30 mm range.

7. A method according to Claim 1, wherein said cooling step by blowing (3) is carried out at a temperature in the 10°C to 22°C range, and at a relative humidity in the 50 % to 70 % range.

8. A method according to Claim 1, wherein the flow rate of air used in said cooling step by blowing is 100 to 300 m^3/hour.

9. A method according to claim 1, characterized in that said drawing step (4) is effected in two stages at different temperatures, the first stage at a temperature of 40 to 80°C, and the second stage at a temperature of about 110 to 150°C.

10. A method according to Claim 9, characterized in that said second drawing stage takes place within a heating means provided to maintain the yarn temperature during said stage at constant values.

11. A method according to Claim 1, wherein said heat setting step (5) is carried out in two stages, the initial or preparation stage being conducted at a temperature of 100 to 150°C, and the final stage at a temperature of 180 to 210°C.

12. A method according to any of the preceding

claims, characterized in that it is a continuous method.

13. A method according to any of the preceding claims, wherein the dried and crimped filaments are cut into lengths of 40 to 180 mm.

14. Fiber prepared by a method according to Claim 1, characterized in that in an environment having a relative humidity of 33 %, 65 %, 93 %, said fiber absorbs humidity in amounts comprised respectively from 4 % to 5 %; 6.5 % to 9 %; and 16 % to 20 % of the weight of the fiber.