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TERUMICHI ONO ETAL

3,379,810

PROCESS FOR THE MANUFACTURE OF HIGH TENACITY NYLON FILAMENTS

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Fig. 1

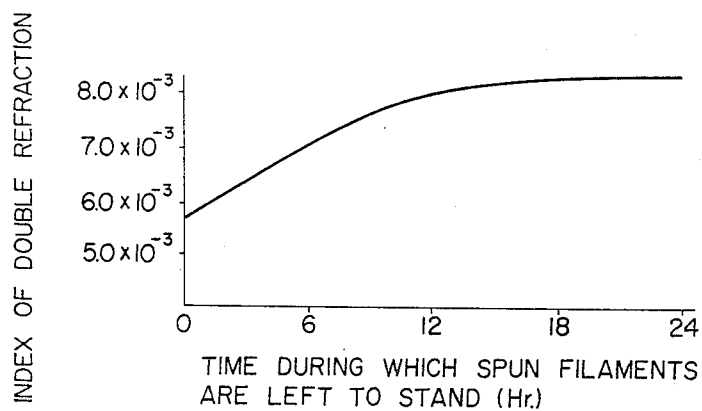
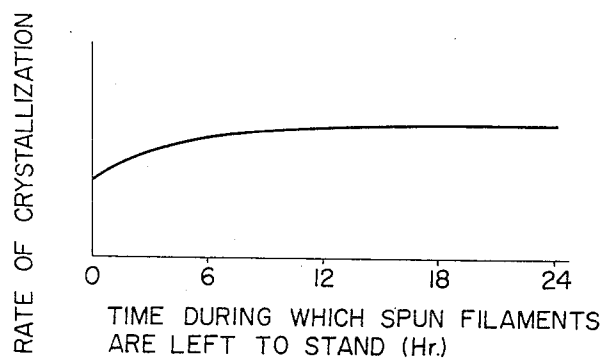


Fig. 2



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PROCESS FOR THE MANUFACTURE OF HIGH TENACITY NYLON FILAMENTS

Terumichi Ono, Tsuneo Haga, and Rokuro Sakai, Okazaki-shi, Aichi-ken, Japan, assignors to Toyo Rayon Kabushiki Kaisha, Tokyo, Japan, a corporation of Japan

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4 Claims. (Cl. 264—210)

ABSTRACT OF THE DISCLOSURE

A method of obtaining high tenacity nylon filaments by subjecting nylon filaments which have been melt-spun, cooled and solidified to a drawing operation in at least two stages immediately after solidification and wherein the following conditions are satisfied:

(i) The solidified filaments to be subjected to a first stage drawing operation should have a birefringence of not more than 0.008 as measured after the filaments have been left to stand for 10 to 20 minutes at a temperature of 20° C. and RH of 65%, and

(ii) The first stage drawing should be carried out at a draw ratio in the range of 1.25 to 2.40.

This invention relates to a process for the manufacture of strong nylon filaments. More particularly, this invention concerns a process for producing strong nylon filaments which comprises melt spinning polyamide and, immediately thereafter, drawing hardened filaments in a step comprising at least two stages, and which is characterized in that said hardened filaments have an index of double refraction (index of birefringence) not greater than 0.008 measured at the end of 10 to 20 minutes' period during which they are left to stand in a state of 28° C., 65% RH, immediately following hardening, and that the first-stage drawing in the said drawing step is conducted at a drawing ratio in the range of 1.25 to 2.40 times.

Generally speaking, synthetic fiber filaments obtained in a melt spinning step are not oriented, and therefore they are liable to elongate even under the application of slight force, which makes them unfit for practical purposes. In order to turn them into serviceable filaments, it is necessary to orient them by drawing 5 or 6× at room temperature or under heated conditions. A method heretofore employed comprises winding up the spun filaments and thereafter drawing the filaments as they are unwound. However, such a process which comprises two independent and separate steps of spinning and drawing requires two kinds of mechanical equipment. This involves a complexity of working steps and a large work force. This process is therefore disadvantageous commercially.

Attempts to produce filaments in a spinning process alone by ultra high speed spinning in order to overcome the above-mentioned defects are known in French Patent No. 976,505 and United States Patent No. 2,957,747. However, in these spinning processes, a very high draft (ratio of take-up speed to delivery speed at the spinneret) is imparted to form filaments, and there is no definite

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drawing step. Therefore, filaments obtained by this process have a low tenacity and a very high stretchability.

Therefore, the above attempts are not suitable for obtaining filaments necessitating high strength.

An object of this invention is to provide a process for obtaining filaments having high strength by continuously effecting two separate steps of spinning and drawing, i.e., drawing filaments continuously right after they have been spun.

After extensive studies in an attempt to achieve such an object, we have found that if melt-spun polyamide is continuously drawn immediately after hardening (in this case, the filaments have an index of double refraction not greater than 0.008 measured under the above-mentioned conditions), and drawing is carried out in more than two stages, the first drawing being effected at a small drawing ratio of 1.25 to 2.40 times, a melt-spinning step and a drawing step can be successively practised to advantage. In the conventional method which is a mere combination of a step in which filaments are pressed out from the spinneret, hardened upon cooling and wound up on a spool after application of an oil agent and water for controlling moisture, lubrication and eliminating static electricity and a step of drawing the filaments, which have been unwound from the spool, in one stage by means of two rollers having different surface speeds in a ratio corresponding to that of the surface speeds, the filaments are often broken in the drawing step and the stability of drawing is very low. It has been therefore completely impossible to obtain nylon filaments having high strength.

However, in accordance with the drawing step of this invention which has more than two stages, a direct spinning drawing is very smoothly carried out, thus solving the above-mentioned problems. The structure of the filaments just spun is unstable as compared with the set filaments and the filaments are viscous. These properties are causes of unstableness of drawing. This unstableness is, to some extent, removed in the first stage, thus making it easy to effect drawing in the second and subsequent stages. If a drawing step is successively practised immediately after a spinning step by such a process, however, it is possible to effectively overcome the difficulties encountered in the conventional process which is a mere combination of a spinning step and a drawing step.

This is explained in further detail with reference to the accompanying drawing, wherein:

FIG. 1 is a diagram showing a relation between an index of double refraction of undrawn polycapromide filaments (210 denier) and the time during which the spun filaments are left to stand, and FIG. 2 is a diagram showing a relation between the rate of crystallization of polycapromide filaments (210 denier) and the time during which the spun filaments are left to stand.

The unstableness of the inner structure of filaments immediately after they have been spun can be substantiated from the fact that when the filaments are left to stand as they are after spinning, an index of double refraction showing an orientation of the polymer that makes up the filaments increases with the passage of time as shown in FIG. 1 and the rate of crystallization increases likewise with the passage of time as shown in FIG. 2.

The drawing is only intended to show an example of the change of double refraction index and degree of crystallization with the passage of time. Actually, the

double refraction index in particular is greatly dependent upon the spinning conditions. These facts prove that filaments which have just been spun are not only different from the set filaments which remain after a period of time in such properties as crystallization and orientation, but also exhibits unique properties with respect to viscosity as compared with such set filaments. When filaments just spun are drawn continuously in a single stage, the conditions of filament-making are worsened as compared to a process in which spun filaments are wound up on a spool and drawn after having been pulled out again. To cite an example, filaments are brought into contact with a hot plate at the time of hot drawing. The above-mentioned facts are probably the causes of such a bad condition.

In order to draw the filaments just spun as excellently as the set filaments, therefore, it is necessary to adjust such properties as crystallization and orientation to the same degree as in the set filaments by establishing some particular conditions. In this invention, a drawing step is divided into more than two stages and the first stage drawing is conducted at a ratio of as low as 1.25 to 2.40 times.

By "drawing hardened filaments immediately," it is meant that the time from the end of the spinning to the beginning of drawing is short; it is preferable usually that this time is within 20 seconds. It is necessary in this invention that the index of double refraction of the filaments to be subjected to the first-stage drawing should be not greater than 0.008 measured under the conditions hereinabove described. This is an essential condition for carrying out drawing effectively and smoothly. An index of double refraction of the hardened filaments sometimes exceeds 0.008 at the time of measuring under said conditions depending upon the change of conditions such as draft and hardening. However, such a spinning condition is not employed in this invention.

The strong nylon filaments as described in this invention are not limited by numerical figures, but generally include those filaments having a fineness of more than 210 denier and a strength of more than 6 g./d. In order

total to 5.0 times. As seen from Table 1, filament breakage at the time of drawing showed good results when a drawing ratio in the first stage was 1.4 to 2.4; extensibility was very unstable when the ratio at the first stage was less than 1.2 and greater than 2.9.

TABLE 1

	Total drawing ratio	Drawing ratio at 1st stage	Drawing ratio at 2nd stage	Number of filament breakage/10 kg.	Fineness (denier)	Strength (g./d.)
10	5.00	1.10	4.55	(1)	-----	-----
	5.00	1.20	4.17	(1)	-----	-----
	5.00	1.30	3.85	11	858	9.00
	5.00	1.40	3.57	9	861	8.78
	5.00	1.50	3.33	2	868	9.07
	5.00	1.60	3.12	0	866	8.98
15	5.00	2.00	2.50	4	869	8.66
	5.00	2.40	2.80	10	865	8.73
	5.00	2.50	2.00	15	867	8.88
	5.00	2.60	1.93	19	854	8.97
	5.00	2.70	1.85	11	874	8.86
	5.00	2.80	1.79	24	844	8.94
	5.00	2.90	1.72	(1)	-----	-----
20	5.00	3.00	1.67	(1)	-----	-----
	5.00	3.20	1.56	(1)	-----	-----

¹ Difficult drawing.

In the example, a drawing pin and a hot plate were not used in the first stage, but in the second stage. In this case, it took about 3 seconds from the end of the spinning to the beginning of the drawing. An index of double refraction of the filaments prior to the first stage drawing was 0.00614.

Example 2

Granular polycapramide having an average molecular weight of about 25,000 was melt spun at a temperature of 300° C. and a rate of 360 m./min. and, immediately after hardening, drawn at a rate of 1870 m./min. with a drawing ratio differing from the first stage to the second stage and amounting in total to 5.20 times. Thereafter, the number of filament breakage at the time of drawing was examined. Also, rate of crystallinity and the index of double refraction of the filaments which were melt spun and drawn in the first stage were measured (in accordance with a density gradient tube method). Results are shown in Table 2.

TABLE 2

Total draw-drawing ratio	Drawing ratio at 1st stage	Rate of crystallinity (Percent)	Index of double refraction	Drawing ratio at 2nd stage	Filament breakage per 10 kg.	Fineness (denier)	Strength (g./d.)
5.20	1.20	26	5.44×10^{-3}	4.33	(1)	-----	-----
5.20	1.60	28	7.53×10^{-3}	3.25	4	860	9.05
5.20	2.00	29	12.35×10^{-3}	2.60	5	848	8.87
5.20	2.40	31	22.76×10^{-3}	2.17	0	869	9.12
5.20	2.80	34	29.15×10^{-3}	1.86	12	872	9.00
5.20	3.20	36	41.60×10^{-3}	1.63	(1)	-----	-----

¹ Difficult drawing.

to obtain such nylon filaments, it is necessary that a total drawing ratio in the first stage and those after the second stage combined should be more than 4 times. In most cases, if such a drawing ratio is attained in a drawing effected in two stages, sufficiently strong filaments can be obtained.

Furthermore, it is preferable to use a hot plate not in the first stage drawing but in the second stage and subsequently. This is because in the first stage drawing, the melt-spun and hardened filaments are still viscous and prone to undergo breakage or fusion on the hot plate, whereas those filaments to be subjected to drawing in the second stage and afterwards do not show such phenomena owing to their accelerated crystallization and orientation in the first drawing.

Example 1

Granular polycapramide having an average molecular weight of about 25,000 was melt spun at a temperature of 290° C. and a rate of 360 m./min. and drawn immediately after hardening. Drawing was effected at a rate of 1800 m./min. with a drawing ratio differing from the first stage to the second stage which amounted in

Rate of crystallinity was begun to be measured at the end of 24 hours in an atmosphere of 20° C. 65% after sampling. An index of double refraction was measured under the said conditions.

As seen from Table 2, drawing was effective when the drawing ratio at the first stage was between 1.6 and 2.8. In this example, a drawing pin and a hot plate were not used in the first drawing stage, but in the second drawing stage. In this case, it took about 15 seconds from the end of the spinning to the beginning of drawing. An index of double refraction of the filament prior to the first stage drawing measured under the said conditions was 0.00523.

Example 3

Granular polycapramide having an average molecular weight of about 25,000 was melt spun at a temperature of 300° C. and a rate of 360 m./min. and subsequently drawn at a rate of 1800 m./min. with a total drawing ratio of 5.00 times, the first stage and second stage drawing ratio being 2.37 and 2.11 times, respectively. There was hardly any filament breakage at the time of drawing. The obtained filament had a fineness of 840 denier and a strength of 9.2 g./d. A drawing pin and a hot plate were

not used in the first stage but in the second stage. In this case, it took about 5 seconds from the end of spinning to the beginning of drawing. The index of double refraction of the filaments prior to the first stage drawing was found to be 0.00751 on measurement under the said conditions.

Example 4

Granular polyhexamethylenedipamide having an average molecular weight of about 24,000 was melt spun at a temperature of 310° C. and a rate of 360 m./min. and thereafter drawn at a rate of 1800 m./min. with a total drawing ratio of 5.00 times, the first stage and second stage drawing ratio being 2.0 and 2.5 times, respectively. There was no breakage of filaments at the time of drawing, and the filaments had a fineness of 835 denier and a strength of 9.5 g./d.

A drawing pin and a hot plate were not used in the first stage drawing but in the second stage drawing. In this case, it took about 4 seconds from the end of spinning to the beginning of drawing. An index of double refraction of the filaments prior to the first stage drawing measured under the said conditions was 0.00693.

In accordance with this invention, it is possible to prevent filament breakage in a drawing step to a negligible degree and to produce extremely strong nylon filaments by a stable drawing operation. Since drawing is effected immediately after melt spinning, the operational steps are simplified and the man power need reduced, thus making it possible to obtain strong nylon filaments which are very advantageous industrially.

What we claim is:

1. A process for the manufacture of strong nylon filaments which comprises melt spinning a polyamide which cools and solidifies to form a hardened filament and, immediately thereafter, drawing the thus hardened filaments in at least two stages, said filaments having a birefringence not greater than 0.008 as measured after the filament has been left to stand for 10 to 20 minutes immediately after hardening at a temperature of 20° C. and 65% RH, the first stage drawing being effected at a drawing ratio within the range of 1.25 to 2.40 times.

2. A process as claimed in claim 1 wherein the total drawing ratio of the filaments in said stages is at least 4.0.

3. A process as claimed in claim 2 wherein the filaments are drawn in the second and any subsequent stages with a draw pin and hot plate, whereas in the first stage the pin and hot plate are not employed.

4. A process as claimed in claim 1 wherein the first stage drawing is carried out within 20 seconds from the melt spinning.

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JAMES A. SEIDLECK, *Primary Examiner*.

ALEXANDER H. BRODMERKEL, *Examiner*.

L. M. GARRETT, *Assistant Examiner*.