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(71) Applicant: **Toray Industries, Inc.**
**Tokyo 103-8666 (JP)**

(72) Inventors:
- **MINAI, Kazushi**
  **Okazaki-shi, Aichi 444-8522 (JP)**
- **SHIGENO, Hisao**
  **Okazaki-shi, Aichi 444-8522 (JP)**
- **URUMA, Takashi**
  **Okazaki-shi, Aichi 444-8522 (JP)**

(74) Representative: **Mewburn Ellis LLP**
**Aurora Building**
**Counterslip**
**Bristol BS1 6BX (GB)**

# (54) POLYAMIDE MULTIFILAMENT, AND METHOD FOR MANUFACTURING SAME

(57) A problem to be solved by the present invention is to provide a polyamide multifilament having a low fineness, a high tenacity, and good fluffy quality. To solve this problem, a main object of the present invention is that the polyamide multifilament is characterized by being composed of a polyamide resin and having a total fineness of 30.0 to 150.0 dtex, a tenacity of 7.5 to 10.0 cN/dtex, and an elongation at break of 15.0 to 35.0%.

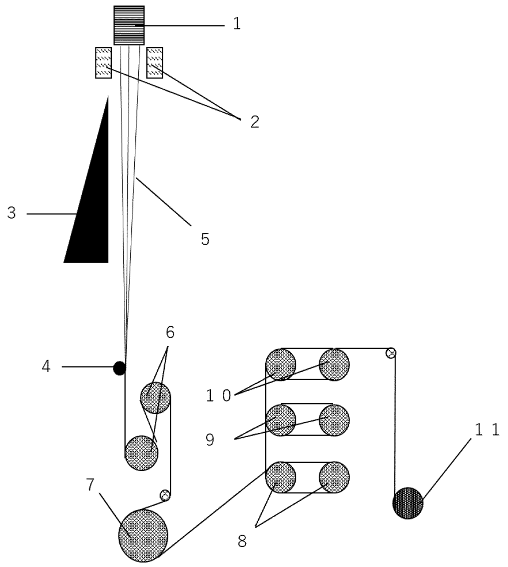

Fig. 1

## Description

### Technical Field

**[0001]** The present invention relates to a polyamide multifilament.

### Background Art

**[0002]** A multifilament of polyamide 6 (also known as "polycaprolactam") or polyamide 66 (also known as "polyhexamethylene adipamide") has higher tenacity and elongation and better fluffy quality than a general-purpose multifilament of polyester, polypropylene, or the like, and thus, is used in various industrial applications such as air bags, catgut for sports rackets, ropes, fishing nets, and belts for bags.

**[0003]** Here, an air bag is taken for example. Air bags, which have been rapidly increasing in the ratio of installation as essential safety devices for protecting passengers in a collision between vehicles, are continuing to increase in terms of the points of installation, for example, an air bag for protection of the knee, an air bag built in a sheet for a seat to protect the chest, and an air bag built in the ceiling above the window to protect the head, in addition to air bags that have been installed for protection of a driver and a front passenger since the initial days. An increase in the number of the points of installation, a demand for lower fuel consumption to be improved year after year, and a recent years' tendency to expand the space in a vehicle are accompanied by various studies to develop a lighter and more compact base cloth to be used for an air bag.

**[0004]** The total fineness of a polyamide 66 yarn to be used for an air bag base cloth was generally 940 dtex in the past, but in recent years, a 470 dtex yarn has been used mainly, and furthermore, a yarn having a fineness as low as 235 dtex or less is also used.

**[0005]** Patent Literature 1 and Patent Literature 2 disclose a technology that affords a polyamide fiber that has a low fineness and a high tenacity with high fluffy quality and can provide an air bag having excellent compactness. The total fineness proposed is 100 to 250 dtex in Patent Literature 1 and 50 to 470 dtex in Patent Literature 2. As seen in the Examples section, however, only a fiber having a fineness down to 175 dtex at best was obtained, and the section does not describe any specific example of a yarn the total fineness of which is made as low as 150 dtex or less, furthermore 100 dtex or less, and which has a high tenacity and thus can be used as an industrial fiber. The reason for this is, as described in Patent Literature 1 (paragraph [0021]), that a lower total fineness makes it more difficult to stably obtain a fiber having a high tenacity.

**[0006]** The reason for this is that obtaining a multifilament having a low total fineness involves increasing the influence of the retention of the molten state of polyamide chips melted before being spun and the influence of defects such as voids remaining in the fiber being spun. That is, no consideration is taken of a problem that will be caused when a multifilament having a fineness of 100 dtex or less is produced using an apparatus such as a direct drawing machine that is included in common facilities for producing fibers for industrial materials having a high tenacity, such as a yarn for air bags, and that is a combination of a spinning unit for a high total fineness of approximately 235 to 2000 dtex and a multistage heat-drawing machine for developing a high tenacity. More specifically, in cases where the fineness is made as low as 100 dtex or less in common facilities for producing fibers for industrial materials, viscosity increase, thermal degradation, and gelation due to the long-time retention of a polymer lead to generating minute foreign matter, and the foreign matter leaks into the yarn, and thus causes an obstruction to yarn-making, making it not possible to afford a polyamide fiber having a high tenacity and good fluffy quality, as described in Patent Literature 3 (paragraphs [0005] to [0008]) and Patent Literature 4 (paragraph [0013]). As above-mentioned, it is extremely difficult to produce a polyamide fiber that is a multifilament having a low fineness, and nonetheless has a high tenacity and good fluffy quality.

**[0007]** On the other hand, in the field of high-tenacity polyamide multifilaments for clothing, a cloth for existing high-order processed articles have been desired to have an even higher tenacity, be made of thinner cloth, and be more compact in recent years, and accordingly, yarns have been desired to have a higher tenacity and the accompanying lower fineness.

**[0008]** In response to such a demand for a higher tenacity for polyamide fibers for clothing, Patent Literature 5 discloses a means by which yarn is drawn and heat-fixed, or heat-drawn, once wound on a package, further brought in contact with a hot plate heated to 170 to 205°C, and thereby heat-drawn 1.15-fold or more. However, this means involves a method including two separated processes, i.e. a spinning process and a drawing process. Thus, not only are the processes complicated but also the production speed is low with the winding speed at approximately 1000 m/min, thus causing concern about higher cost. In addition, according to Patent Literature 6, a product obtained by a step that could be commercialized was only a polyamide multifilament having a tenacity up to 7.3 cN/dtex at best, and the tenacity was insufficient for an attempt to enhance the durability of fibers for industrial materials such as air bags and that of textiles for clothing.

## Citation List

### Patent Literature

**[0009]**

Patent Literature 1: JP 2017-222939 A
Patent Literature 2: JP 2003-20566 A
Patent Literature 3: JP 2007-254945A
Patent Literature 4: JP 2008-133566 A
Patent Literature 5: JP H11-247022 A
Patent Literature 6: JP 2002-88577 A

## Summary of Invention

### Technical Problem

**[0010]** An object of the present invention is to solve the above-mentioned problems, to provide a polyamide multifilament having a low fineness, a high tenacity, excellent toughness, and in addition, good fluffy quality, and to provide a polyamide multifilament that makes it possible to attempt to decrease the weight of an industrial material such as an air bag, and to enhance the durability of a textile for clothing, as above-mentioned. In addition, the present invention makes it possible to obtain a polyamide multifilament which is good also in terms of production efficiency.

### Solution to Problem

**[0011]** The present invention has been made through a vigorous study to solve the above-mentioned problems, and is mainly composed of the following constituents.

(1) A polyamide multifilament including a polyamide and having a total fineness of 30 to 150 dtex, a tenacity of 7.5 to 10.0 cN/dtex, and an elongation at break of 15.0 to 35.0%.
(2) A polyamide multifilament including a polyamide and having a total fineness of 50 to 120 dtex, a tenacity of 8.0 to 9.7 cN/dtex, an elongation at break of 17.0 to 30.0%, a variation coefficient of elongation of 1.00% or less at 3 cN/dtex.
(3) The polyamide multifilament according to (1) or (2), including 0 to 3 fluffs/10,000 m.
(4) The polyamide multifilament according to any one of (1) to (3), wherein the number of voids contained in a polyamide filament as a constituent of the polyamide multifilament is 50 voids/cm or less.
(5) A method of producing the polyamide multifilament according to (1) or (2), including the steps of: providing polyamide chips; and making yarn using a method of direct spinning and drawing, wherein $|\eta a - \eta b| < 0.3$ is satisfied, assuming that sulfuric acid relative viscosity of the polyamide chips is $\eta a$, and that sulfuric acid relative viscosity of the polyamide multifilament made into yarn is $\eta b$.

### Advantageous Effects of Invention

**[0012]** The present invention can provide a polyamide multifilament having a low fineness and nonetheless having a high tenacity, excellent toughness, and in addition, good fluffy quality, and makes it possible to attempt to decrease a weight of an industrial material, and to enhance durability of a textile for clothing.

## Brief Description of Drawings

**[0013]** Fig. 1 is a schematic diagram for describing an example of a step of producing a polyamide multifilament according to the present invention.

## Description of Embodiments

**[0014]** The present invention will now be described with reference to examples. However, the present invention should not be construed as limited to the specific examples described below.

**[0015]** Examples of a raw material to be used for a polyamide multifilament according to the present invention include: nylon 6; nylon 66; nylon 12; nylon 46; nylon 56; nylon 610; copolymer polyamides of nylon 6 and nylon 66; copolymer polyamides obtained by copolymerizing nylon 6 with a polyalkylene glycol, dicarboxylic acid, diamine or other copolymerizable monomers. These are known as polymers containing repeating units linked via an amide bond. Such a material is subject to no particular limitation as long as the material is a polyamide. Among these materials, polyamide 66 having excellent impact resistance and heat resistance is preferably used. As needed, a polyamide multifilament according to the present invention may contain a component other than a polyamide. Examples of such a component include: end-capping agents such as monocarboxylic acids; delustering agents such as titanium oxide; polymerization catalysts and heat resistance promoters, such as phosphorus compounds; and antioxidants and heat stabilizers, such as copper compounds and halides of alkali metals or alkaline earth metals. The content ratio of the polyamide in the polyamide multifilament is preferably 95 wt% or more, more preferably 97 wt% or more. In cases where the content ratio of the polyamide is less than 95 wt%, the heat resistance is decreased in some cases.

**[0016]** A polyamide multifilament according to the present invention has a total fineness of 30 to 150 dtex. The total fineness is more preferably in the range of from 50 to 120 dtex. With the total fineness of less than 30 dtex, it is difficult to achieve a sufficient value as the total strength of the multifilament, and, in addition, drawing at a high ratio to obtain a high tenacity tends to cause single yarn breakage, and also has a higher possibility of generating fluffs. The total fineness of more than 150 dtex does not lead to decreasing the weight of an industrial material or to enhancing the durability of a textile for clothing.

**[0017]** The tenacity of a polyamide multifilament according to the present invention is 7.5 to 10.0 cN/dtex, more preferably 8.0 to 9.7 cN/dtex. Having a tenacity in such a range allows the polyamide fiber to be suitable for industrial materials such as air bags and for textiles for clothing that have excellent durability. The tenacity of less than 7.5 cN/dtex is insufficient to enhance the durability of fibers for industrial materials such as air bags and that of textiles for clothing. Bringing the tenacity of the polyamide fiber to more than 10.0 cN/dtex involves mechanical drawing at a high ratio, tends to generate single yarn breakage, and in addition, degrades fluffy quality. Such a polyamide multifilament is unsuitable for quality-demanding fibers for industrial materials such as air bags.

**[0018]** In addition, a polyamide multifilament according to the present invention has an elongation at break of 15.0% to 35.0%, more preferably 17.0% to 30.0%. The higher the elongation at break, the more preferable. The elongation at break is realistically 35.0% or less to obtain a predetermined tenacity with a polyamide. This range makes it possible to increase the toughness of the polyamide multifilament and the amount of work to break, and to maintain excellent durability.

**[0019]** Although depending on the total fineness and the single-filament fineness, the product of a tenacity and a square root of elongation is preferably 38 cN/dtex. ($\%^{1/2}$) or more, more preferably 40 cN/dtex·($\%^{1/2}$) or more. Having a high product of a tenacity and a square root of elongation makes it possible to inhibit generation of fluffs, yarn breakage, and the like, and thus, to obtain a polyamide multifilament having a high tenacity and nonetheless having extremely high quality. In this regard, the tenacity (cN/dtex) and the elongation at break (%) each refer to a value measured under the conditions of constant rate of elongation that are described in JIS L1013 (1999), 8.5.1 Standard-condition Test. The product of a tenacity and a square root of elongation is a value calculated in accordance with [tenacity $\times$ $\sqrt{}$(elongation at break)]. The upper limit is subject to no particular limitation, and is practically 50.0 cN/dtex. ($\%^{1/2}$) or less.

**[0020]** A polyamide multifilament according to the present invention preferably has an uster unevenness (U%) of 1.2% or less. The user unevenness is still more preferably 1.0% or less, particularly preferably 0.8% or less. Bringing the U% to 1.2% or less allows a textile for clothing to generate no uneven dyeing or no streak during dyeing, and to have good appearance and excellent product quality. The lower limit is subject to no particular limitation, and is practically 0.3% or more.

**[0021]** In addition, a polyamide multifilament according to the present invention preferably has a variation coefficient of elongation of 1.00% or less at 3 cN/dtex. The variation coefficient of elongation at 3 cN/dtexs is still more preferably 0.80% or less, particularly preferably 0.50% or less. Having the variation coefficient of 1.00% or less makes it possible that, when a given load is applied to an industrial textile such as an air bag, the elongation at break of the multifilament is uniformized. Thus, such a variation coefficient is advantageous from the viewpoint of inhibiting the distortion and slippage of the yarn. In addition, the variation coefficient is due to the irregularity of the crystal structure, and thus, is related to inhibiting uneven dyeing in the case of a textile for clothing. A simple means of decreasing the variation coefficient of elongation at 3 cN/dtex to 1.00% or less is to control a difference between sulfuric acid relative viscosity of polyamide chips to be used and sulfuric acid relative viscosity of the resulting polyamide multifilament. In some of the cases where this difference in viscosity is large, the raw material chips before being made into yarns undergo local viscosity increase or hydrolysis due to thermal cross-linking or the like. Such viscosity increase generates spots at which the crystalline orientation is locally increased in the longitudinal direction of the fibers, and such hydrolysis generates spots at which the crystalline orientation is locally decreased in the longitudinal direction of the fibers. In either case, the elongation at break is prone to undergo irregularity. In this regard, the variation coefficient of elongation at 3 cN/dtex is determined by the method described in the Examples section.

**[0022]** A polyamide multifilament according to the present invention preferably has 0 to 3 fluffs/10,000 m or less, particularly 0 to 2 fluffs/10,000 m, more preferably 0 to 1 fluff/10,000 m. Having a smaller number of fluffs makes it possible to expand the uses to articles desired to have excellent fluffy quality, such as air bags. In this regard, the number of fluffs refers to a value converted to the number of fluffs/10,000 m from the total number of fluffs measured along a filament having a length of 100,000 m or more while the filament is rewound at a rate of 150 m/minute.

**[0023]** For a polyamide multifilament according to the present invention, the number of voids contained in the polyamide filament as a constituent of the polyamide multifilament is preferably 50 voids/cm or less, that is, 0 to 50 voids/cm, particularly 0 to 30 voids/cm, more preferably 0.2 to 20 voids/cm. In cases where the number of voids contained in the polyamide filament is more than 50 voids/cm, the tenacity of the single filament containing the voids is decreased. This means that the voids in the single filament inhibit drawing. In addition, the polymer being melted excessively takes in moisture from the air, and thereby causes hydrolysis, causes a decrease in the viscosity of the polyamide, and leaves the crystalline orientation insufficient, resulting in a decrease in the tenacity. In addition, the fluffy quality is degraded. On the other hand, in cases where the number of fluffs is 0.2 or more, allowing the polymer being melted to take in moisture from the air makes it possible to obtain a yarn having good fluffy quality. Examples of a method of decreasing the voids include a method in which the pressure at which polyamide is extruded by an extruder is adjusted to 20.0 to 80.0 kPa.

**[0024]** Fig. 1 is a schematic diagram of a direct spinning and drawing machine to be preferably used in the present invention.

**[0025]** Below, a method of producing a polyamide multifilament according to the present invention will be described with reference to Fig. 1.

**[0026]** First, raw material chips of polyamide are provided as a raw material for a polyamide multifilament according to the present invention. As a method of polymerizing a polyamide, any known polymerization method can be used.

**[0027]** Sulfuric acid relative viscosity of the polyamide raw material chips to be used for a polyamide multifilament according to the present invention (hereinafter referred to simply as "viscosity" in some cases) is preferably 2.8 to 3.9, more preferably 3.3 to 3.9. With the chips having a viscosity of 4.0 or more, bringing the total fineness into a range prescribed in the present invention leads to generating minute foreign matter due to viscosity increase, thermal degradation, and gelation caused by the long-time retention of the polymer, and thus, degrading the fluffy quality. With the chips having a viscosity of less than 2.8, it is difficult to obtain a polyamide multifilament having a tenacity prescribed in the present invention. In this regard, the sulfuric acid relative viscosity refers to a value obtained by using an Ostwald viscometer at 25°C to measure the viscosities of the following: a solution of 1 g of chips dissolved in 100 ml of 98% sulfuric acid; and 98% sulfuric acid having no chip dissolved therein. The details of the measurement are as described in the Examples section.

**[0028]** In the production of a polyamide multifilament according to the present invention, it is preferable that a method of direct spinning and drawing is used, and that $|\eta a - \eta b| < 0.3$ is satisfied, assuming that sulfuric acid relative viscosity of polyamide chips used as a raw material is $\eta a$, and that sulfuric acid relative viscosity of a polyamide multifilament made into yarn is $\eta b$. Preferably, $|\eta a - \eta b|$ is less than 0.2. For example, a polyamide multifilament produced in accordance with $|\eta a - \eta b| < 0.3$ has extremely good fluffy quality and high tenacity and elongation, and can be obtained as a polyamide multifilament having less irregularity in the elongation at break when elongated 3%. Although the details are unclear, this is considered to be because satisfying $|\eta a - \eta b| < 0.3$ inhibits viscosity increase and thermal degradation due to the long-time retention of the polymer, or inhibits the hydrolysis of the polyamide. In addition, the polyamide multifilament produced can be sorted out in an inspection step after the production, if such sorting-out is permissible from a productivity point of view.

**[0029]** Next, an example of a production method that satisfies $|\eta a - \eta b| < 0.3$ will be described. Polyamide chips having above-mentioned sulfuric acid relative viscosity are provided, dried, then supplied into an extruder type spinning machine, led into a spinneret with a measuring pump, and melt-spun. When this is done, the pressure at the feeding unit in the extruder is preferably not a vacuum (having a pressure of 0.0 kPa) but 20.0 to 80.0 kPa in order to inhibit the viscosity increase, thermal degradation, and gelation of the polymer. The pressure is more preferably 40.0 to 60.0 kPa. In some of the cases where the pressure at the feeding unit in the extruder is less than 20.0 kPa, the fluffy quality is degraded owing to the viscosity increase, thermal degradation, and gelation of the polymer, and in addition, yarn having a high tenacity cannot be obtained. In cases where the pressure at the feeding unit in the extruder is 80.0 kPa or more, the number of voids contained in the polyamide filament is increased, and furthermore, the hydrolysis reaction of the polymer is predominant, thus making it not possible to obtain a yarn having a high tenacity.

**[0030]** Referring to Fig. 1, polyamide spun through a spinneret 1 is preferably allowed to pass through a heating hood 2 surrounding the range 5 to 300 cm immediately below the spinneret. In a preferable embodiment, the temperature in this heating hood is -30 to +30°C, more preferably -15 to +15°C, with respect to the melting point of the polymer polyamide. The yarn spun out is not immediately cooled but slowly cooled through the high-temperature atmosphere surrounded by the above-mentioned heating hood. Thereby, the orientation of the polyamide molecules melt-spun is relaxed, and makes it possible to enhance the molecular orientation uniformity between single filaments, thus enabling the polyamide multifilament to have a higher tenacity. On the other hand, if the yarn is immediately cooled without passing through the high-temperature atmosphere, the orientation of the yarn undrawn is increased, and the irregularity in the degree of orientation is increased among the single filaments. Heat-drawing such an undrawn yarn results in a possibility of failing to obtain a polyamide multifilament having a high tenacity.

**[0031]** An undrawn yarn 5 that has passed through the high-temperature atmosphere is then blown by air at 10 to 80°C, preferably 10 to 50°C, using a cross flow cooling device 3, and thus cooled and solidified. Additionally, in cases where the cooling air is more than 80°C, the single filaments are more shaken during spinning, and thus, for example, the single filaments collide thereamong, resulting in difficulty in yarn production.

**[0032]** Then, the cooled yarn is provided with an oil agent using a known oil supply device 4, taken up on a take-up roll 6, drawn, and then can be wound up. As the oil agent, any known oil can be used. The amount of application is preferably 0.3 to 1.5 wt%, still more preferably 0.5 to 1.0 wt%, to inhibit the entwinement of the single yarns around the take-up roll 6.

**[0033]** In addition, the spinning rate defined by the rotational speed of the take-up roll 6 is preferably 500 to 1200 m/minute, more preferably 600 to 800 m/minute. With the spinning rate at 500 m/minute or more, the final production speed is sufficient, and the polyamide multifilament can be produced inexpensively with high production efficiency. The spinning rate of 1200 m/minute or less makes it possible to inhibit a generation of fluff and yarn breakage, and thus, is preferable. In addition, the drawing rate represented by the maximum speed of the drawing roll is preferably 2800 m/minute or more, more preferably 3000 m/minute or more.

**[0034]** Such an undrawn yarn obtained by the above-mentioned method can be drawn, undergo relaxation heat treatment, be wound up, and so on using a known method. Here, a specific example of two-stage drawing is described. The undrawn yarn taken up on the take-up roller 6 (1FR) is wound up on a feeding roller 7 (2FR), a first drawing roller 8 (1DR), a second drawing roller 9 (2DR), and a relaxing roller 10 (RR) in this order, heat-treated and drawn, and wound up on a winder 11.

**[0035]** A prestretch drawing is performed between 1FR and 2FR, a 1st-stage drawing is performed between 2FR and 1DR, and a 2nd-stage drawing is performed between 1DR and 2DR. It is preferable that the temperature for 2FR is set at 30 to 50°C, that the temperature for 1DR is set at 100 to 225°C, and that the prestretch drawing and the 1st-stage drawing are heat-drawing processes performed at and around the glass transition temperature. The remaining drawing and heat-setting processes are usually preferably performed in the temperature range of from 180 to 240°C. The temperature is more preferably 200 to 220 °C.

**[0036]** For a total draw ratio (hereinafter referred to simply as a "draw ratio"), that is, a ratio at which drawing is performed between the take-up roller 6 and the second drawing roller 9, a high draw ratio is preferably adopted to afford a polyamide multifilament having a high tenacity, and the draw ratio has only to be 3.8 to 5.0 as long as the ratio allows the fineness to fall within the range described in the present invention. The winding speed is usually preferably 2000 to 5000 m/minute, more preferably 2500 to 4500 m/minute. In addition, the yarn is preferably wound up in the form of a cheese stripe using a winding device under conditions where the winding tension is 20 to 250 gf.

**[0037]** Using the method described above makes it possible to inhibit the viscosity increase, thermal degradation, gelation, and hydrolysis of the polyamide polymer, lessen the influence of the voids on the mechanical properties, and obtain a polyamide multifilament having a low fineness of 150 dtex or less as a total fineness, and nonetheless having a high tenacity and high elongation at break, that is, high toughness, and in addition, good quality.

## Examples

**[0038]** The present invention will now be described in detail with reference to Examples. However, the present invention should not be construed as limited to the aspects specifically described in the Examples. The definition of the characteristics and the measurement methods in the present invention are as below-mentioned.

**[0039]**

(1) Sulfuric acid relative viscosity ($\eta r$): determined in accordance with the below-mentioned formula, for which measurements were made with 0.25 g of a sample dissolved in 25 ml of 98% sulfuric acid, using an Ostwald viscometer at 25°C, wherein the sample used was polymer chips or yarns. The measurement values were each determined from the average value of five samples.

$$\eta r = \text{number of seconds during downflow of sample solution/number of seconds during downflow of sulfuric acid}$$

(2) Total fineness: measured in accordance with JIS L1090 (1999)

(3) Number of single filaments: calculated using the method in JIS L1013 (1999), 8.4

(4) Single-filament fineness: calculated by dividing the total fineness by the number of single filaments

(5) Strength,·tenacity, and elongation at break: measured under the conditions of constant rate of elongation that are described in JIS L1013 (1999), 8.5.1 Standard-condition Test. A multifilament sample was used in a "TENSILON" (TENSILON) UCT-100 manufactured by Orientec Corporation. The distance between grips was 25 cm, and the tension rate was 30 cm/minute. The strength was determined from the maximum strength on an S-S curve. The elongation at break was determined from the elongation at the point representing the maximum strength on the S-S curve. The tenacity was determined by dividing the strength by the total fineness. Multifilament samples were sampled at 1 m intervals in the longitudinal direction, measurements were made at five points, and the average value was determined from the measurement data.

(6) Uster unevenness (U%): measured in terms of 1/2 Inert with a sample having a length of 500 m and at a measurement yarn rate of 25 m/min, using an USTER TESTER IV manufactured by Zellweger Uster AG

(7) Variation coefficient of elongation at 3 cN/dtex: based on an elongation at break with a load of 3 cN/dtex applied, in which the elongation was determined from an S-S curve determined under the same conditions as the strength and the elongation at break were measured in the above-mentioned (5). In addition, multifilament samples to be used for measurement were sampled at 1 m intervals in the longitudinal direction of the fiber, and measurements were made at ten points. The average value and the standard deviation were calculated from the measurement data, and the variation coefficient was determined in accordance with the following formula.

$$\text{Variation coefficient} = [\text{standard deviation}] / [\text{average value}] \times 100\ (\%)$$

(8) Number of fluffs: evaluated as the total number of fluffs detected in the resulting fiber package being rewound at a rate of 150 m/minute, using a laser fluff detector "FLYTECH V" manufactured by Heberlein, in which the detector was placed 2 m apart from the yarn being rewound. Multifilaments 100,000 m or longer were evaluated, and the number obtained was expressed in terms of the number of fluffs/10,000 m.

(9) Number of voids: evaluated on the basis of the number of voids observed under a microscope "VHX-5000" manufactured by Keyence Corporation using a 1000-fold magnifying lens. In cases where there is a void in the fiber, a portion starting from the void is inhibited from being drawn. For this reason, the voids were observed under a 1000-fold magnifying optical lens, and then, portions inhibited from being drawn were recognized with a polarizing lens to verify the presence of voids. Samples were made by cutting out fibers having the same length from all the polyamide filaments that constituted a polyamide multifilament. Here, the samples were made in such a manner that the total length of the fibers cut out was 100 cm. The samples cut out were observed, and the total number of voids was expressed in terms of the number of voids/cm. In this regard, the samples to be cut out do not have to be strictly 100 cm as long as the total length of 100 cm is secured for measurement.

(Example 1)

**[0040]** An aqueous solution of 5 wt% copper acetate as an antioxidant was added to and mixed with nylon 66 chips obtained by liquid-phase polymerization, so that copper was added and adsorbed at 68 ppm with respect to the weight of the polymer. Next, an aqueous solution of 50 wt% potassium iodide and an aqueous solution of 20 wt% potassium bromide were added to and adsorbed in the polymer chips in such a manner that the ratio of the potassium from each solution was 0.1 part by weight with respect to 100 parts by weight of the polymer chips. The resulting mixture was solid-phase-polymerized using a batch type solid-phase polymerization device to obtain nylon 66 pellets having a sulfuric acid relative viscosity of 3.75. The resulting nylon 66 pellets were supplied into an extruder having a diameter of 110 mm, and melted in an atmosphere where the melting temperature was 300°C, and where the pressure at the feeding unit in the extruder was 50.0 kPa. The amount of the molten polymer was adjusted with a measuring pump so as to afford a multifilament having a total fineness of 80 dtex, and the molten polymer was fed into a spinning pack. Then, the molten polymer was filtrated through a metal nonwoven filter having a coarseness of 40 μm in the spinning pack, allowed to pass through a spinneret having 24 circular holes, and spun using an apparatus configured as depicted in Fig. 1. A heating hood having a length of 20 cm was disposed 3 cm below the spinneret face, and heated in such a manner that the atmospheric temperature in the hood was 250°C. Here, the atmospheric temperature in the hood is the temperature of the air in the portion that is in the center of the heating hood in the length direction and is 1 cm apart from the inner wall. Immediately below the heating hood, a cross flow type chimney for blowing air unidirectionally was attached, and the yarn was blown by cold air at 18°C at a rate of 35 m/minute to be cooled and solidified. Then, the yarn was provided with an oil.

**[0041]** The undrawn yarn provided with the oil was wound and taken up on the 1FR rotating at a surface speed of 800 m/minute, and then drawn at a total draw ratio of 4.3. Without being once wound up, the yarn taken up was continuously stretched 5% between the take-up roller and the 2FR, subsequently drawn at a rotational speed ratio of 2.80 in the 1st stage, then drawn at a rotational speed ratio of 1.46 in the 2nd-stage, and wound up at a rate of 3400 m/minute. The surface of each of the 1FR and 2FR rollers was mirror-finished, and that of each of the 1DR, 2DR, and RR was satin-finished. In addition, the temperature of each roller was set as follows: 1FR, non-heated; 2FR, 40°C; 1DR, 150°C; 2DR, 225°C; and RR, 150°C. Such melt-spinning and drawing afforded a nylon 66 multifilament. In an entangling process, high-pressure air was sprayed perpendicularly onto the yarn running through an entangling device. Before and after the entangling device, there was provided a guide for regulating the running yarn. The pressure of the air to be sprayed was set constantly at 0.2 MPa.

(Examples 2 to 5)

**[0042]** These Examples were performed in the same manner as in Example 1 except that the total fineness and total draw ratio of the polyamide multifilament were changed as in Table 1.

(Examples 6 to 8)

**[0043]** These Examples were performed in the same manner as in Example 1 except that the number of single filaments of the polyamide multifilament was changed as in Table 1.

(Examples 9 to 10)

**[0044]** These Examples were performed in the same manner as in Example 1 except that the total draw ratio was changed as in Table 1.

(Examples 11 to 12)

**[0045]** These Examples were performed in the same manner as in Example 1 except that the pressure at the feeding unit in the extruder and the total draw ratio were changed as in Table 1.

**[0046]** The results obtained by evaluating the properties of these polyamide multifilaments obtained in Examples 1 to 12 are tabulated in Table 1.

[Table 1]

| Item | Example 1 | Example 2 | Example 3 | Example 4 | Example 5 | Example 6 | Example 7 | Example 8 | Example 9 | Example 10 | Example 11 | Example 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Total fineness (dtex) | 80 | 50 | 110 | 30 | 135 | 80 | 80 | 80 | 80 | 80 | 80 | 80 |
| Number of filaments | 24 | 24 | 24 | 24 | 24 | 72 | 10 | 136 | 24 | 24 | 24 | 24 |
| Single-filament fineness (dtex) | 3.3 | 2.1 | 4.6 | 1.3 | 5.6 | 1.1 | 8.0 | 0.6 | 3.3 | 3.3 | 3.3 | 3.3 |
| Pressure at feeding unit in extruder (kPa) | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 50.0 | 25.0 | 75.0 |
| Total draw ratio | 4.3 | 4.3 | 4.3 | 4.1 | 4.3 | 4.3 | 4.3 | 4.3 | 3.6 | 3.9 | 4.0 | 4.3 |
| Winding speed (m/min) | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 |
| Tenacity (cN/dtex) | 8.9 | 9.2 | 8.6 | 9.1 | 8.5 | 9.1 | 8.3 | 9.0 | 7.5 | 8.1 | 8.7 | 8.2 |
| Elongation at break (%) | 22.5 | 21.3 | 23.8 | 22.1 | 23.9 | 21.9 | 22.0 | 23.5 | 31.1 | 26.9 | 20.3 | 22.8 |
| Product of tenacity and square root of elongation (cN/dtex·(%$^{1/2}$)) | 42.2 | 42.5 | 42.0 | 42.8 | 41.6 | 42.6 | 38.9 | 43.6 | 41.8 | 42.0 | 39.2 | 39.2 |
| Uster unevenness (%) | 0.76 | 0.94 | 0.71 | 1.15 | 0.70 | 0.92 | 0.78 | 1.38 | 0.79 | 0.77 | 0.79 | 0.78 |
| Variation coefficient of elongation (%) at 3 cN/dtex | 0.69 | 0.84 | 0.60 | 0.96 | 0.60 | 0.70 | 0.72 | 0.71 | 0.83 | 0.75 | 1.07 | 1.05 |
| Number of fluffs (Number/10,000 m) | 0.3 | 0.8 | 0.1 | 1.0 | 0.1 | 1.3 | 0.3 | 3.4 | 0.0 | 0.0 | 2.7 | 2.5 |

(continued)

| Item | Example 1 | Example 2 | Example 3 | Example 4 | Example 5 | Example 6 | Example 7 | Example 8 | Example 9 | Example 10 | Example 11 | Example 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Number of voids (Number/cm) | 0.50 | 0.43 | 0.67 | 0.32 | 0.72 | 0.46 | 0.51 | 0.49 | 0.48 | 0.52 | 0.18 | 24.30 |
| Relative viscosity of polyamide chip | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 |
| Relative viscosity of polyamide multifilament | 3.80 | 3.87 | 3.77 | 3.94 | 3.75 | 3.79 | 3.84 | 3.80 | 3.88 | 3.85 | 4.00 | 3.64 |
| Relative viscosity difference between chip and multifilament (absolute value) | 0.05 | 0.12 | 0.02 | 0.19 | 0.00 | 0.04 | 0.09 | 0.05 | 0.13 | 0.10 | 0.25 | 0.11 |

**[0047]** As obvious from Table 1, the polyamide multifilament according to the present invention has a low fineness and a high tenacity, and nonetheless has good fluffy quality.

**[0048]** In Examples 1 to 5, different polyamide multifilaments according to the present invention that have different total finenesses were produced. Compared with Examples 11 and 12 and the below-mentioned Comparative Examples 1 to 5, melting the polymer in an atmosphere having a pressure of 50.0 kPa at the feeding unit in the extruder made it possible to inhibit viscosity increase, and to obtain a polyamide multifilament of interest. In addition, as the total fineness was decreased, and as the single-filament fineness was lowered, cooling was more advantageous, and thus, the product of a tenacity and a square root of elongation of the polyamide multifilament tended to be increased. On the other hand, lowering the single-filament fineness excessively as in Example 8 resulted in lacking in uniform coolability with chimney air, and exhibited influence on the uster unevenness (U%). Examples 11 to 12 are examples in which yarn was made with the pressure set at 25.0 kPa and 75.0 kPa respectively at the feeding unit in the extruder. Viscosity increase or hydrolysis tended to occur in the process from the chips to the multifilament, and influence on the variation coefficient of elongation at 3 cN/dtex was observed.

(Reference Example 1)

**[0049]** This Reference Example was performed in the same manner as in Example 1 except that the total fineness of the polyamide multifilament was changed to 175 dtex, and that the pellets were melted in an atmosphere having a pressure of 0.0 kPa at the feeding unit in the extruder.

(Comparative Examples 1 to 2)

**[0050]** These Comparative Examples were performed in the same manner as in Reference Example 1 except that the total fineness of the polyamide multifilament was changed to 110 dtex, and that total draw ratio was changed as in Table 2.

(Comparative Examples 3 to 4)

**[0051]** These Comparative Examples were performed in the same manner as in Reference Example 1 except that the total fineness of the polyamide multifilament was changed to 80 dtex, and that total draw ratio was changed as in Table 2.

(Comparative Example 5)

**[0052]** This Example was performed in the same manner as in Comparative Example 4 except that the pressure at the feeding unit in the extruder and the total draw ratio were changed as in Table 2.

(Reference Example 2)

**[0053]** The properties of a multifilament produced in accordance with Example 1 in WO2016/076184, which is a common polyamide multifilament for clothing, are listed as Reference Example 2 in Table 2.

[Table 2]

| Item | Reference Example 1 | Comparative Example 1 | Comparative Example 2 | Comparative Example 3 | Comparative Example 4 | Comparative Example 5 | Reference Example 2 |
|---|---|---|---|---|---|---|---|
| Total fineness (dtex) | 175 | 110 | 110 | 80 | 80 | 80 | 5.9 |
| Number of filaments | 24 | 24 | 24 | 24 | 24 | 24 | 5 |
| Single-filament fineness (dtex) | 7.3 | 4.6 | 4.6 | 3.3 | 3.3 | 3.3 | 1.2 |
| Pressure at feeding unit in extruder (kPa) | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 101.3 | - |
| Total draw ratio | 4.3 | 4.3 | 3.6 | 3.6 | 3.2 | 4.3 | 2.1 |
| Winding speed (m/min) | 3600 | 3600 | 3600 | 3600 | 3600 | 3600 | 3500 |
| Tenacity (cN/dtex) | 8.5 | N.D. | 7.4 | N.D. | 7.2 | 7.1 | 5.6 |
| Elongation at break (%) | 24.0 | N.D. | 19.1 | N.D. | 17.8 | 23.8 | 46.0 |
| Product of tenacity and square root of elongation (cN/dtex·(%$^{1/2}$)) | 41.6 | N.D. | 32.3 | N.D. | 30.4 | 34.6 | 38.0 |
| Uster unevenness (%) | 1.05 | N.D. | 1.37 | N.D. | 1.60 | 1.40 | 0.55 |
| Variation coefficient of elongation (%) at 3 cN/dtex | 0.96 | N.D. | 1.30 | N.D. | 1.73 | 1.47 | 1.68 |
| Number of fluffs (Number/10,000 m) | 0.1 | N.D. | 21.3 | N.D. | 24.8 | 14.7 | - |
| Number of voids (Number/cm) | 0.17 | N.D. | 0.14 | N.D. | 0.10 | 52.10 | - |
| Relative viscosity of polyamide chip | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 3.75 | 2.20 |
| Relative viscosity of polyamide multifilament | 4.00 | N.D. | 4.08 | N.D. | 4.11 | 3.55 | 2.10 |
| Relative viscosity difference between chip and multifilament (absolute value) | 0.25 | N.D. | 0.33 | N.D. | 0.36 | 0.20 | 0.10 |

**[0054]** The results obtained by evaluating the properties of the polyamide multifilaments obtained in Comparative Examples 1 to 5 and Reference Examples 1 and 2 are tabulated in Table 2.

**[0055]** In Reference Example 1, a polyamide multifilament having a total fineness of 175 dtex was produced, and the polymer was melted under vacuum (in an atmosphere having a pressure of 0.0 kPa at the feeding unit in the extruder), and thus, the polymer was a little increased in viscosity, but it is understood that this total fineness made it possible to obtain a polyamide multifilament having such a level of tenacity as desired for fibers for industrial uses. However, the total fineness is large, and thus, not sufficient to achieve that object of the present invention which is to make attempts to decrease the weight of an industrial material such as an air bag, and to enhance the durability of a textile for clothing, both with high production efficiency.

**[0056]** In Comparative Example 1, a polyamide multifilament having a total fineness of 110 dtex was produced in the same manner as in Reference Example 1, but in this case, it was not possible to make yarn. In Comparative Example 2, a polyamide multifilament was made into yarn in the same manner as in Comparative Example 1 except that the draw ratio was 3.6. It was possible to make yarn. However, the resulting polyamide multifilament underwent viscosity increase and thermal degradation due to the long-time retention of the polymer, lost tenacity, and generated many fluffs.

**[0057]** In Comparative Example 3, a polyamide multifilament having a total fineness of 80 dtex was produced under the same yarn-making conditions as in Comparative Example 2, but in this case, it was not possible to make yarn. In Comparative Example 4, a polyamide multifilament was made into yarn in the same manner as in Comparative Example 3 except that the draw ratio was decreased to 3.2. It was possible to make yarn. However, the resulting polyamide multifilament underwent viscosity increase and degradation due to the long-time retention of the polymer, lost tenacity, and generated many fluffs. Compared with Comparative Examples 1 and 2, the fineness was lowered in Comparative Examples 3 to 4, the retention time of the polymer was accordingly increased, the polymer was further markedly increased in viscosity, and thus, the polymer lost drawability, revealing that it was not possible to make yarn at a high draw ratio.

**[0058]** In Comparative Example 5, a multifilament was produced in the same manner as in Example 1 except that the polymer was melted in an atmosphere having a pressure of 101.3 kPa at the feeding unit in the extruder, but in this case, the hydrolysis reaction of the polymer was markedly significant, with the result that the viscosity of the multifilament was much below the viscosity of the chips. Furthermore, an increase in the amount of voids in the yarn caused many fluffs to be generated, thus making it not possible to achieve a tenacity prescribed in the present invention.

**[0059]** Reference Example 2 is an example in which a polyamide multifilament was produced in accordance with WO2016/076184 in common facilities for producing fibers for clothing. In this case, it is understood that, compared with the cases where common facilities for producing fibers for industrial materials are used, the retention time of the polymer was short, and thus the polymer was hardly increased in viscosity. On the other hand, the number of drawing stages was one, and in addition, the draw ratio was low. Thus, the tenacity was not sufficient, and in addition, the crystal structure tended to cause irregularity in the longitudinal direction of the fiber, exhibiting a large influence on the variation coefficient of elongation at 3 cN/dtex.

## Industrial Applicability

**[0060]** A polyamide multifilament according to the present invention has a low fineness and a high tenacity and good fluffy quality, and thus, is suitable mainly in attempts to decrease the weight of an industrial material such as an air bag, and to enhance the durability of a textile for clothing.

## Reference Signs List

**[0061]**

1: Spinneret
2: Heating hood
3: Cross flow cooling device
4: Oil supply device
5: Yarn
6: Take-up roller (1FR)
7: Feeding roller (2FR)
8: First drawing roller (1DR)
9: Second drawing roller (2DR)
10: Relaxing roller (RR)
11: Winder

## Claims

1. A polyamide multifilament comprising a polyamide and having a total fineness of 30 to 150 dtex, a tenacity of 7.5 to 10.0 cN/dtex, and an elongation at break of 15.0 to 35.0%.

2. A polyamide multifilament comprising a polyamide and having a total fineness of 50 to 120 dtex, a tenacity of 8.0 to 9.7 cN/dtex, an elongation at break of 17.0 to 30.0%, a variation coefficient of elongation of 1.00% or less at 3 cN/dtex.

3. The polyamide multifilament according to claim 1 or 2, comprising 0 to 3 fluffs/10,000 m.

4. The polyamide multifilament according to any one of claims 1 to 3, wherein the number of voids contained in a polyamide filament as a constituent of said polyamide multifilament is 50 voids/cm or less.

5. A method of producing the polyamide multifilament according to claim 1 or 2, comprising the steps of: providing polyamide chips; and making yarn using a method of direct spinning and drawing, wherein $|\eta a - \eta b| < 0.3$ is satisfied, assuming that sulfuric acid relative viscosity of said polyamide chips is $\eta a$, and that sulfuric acid relative viscosity of said polyamide multifilament made into yarn is $\eta b$.

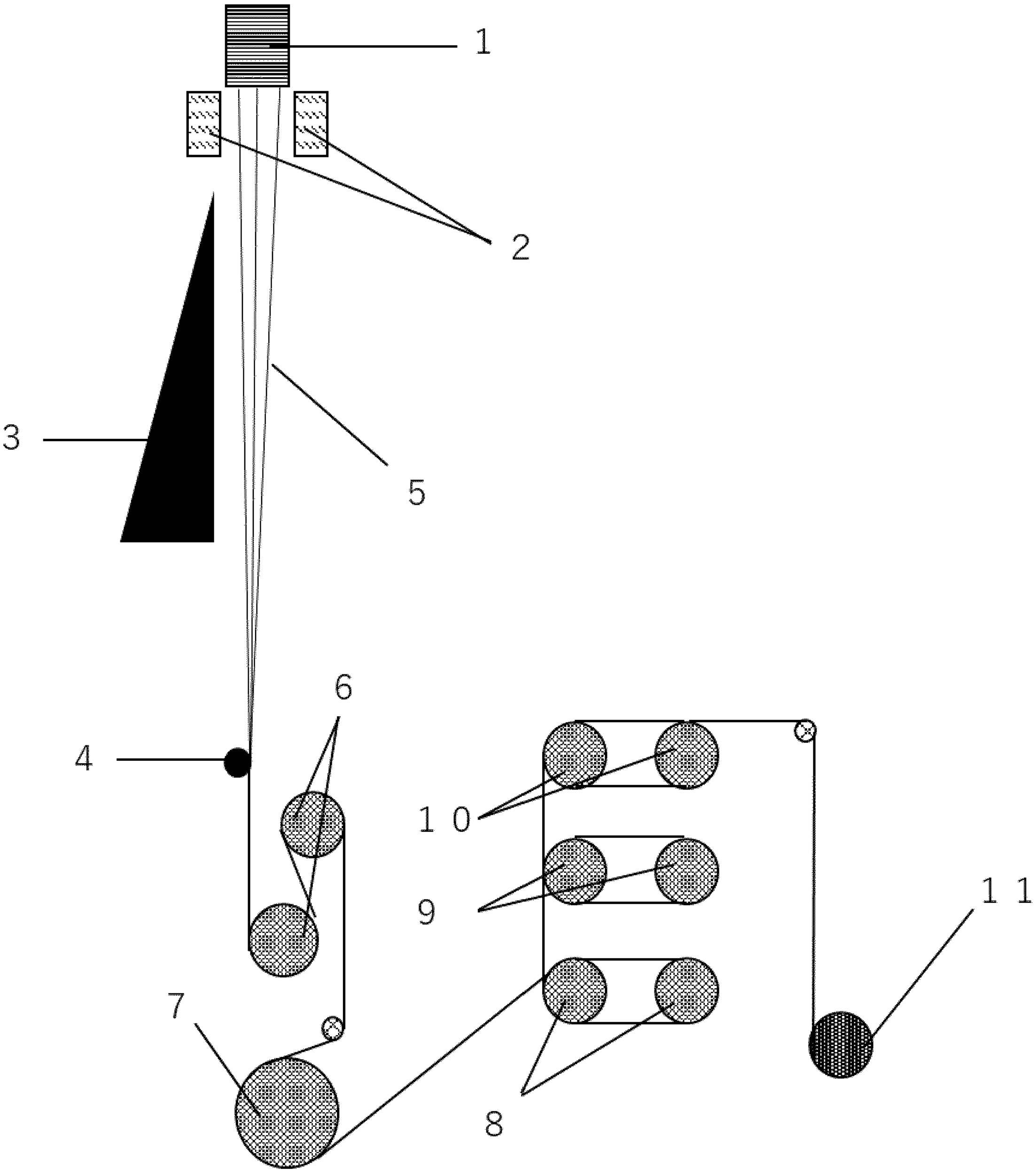

Fig. 1

## INTERNATIONAL SEARCH REPORT

International application No.

**PCT/JP2021/029073**

### A. CLASSIFICATION OF SUBJECT MATTER

***D01F 6/60***(2006.01)i
FI: D01F6/60 321Z; D01F6/60 301E; D01F6/60 351B

According to International Patent Classification (IPC) or to both national classification and IPC

### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

D01F1/00-6/96

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2021
Registered utility model specifications of Japan 1996-2021
Published registered utility model applications of Japan 1994-2021

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
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| X | JP 2005-527714 A (INVISTA TECHNOLOGIES S.À R.L.) 15 September 2005 (2005-09-15)<br>claims 1-2, 17-20, paragraphs [0001], [0020]-[0041], examples 1, 2, fig. 1 | 1-2 |
| Y | JP 2020-12215 A (TEIJIN LTD) 23 January 2020 (2020-01-23)<br>paragraphs [0024]-[0025] | 4 |
| Y | JP 48-46696 A (TEIJIN LTD) 03 July 1973 (1973-07-03)<br>page 3, lower left column, lines 8-17 | 4 |
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| X | JP 6699381 B2 (TORAY INDUSTRIES ) 27 May 2020 (2020-05-27)<br>claims 1, 3, paragraphs [0001], [0019]-[0030] | 1-3 |

☑ Further documents are listed in the continuation of Box C. ☑ See patent family annex.

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

| | |
|---|---|
| Date of the actual completion of the international search<br>**14 October 2021** | Date of mailing of the international search report<br>**26 October 2021** |
| Name and mailing address of the ISA/JP<br>**Japan Patent Office (ISA/JP)**<br>**3-4-3 Kasumigaseki, Chiyoda-ku, Tokyo 100-8915**<br>**Japan** | Authorized officer<br><br>Telephone No. |

## INTERNATIONAL SEARCH REPORT

International application No.
**PCT/JP2021/029073**

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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**Information on patent family members**

International application No.
**PCT/JP2021/029073**

| Patent document cited in search report | Publication date (day/month/year) | Patent family member(s) | Publication date (day/month/year) |
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