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STRETCHING PROCESS

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This invention relates to a process for stretching melt spun filaments of a polymer of metaxylylene diamine and adipic acid. Throughout this specification and the appended claims, the phrases "polymer of metaxylylene diamine and adipic acid" or "polymetaxylylene adipamide" are intended to include a copolymer of metaxylylene diamine, adipic acid and not more than 10% (expressed as a percentage of the total diamines) of paraxylylene diamine.

Melt spun filaments of these polymers may be produced by melting at a temperature above the melting point but below their decomposition temperature and preferably between 285° C. and 292° C. extruding the molten polymer through a spinneret and then passing the filaments so produced through a steam conditioning tube to a wind-up bobbin, preferably rotating at a surface speed in excess of 2,000 feet per minute. These filaments are in the amorphous, i.e., non crystalline, state.

In order that useful textile yarns and fibres may be prepared from the melt spun filaments produced as described above, it is necessary that the filaments should be heated above their apparent second order transition temperature and stretched by at least 300% to provide highly crystalline oriented filaments with good tensile properties.

We have found that, as is the case with other synthetic polymeric filaments, it is preferable that the heating of the filament during the drawing operation should take place around a heated pin and that the temperature of the pin should be fixed in order to provide a smooth running process giving drawn filaments with constant properties.

We have also found, however, that it is essential to control the moisture content of the spun filaments prior to and during drawing and that there exists a relationship between the fixed temperature of the pin around which the yarn is drawn and the moisture content of said filaments which relationship must be fulfilled if optimum properties, uniformity of product and smooth running of the drawing process are to be obtained. The moisture content of the filaments at any particular temperature is suitably expressed and measured as a function of the percent relative humidity of the atmosphere surrounding the filaments. To a first approximation, between 0 and 66% RH at 70° F. the relation is $M=0.09 \times RH$ (where M is the percentage moisture content of the fibre).

It is well known in the art of drawing of crystallisable synthetic fibres that considerable advantage is to be gained from the use of a heated plate in conjunction with a hot pin in the process. This plate may either be placed between the pin and the draw roll, with the yarn running over the plate, or, if so desired, the plate may be placed after the draw roll, in which case a second such roll is needed and the yarn may be allowed to undergo a controlled shrinkage, or may be left at constant length or may be further stretched whilst passing over the plate. If so desired the yarn may be made to pass over hot plates associated with the first and second draw rolls. Alternatively, if desired, the heat treatment obtained by use of the hot plate, may be carried out after processing in an

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oven with or without the use of a hot plate in the drawing process. The advantages to be gained from using such devices arise from the fact that the increased crystallisation induced by one or both of such treatments can result in higher tenacities and reduced shrinkage at elevated temperatures. The use of such multi-stage drawing processes is of course not necessarily limited in principle to two stages.

In the same way as the moisture content of the yarn affects its drawing behaviour at various pin temperatures, so also it does at various plate temperatures and the optimum temperature of the hot plate is defined by the moisture content of the yarn, and hence the temperature and relative humidity of the atmosphere in which the yarn is stored prior to and during drawing.

We have discovered also that in order to produce useful textile filaments and yarns from substantially amorphous melt spun filaments made from polymers of metaxylylene diamine and adipic acid, the environment in which the melt spun fibres are kept until they have been heated and stretched, must be rigidly controlled with respect to relative humidity and temperature and that there is a strict relationship between the relative humidity and, therefore, the moisture content of the fibres, and the temperature of the spun fibres such that if the moisture content or temperature is too high the fibres spontaneously crystallise to such an extent that subsequent stretching is difficult.

According to our invention we provide a drawing process for melt spun filaments made from a polymer of metaxylylene diamine and adipic acid as hereinbefore defined where the melt spun filaments are drawn while passing through a heating zone which may be a heated pin or roll on which the filaments are drawn to at least 3 times their original length, characterised in that the minimum temperature of the heating zone (T_p) satisfies, to a first approximation the following equation:

$$T_p^\circ \text{ C.} = 109 - 9.25M \quad (1)$$

where M is the percent moisture content of the spun yarn which is to be drawn. As the temperature and relative humidity of the atmosphere control the moisture content of the yarn it is convenient to express Equation 1 in terms of relative humidity. The appropriate equation for yarn conditioned at 70° F. is

$$T_p^\circ \text{ C.} = 109 - 0.83H \quad (1a)$$

where H is the relative humidity at 70° F. in which the yarn is conditioned prior to drawing.

It is to be understood that it is the moisture content M of the spun yarn which governs the pin temperature and that a given value of M can be obtained by any suitable choice of atmospheric temperature and relative humidity: Equation 1a merely presents Equation 1 in a form particularly convenient for working with and storage of the yarn at a comfortable ambient temperature.

In a preferred embodiment of our invention, we provide a drawing process for melt spun filaments made from a polymer of metaxylylene diamine and adipic acid wherein the melt spun filaments are drawn through a heating zone comprising a heated pin or roll, the temperature of which (T_p) satisfies to a first approximation the equation $T_p^\circ \text{ C.} = 109 - 9.25M$ where M is the percent moisture content of the spun yarn being drawn, and then over a heated plate the temperature of which (T_{p1}) is not lower than that given by the equation:

$$T_{p1}^\circ \text{ C.} = 172 - 11.1M \quad (2)$$

where M is the percent moisture content of the spun yarn. It is convenient to express this Equation 2 as

$$T_{p1}^\circ \text{ C.} = 172 - H \quad (2a)$$

where H is the percent relative humidity at 70° F. of the

atmosphere in which the yarn is conditioned prior to drawing.

In another embodiment of our invention we provide a drawing process for melt spun filaments made from a polymer of metaxylylene diamine and adipic acid wherein the maximum relative humidity of the atmosphere surrounding the bobbin of collected spun yarn, from substantially the time that said bobbin is removed from the spinning machine to the time when the spun yarn leaves the bobbin to be fed to a drawing system shall satisfy the equation:

$$RH_{\max}=118-0.55T \quad (3)$$

where $RH_{\max}$ is the maximum percent relative humidity and T is the ambient temperature in ° F. and preferably, the conditions should satisfy the equation:

$$RH_{\max}=108-0.55T \quad (3a)$$

We have found that the maximum tenacity is achieved by operating under conditions defined by Equations 1 and/or 2. If the pin temperature at a given relative humidity, falls significantly below the value as calculated from the equation, then running of the drawing process becomes difficult and tenacities obtained are also reduced. If the operating conditions are such that the pin temperature is above that defined by the equation, then again the tenacity obtained is lower but running of the process, and the attaining of a high quality product is easier, and it is frequently preferred to surrender a portion of the available strength in order to gain ease of operation of the drawing process. Accordingly, we prefer to operate the drawing process with the pin temperatures fixed to be not more than 2° C. below the value given by the above algebraic Equation 1 and preferably lying just above that calculated value, preferably within 5° C. of it. Alternatively, if a pin temperature is chosen then there is one optimum relative humidity and it is preferred to operate with the humidity lying slightly above the value determined by our data, for the sake of smooth operation of the process.

The moisture content of the spun yarns may be controlled by regulating the percent relative humidity and temperature of the rooms in which the yarns are processed but we prefer to store the spun yarn in cabinets the internal percent relative humidity and temperature of which is more easily controlled. It has been found that the moisture content of the spun yarn reaches an equilibrium value after it has been stored in an atmosphere of substantially constant percent relative humidity for about 12 hours.

Yet another method of controlling the moisture content of the spun yarn so that the drawing process may be operated according to our invention is to feed the spun yarn directly from the spinning unit, into the feed roll system of a drawing machine, without contacting the yarn with any supply of aqueous spin finish. The yarn which has been spun from vigorously dried polymer will therefore contain a negligible amount of moisture on entering the drawing machine, as the time which would normally elapse between the yarn leaving the spinneret and entering the feed system of the drawing unit will normally be insufficient for significant pick up of atmospheric moisture. The pin and plate temperature for drawing will then be defined as in Equation 1 and 2, with $M=0$.

In the following examples which illustrate but do not limit our invention, the drawn yarn was conditioned for 12 hours in an atmosphere of 65% RH and 70° F. prior to testing and testing was carried out in a similar atmosphere.

EXAMPLE I

A substantially amorphous five filament yarn of melt spun polymetaxylylene adipamide on a bobbin was fed to a draw twisting machine from within a constant hu-

midity chamber in which it had been allowed to equilibrate. The yarn was fed between a driven feed roll and a rubber covered nip roll and passed round a thermostatically controlled heated metal pin of 1½" diameter to a draw roll provided with a separating device so that two or more turns of yarn could be wound around it. The feed roll was of 12" circumference and its speed could be varied, the pin temperature could be adjusted in the range 20° C.-150° C. and the draw roll was of 25" circumference and the draw speed was 500 ft. per minute.

A series of experiments were carried out drawing the yarn at different pin temperatures at five levels of relative humidity within the chamber and/or a constant draw ratio of 3.8:1.

The results obtained are shown in Table 1 below.

Table 1

RH, percent at 70° F.	Pin temperature, ° C.	Tenacity, g./den.
39	70	(1)
	75	5.4
	*80	5.5
	85	4.5
	90	3.9
47	100	3.1
	60	(1)
	*70	5.4
	75	5.1
	80	4.5
58	85	4.7
	90	3.3
	95	3.3
	100	3.4
	50	(1)
66	*60	5.6
	70	5.2
	75	4.4
	80	4.2
	40	(1)
	*50	5.3
	60	4.4
	65	4.4
	70	4.5
	75	3.8
	80	3.7
	.85	3.3

¹ Would not run.

From Table 1 it is seen that optimum running conditions marked * were obtained when the pin temperature/relative humidity relationship approximated to Equation 1a.

EXAMPLE II

Spun yarn similar to that used in Example 1 was conditioned at various relative humidities and with fixed pin temperatures the maximum tenacity obtainable at a draw ratio of 3.8:1 was determined. The results are shown in Table 2 for fixed pin temperatures of 70° C. and 80° C.

Table 2

RH at 70° F.-----	12%	39%	47%	58%	66%
Pin:	G.p.d.	G.p.d.	G.p.d.	G.p.d.	G.p.d.
70° C.-----	(1)	(1)	5.4	5.2	4.5
80° C.-----	3.5	5.4	4.8	4.3	4.6

¹ Would not run.

EXAMPLE III

A sample of polymetaxylylene adipamide spun yarn similar to that used in Example 1 was drawn with the threadline running in contact with a heated plate 8" long situated between the pin and the draw roll. The yarn was drawn from a constant humidity box maintained at various relative humidities and with the pin temperature appropriate to that humidity (Equation 1a) the plate temperature was varied. The drawn yarn tenacities and shrinkages in water at 100° C. were obtained and the results are recorded in Table 3.

Table 3

Pin Temp., ° C.	Relative humidity, percent at 70° F.	Draw ratio	Plate Temp., ° C.	Tenacity, g./den.	Shrinkage percent in water at 100° C.
99	12	3.7	100	5.0	21.6
			120	4.8	16.0
			140	5.66	14.9
			160	5.13	12.7
			180	5.69	11.15
77	39	3.8	200	5.51	10.5
			80	4.83	22.1
			100	5.45	16.8
			120	5.76	15.4
			140	5.90	13.5
61	58	3.8	160	4.9	12.4
			180	5.2	10.2
			60	3.88	19.4
			80	5.45	15.9
			100	-----	14.05
			120	5.45	14.05
			140	5.19	12.95
			160	4.76	12.0
			180	4.26	11.0
			200	4.41	10.8

EXAMPLE IV

A sample of polymetaxylene adipamide, after careful drying, was spun into fibre by melting and pumping through a sand filter and then through a stainless steel spinneret having 7 holes of 0.015 inch diameter, and wound up at 1000 f.p.m. This yarn was lustrous in appearance and X-ray examination showed that the yarn was amorphous. On storing in a constant humidity chamber having an atmospheric relative humidity of 66% at 70 F., the yarn was seen after 24 hours to have remained lustrous. X-ray examinations showed that no change in the internal structure had occurred and the yarn could be drawn easily under conditions prescribed by Equations 1 and 2. Yarns made in a similar manner but stored for 24 hours at 88% RH at 70° F. became non-lustrous in appearance and X-ray examination showed that crystallisation had occurred. These yarns could not be drawn, even when the drawing conditions satisfied Equations 1 and 2.

EXAMPLE V

Spun yarn produced as for Example IV was wound up at 3000 f.p.m. As in Example IV the yarn was lustrous

in appearance and was shown by X-rays to be amorphous. Once again when samples of these yarns were stored and treated as for Example IV the same results were obtained.

What we claim is:

- 5 1. A drawing process for melt spun filaments made from a polymer of metaxylylene diamine and adipic acid where the melt spun filaments are drawn while passing through a heating zone in which the filaments are drawn to at least 3 times their original length, characterized in that the temperature of the heating zone (T_p) is within the range of 2° C. lower than to 5° C. above that calculated from the following equation:

$$T_p^{\circ} \text{C.} = 109 - 9.25M$$

- 15 where M is the percent moisture content of the spun yarn which is to be drawn and is controlled by conditioning the spun yarn, prior to drawing, in an environment of controlled temperature and relative humidity such that the relative humidity does not exceed that defined by the equation:

$$RH_{(\max)} = 118 - 0.55T$$

where $RH_{(\max)}$ is the maximum percent relative humidity and T is the ambient temperature of the conditioning environment in degrees Fahrenheit.

- 25 2. A drawing process according to claim 1 wherein the filaments after drawing through the heating zone are then passed over a heated plate, the temperature of which (T_{p1}) is not lower than that given by the equation:

$$T_{p1}^{\circ} \text{C.} = 172 - 11.1M$$

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