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PATENT SPECIFICATION

NO DRAWINGS

1,111,305

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COMPLETE SPECIFICATION

Improvements in or relating to Polyamide Films

We, NIPPON RAYON KABUSHIKI KAISHA, a Japanese Body Corporate of No. 5, Ujimonouchi, Uji-shi, Kyoto-fu, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for improving polyamide films. More particularly this invention relates to a process for improving polyamide films by means of a concurrent biaxial drawing and subsequent shrinkage technique whereby very thin polyamide films having high purity and improved properties can be achieved, and is in particular concerned with an improvement in or modification of the process described and claimed in copending Application No. 4004/66 (Serial No. 1110623).

Films of thermoplastic resins such as polyesters, polypropylene, polyvinyl chloride, polyvinylidene chloride, polystyrene and polycarbonates, have already been prepared by biaxial drawing techniques. However, it is difficult to obtain polyamide films by concurrent biaxial drawing techniques on a commercial scale owing to the various difficult properties of polyamides, for example those due to strong hydrogen bonding and a tendency for necking-out during drawing.

Our co-pending Application No. 4004/66 (Serial No. 1110623) describes and claims a process for drawing a polyamide film in which the film is drawn simultaneously in the longitudinal and transverse directions at a draw speed of from 6,000 to 100,000 %/min and at a temperature within the range of from 70°C to 180°C which is at least 35°C below the melting point of the film, the ratio of the longitudinal and transverse draw speeds being from 2:1 to 0.5:1, and the draw ratio (as hereinafter defined) being from 16:1 to 4:1.

The films obtained according to the process described and claimed in Application No.

(Serial No. 1110623) 4004/66 are useful industrially in for example the packaging and textile industries, the films having desirable properties such as transparency, high tensile strength, soft feel, brightness, air and vapour permeability, oil resistance, good electrical properties, and high dimensional stability.

The present invention is based upon the discovery that polyamide films having particularly even thickness and uniform physical properties can be produced by the process of Application No. (Serial No. 1110623) 4004/66 if the drawing is effected at a temperature of from 100°C to 180°C and the film prior to drawing is pretreated with water and preheated at a temperature higher than the draw temperature. The polyamide films obtained may for example be useful in the manufacture of bags, tapes, strips, bands and metallised films.

According to the present invention, we provide a process for drawing a polyamide film in which the film is drawn simultaneously in the longitudinal and transverse directions at a draw speed of from 6,000 to 100,000 %/min and at a temperature within the range of from 100°C to 180°C which is at least 35°C below the melting point of the film, the ratio of the longitudinal and transverse draw speeds being from 2:1 to 0.5:1 and the draw ratio (as herein defined) being from 16:1 to 4:1, the said film being pretreated with water and preheated at a temperature higher than the draw temperature prior to drawing whereby water is absorbed.

In the process according to the invention, the pretreatment with water is preferably carried out such that the film absorbs at least 2% of water (based upon the weight of the film). The preheating is advantageously effected at a temperature which is below 200°C and which is from 10 to 50°C higher than the draw temperature.

The process according to the invention is conveniently applied to substantially amorphous



ous polyamide films, and is particularly useful when applied to polyamide films comprising poly-ε-caproamide, poly-hexamethylene-adipamide, polyhexamethylene-sebacamide, poly-11-amino-undecanamide, or a copolymerized polyamide, and mixtures of two or more such polyamides. By the term "substantially amorphous polyamide film" we mean a film having a crystallinity of not more than 30% preferably not more than 25% measured before drawing in the process according to the present invention; polyamides having a crystallinity of more than 30% are more liable to disadvantages such as breakage during draw-

ing. Crystallinity is defined as follows:

$$\text{crystallinity} = \frac{(d_a - d)}{(d_a - d_c)} \times \frac{(d_c)}{(d)} \times 100\%;$$

in which d is the density of the film; d_c is the density of the crystalline structure; and d_a is the density of the non-crystalline structure in the film.

The following table gives examples of linear polyamides having a crystallinity of not more than about 25% or 30% measured at 25°C.

	d_c	d_a	d (30%)	d (25%)
poly-hexamethylene-adipamide	1.24	1.09	<1.131	<1.124
poly-ε-caproamide	1.212	1.113	<1.141	<1.135
poly-11-amino-undecanamide	1.12	1.01	<1.040	<1.035
poly-hexamethylene-sebacamide	1.157	1.041	<1.073	<1.067

Polyamide films useful in the process of the present invention can in general be prepared by the following processes:

1. Extrusion of molten polymer (e.g. T-die process, inflation process, etc.);
2. Extrusion of polymer solution (e.g. dry processes or wet processes).

Draw Speed

The draw speeds used in the process according to the invention may be calculated as follows:—

$$\text{draw speed} = (d_1/d_2) \times 100 \% / t$$

in which d_2 is thickness of the drawn film, d_1 is thickness of the film before drawing, and t is the time in minutes necessary to draw the film.

According to the present invention the draw

$$(t/l_1) \times 100 \% / l_1 \text{ and } (t/h_1) \times 100 \% / h_1$$

where l_1 and h_1 are respectively the length and width of the drawn film, which are measured after t minutes from the beginning of the drawing at an optional point on the film track.

Draw ratio

In industrial practice of biaxial and simultaneous drawing, it is preferred to define the draw ratio as d_1/d_2 , in which d_2 is the thickness of a drawn film and d_1 is the thickness of said film before drawing. The term is used in this sense herein.

Percent gauge variation

Percent gauge variation (hereinafter design-

speed of the polyamide film is conveniently about 6,000 to 100,000 %/min, and especially good results can be obtained at draw speeds of from 10,000 to 60,000 %/min.

Ratio of draw speeds

Ratio of draw speeds is defined as follows: ratio of draw speeds = ratio of speed of stretching in a longitudinal direction to that in a transverse direction. The longitudinal and transverse draw speeds used in the calculation are normal or mean values of draw speeds in the longitudinal and transverse directions from the beginning of drawing to any chosen point reached in the drawing process. Draw speeds are measured at the said point. In other words, when a film having a length l_1 and width h_1 is drawn, the longitudinal draw speed and transverse draw speed can be defined respectively as follows:

ated as %GV) indicates the evenness of a polyamide film, and is defined as follows:

$$\%GV = \frac{t_2 - t_1}{t_a} \times 100 \%$$

where t_1 , t_2 and t_a are respectively the minimum, maximum and average thickness of a polyamide film, and can be measured on any line from one side to another side of the polyamide film in the transverse direction.

A smaller %GV value generally indicates better evenness.

Polyamide films produced by conventional



extruding processes are in general liable to have a higher %GV value when drawn by conventional drawing techniques. However, it is possible to obtain polyamide films having a %GV value of less than about 20% when materials having a %GV of about 10% are treated by the process according to the present invention. On the other hand, it is observed that the %GV value of a polyamide film does depend upon the particular condition under which is treated.

Gauge magnification

Gauge magnification (hereinafter defined as GM) indicates the relationship between %GV values before and after drawing. The term is defined as follows:

$$GM = V_s/V_i;$$

where V_s and V_i represent %GV values after and before drawing respectively.

A GM of 1 indicates that the drawing operation has not affected the %GV value during the drawing, whilst a GM of less than 1 indicates that the %GV value has been improved during the drawing operation.

Bowing defects

When a thermoplastic film is drawn its central portions are liable to a lag behind its edges.

This phenomenon is known as "bowing", and can give rise to various disadvantages

such as unbalanced tensile properties and unbalanced dimensional stability in the drawn film. The bowing degree can be estimated by bowing curvature (hereinafter designated as BC), which is defined as follows: $BC = (\text{distance by which the central portion of the drawn film lags})/(\text{breadth of the drawn film})$.

No satisfactory method for restraining bowing defects of biaxially drawn polyamide films has been reported since no biaxial method for improving polyamide films has yet been commercially established. However, such defects can with advantage be restrained according to the present invention, whereby improved polyamide films have a %GV of less than about 20% and a BC of less than about 0.01 can advantageously be obtained.

In the prior art processes of drawing various thermoplastic films, the influence of water contained in the film before drawing upon various properties of the drawn film has been completely overlooked. The basic discovery upon which the present invention is based is that the water content of a polyamide film prior to drawing is significant, and that the pretreatment with water followed by preheating in accordance with the present invention gives a beneficial result. Preferred amounts of water which are desirably added in the pretreatment of the polyamide films in the process according to the invention vary according to the types of polyamide film and can be exemplified as follows (approximate values):

Polyamide	Preferred water content (% by weight of film)
Poly-ε-caproamide	2—12% (advantageously 2—10%)
Polyhexamethylene-adipamide	2—10%
Polyhexamethylene-sebacamide	2—5%
Poly-11-amino-undecanamide	2—3%

These values may however vary largely according to the temperature and other conditions under which the film is treated.

A very high water content (for example more than about 10% in poly-ε-caproamide or poly-hexamethylene-adipamide films) can give rise to disadvantages, for example breakage of the film during drawing or inferior properties in the drawn film. On the other hand, a water content less than about 2% can cause various difficulties such as unevenness and necking-out.

Water can be added to extruded polyamide film, which is absolutely dry, immediately after extrusion, for example by means of a water bath or water spray.

Larger amounts of water can be added to polyamide films in a shorter period of time at higher temperatures, but the properties of the

drawn films may deteriorate owing to increased crystallization of the film.

Water can also be added after sheet formation. Moreover, if desired, the water may contain colouring matter or an antistatic agent, etc.

After addition of the desired amount of water, the film is pre-heated in a pre-heating zone, where the atmosphere is preferably maintained at a temperature of about 10—50°C above that of the subsequent drawing zone.

The following Table 1 shows the results obtained from various experiments, in which various amounts of water were first added to a substantially amorphous film of poly-ε-caproamide (0.1 mm in average thickness; % gauge variation 8), and the film was then pre-heated in a preheating zone where the atmosphere was maintained at 180°C, and was

drawing

afterwards simultaneously and biaxially drawn in a drawing zone, where the atmosphere was maintained at 140°C, with a draw speed of about 24,000%/min and at a ratio of draw speeds of about 1:1, to yield a film having a thickness of about 0.01 mm.

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TABLE 1

Effect of water content on film properties

Test No.	Water content %	% GV	GM	BC x 525
1	8.3	7	0.9	0
2	6.0	8	1.0	1
3	4.5	9	1.1	1
4	2.0	15	1.9	3
5	1.2	22	2.5	6
6	0.8	24	3.0	15

From Table 1, it appears that a water content of more than 2% can yield improved films having a very even thickness and a low BC value.

The following Table 2 shows the results obtained from various experiments, in which water was first added to a substantially amorphous film of poly-ε-caproamide (0.1 mm in average thickness; 1% gauge variation 8, density 1.130 g/cc) in an amount of 4.5% by

weight, and the film was then pre-heated in a pre-heating zone where the atmosphere was maintained in the range of 100—200°C, and afterwards was drawn simultaneously and biaxially in a drawing zone where the temperature of the atmosphere ranged between 100 and 180°C, with a draw speed of about 24,000%/min at a ratio of draw speeds of about 1:1, to yield a film having a thickness of about 0.01 mm.

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TABLE 2

Effect of pre-heat and draw temperature on film properties

Temperature of

Test No.	pre-heating Zone	drawing Zone	% GV	GM	BC x 525
7	200	160	9	1.1	2
8	180	160	12	1.5	3
9	180	140	9	1.1	1
10	160	140	13	1.6	3
11	160	120	12	1.5	4
12	120	100	14	1.7	5
13	180	180	21	2.6	2
14	160	160	23	2.9	3
15	140	140	25	3.1	5



From Table 2, it is observed that films according to the present invention have even thickness and a low BC value.

According to the present invention, the draw temperature is maintained from about 100°C to a temperature which is not higher than 180°C and which is at least 35°C below the melting point of the film. Particularly good results can generally be achieved at about 100 to 180°C. By drawing at temperatures of from 100 to 180°C, polyamide films can with ad-

vantage be drawn from the very first with larger magnifications and with large draw speeds. Whereas some polyamide films can be drawn at a draw temperature above 180°C, various disadvantages such as heat deterioration will occur at such temperatures.

Melting points of several polyamides are given on page 34 of "Mechanical Properties of Polymers" by Lawrence E. Nielsen (Reinhold Publishing Corp., New York, U.S.A., 1962,) as follows:

Poly-ε-caproamide	225° C.
Polyhexamethylene adipamide	265° C.
Poly-11-amino-undecanamide	194° C.
Polyhexamethylene sebacamide	227° C.

transverse

The draw speeds in the process according to the invention are from 6,000 to 100,000 %/min. A draw speed less than about 6,000 %/min can give rise to disadvantages such as for example necking-out and even breakage. A draw speed more than 100,000 %/min can also give rise to breakages or necking-out. At any point on the surface of the polyamide film during drawing the ratio of the longitudinal and transverse draw speeds must be from 2:1 to 0.5:1 in the process according to the invention. A polyamide film can be drawn at various ratios of draw speeds, but the ratio must in general be maintained within a range from 0.5 to 2.0 when the ratio of the thickness of a drawn film to that of the material before drawing is to be less than 0.25. The draw ratio is from 16:1 to 4:1. A draw ratio higher than about 16:1 can give rise to non-uniformity or partial breakage of the films obtained, while a ratio less than about 4:1 can also give rise to various difficulties. In addition, such a low draw ratio does not yield films having particularly desirable properties, and is not therefore useful for drawing polyamides on a commercial scale.

The present invention also provides a further series of treatment steps including heat-shrinking and heat-setting, the heat-shrinking being preceded if desired by an additional heat-setting step, whereby improved properties and in particular good dimensional stability can be obtained.

According to a further feature of the present invention, a film which has been drawn as hereinbefore described is heated, preferably for about 1 second to 1 minute, at about 120°C to a temperature not higher than 10°C below the melting point of the film to heat-set the film and improve its properties. The period of time for heat-setting depends upon the thickness of the drawn film, temperature of

heat-setting and desired properties of the film, etc.

Such a drawn film has improved properties, although it is difficult to remove distortions from the resulting film by the above mentioned heat-setting process, in particular where improved dimensional stability is required.

According to yet a further feature of the present invention, the shrinking step for the removal of distortion of the drawn film comprises shrinking a drawn film simultaneously and biaxially at a ratio of shrinking speeds of about 0.5:1 to 2.0:1, to an extent of about 1—10%, and from about 120°C to a temperature about 10°C below the melting point of the film.

The ratio of shrinking can be defined as follows;

$$\text{ratio of shrinking} = \frac{d_4 - d_3}{d_3} \times 100\%$$

in which d_3 and d_4 are the thicknesses of the polyamide film before and after shrinking, respectively.

The films obtained according to the present invention can be utilized without any after-treatment or finishing process for example in the packaging and textile industries. Films prepared according to the present invention may have desirable properties such as transparency, mechanical properties such as high tensile strength, soft feel, brightness, air and vapour permeability, oil resistance, good electrical properties and high dimensional stability.

The following Examples illustrate the invention:

EXAMPLE 1

A substantially amorphous film (average thickness 0.1 mm; % gauge variation 8; no beaded edges) of poly-ε-caproamide (rela-



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shrinkage

5 tive viscosity 2.7 when measured at 25°C in 96% sulphuric acid) was extruded at an extruding speed of 12 m/min by means of a conventional T-die process using a 65 mm extruder, and was immediately dipped into a water bath, which was maintained at 45°C and which had an effective length of 36 m, to give the film a water content of about 8.3% (measured by Karl Fischer's method) and a density of 1.130 g/cc. Immediately after this the film was pre-heated by passing at a through-put speed of 12 m/min through a pre-heating zone 2 m in length, where the atmosphere was maintained at 180°C by conventional infra-red heaters. The pre-heated film was then simultaneously and biaxially drawn with a draw speed of about 24,000 %/min (as hereinbefore defined) for about 2.2 seconds through a drawing zone about 0.9m in length, where the atmosphere was maintained at 140°C, to draw a film with a magnification of about 3 in both the longitudinal and transverse directions at a ratio of draw speeds of about 1:1.

25 The drawn film was passed at a through-put speed of 36 m/min through a heat-setting zone 1 m in length, where the atmosphere was maintained at 180°C, under tension such that the transverse dimension would be kept constant, and was then passed through a shrinkage zone 4 m in length, where the atmosphere was maintained at 200°C by means of hot air,

with a ratio of shrinkage speeds of about 1:1 to give a shrinkage value of 6% in both the longitudinal and transverse directions, and was then heat-set by passing through a heat-setting zone 1 m in length, where the atmosphere was maintained at 200°C by means of hot air, at a through-put speed of about 36 m/min under tension such that the transverse dimension would be kept constant, and was then air-cooled to room temperature to give a biaxially drawn film having an average thickness of 0.01 mm.

The atmospheric temperatures were measured 0.5 cm above the film tracks in the pre-heating and drawing zones. The film obtained had an even thickness and well balanced properties (% gauge variation 7; gauge magnification 0.9; bowing curvature 0.). The film, metalized in vacuum with aluminium by a conventional technique, possessed excellent surface properties.

For comparison purposes, another biaxially drawn film was prepared under similar conditions to those described above except that it had a water content of 1.2% before the pre-heating zone.

The comparative film had uneven thickness (% gauge variation 22; gauge magnification 2.5; bowing curvature 6/525) and other inferior properties.

The following table shows the properties of the resulting film according to this example.

Properties

Tensile Strength:

longitudinal	2000 kg/cm ²
transverse	2200 kg/cm ²

Elongation

longitudinal	90%
transverse	100%

Shrinkage value (in air at 130° C.)	>1%
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Shrinkage value is the ratio of shrinking per unit area, obtained by leaving a film for 30 minutes in hot air to give free shrinkage without any tension.

EXAMPLE 2

A substantially amorphous film (average thickness 0.1 mm; % gauge variation 10; no beaded edges) of poly-ε-caproamide (relative viscosity 2.7 when measured at 25°C in 96% sulphuric acid) which had been extruded

in a similar manner to that described in Example 1, was dipped in water at 45°C to give a water content of 4.5% and density of 1.130 g/cc and was then passed at a through-put speed of 15 m/min through a pre-heating zone 2 m in length, where the atmosphere was maintained at 180°C with conventional hot air heaters. The film was drawn biaxially and simultaneously with a draw speed of about 30,000 %/min through a drawing zone 2 m

shrinkage

in length, where the atmosphere was maintained at 140°C by conventional hot air heaters, to draw a film with a magnification of about 4 in both longitudinal and transverse directions at a ratio of draw speeds of about 1.0:1 to 1.3:1.

The drawn film was passed at a through-put speed of 60 m/min through a heat-setting zone 10 m in length, where the atmosphere was maintained at 180°C by means of hot air, under tension such that the transverse dimension would be kept constant, and was then air-cooled to room temperature to give a biaxially drawn film having an average thickness of 0.006 mm, % gauge variation 14, and bowing curvature 2/525. The obtained film had an even thickness as well as other improved properties.

EXAMPLE 3

A substantially amorphous film (average thickness 0.1 mm; gauge variation 8; no beaded edges) of poly - 11 - amino - undecanamide (relative viscosity 2.7 when measured at 25°C in 96% sulphuric acid) was made with a conventional T-die process and was dipped in water at 50°C to give the film a water content of about 2.1% and a density of 1.030 g/cc. The film was immediately passed at a through-put speed of 20 m/min through a pre-heating zone 2 m in length, where the atmosphere was maintained at 160°C. The pre-heated film was biaxially and simultaneously drawn with a draw speed of about 17,000 %/min through a drawing zone 2.5 m in length, where the atmosphere was maintained at 140°C, to draw a film with a magnification of about 3.3 in both longitudinal and transverse directions at a ratio of draw speeds of about 0.8:1 to 1.2:1.

The drawn film was passed at a through-put speed of 66 m/min through a heat-setting zone 2 m in length, where the atmosphere was maintained at 145°C, under tension such that the transverse dimension would be kept constant, and was then cooled to room temperature by blowing cool air thereon. There was obtained a biaxially drawn film having an average thickness of 0.01 mm, % gauge variation of 9; and bowing curvature of 1/525. This film had well balanced properties and an even thickness so that it could be easily wound up on a spool and could be treated very easily.

EXAMPLE 4

A substantially amorphous poly - ε - caproamide film 0.1 mm thick (density of 1.130 g/cc; % gauge variation 8;) was dipped in water to give it a water content of 4.5% and was immediately passed at a through-put speed of 15 m/min through a pre-heating zone 1.5 m in length, where the atmosphere was maintained at 200°C, and was then drawn with a draw speed of about 27,000 %/min for about 2 seconds through a drawing zone 1.0 m in length, where the atmosphere was maintained

at 160°C, to draw a film with a magnification of about 3 in both the longitudinal and transverse directions at a ratio of draw speeds of about 1:1.

The drawn film was passed at a through-put speed of 45 m/min through a heat-setting zone, where the atmosphere was maintained at 200°C with hot air, under tension such that the transverse dimension would be kept constant, and was then air-cooled. The film obtained had an average thickness of about 0.01 mm, % gauge variation of 9; and bowing curvature 2/525. These values show that the film obtained has an improved evenness as well as other improved properties.

EXAMPLE 5

A biaxially drawn film was obtained in a similar manner to that described in Example 4 except that the atmospheric temperatures in the pre-heating and drawing zones were maintained at 160°C and 140°C respectively to yield a drawn film having a % gauge variation of 13 and bowing curvature of 5/525. This Example shows that any possible increase in uneven thickness due to bowing trouble can be sufficiently controlled by the process according to the present invention.

EXAMPLE 6

A substantially amorphous film (average thickness 0.2 mm; % gauge variation 8; water content 8.6%; density of 1.132 g/cc, no beaded edges) of poly - ε - caproamide (relative viscosity 2.7 when measured at 25°C in 96% sulphuric acid), which had been prepared by a conventional T-die process and to which water had been added in a similar manner to that described in Example 1, was passed at a through-put speed of 12 m/min through a pre-heating zone 2 m in length, where the atmosphere was maintained at 180°C by conventional infra-red heaters, and was simultaneously and biaxially drawn with a draw speed of about 24,000 %/min for about 2.2 seconds through a drawing zone 0.9 m in length, where the atmosphere was maintained at 160°C by conventional infra-red heaters, to draw a film with a magnification of 3 in both the longitudinal and transverse directions at a ratio of draw speeds of about 1:1.

The draw film was passed at a through-put speed of 36 m/min through a heat-setting zone 6 m in length, where the atmosphere was maintained at 200°C, under tension such that the transverse dimension would be kept constant, and was then air-cooled to yield a biaxially drawn film having an average thickness of 0.022 mm, % gauge variation of 7; and bowing curvature of 0. These values show that this film has well balanced properties and very even thickness. It is apparent that the increase in unevenness due to the drawing can be with advantage prevented by the process according to the present invention.



EXAMPLE 7

A substantially amorphous film of poly-
ε-caproamide similar to that described in
Example 6 was treated in an analogous manner
to that described in Example 6 except that it
was given a water content of 2.1% to yield a
film having an average thickness of 0.022 mm;
% gauge variation of 11; and bowing curva-
ture of 5/525. The film obtained showed that
the increase in the uneven thickness could be
remarkably well controlled by the process ac-
cording to the present invention.

WHAT WE CLAIM IS:—

1. A process for drawing a polyamide film
in which the film is drawn simultaneously in
the longitudinal and transverse directions at a
draw speed of from 6,000 to 100,000 %/min
and at a temperature within the range of from
100°C to 180°C which is at least 35°C be-
low the melting point of the film, the ratio of
the longitudinal and transverse draw speeds
being from 2:1 to 0.5:1, and the draw ratio
(as herein defined) being from 16:1 to 4:1,
the said film being pretreated with water and
pre-heated at a temperature higher than the
draw temperature prior to drawing whereby
water is absorbed.

2. A process according to claim 1 wherein
said film is a linear polyamide film.

3. A process according to either of claims 1
and 2 wherein the said film comprises poly-
ε-caproamide, polyhexamethylene-adip-
amide, poly-hexamethylene-sebacamide,
poly-11-aminodecanamide, or a copoly-
merized polyamide, or a mixture of two or
more thereof.

4. A process according to any of the pre-
ceding claims wherein the said film is pre-
treated and pre-heated so as to absorb at least
2% of water (based upon the weight of the
film).

5. A process according to either of claims
1 and 2 wherein a film of poly-ε-capro-
amide or poly-hexamethylene-adipamide
is pretreated and preheated so as to absorb
from 2 to 10% of water (based upon the
weight of the film).

6. A process according to either of claims 1
and 2 wherein a film of poly-hexamethylene-
sebacamide is pretreated and pre-heated so as
to absorb from 2 to 5% of water (based upon
the weight of the film).

7. A process according to either of claims 1
and 2 wherein a film of poly-11-aminodecan-
amide is pretreated and pre-heated so as to
absorb from 2 to 3% of water (based upon
the weight of the film).

8. A process according to any of the pre-
ceding claims wherein the said film is pre-
heated to a temperature which is below 200°C
and which is from 10 to 50°C higher than the
draw temperature.

9. A process according to any of the preced-
ing claims wherein the said film is drawn with
a draw speed of from 10,000 to 60,000
%/min.

10. A process according to any of the pre-
ceding claims wherein the drawn film is heat-
set at a temperature not higher than 10°C
below the melting point of the film while said
film is maintained under tension such that the
transverse dimension is kept substantially con-
stant.

11. A process according to claim 10 wherein
the heat-set film is shrunk simultaneously in
both the longitudinal and transverse directions
at a temperature from 120°C up to 10°C
below the melting point of the film by an
amount in each case of from 1 to 10%, the
ratio of shrinkage speeds in the longitudinal
and transverse directions being from 0.5:1 to
2.0:1.

12. A process according to claim 11 wherein
the shrunk film is heat-set at a temperature
of from 120°C to a temperature not higher
than 10°C below the melting point of the film
while maintaining said film under tension to
keep the transverse dimension substantially
constant.

13. A process according to any of claims 1
to 9 wherein the drawn film is shrunk simul-
taneously in both the longitudinal and trans-
verse directions at a temperature from 120°C
up to 10°C below the melting point of the
film by an amount in each direction of from
1 to 10%, the ratio of shrinking speeds in
the longitudinal and transverse directions being
from 0.5:1 to 2.0:1.

14. A process according to claim 13 wherein
the shrunk film is heat-set at a temperature
of from 120°C to a temperature not higher
than 10°C below the melting point of the film
while said film is maintained under tension
to keep the transverse dimension substantially
constant.

15. A process as claimed in any of the pre-
ceding claims wherein the process is carried
out continuously.

16. A process as claimed in any of the pre-
ceding claims wherein the said polyamide film
is a linear polyamide film having a crystal-
linity of not more than 30%.

17. A process as claimed in claim 16 where-
in the said polyamide film is a linear poly-
amide film having a crystallinity of not more
than 25%.

18. A process according to claim 1 sub-
stantially as herein described.

19. A process according to claim 1 sub-
stantially as herein described with reference to
any of the Examples.

20. Drawn polyamide film whenever pre-
pared by a process as claimed in any of the
preceding claims.

aminoundecanamide

shrinking

polyamide



EXAMPLE 7

A substantially amorphous film of poly-ε-caproamide similar to that described in Example 6 was treated in an analogous manner to that described in Example 6 except that it was given a water content of 2.1% to yield a film having an average thickness of 0.022 mm; % gauge variation of 11; and bowing curvature of 5/525. The film obtained showed that the increase in the uneven thickness could be remarkably well controlled by the process according to the present invention.

WHAT WE CLAIM IS:—

1. A process for drawing a polyamide film in which the film is drawn simultaneously in the longitudinal and transverse directions at a draw speed of from 6,000 to 100,000 %/min and at a temperature within the range of from 100°C to 180°C which is at least 35°C below the melting point of the film, the ratio of the longitudinal and transverse draw speeds being from 2:1 to 0.5:1, and the draw ratio (as herein defined) being from 16.1 to 4:1, the said film being pretreated with water and pre-heated at a temperature higher than the draw temperature prior to drawing whereby water is absorbed.

2. A process according to claim 1 wherein said film is a linear polyamide film.

3. A process according to either of claims 1 and 2 wherein the said film comprises poly-ε-caproamide, polyhexamethylene-adipamide, poly-hexamethylene-sebacamide, poly-11-aminodecanamide, or a copolymerized polyamide, or a mixture of two or more thereof.

4. A process according to any of the preceding claims wherein the said film is pretreated and pre-heated so as to absorb at least 2% of water (based upon the weight of the film).

5. A process according to either of claims 1 and 2 wherein a film of poly-ε-caproamide or poly-hexamethylene-adipamide is pretreated and preheated so as to absorb from 2 to 10% of water (based upon the weight of the film).

6. A process according to either of claims 1 and 2 wherein a film of poly-hexamethylene-sebacamide is pretreated and pre-heated so as to absorb from 2 to 5% of water (based upon the weight of the film).

7. A process according to either of claims 1 and 2 wherein a film of poly-11-aminoundecanamide is pretreated and pre-heated so as to absorb from 2 to 3% of water (based upon the weight of the film).

8. A process according to any of the preceding claims wherein the said film is pre-heated to a temperature which is below 200°C and which is from 10 to 50°C higher than the draw temperature.

9. A process according to any of the preceding claims wherein the said film is drawn with a draw speed of from 10,000 to 60,000 %/min.

10. A process according to any of the preceding claims wherein the drawn film is heat-set at a temperature not higher than 10°C below the melting point of the film while said film is maintained under tension such that the transverse dimension is kept substantially constant.

11. A process according to claim 10 wherein the heat-set film is shrunk simultaneously in both the longitudinal and transverse directions at a temperature from 120°C up to 10°C below the melting point of the film by an amount in each case of from 1 to 10%, the ratio of shrinkage speeds in the longitudinal and transverse directions being from 0.5:1 to 2.0:1.

12. A process according to claim 11 wherein the shrunk film is heat-set at a temperature of from 120°C to a temperature not higher than 10°C below the melting point of the film while maintaining said film under tension to keep the transverse dimension substantially constant.

13. A process according to any of claims 1 to 9 wherein the drawn film is shrunk simultaneously in both the longitudinal and transverse directions at a temperature from 120°C up to 10°C below the melting point of the film by an amount in each direction of from 1 to 10%, the ratio of shrinking speeds in the longitudinal and transverse directions being from 0.5:1 to 2.0:1.

14. A process according to claim 13 wherein the shrunk film is heat-set at a temperature of from 120°C to a temperature not higher than 10°C below the melting point of the film while said film is maintained under tension to keep the transverse dimension substantially constant.

15. A process as claimed in any of the preceding claims wherein the process is carried out continuously.

16. A process as claimed in any of the preceding claims wherein the said polyamide film is a linear polyamide film having a crystallinity of not more than 30%.

17. A process as claimed in claim 16 wherein the said polyamide film is a linear polyamide film having a crystallinity of not more than 25%.

18. A process according to claim 1 substantially as herein described.

19. A process according to claim 1 substantially as herein described with reference to any of the Examples.

20. Drawn polyamide film whenever prepared by a process as claimed in any of the preceding claims.

aminoundecanamide

1-1

1-1

61

70

75

80

85

90

95

100

105

110

115

120

shrinking

polyamide



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