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PATENT SPECIFICATION

(11) 1 487 843

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- (21) Application No. 41184/74 (22) Filed 20 Sept. 1974
(31) Convention Application No. 400 863
(32) Filed 26 Sept. 1973 in
(33) United States of America (US)
(44) Complete Specification published 5 Oct. 1977
(51) INT CL² D01D 5/10; D01F 6/62
(52) Index at acceptance

B5B 230 35Y 360 401 412 422 510 714 720 725 72Y 732 783
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(54) PROCESS FOR THE EXPEDITIOUS FORMATION AND STRUCTURAL MODIFICATION OF POLYESTER FIBRES AND FILMS

(71) We, CELANESE CORPORATION, a corporation organised and existing under the laws of the State of Delaware, United States of America of 1211 Avenue of the Americas, New York, New York 10036, United States of America, 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:—

Polymeric filamentary materials and films have been produced in the past under a variety of melt extrusion conditions. Both high stress and low stress spinning processes have been employed. Under high stress conditions the as-spun filamentary material is withdrawn from the spinneret under conditions whereby substantial orientation is imparted to the same soon after it is extruded and prior to its complete solidification. See, for instance, United States Patent Nos. 2,604,667 and 2,604,689. Such high stress conditions of the prior art commonly yield a non-uniform filamentary material wherein substantial radial non-homogeneity exists across the fiber diameter leading to self-crimping characteristics upon heating, or less than desired tensile properties.

Melt spinning processes have also been proposed wherein the cooling of the extruded filamentary material has been retarded (i.e., prolonged) prior to complete solidification so as to alter the properties thereof. See, for instance, United States Patent Nos. 2,323,383; 3,053,611 and 3,361,859.

Heretofore, polymeric fibers, e.g., polyester fibers, following extrusion and solidification have commonly been drawn while at an elevated temperature to further enhance their tensile properties. Such drawing may be conducted in an inline fashion following fiber formation wherein the fiber is passed about appropriate drawing equipment or after the as-spun fiber is unwound from an intermediate collection device. Such drawing is commonly conducted upon contact with an appropriate heating device, heated gaseous atmosphere, or heated liquid medium. Also, it has been known that previously drawn polyester fibers may be heat treated with or without allowed shrinkage (i.e., post-annealed) in order to modify their physical properties.

According to the present invention there is provided a process for expeditiously forming and structurally modifying a polyester filamentary material or film comprising:

(a) extruding a molten melt-spinnable polyester material capable of undergoing crystallization through a shaped orifice to form a molten filamentary material or film,

(b) passing the resulting molten filamentary material or film in the direction of its length through a solidification zone provided with a gaseous atmosphere at a temperature below the glass transition temperature thereof wherein the molten filamentary material or film is solidified,

(c) passing the resulting filamentary material or film in the direction of its length through a conditioning zone provided with a gaseous atmosphere at a temperature above the glass transition temperature thereof and below the melting temperature thereof for a residence time of 0.0001 to 0.8 second wherein substantial crystallization of said previously solidified filamentary material or film takes place, and

(d) withdrawing the resulting filamentary material or film from the conditioning zone at a rate of 1000 to 6000 meters per minute while under a stress of about 0.1 to 1.0 gram per denier; with the processing of the polyester filamentary material or film following the extrusion being conducted while exerting a constant tension thereon in the absence of stress isolation along the length of the same intermediate the shaped orifice and the point of withdrawal from said conditioning zone (i.e., the filamentary material or film is axially suspended in the absence of external stress isolation devices in the region intermediate the shaped orifice and the point of withdrawal from the conditioning zone).

The drawing is a schematic presentation of an apparatus arrangement capable of carrying out the process of the present invention.

Polyester materials which are melt-spinnable and capable of undergoing crystallization, i.e., when heated between their glass transition temperature and their melting temperature, may be selected for use in the present process.

For instance, the melt-spinnable polyester selected for use in the present process may be principally polyethylene terephthalate, and contain at least 85 mol percent polyethylene terephthalate, and preferably at least 90 mol percent polyethylene terephthalate. In a particularly preferred embodiment of the process the melt-spinnable polyester is substantially all polyethylene terephthalate. Alternatively, during the preparation of the polyester minor amounts of one or more ester-forming ingredients other than ethylene glycol and terephthalic acid or its derivatives may be copolymerized. For instance, the melt-spinnable polyester may contain 85 to 100 mol percent (preferably 90 to 100 mol percent) polyethylene terephthalate structural units and 0 to 15 mol percent (preferably 0 to 10 mol percent) copolymerized ester units other than polyethylene terephthalate. Illustrative examples of other ester-forming ingredients which may be copolymerized with the polyethylene terephthalate units include glycols such as diethylene glycol, tetramethylene glycol, hexamethylene glycol, etc., and dicarboxylic acids such as hexahydroterephthalic acid, bibenzoic acid, adipic acid, sebacic acid, azelaic acid, etc.

The melt-spinnable polyethylene terephthalate selected for use in the process preferably exhibits an intrinsic viscosity, i.e., I.V., of 0.45 to 1.0, and an I.V. of 0.6 to 0.95 in a particularly preferred embodiment of the process. The I.V. of the melt-spinnable polyester may be conveniently determined by the equation

$$\lim_{c \rightarrow 0} \frac{\ln_r r}{c}$$

where r is the "relative viscosity" obtained by dividing the viscosity of a dilute solution of the polymer by the viscosity of the solvent employed (measured at the same temperature), and c is the polymer concentration in the solution expressed in grams/100 ml. The polyethylene terephthalate additionally commonly exhibits a glass transition temperature of 75 to 80°C. and a melting point of 250 to 265°C., e.g., about 260°C.

The extrusion orifice may be selected from among those commonly utilized during the melt extrusion of fibers or films. For instance, the shaped extrusion orifice may be in the form of a rectangular slit when forming a film. When forming a filamentary material the spinneret selected for use in the process may contain one or preferably a plurality of extrusion orifices. For instance, a standard conical spinneret containing 1 to 200 holes (e.g., 6 to 200 holes), such as commonly used in the melt spinning of polyethylene terephthalate, having a diameter of 10 to 60 mils (e.g., 10 to 40 mils) may be utilized in the process. Yarns of 20 to 36 continuous filaments are commonly formed. The melt-spinnable polyester material is supplied to the extrusion orifice at a temperature above its melting point.

A molten polyester consisting principally of polyethylene terephthalate is preferably at a temperature of 270 to 310°C., and most preferably at a temperature of 285 to 305°C. (e.g., 300°C.) when extruded through the spinneret.

Subsequent to extrusion through the shaped orifice the resulting molten filamentary material or film is passed in the direction of its length through a solidification zone provided with a gaseous atmosphere at a temperature below the glass transition temperature thereof wherein the molten filamentary material or film is transformed to a solid filamentary material or film. When the filamentary

material or film is principally polyethylene terephthalate the gaseous atmosphere of the solidification zone is provided at a temperature below about 80°C. Within the solidification zone the molten material passes from the melt to a semi-solid consistency, and from the semi-solid consistency to a solid consistency. While present in the solidification zone the material undergoes substantial orientation while present as a semi-solid as discussed hereafter. The solidification zone could also be termed a "quench zone". The gaseous atmosphere present within the solidification zone preferably circulates so as to bring about more efficient heat transfer. In a preferred embodiment of the process the gaseous atmosphere of the solidification zone is provided at a temperature of 10 to 40°C., and most preferably at about room temperature (e.g., at about 25°C.). The chemical composition of the gaseous atmosphere is not critical to the operation of the process provided the gaseous atmosphere is not unduly reactive with the polyester filamentary material or film. In a particularly preferred embodiment of the process the gaseous atmosphere of the solidification zone is air. Other representative gaseous atmospheres which may be selected for utilization in the solidification zone include inert gases such as helium, argon, nitrogen, etc.

The gaseous atmosphere of the solidification zone preferably impinges upon the extruded polyester material so as to produce a uniform quench wherein no substantial radial non-homogeneity exists across the product. The uniformity of the quench may be demonstrated with a filamentary material through its ability to exhibit no substantial tendency to undergo self-crimping upon the application of heat. A flat yarn accordingly is produced in a preferred embodiment of the process.

The solidification zone is preferably disposed immediately below the shaped extrusion orifice and the extruded polyester material is present while axially suspended therein for a residence time of preferably 0.0008 to 0.4 second, and most preferably for a residence time of 0.033 to 0.14 second. Commonly the solidification zone possesses a length of 0.25 to 20 feet, and preferably a length of 1 to 7 feet. The gaseous atmosphere is also preferably introduced at the lower end of the solidification zone and withdrawn along the side thereof with the moving continuous length of the polyester material passing downwardly therethrough from the spinneret. A center flow quench or any other technique capable of bringing about the desired quenching alternatively may be utilized. If desired, a hot shroud may be positioned intermediate the shaped orifice and the solidification zone.

The resulting filamentary material or film is next passed in the direction of its length through a conditioning zone provided with a gaseous atmosphere at a temperature above the glass transition temperature thereof and below the melting temperature thereof for a residence time of .0001 to 0.8 second, wherein substantial crystallization of the previously solidified filamentary material or film takes place. In a preferred embodiment wherein the filamentary material or film is principally polyethylene terephthalate and conditioning zone is provided with a gaseous atmosphere at a temperature of 90 to 180°C. (e.g., 90 to 140°C.) and the previously solidified material is present therein for a residence time of 0.001 to 0.8 second. In particularly preferred embodiment the conditioning zone is provided with a gaseous atmosphere at a temperature of 100 to 120°C. The preferred residence time for the filamentary material or film which is principally polyethylene terephthalate within the conditioning zone is 0.0016 to 0.6 second, and most preferably 0.03 to 0.09 second. If residence times much below 0.0001 second are employed, then a stable achievement of the desired property levels commonly does not result. The optimum residence time required to produce substantial crystallization may vary with the polyester material involved. Longer residence times may be utilized with no commensurate advantage.

The chemical composition of the gaseous atmosphere provided within the conditioning zone is not critical to the operation of the process provided the gaseous atmosphere is not unduly reactive with the polyester filamentary material or film. Static air or steam conveniently may be selected. Other representative gaseous atmospheres which may be employed in the conditioning zone include helium, argon, nitrogen, etc. Band heaters or any other heating means may be provided so as to maintain the conditioning zone at the required temperature. The conditioning zone commonly has a length of 0.5 to 30 feet, and preferably a length of 5 to 12 feet.

The resulting filamentary material or film is withdrawn from the conditioning zone at a rate of 1000 to 6000 meters per minute (preferably 2500 to 3500 meters

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per minute) while under a stress of 0.1 to 1 gram per denier (preferably 0.15 to 0.6 gram per denier and most preferably 0.2 to 0.4 gram per denier). Following extrusion the filamentary material or film is maintained under constant tension and throughout the process no stress isolation is utilized along the length of the filamentary material or film intermediate the shaped orifice (e.g., spinneret) and the point of withdrawal from the conditioning zone (e.g., a yarn is axially suspended in the absence of external contact in the regions intermediate the spinneret and the point of withdrawal from the conditioning zone). When withdrawn from the conditioning zone the filamentary material commonly exhibits a denier per filament of 1 to 15, e.g., 1.5 to 5.

The improved melt extrusion process of the present invention may be conveniently carried out in conventional nylon equipment provided with a heated conditioning chamber of adequate length below the quench zone and having the required high stress take-up equipment. The results achieved with a melt-spinnable polyester material described herein are considered to be unexpected to those skilled in melt spinning technology.

While present in the conditioning zone, the filamentary material or film is heat treated under constant tension. During this heat treatment small amounts of thermally induced elongation may occur, but this process is differentiated from a draw process because of the constant tension rather than constant strain. The level of tension on the filamentary material or film in the conditioning zone is extremely critical to the development of the desired structure and properties and primarily is influenced by the rate of withdrawal from the conditioning zone rather than friction with the surrounding gaseous atmosphere. No stress isolation results along the filamentary material or film intermediate the shaped orifice and the point of withdrawal from the conditioning zone (e.g., the filamentary material is axially suspended in the absence of external stress isolating devices in the region intermediate the spinneret and the point of withdrawal from the conditioning zone). Should one omit the passage of the filamentary material through the conditioning zone, the denier and cross sectional dimension of the filamentary material commonly are found to be unaltered.

In the high stress melt spinning process of the present invention the extruded filamentary material or film intermediate the point of its maximum die swell area and its point of withdrawal from the conditioning zone commonly exhibits a substantial drawdown. For instance, a filamentary material may exhibit a drawdown ratio of 100:1 to 2000:1, and most commonly a drawdown ratio of 600:1 to 1700:1. The "drawdown ratio" as used above is defined as the ratio of the maximum die swell cross sectional area to the cross sectional area of the filamentary material as it leaves the conditioning zone. Such substantial change in cross sectional area occurs almost exclusively in the solidification zone prior to complete quenching. In some embodiments of the process, however, up to a 4:1 reduction in cross sectional area of the filamentary material is observed in the condition zone via heat induced elongation as discussed above.

The passage of the filamentary material or film through the conditioning zone in the precise manner described surprisingly has been found to beneficially enhance the same through the modification of its internal structural morphology. More specifically, the tensile properties are surprisingly improved and may render a conventional hot drawing step unnecessary. The tensile strength and modulus are improved and the shrinkage characteristics are diminished.

A resulting polyester filament is claimed in our co-pending U.K. application No. 41185/74, Serial No. 1,487,844, entitled "Polyester Fiber" filed concurrently herewith, and differs structurally from polyester fibers heretofore produced in that it has an interconnected highly oriented crystalline microstructure coextensive with its length coexisting with an interdispersed substantially disoriented non-crystalline phase, and exhibits a propensity to undergo a low degree of shrinkage with a high degree of force at an elevated temperature as evidenced by a modulus ratio of at least 0.1. Also the filamentary material exhibits a relatively high initial modulus, coupled with a relatively high crystalline orientation function, and a relatively low amorphous orientation function, i.e., a mean initial modulus when present in a multifilament yarn at 25°C. of at least 55 grams per denier, a birefringence of 0.10 to 0.14, a crystalline orientation function of at least 0.88, and an amorphous orientation function of not more than 0.35. See our concurrently filed application No. 41185/74 Serial No. 1,487,844 for an amplified discussion of the resulting polyester filament.

The theory whereby the present process is capable of producing a polyester

filamentary material or film exhibiting the properties recited is considered complex and incapable of simple explanation. It is believed, however, that the stress exerted upon the semi-solid filamentary material or film in the solidification zone produces an oriented crystalline fibrillar microstructure of polymer molecules within the same which serve to nucleate the epitaxial growth of polymer crystals intermediate adjoining fibrils. As the resulting filamentary material or film next passes through a conditioning zone, as defined, substantial epitaxial crystallization spontaneously occurs onto the oriented fibrillar structure. Such rapid crystallization is believed to form a lamella overgrowth on the existing fibrillar structure with lamellar crystals extending between fibrils and with the lamellar crystals being joined by tie molecules.

The resulting filamentary material or film is amenable to further processing through the use of additional processing equipment or it may be used directly in applications requiring a continuous filament commercial yarn. If desired, the filamentary material subsequently may be converted from a flat yarn to a textured yarn, e.g., through the utilization of known false twist texturing conditions. Illustrative conditions for a yarn of 150 denier employ a yarn speed of 125 meters per minute, a feed roll heater plate temperature of 215°C., an over feed into the heater of 3.5 percent, and a turn per inch of 60.

The following examples are given as specific illustrations of the process. It should be understood, however, that the invention is not limited to the specific details set forth in the examples. Reference is made in the examples to the apparatus arrangement illustrated in the drawing. The claimed invention is not restricted to the utilization of the apparatus illustrated in the drawing.

Example I.

Polyethylene terephthalate having an intrinsic viscosity (I.V.) of 0.67 was selected as the starting material. The intrinsic viscosity was determined from a solution of 0.1 gram of the polymer in 100 ml. of ortho-chlorophenol at 25°C.

The polyethylene terephthalate polymer while in particulate form was placed in hopper 1 and was advanced toward spinneret 2 by the aid of screw conveyor 4. Heater 6 caused the polyethylene terephthalate particles to melt to form a homogenous phase which was further advanced toward spinneret 2 by the aid of pump 8.

The spinneret 2 had a standard conical entrance and possessed a ring of 20 extrusion holes, each having a diameter of 20 mils. The molten polyethylene terephthalate was at a temperature of about 300°C. when extruded through spinneret 2.

The resulting extruded polyethylene terephthalate 10 passed directly from the spinneret 2 through solidification zone 12. The solidification zone 12 had a length of 6 feet and was vertically disposed. Air at room temperature (i.e., about 25°C.) was continuously introduced into solidification zone 12 at 14 which was supplied via conduit 16 and fan 18. The air was continuously withdrawn through elongated conduit 20 vertically disposed in communication with the wall of solidification zone 12, and was continuously withdrawn through conduit 22. While passing through the solidification zone the extruded polyethylene terephthalate was uniformly quenched and was transformed into a continuous yarn of as-spun polyethylene terephthalate. The polymeric material was first transformed from a molten to a semi-solid consistency, and then from a semi-solid consistency to a solid consistency while passing through solidification zone 12. The extruded polyethylene terephthalate was present in the solidification zone 12 for a residence time of about 0.045 second.

Upon being withdrawn from solidification zone 12 the continuous yarn 24 of polyethylene terephthalate was next immediately passed through vertically disposed conditioning zone 26 having a length of 12 feet. A static air atmosphere was maintained in conditioning zone 26 at a temperature of 120°C. by the aid of band heater 28 which surrounded the walls of the same. The polyethylene terephthalate yarn was present in the conditioning zone 26 for a residence time of about 0.09 second where it was structurally modified.

The resulting polyethylene terephthalate yarn was under a constant tension following extrusion and was withdrawn from conditioning zone 26 at a rate of 2500 meters per minute while under a stress of about 0.2 gram per denier. The extruded filamentary material intermediate the point of its maximum die swell area and its point of withdrawal from the conditioning zone was drawn down at a ratio of about 1400:1. The resulting polyethylene terephthalate yarn exhibited a denier per

filament of 2, and was packaged at 30 after passing around godets 32 and 34, and contacting roller 36 which applied an anti-static lubricant.

The polyethylene terephthalate yarn was axially suspended in the absence of external contact intermediate the spinneret and the point of its withdrawal from conditioning zone 26. There was accordingly no stress isolation along the length of the same in this region and the fibrous material was under substantial stress throughout its processing which was exerted by rotation of packaging equipment 30.

For comparative purposes, Example I was repeated with the exception that the static air atmosphere of the conditioning zone 26 was provided at room temperature (i.e., about 25°C.) instead of 120°C. The extruded filamentary material intermediate the point of its maximum die swell area and its point of withdrawal from the conditioning zone was drawn down at a ration of about 1400:1. The resulting yarn upon withdrawal from the conditioning zone 26 exhibited a denier per filament of 2.

Summarized below are the properties of the resulting polyethylene terephthalate product. See out co-pending U.K. application No. 41185/74, Serial No. 1,487,844, entitled "Polyester Fiber" filed concurrently herewith, for a more detailed discussion of how the reported properties were determined.

	With Invention (Conditioning Tube at 120°C.)	Without Invention (Conditioning Tube at 25°C.)	
Denier Per Filament	2	2	
Mean Yarn Tenacity (grams per denier)	3.7	1.92	
Mean Yarn Elongation (per cent)	56	175	
Mean Yarn Initial Modulus (grams per denier)	70	22.5	
Mean Yarn Shrinkage at 100°C. (per cent)	3.7	33.0	
Mean Yarn Shrinkage at 175°C. (per cent)	6.6	16.5	
Mean Yarn Internal Tension at 100°C. (grams/denier)	0.36	0.026	
Mean Yarn Internal Tension at 175°C. (grams/denier)	0.25	0.005	
Maximum Yarn Internal Tension (grams/denier)	0.37	0.039	
Shrinkage Modulus at 100°C. (grams/denier)	10.0	0.079	
Shrinkage Modulus at 175°C. (grams/denier)	3.79	0.030	
Modulus Ratio	0.143	0.0036	
Birefringence	0.1188	0.025	
Crystalline Orientation Function	0.92	*	
Amorphous Orientation Function	0.30	0.10	

* = Not crystalline enough to yield useful diffraction

Example II.

Example I was repeated with the exception that the resulting polyethylene terephthalate yarn was withdrawn from conditioning zone 26 at a rate of 3000 meters per minute while under stress of about 0.25 gram per denier. The extruded polyethylene terephthalate yarn was present in the solidification zone 12 for a residence time of about 0.036 second. The polyethylene terephthalate yarn was present in the conditioning zone 26 for a residence time of about 0.07 second. The extruded filamentary material intermediate the point of its maximum die swell area and its point of withdrawal from the conditioning zone was drawn down at a ratio of about 1500:1. The resulting yarn upon withdrawal from conditioning zone 26 exhibited a denier per filament of about 2.

For comparative purposes, Example II was repeated with the exception that the static air atmosphere of the conditioning zone 26 was provided at room temperature (i.e., about 25°C.) instead of 120°C. The extruded filamentary material intermediate the point of its maximum die swell area and its point of withdrawal from the conditioning zone was drawn down at a ratio of about 1500:1. The resulting yarn upon withdrawal from conditioning zone 26 exhibited a denier per filament of 2.

Summarized below are the average single filament properties of the resulting polyethylene terephthalate yarns achieved. See out co-pending U.K. application No. 41185/74, Serial No. 1,487,844, entitled "Polyester Fiber" filed concurrently herewith, for a more detailed discussion of how the reported properties were determined.

	With Invention (Conditioning Tube at 120°C.)	Without Invention (Conditioning Tube at 25°C.)	
25			25
	Denier Per Filament	2	2
	Mean Yarn Tenacity grams per denier)	4	2.36
30	Mean Yarn Elongation (per cent)	50	133
	Mean Yarn Initial Modulus (grams per denier)	76	24.1
35	Mean Yarn Shrinkage at 100°C. (per cent)	3.8	33.0
	Mean Yarn Shrinkage at 175°C. (per cent)	7.8	22.0
	Mean Yarn Internal Tension at 100°C. (grams/denier)	0.41	0.033
40	Mean Yarn Internal Tension at 175°C. (grams/denier)	0.35	0.011
	Maximum Yarn Internal Tension (grams/denier)	0.42	0.052
45	Shrinkage Modulus at 100°C. (grams/denier)	10.8	0.10
	Shrinkage Modulus at 175°C. (grams/denier)	4.49	0.050
	Modulus Ratio	0.142	0.00417
	Birefringence	0.1240	0.046
50	Crystalline Orientation Function	0.94	*
	Amorphous Orientation Function	0.28	0.17

* = Not crystalline enough to yield useful diffraction

It can be seen from the preceding data of Examples I and II that the process of the present invention is capable of yielding a polyethylene terephthalate fiber of substantially increased tenacity and modulus in combination with a significantly reduced shrinkage. Conventional polyester fiber hot drawing procedures are rendered unnecessary when such a fiber is produced.

As indicated by the data present in our co-pending U.K. application No. 41185/74, Serial No. 1,487,844, entitled "Polyester Fiber", filed concurrently herewith, at Comparative Examples 8 and 9, these results cannot be achieved if one should attempt to divide the presently claimed process by collection of the filamentary material after it leaves the solidification zone, and by subsequent passage of the same while under a comparable stress through the conditioning zone provided at a comparable temperature. Accordingly, the process of the present invention is capable of producing unexpected results which cannot be duplicated by the subsequent passage of a filamentary material or film resulting from a high stress spinning operation through an annealing zone where stress isolation exists between zones.

Although the invention has been described with preferred embodiments, it is to be understood that variations and modifications may be resorted to as will be apparent to those skilled in the art.

WHAT WE CLAIM IS:—

1. A process for forming a polyester filamentary material or film which comprises

(a) extruding a molten melt-spinnable polyester material capable of undergoing crystallization through a shaped orifice to form a molten filamentary material or film,

(b) passing the resulting molten filamentary material or film in the direction of its length through a solidification zone provided with a gaseous atmosphere at a temperature below the glass transition temperature thereof wherein the molten filamentary material or film is solidified

(c) passing said resulting solidified filamentary material or film in the direction of its length through a conditioning zone provided with a gaseous atmosphere at a temperature above its glass transition temperature and below the melting temperature for a residence time of 0.0001 to 0.8 second whereby substantial crystallization of said previously solidified filamentary material or film takes place, and

(d) withdrawing the resulting filamentary material or film from said conditioning zone at a rate of 1000 to 6000 meters per minute while under a stress of 0.1 to 1.0 gram per denier; a constant tension being maintained on said polyester filamentary material or film in the absence of stress isolation along its length between said orifice and the point of withdrawal from said conditioning zone.

2. A process as claimed in claim 1 wherein said polyester contains 85 to 100 ml % of polyethylene terephthalate and 0 to 15 mol % of copolymerized ester units other than polyethylene terephthalate

3. A process as claimed in claim 1 wherein said polyester is substantially all polyethylene terephthalate.

4. A process as claimed in any preceding claim wherein said gaseous atmosphere in said solidification zone has a temperature below 80°C.

5. A process as claimed in claim 4 wherein the temperature is from 10 to 40°C.

6. A process as claimed in any preceding claim wherein said gaseous atmosphere in said solidification zone is air.

7. A process as claimed in any preceding claim wherein said molten filamentary material or film is uniformly quenched by said gaseous atmosphere in said solidification zone.

8. A process as claimed in any preceding claim wherein said gaseous atmosphere in said conditioning zone is air.

9. A process as claimed in any preceding claim wherein said filamentary material or film is present in said conditioning zone for a residence time of 0.0016 to 0.6 second.

10. A process as claimed in claim 9 wherein the residence time is from 0.03 to 0.09 seconds.

11. A process as claimed in any preceding claim wherein temperature of said gaseous atmosphere in said conditioning zone is from 90 to 180°C.

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As indicated by the data present in our co-pending U.K. application No. 41185/74, Serial No. 1,487,844, entitled "Polyester Fiber", filed concurrently herewith, at Comparative Examples 8 and 9, these results cannot be achieved if one should attempt to divide the presently claimed process by collection of the filamentary material after it leaves the solidification zone, and by subsequent passage of the same while under a comparable stress through the conditioning zone provided at a comparable temperature. Accordingly, the process of the present invention is capable of producing unexpected results which cannot be duplicated by the subsequent passage of a filamentary material or film resulting from a high stress spinning operation through an annealing zone where stress isolation exists between zones.

Although the invention has been described with preferred embodiments, it is to be understood that variations and modifications may be resorted to as will be apparent to those skilled in the art.

WHAT WE CLAIM IS:—

1. A process for forming a polyester filamentary material or film which comprises

(a) extruding a molten melt-spinnable polyester material capable of undergoing crystallization through a shaped orifice to form a molten filamentary material or film,

(b) passing the resulting molten filamentary material or film in the direction of its length through a solidification zone provided with a gaseous atmosphere at a temperature below the glass transition temperature thereof wherein the molten filamentary material or film is solidified

(c) passing said resulting solidified filamentary material or film in the direction of its length through a conditioning zone provided with a gaseous atmosphere at a temperature above its glass transition temperature and below the melting temperature for a residence time of 0.0001 to 0.8 second whereby substantial crystallization of said previously solidified filamentary material or film takes place, and

(d) withdrawing the resulting filamentary material or film from said conditioning zone at a rate of 1000 to 6000 meters per minute while under a stress of 0.1 to 1.0 gram per denier; a constant tension being maintained on said polyester filamentary material or film in the absence of stress isolation along its length between said orifice and the point of withdrawal from said conditioning zone.

2. A process as claimed in claim 1 wherein said polyester contains 85 to 100 ml % of polyethylene terephthalate and 0 to 15 mol % of copolymerized ester units other than polyethylene terephthalate

3. A process as claimed in claim 1 wherein said polyester is substantially all polyethylene terephthalate.

4. A process as claimed in any preceding claim wherein said gaseous atmosphere in said solidification zone has a temperature below 80°C.

5. A process as claimed in claim 4 wherein the temperature is from 10 to 40°C.

6. A process as claimed in any preceding claim wherein said gaseous atmosphere in said solidification zone is air.

7. A process as claimed in any preceding claim wherein said molten filamentary material or film is uniformly quenched by said gaseous atmosphere in said solidification zone.

8. A process as claimed in any preceding claim wherein said gaseous atmosphere in said conditioning zone is air.

9. A process as claimed in any preceding claim wherein said filamentary material or film is present in said conditioning zone for a residence time of 0.0016 to 0.6 second.

10. A process as claimed in claim 9 wherein the residence time is from 0.03 to 0.09 seconds.

11. A process as claimed in any preceding claim wherein temperature of said gaseous atmosphere in said conditioning zone is from 90 to 180°C.



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12. A process as claimed in claim 11 wherein the temperature in said conditioning zone is from 90 to 140°C.

13. A process as claimed in claim 11 or 12 wherein the temperature in said conditioning zone is from 100 to 120°C.

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14. A process as claimed in any preceding claim wherein said filamentary material or film is withdrawn from said conditioning zone at a rate of from 2500 to 3500 meters per minute.

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15. A process as claimed in any preceding claim wherein the molten polyester material has a temperature of 270 to 310°C. when extruded through the spinneret.

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16. A process as claimed in claim 15 wherein the temperature of the polyester material is from 285 to 305°C.

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17. A process as claimed in any preceding claim wherein a film is formed by extrusion of said polyester material through an appropriately shaped orifice.

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18. A process as claimed in any of claims 1 to 16 wherein a filamentary material is formed by extrusion of said polyester material through an appropriately shaped orifice.

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19. A process as claimed in claim 18 wherein said filamentary material is yarn consisting of 6 to 200 filaments.

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20. A process as claimed in claim 18 or 19 wherein said filamentary material when withdrawn from said conditioning zone exhibits a denier per filament of 1 to 15.

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21. A process as claimed in any of claims 18 to 20 wherein the filamentary material is withdrawn from said conditioning zone at a rate of 2500 to 3500 meters per minute while under a stress of 0.15 to 0.6 grams per denier.

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22. A process as claimed in claim 21 wherein the stress is from 0.2 to 0.4 grams per denier.

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23. A process as claimed in any of claims 18 to 22 wherein filaments are extruded through a plurality of holes having a diameter of 10 to 60 mils.

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24. A process as claimed in claim 23 wherein the yarn is withdrawn from said conditioning zone at a denier per filament of 1 to 15.

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25. A process for forming a polyester filamentary material substantially as hereinbefore described with reference to the examples of the invention.

26. Polyester filamentary material or film when formed by a process as claimed in any preceding claim.

D. VINCENT,
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