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(54) Titre : PROCEDE POUR FILER ET ENROULER DES FILAMENTS DE POLYESTER EN UTILISANT DES ADDITIFS DE FILAGE, FILAMENTS DE POLYESTER OBTENUS PAR CE PROCEDE DE FILAGE, TEXTURATION PAR ETIRAGE DES FILAMENTS DE POLYESTER ET FILAMENTS DE POLYESTER GONFLANTS OBTENUS PAR CETTE TEXTURATION PAR ETIRAGE

(54) Title: METHOD FOR THE SPINNING AND WINDING OF POLYESTER FILAMENTS USING A SPINNING ADDITIVE, POLYESTER FILAMENTS OBTAINED BY THE SPINNING METHOD, DRAW TEXTURING OF THE POLYESTER FILAMENTS AND BULKED POLYESTER FILAMENTS OBTAINED BY DRAW TEXTURING

(57) Abrégé/Abstract:
The invention relates to a method for the production and winding of pre-oriented polyester filaments, comprising at least 90 wt. %, based on the total weight of polyester filament, polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), preferably PTMT, characterised in that a) the spinning draft is set in the range 70 to 500, b) directly after exiting the spinning nozzle, the filaments run through a cooling delay zone of 30 mm to 200 mm in length, c) the filaments are cooled to below the solidification temperature, d) the filaments are bundled at a separation of between 500 mm and 2500 mm from the bottom face of the nozzle, e) the thread tension, before and between the drawing galettes is set to between 0.05 cN/dtex and 0.20 cN/dtex, f) the threads are wound with a thread tension of between 0.025 cN/dtex and 0.15 cN/dtex, g) the winding speed is set to between 2200 m/min and 6000 m/min and h) a polyester is used with 0.05 wt. % to 2.5 wt. %, based on the total weight of the filament, of added additive polymer as extensibility improver.

(57) Abstract: The invention relates to a method for the production and winding of pre-oriented polyester filaments, comprising at least 90 wt. %, based on the total weight of polyester filament, polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), preferably PTMT, characterised in that a) the spinning draft is set in the range 70 to 500, b) directly after exiting the spinning nozzle, the filaments run through a cooling delay zone of 30 mm to 200 mm in length, c) the filaments are cooled to below the solidification temperature, d) the filaments are bundled at a separation of between 500 mm and 2500 mm from the bottom face of the nozzle, e) the thread tension, before and between the drawing galettes is set to between 0.05 cN/dtex and 0.20 cN/dtex, f) the threads are wound with a thread tension of between 0.025 cN/dtex and 0.15 cN/dtex, g) the winding speed is set to between 2200 m/min and 6000 m/min and h) a polyester is used with 0.05 wt. % to 2.5 wt. %, based on the total weight of the filament, of added additive polymer as extensibility improver.

Method for the spinning and winding of polyester filaments using a spinning additive, polyester filaments obtained by the spinning method, draw texturing of the polyester filaments and bulked polyester filaments obtained by draw texturing

The present invention relates to processes for spinning and winding POY polyester filaments not less than 90% by weight, based on the total weight of the polyester filament, polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), preferably PTMT, by using spinning additives and also to the POY polyester filaments obtainable by the process. The present invention further also relates to processes for draw texturing the spun and wound polyester filaments and also to the bulky polyester filaments obtainable thereby.

The production of continuous polyester filaments and especially of polyethylene terephthalate (PET) filaments in a two-step process is already known. In this process, the first step comprises spinning and winding flat POY filaments which are fully drawn and heatset or draw-textured to bulky filaments in a second step.

An overview of this field is given by the book Synthetische Fasern by F. Fourné (1995), published by Hanser, Munich. However, only the production of PET fibers is described and no unified spinning technology is presented, only an overview describing the most diverse features.

Fiber production from various spinnable polymers, including polypropylene, polyamides, polyester, etc., forms part of the subject matter of DE-A 38 19 913. However, only the production of PET fibers is described

in the examples, as is discernible from the temperature at which the polymer is processed.

The prob lem with producing continuous polytrimethylene terephthalate (PTMT) or polyb utylene terephthalate (PBT) filaments is that POY filaments, not only directly after spinning and in winding b ut also for several hours after winding, in the course of storage at room temperature, exhibit a considerable tendency to shrink, which leads to yarn shortening. The package is compressed as a result, so that, in the extreme case, the package will shrink solid onto the winding mandrel and can no longer be removed. Furthermore, the package will develop a so-called saddle with hard edges and a waisted center portion. As a result, textile data of the filaments, for example the Uster value, become less uniform and prob lems develop when unwinding the packages. Such problems do not arise in the processing of PET fibers.

It is further ob served that, in contradistinction to PET filaments, POY PBT or PTMT filaments age fast in the course of storage. Structure hardening occurs and causes the b oiloff shrinkage to decrease to such a large extent that aftercrystallization can be detected. Such PBT or PTMT filaments are only partially suitable for further processing in that they lead to defects in draw texturing and to a significant reduction in the breaking strength of the textured yarn.

These differences b etween PET on the one hand and PBT and PTMT on the other are attrib utab le to structural and property differences, as reported for example in Chemical Fib ers Int., p. 53, vol. 50 (2000) and discussed at the 39th International Manmade Fib re Congress at Dornb irn from Septemb er 13 to 15. It is accordingly b elieved that different chain formations

are responsible for the property differences.

The first approaches to solving these prob lems are describ ed in WO 99/27168 and EP 0,731,196 B1. WO 99/27168 discloses a polyester fiber which is at least 90% by weight polytrimethylene terephthalate and has a b oiloff shrinkage b etween 5% and 16% and a b reaking extension of 20% to 60%. The polyester fiber described in WO 99/27168 is produced by spinning and drawing. The maximum spinning takeoff speed reported is 2 100 m/min. The process is uneconomical because of the low spinning speed. In addition, the polyester fibers obtained are, as the reported parameters document, highly crystalline and hence only partially suitab le for draw-texturing processes.

EP 0,731,196 B1 describ es a process for spinning, drawing and winding a synthetic yarn by subjecting the yarn to a heat treatment after drawing and b efore winding to reduce its tendency to shrink. Synthetic fib ers which can b e used include polytrimethylene terephthalate fib ers. In EP 0,731,196 B1, the heat treatment is effected b y the synthetic yarn b eing guided in close vicinity to b ut essentially contactlessly along an elongate heating surface. The application of a heat treatment adds to the cost of the process and, what is more, provides synthetic yarns of high crystallinity which are only partially suitab le for draw-texturing processes.

The article b y Dr. H.S. Brown and H.H. Chuah; "Texturing of textile filament yarns b ased on polytrimethylene terephthalate" Chemical Fib ers International, Volume 47, Feb r. 1997, p. 72-74 describ es the draw texturing of POY polytrimethylene terephthalate filaments at texturing speeds of 450 m/min and 850 m/min. According to this disclosure, the

lower texturing speed of 450 m/min is more suitable for polytrimethylene terephthalate filaments, since fibers having better material properties are obtained in this case. The breaking strength of the polytrimethylene terephthalate fibers is reported as 26.5 cN/tex (texturing speed 450 m/min) and 29.15 cN/tex (texturing speed 850 m/min) and the breaking extension as 38.0% (texturing speed 450 m/min) and 33.5% (texturing speed 850 m/min).

WO 01/04393 describes PTMT filaments having a boiloff shrinkage in the range from 3 to 40%. However, this value is determined immediately after the filaments have been formed. This value decreases to below 20% in the course of 4 weeks of storage under standard conditions, as documented by the accompanying figure 1.

Figure 1 describes the change in the boiloff shrinkage for three PTMT POY bobbins as a function of the storage time under standard conditions. The three bobbins investigated had different initial values. Bobbins #16 and 17, having a high initial value of > 40%, have a boiloff shrinkage after 4 weeks of above 30% and preferably of above 40%. However, when the initial boiloff shrinkage value is less than 40%, it is evident from bobbin 18 that the boiloff shrinkage value will drop to below the critical value of 30% after a storage time of 4 weeks.

The boiloff shrinkage is a measure of the processibility and the crystallinity of the fibers. The fibers described in WO 01/04393 comprise plastics having a comparatively high crystallinity, which are significantly more difficult to process and can only be processed at a lower draw ratio and/or at a lower texturing speed.

It is an object of the present invention to provide a process for spinning and winding POY polyester filaments not less than 90% by weight, based on the total weight of the filaments, PBT and/or PTMT whereby POY polyester filaments are simple to produce and wind up. More particularly, the POY polyester filaments shall have breaking extension values in the range of 90%-165%, a high uniformity with regard to filament parameters and also a low crystallinity.

It is a further object of the present invention to provide an economical industrial process for spinning and winding POY polyester filaments. The process of the invention shall permit very high spinning takeoff speeds, preferably above 2 200 m/min, and high yarn weights on the package of more than 4 kg.

It is yet a further object of the present invention to improve the storability of the POY polyester filaments obtainable by the process of the invention. They shall be storable for a prolonged period, for example 4 weeks. Ideally, the package shall not compact in the course of storage, especially shall not shrink solid on the winding mandrel and form a saddle having hard edges and waisted center portion, so that there shall be no problems unwinding from the package.

According to the invention, the POY polyester filaments shall be simple to further process in a drawing or draw-texturing operation, especially at high texturing speeds, preferably above 450 m/min. The filaments obtainable by draw texturing shall have excellent material properties, for example a high breaking strength of more than 26 cN/tex and a high breaking extension of more than 30% for HE filaments or more than 36% for SET filaments.

These and other ob jects not explicitly mentioned b ut readily derivab le or apparent from the related matters discussed herein at the b eginning are achieved b y a process for spinning and winding that comprises all the features of claim 1. Advantageous modifications of the process according to the invention are protected in sub claims appendant to claim 1. The POY polyester filament ob tainab le b y the spinning process is describ ed in an independent product claim. The draw texturing of the POY polyester filament is claimed in process claim 7, whereas product claims 8 and 9 relate to the b ulky filaments ob tainab le b y the draw texturing.

The present invention accordingly provides a process for producing and winding POY filaments not less than 90% b y weight, b ased on the total weight of the polyester filament, polyb utylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), preferably PTMT, characterized in that it comprises

a) setting the spinline extension ratio in the range from 70 to 500,
b) passing the filaments directly upon exit from the spinneret through a quench delay zone 30 mm to 200 mm in length,
c) quenching the filaments to b elow the solidification temperature,
d) converging the filaments at a distance between 500 mm and 2 500 mm from the underface of the spinneret,
e) setting the yarn tension ab ove and b etween the takeoff godets b etween 0.05 cN/dtex to 0.20 cN/dtex,
f) taking the yarn up at a yarn tension between 0.025 cN/dtex to 0.15 cN/dtex,
g) setting the takeup speed between 2 200 m/min and 6

000 m/min

h) and using a polyester which contains 0.05% b y weight to 2.5% b y weight, b ased on the total weight of the filament, of additive polymer extensibility enhancer in admixture.

This unforeseeab le process provides POY polyester filaments which maintain their excellent material properties even after 4 weeks of storage under standard conditions. No significant worsening in the uniformity values of the yarn due to aging and no shrinkage of the spun fiber on the bobbin are observed.

At the same time, the process of the invention has a number of further advantages. These include:

⇒ The process of the invention is simple and economical to practice on a large industrial scale. More particularly, the process permits spinning and winding at high takeoff speeds of at least 2 200 m/min and the production of packages holding high yarn weights of more than 4 kg.

⇒ The use of spinning additives makes it possible to achieve takeoff speeds of up to 6 000 m/min. The equipment can be operated particularly economically as a result.

⇒ The POY polyester filaments ob tainab le b y the process can thus b e further processed in a drawing or draw-texturing operation simply, economically and on a large industrial scale. In the operation, the texturing can b e carried out at speeds ab ove 450 m/min.

⇒ Owing to the high uniformity of the POY polyester filaments ob tainab le b y the process, it is simple

to achieve good package build to ensure uniform and sub stantially defect-free dyeing and further processing of the POY polyester filament.

⇒ The filaments obtainable by the draw texturing have a high breaking strength of more than 26 cN/tex and a high breaking extension of more than 30% for HE filaments and more than 36% for SET filaments.

The present invention provides a process for producing and for winding POY polyester filaments not less than 90% by weight polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), based on the total weight of the filament. Polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT) are known to one skilled in the art. Polybutylene terephthalate (PBT) is obtainable by polycondensation of terephthalic acid with equimolar amounts of 1,4-butanediol and polytrimethylene terephthalate is obtainable by polycondensation of terephthalic acid with equimolar amounts of 1,3-propanediol. Mixtures of the two polyesters are also conceivable. According to the invention, PTMT is preferred.

The polyesters may be homopolymers or copolymers. Useful copolymers include especially copolymers which, as well as PTMT and/or PBT repeat units, contain up to 15 mol%, based on all the repeat units of the polyesters, of repeat units of customary comonomers, for example ethylene glycol, diethylene glycol, triethylene glycol, 1,4-cyclohexanedimethanol, polyethylene glycol, isophthalic acid and/or adipic acid. For the purposes of the present invention, however, polyester homopolymers are preferred.

The polyesters of the invention may include customary

amounts of further additives as admixtures, such as catalysts, stab ilizers, antistats, antioxidants, flame retardants, dyes, dye uptake modifiers, light stab ilizers, organic phosphites, optical b righteners and delusterants. Preferab ly, the polyesters include 0 to 5% by weight of additives, based on the total weight of the filament.

The polyesters may further include a small fraction, preferab ly up to 0.5% b y weight, b ased on the total weight of the filament, of b rancher components. Preferred b rancher components according to the invention include polyfunctional acids, such as trimellitic acid, or pyromellitic acid, or tri- to hexavalent alcohols, such as trimethylolpropane, pentaerythritol, dipentaerythritol, glycerol or corresponding hydroxyacids.

In the context of the present invention, the PBT and/or PTMT are admixed with 0.05% b y weight to 2.5% b y weight, b ased on the total weight of the filament, of additive polymers as extensib ility enhancers. Particularly useful additive polymers for the purposes of the invention include the hereinb elow specified polymers and/or copolymers:

1. A copolymer containing the following monomer units:

   A = acrylic acid, methacrylic acid or CH_2 =

   CR-COOR', where R is an H atom or a CH_3 group

   and R' is a C_{1-15}-alkyl radical or a C_{5-12}

   -cycloalkyl radical or a C_{6-14}-aryl radical,

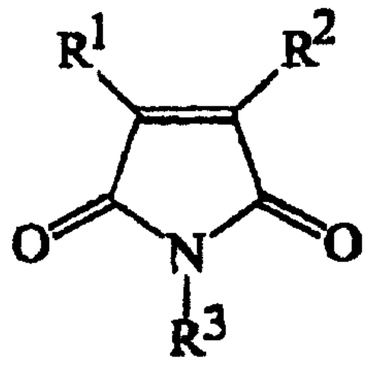
B = styrene or C_{1-3}-alkyl-substituted styrenes,

the copolymer consisting of 60 to 98% by weight of A and 2 to 40% by weight of B, preferably of 83 to 98% by weight of A and 2 to 17% by weight of B, and more preferably of 90 to 98% by weight of A and 2 to 10% by weight of B (sum total = 100% by weight).

2. A copolymer containing the following monomer units:

C = styrene or C_{1-3}-alkyl-substituted styrenes,

D = one or more monomers of the formula I, II or III



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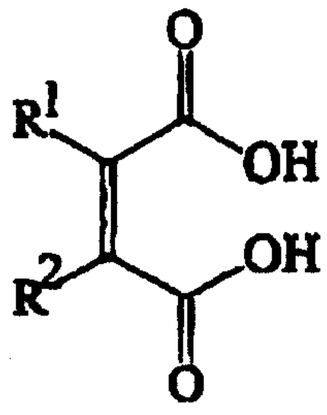

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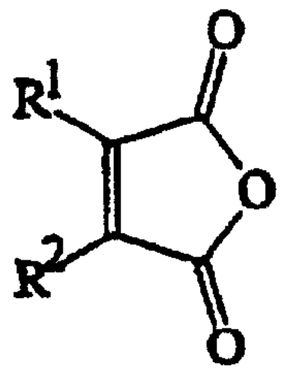


(III)

where R^1, R^2 and R^3 are each an H atom or a C_{1-15}-alkyl radical or a C_{6-14}-aryl radical or a C_{5-12}-cycloalkyl radical,

the copolymer consisting of 15 to 95% by weight of C and 2 to 80% by weight of D, preferably of 50 to 90% by weight of C and 10 to 50% by weight of D and more preferably of 70 to 85% of C and 15 to 30% by weight of D, the sum total of C and D being

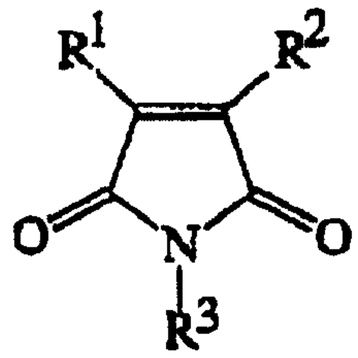
100% by weight.

3. A copolymer containing the following monomer units:

E = acrylic acid, methacrylic acid or CH_2 = CR-COOR', where R is an H atom or a CH_3 group and R' is a C_{1-15}-alkyl radical or a C_{5-12} -cycloalkyl radical or a C_{6-14}-aryl radical,

F = styrene or C_{1-3}-alkyl-substituted styrenes,

G = one or more monomers of the formula I, II or III



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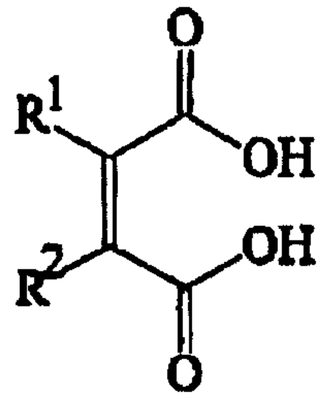

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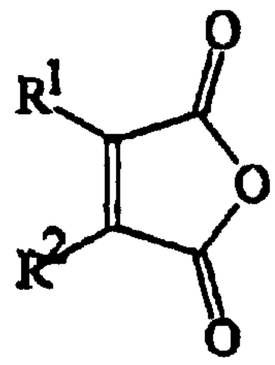


(III)

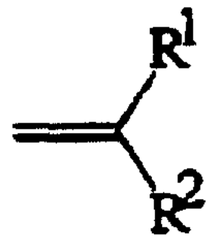
where R^1, R^2 and R^3 are each an H atom or a C_{1-15}-alkyl radical or a C_{5-12}-cycloalkyl radical or a C_{6-14}-aryl radical,

H = one or more ethylenically unsaturated monomers which are copolymerizab le with E and/or with F and/or G and are selected from

the group consisting of α-methylstyrene, vinyl acetate, acrylic esters, methacrylic esters other than E, vinyl chloride, vinylidene chloride, halogen-sub stituted styrenes, vinyl ethers, isopropenyl ethers and dienes,

the copolymer consisting of 30 to 99% by weight of E, 0 to 50% by weight of F, >0 to 50% by weight of G and 0 to 50% by weight of H, preferably of 45 to 97% by weight of E, 0 to 30% by weight of F, 3 to 40% by weight of G and 0 to 30% by weight of H and more preferably of 60 to 94% by weight of E, 0 to 20% by weight of F, 6 to 30% by weight of G and 0 to 20% by weight of H, the sum total of E, F, G and H being 100% by weight.

4. A polymer of the following monomer unit:



where R^1 and R^2 are substituents consisting of the optional atoms C, H, O, S, P and halogen atoms and the sum total of the molecular weights of R^1 and R^2 is at least 40. Exemplary monomer units include acrylic acid, methacrylic acid and CH_2 = CR-COOR', where R is an H atom or a CH_3 group and R' is a C $_{1-15}$-alkyl radical or a C_{5-12}-cycloalkyl radical

or a C_{6-14}-aryl radical, and also styrene and C

$_{1-3}$-alkyl-substituted styrenes.

Details of the production of these sub stances are described in WO 99/07 927.

Particular preference for the purposes of the present invention is given to additive polymers and/or copolymers in the form of bead polymers whose particle size is in a particularly favorab le range. It is preferab le for the additive polymers and/or copolymers which are to b e used according to the invention, for example by mixing into the melt of the fiber polymers, to b e present in the form of particles having an average diameter of 0.1 to 1.0 mm. However, larger or smaller b eads or granules can also b e used. The additive polymers and/or copolymers can also already be included in chips of the matrix polymer, obviating any metered addition.

Preference is further given to additive polymers and/or copolymers which are amorphous and insolub le in the polyester matrix. They preferab ly possess a glass transition temperature of 90 to 200°C, the glass transition temperature b eing determined in a known manner, preferab ly b y differential scanning calorimetry. Further details are discernib le from the prior art, for example from WO 99/07927, the disclosure of which is hereb y expressly incorporated herein b y reference.

Preferab ly, the additive polymer and/or copolymer is selected so that the ratio of the melt viscosities of the additive polymer and/or copolymer and of the matrix polymer is in the range from 0.8:1 to 10:1 and preferab ly in the range from 1.5:1 to 8:1. The melt

viscosity is measured in a known manner using an oscillation rheometer at an oscillation frequency of 2.4 Hz and a temperature equal to the melting temperature of the matrix polymer plus 28°C. For PTMT, the temperature at which the melt viscosity is measured is 255°C. Further details may again b e found in WO 99/07927. The melt viscosity of the additive polymer and/or copolymer is preferab ly higher than that of the matrix polymer, and it has b een determined that the choice of a specific viscosity range for the additive polymer and/or copolymer and the choice of the viscosity ratio contrib utes to optimizing the properties of the yarn product. Given an optimized viscosity ratio, it is possib le to minimize the amount of additive polymer and/or copolymer added and so, inter alia, improve the economics of the process. The polymer b lend to b e spun preferab ly contains 0.05 to 2.5% b y weight and more preferab ly 0.25 to 2.0% b y weight of additive polymer and/or copolymer.

The choice of the favorab le viscosity ratio provides a narrow distrib ution of the particle sizes of the additive polymer and/or copolymer in the polymer matrix comb ined with the desired fib ril structure for the additive polymer and/or copolymer in the fib er. The high glass transition temperature of the additive polymer and/or copolymer compared with the matrix polymer ensures rapid consolidation of this fib ril structure in the spun fiber. The maximum particle sizes of the additive polymer and/or copolymer amount to about 1 000 nm immediately following emergence from the spinneret, while the average particle size is 400 nm or less. The favorable fibril structure is obtained after the fiber has been drawn down, the filaments containing at least 60% by weight of the additive polymer and/or copolymer in the form of fibrils having lengths in the

range from 0.5 to 20 µm and diameters in the range from 0.01 to 0.5 µm.

Useful polyesters for the invention are preferab ly thermoplastically formab le and can b e spun into filaments and wound up. In this context, particularly advantageous polyesters have a limiting viscosity number in the range from 0.70 dl/g to 0.95 dl/g.

A polymer melt can b e taken for example directly from the final reactor of a polycondensation plant or b e produced from solid polymer chips in a melting extruder.

One known way of incorporating the spinning additive is to meter it in molten or solid form into the matrix polymer and disperse it therein homogeneously to form fine particles. Advantageously, an apparatus as described in DE 100 22 889 can be used.

In the process of the invention, the melt or melt mixture of the polyester is pumped by spinning pumps at constant speed, the speed b eing calculated b y a known formula so that the desired fib er linear density is ob tained, into spinneret packs to b e extruded through the holes in the die plate of the pack to form molten filaments.

The melt may be prepared for example from polymer chips in an extruder, in which case it is particularly favorab le for the chips first to b e dried to a water content ≤30 ppm and especially to a water content ≤15 ppm.

The temperature of the melt, which is commonly referred to as the spinning temperature and which is measured ab ove the spinning pump, depends on the melting point of the polymer or polymer b lend used. It is preferably

situated in the range given by formula 1:

Formula 1:

$T_m + 15°C \leq T_{Sp} \leq T_m + 45°C$

where

T_m is the melting point of the polyester [°C]

T_{Sp} is the spinning temperature [°C].

The specified parameters serve to limit the hydrolytic and/or thermal viscosity degradation, which should advantageously b e very low. In the context of the present invention, a viscosity degradation of less than 0.12 dl/g and especially less than 0.08 dl/g is desirable.

Melt homogeneity has a direct influence on the properties of the spun filaments. It is therefore preferab le to use a static mixer having at least one element and installed b elow the spinning pump to homogenize the melt.

Die plate temperature, which depends on the spinning temperature, is controlled b y the plate's secondary heating system. Useful secondary heating systems include for example a spinning b eam heated with "Diphyl" or additional convective or radiative heaters. The temperature of the die plates is customarily equal to the spinning temperature.

A temperature increase at the die plate can be obtained through the pressure gradient in the spinneret pack. Known derivations, for example K. Riggert "Fortschritte in der Herstellung von Polyester-Reifenkordgarn" Chemiefasern **21**, page 379 (1971), describ e a temperature increase of ab out 4°C per 100 b ar of pressure drop.

It is further possible to control die pressure through the use of loose filter media, especially through the use of steel sand having an average particle size between 0.10 mm and 1.2 mm, preferably between 0.12 mm and 0.75 mm and/or filter disks, which can be formed from woven or nonwoven metal fabrics having a fineness ≤40 µm.

In addition, the pressure drop in the die hole contributes to the overall pressure. The die pressure is preferably set between 80 bar and 450 bar, especially between 100 bar and 250 bar.

The spinline extension ratio i_{Sp}, i.e. the ratio of the takeoff speed to the extrusion speed, is calculated in accordance with US 5,250,245 via formula 2 from the density of the polymer or polymer mixture, the spinneret hole diameter and the filament linear density:

Formula 2:

$$i_{Sp} = 2.25 \bullet 10^5 \bullet (\delta \bullet \pi) \bullet D^2 (cm) / dpf (den)$$

where

δ = density of melt [g/cm^3]; for PTMT = 1.12 g/cm^3

D = spinneret hole diameter [cm]

dpf = denier per filament [den].

For the purposes of the present invention, the spinline extension ratio is between 70 and 500, preferably between 100 and 250.

The length/diameter ratio of the spinneret hole is preferably selected to be between 1.5 and 6, especially between 1.5 and 4.

The extruded filaments pass through a quench delay zone. The quench delay zone is configured directly

b elow the spin pack as a recess zone in which the filaments emerging from the spinneret holes are protected from the direct action of the cooling gas and are delayed in spinline extension or cooling. An active part of the recess is constructed as an extension of the spin pack into the spinning b eam, so that the filaments are surrounded b y heated walls. A passive part is formed b y insulating layers and unheated frames. The lengths of the active recess are between 0 to 100 mm and those of the passive part between 20 to 120 mm, sub ject to an overall length of 30 - 200 mm, preferably 30 - 120 mm.

As an alternative to the active recess, a reheater can be disposed below the spinning beam. In contrast to the active recess, this zone of cylindrical or rectangular cross section then comprises at least one heating system independent of the spinning beam.

In the case of radial porous quenching systems which surround the spinline concentrically, the quench delay can be attained using cylindrical shrouds.

The filaments are sub sequently cooled to temperatures below the solidification temperature. For the purposes of the invention, the solidification temperature is the temperature at which the melt passes into the solid state.

In the context of the present invention, it has b een determined to be particularly advantageous to cool the filaments down to a temperature at which they are essentially no longer tacky. It is particularly advantageous to cool the filaments to temperatures b elow their crystallization temperature, especially to temperatures below their glass transition temperature.

Means for quenching or cooling filaments are known from

the prior art. It is particularly useful according to the invention to use cooling gases, especially cooled air. The temperature of the cooling air is preferably in the range from 12°C to 35°C, and especially in the range from 16°C to 26°C. The velocity of the cooling air is advantageously in the range from 0.20 m/sec to 0.55 m/sec.

The filaments may be cooled using for example single end systems comprising single cooling tubes having a perforated wall. Cooling of each individual filament is obtained through active cooling air supply or by utilizing the self-suction effect of the filaments. As an alternative to the individual tubes, it is also possible to use the familiar crossflow quench systems.

In a particular embodiment of the cooling and spinline extension region, the filaments emerging from the delay zone are exposed to cooling air in a zone 10 to 175 cm and preferably 10 - 80 cm in length. A zone 10 - 40 cm in length is particularly suitable for filaments having a linear density at windup ≤1.5 dtex per filament and a zone length of 20 - 80 cm is particularly suitable for filaments having a linear density between 1.5 and 9.0 dtex per filament. The filaments and the accompanying air are subsequently conjointly passed through a channel having a reduced cross section at a ratio of the air to the spinline speed at takeoff in the range from 0.2 to 20:1, preferably in the range from 0.4 to 5:1, by controlling the cross-sectional constriction and the dimensioning in the spinline transportation direction.

After the filaments have been cooled down to temperatures below the solidification point, they are converged to form a yarn bundle. A suitable distance according to the invention for the point of convergence

from the underface of the spinneret can be determined using conventional methods for online measurement of the yarn speed and/or yarn temperature, for example using a laser doppler anemometer from TSI/Germany or an infrared camera from Goratec/Germany type IRRIS 160. It is in the range from 500 to 2 500 mm, preferably from 500 to 1 800 mm. Filaments having an as-spun linear density ≤4.5 dtex are preferably converged into a multifilament bundle at a smaller distance ≤1 500 mm, while thicker filaments are preferably converged at a greater distance.

It is advantageous for the purposes of the present invention that preferably all surfaces which come into contact with the spun filament are fabricated of particularly low-friction materials. This substantially avoids broken filaments and provides higher quality filament yarns. Particularly suitable for this purpose are low-friction surfaces of the "Trib oFil" specification from Ceramtec/Germany.

The filaments are converged in an oiler pin which supplies the yarn with the desired amount of spin finish at a uniform rate. A particularly suitable oiler pin is characterized by an inlet part, the yarn duct with oil inlet orifice and an outlet part. The inlet part is funnellike, so that contact by the still dry filaments is avoided. The contact point of the filaments occurs within the yarn duct after the supply of spin finish. Yarn duct and oil inlet orifice are conformed in width to the yarn linear density and the number of filaments. Orifices and widths in the range from 1.0 mm to 4.0 mm are particularly suitable. The outlet part of the oiler pin is configured as a uniformizing zone, which preferably comprises oil reservoirs. Such oilers are obtainable for example from

Ceramtec/Germany or Goulston/USA.

The uniformity of oil application is of immense importance for the invention. The uniformity can b e determined for example using a Rossa meter as per the method described in Chemiefasern/Textilindustrie, 42/94 November 1992 at page 896. Preferably, such a procedure provides standard deviation values for the oil application which are less than 90 digits and especially less than 60 digits. Particular preference for the purposes of the invention is given to oil application standard deviation values of less than 45 digits and especially less than 30 digits. A standard deviation value of 90 or 45 digits corresponds approximately to 6.2% or 3.1% of the coefficient of variation, respectively.

It is particularly advantageous for the purposes of the present invention to design spin finish lines and pumps to b e self-degassing to avoid gas b ub b les, since gas b ub b les can lead to an appreciab le variation in oil application.

According to the invention, it is particularly preferable for the filaments to be entangled before the yarn is wound up. In the context of the present invention, jets having closed yarn ducts will be found to be particularly suitable, since such systems prevent snubbing of the yarn in the feed slot even at low yarn tension and high air pressure. The entangling jets are preferab ly disposed b etween godets and the yarn exit tension is controlled via the different speeds of the inlet and outlet godets. The yarn exit tension should not exceed 0.20 cN/dtex and should primarily have values b etween 0.05 cN/dtex and 0.15 cN/dtex. The entangling air pressure is between 0.5 and 5.5 bar, or at most 3.0 bar in the case of takeup speeds of up to 3

500 m/min.

The yarns are preferably entangled to node counts of at least 10 n/m. Maximum nodeless gaps of less than 100 cm and node count coefficient variation values below 100% are of particular interest. Advantageously, the employment of air pressures above 1.0 bar provides node counts ≥15 n/m, which are characterized b y high uniformity in that the coefficient of variation is not more than 70% and the maximum nodeless gap is 50 cm. In actual service, systems of the LD type from Temco/Germany, the doub le system from Slack & Parr/USA or Polyjet from Heb erlein have b een found to b e particularly useful.

The circumferential speed of the first godet unit is referred to as takeoff speed. Further godet systems can b e employed b efore the yarn is wound up in the wind assembly to form packages (bobbins) on formers.

Stab le, defect-free packages are a b asic prerequisite for defect-free winding of the yarn and for an ideally defect-free further processing. Therefore, in the context of the present invention, the takeup tension employed is in the range of 0.025 cN/dtex - 0.15 cN/dtex and preferab ly in the range of 0.03 cN/dtex - 0.08 cN/dtex.

An important parameter of the process according to the invention is the yarn tension setting above and between the takeoff godets. As will b e known, this tension is made up essentially of Hamana's actual orientation tension, the frictional tension on the yarn guides and the oiler and the yarn-air frictional tension. For the purposes of the present invention, the yarn tension ab ove and b etween the takeoff godets is in the range from 0.05 cN/dtex to 0.20 cN/dtex and preferably in the

range between 0.08 cN/dtex and 0.15 cN/dtex.

An excessively low tension below 0.05 cN/dtex no longer provides the desired degree of partial orientation. When the tension exceeds 0.20 cN/dtex, this tension will induce a memory effect in the course of winding and storing the bobbins that leads to a deterioration in yarn parameters.

The tension is controlled according to the invention by the distance of the oiler from the jet spinneret, the frictional surfaces and the length of the gap between oiler and takeoff godet. This length is advantageously not more than 6.0 m and preferably less than 2.0 m, the spinning system and the takeoff machine being disposed in such a way by parallel construction as to ensure a straight yarn path.

The geometric parameters also describe the conditioning time of the yarn between converging point and takeup. The fast relaxation during the period has an effect on the quality of package build. The conditioning time so defined is preferably chosen to be between 50 and 200 ms.

The takeup speed of the POY is between 2 200 m/min and 6 000 m/min according to the invention. It is preferable to choose a speed between 2 500 m/min and 6 000 m/min. It is particularly preferable for the polymer blends to be wound up at speeds in the range from 3 500 m/min to 6 000 m/min.

Advantageously, the process according to the invention is carried out by adjusting the environment of the yarn package to be at a temperature ≤45°C, especially between 12 and 35°C, and a relative humidity of 40 - 85%. It is further advantageous to store the POY

packages at 12 to 35°C and a relative humidity of 40-85% for at least 4 hours prior to further processing.

After 4 weeks of storage under standard conditions, the filament according to the invention has

a) a b reaking extension b etween 90% and 165%, preferably between 90 and 135%,
b) a boiloff shrinkage of at least 30%, preferably ≥40%,
c) a normal Uster b elow 1.1%, preferab ly b elow 0.9%,
d) a birefringence between 0.030 and 0.058,
e) a density of less than 1.35 g/cm^3, preferably less than 1.33 g/cm^3,
f) a breaking load coefficient of variation ≤4.5%, preferably ≤2.5% and
g) a b reaking extension coefficient of variation ≤ 4.5%, preferably ≤2.5%

The term "standard conditions" is known to one skilled in the art and defined via the DIN 53802 standard. Under "standard conditions" as per DIN 53802, the temperature is 20±2°C and the relative humidity 65±2%.

It is additionally particularly advantageous for the purposes of the present invention for the b oiloff shrinkage to b e b etween 50 and 65% when measured directly after windup and to b e at least 30% and preferably ≥40% after 4 weeks of storage under standard conditions. It has b een determined that, surprisingly, POY bobbins produced in this way have excellent further processing properties.

Methods for determining the indicated material parameters are well known to those skilled in the art.

They are discernib le from the technical literature. Although most of the parameters can b e determined in various ways, the following methods for determining the filament parameters will prove particularly advantageous for the purposes of the present invention:

The intrinsic viscosity is measured at 25°C in an Ub b elohde capillary viscometer and calculated b y the familiar formula. The solvent used is a 3:2 w/w mixture of phenol and 1,2-dichlorobenzene. The concentration of the solution is 0.5 g of polyester per 100 ml of solution.

The melting point, the crystallization temperature and the glass transition temperature are each determined using a DSC calorimeter from Mettler. The sample is initially heated to 280°C to melt it and then quenched. The DSC measurement is done in the range from 20°C to 280°C at a heating rate of 10 K/min. The reported temperatures are determined by the processor.

Filament density is determined in a density gradient column at a temperature of 23±0.1°C. The reagent used is n-heptane (C_7H_{16}) and tetrachloromethane (CCl_4). The result of the density measurement can b e used to calculate the crystallinity on the basis of the density of the amorphous polyester D_a and the density of the crystalline polyester D_k. The calculation is described in the literature and for PTMT for example the corresponding values are D_a = 1.295 g/cm^3 and D_k = 1.429 g/cm^3.

Linear density is determined in a known manner using a precision reel and weighing means. The pretension used is advantageously 0.05 cN/dtex for POY and 0.2 cN/dtex for DTY.

Breaking strength and breaking extension are determined on a Statimat apparatus under the following conditions: the clamped length is 200 mm for POY and 500 mm for DTY, the rate of extension is 2 000 mm/min for POY and 1 500 mm/min for DTY and the pretension is 0.05 cN/dtex for POY and 0.2 cN/dtex for DTY. The maximum breaking load values are divided by the linear density to determine the breaking strength, and breaking extension is determined at maximum load.

Boiloff shrinkage is determined by treating filament skeins in water at 95±1°C for 10±1 min in a tensionless state. The skeins are prepared by reeling at a pretension of 0.05 cN/dtex for POY and 0.2 cN/dtex for DTY; the length measurement of the skeins before and after the thermal treatment is carried out at 0.2 cN/dtex. The difference in length is used to calculate the boiloff shrinkage in a known manner.

Birefringence is determined by the method described in DE 19,519,898, the disclosure of which is explicitly incorporated herein by reference.

The crimp parameters of the textured filaments are measured in accordance with DIN 53840 Part 1 using a Texturmat apparatus from Stein/Germany at a development temperature of 120°C.

The normal Uster values are determined using a 4-CX Uster tester and are reported as Uster % values. The testing speed is 100 m/min and the testing time 2.5 min.

The POY according to the invention is simple to further process, especially draw texture. In the present invention, draw texturing is preferably carried out at a texturing speed of at least 500 m/min and particularly preferably at a texturing speed of at

least 700 m/min. The draw ratio is preferably at least 1.35:1 and especially at least 1.40:1. It will b e particularly advantageous to draw texture on a high temperature heater type machine, for example an AFK machine from Barmag.

The bulky filaments produced in this way exhibit a low numb er of defects and on dyeing at the b oil with a disperse dye without carrier an excellent depth of shade and uniformity of color.

Bulky SET filaments produced according to the invention preferab ly have a b reaking strength of more than 26 cN/tex and a b reaking extension of more than 36%. In the case of b ulky HE filaments, which are ob tainab le without thermal treatment in a second heater, the breaking strength is preferably more than 26 cN/tex and the breaking extension more than 30%.

The b ulk and elasticity b ehavior of the filaments according to the invention is excellent.

Illustrative emb odiments of the invention will now b e more particularly described without the invention being limited to these examples.

Examples 1 to 3

Spinning and winding

PTMT chips having an intrinsic viscosity of 0.93 dl/g, a melt viscosity of 325 Pa s (measured at 2.4 Hz and 255°C), a melting point of 227°C, a crystallization temperature of 72°C and a glass transition temperature of 45°C were tumble dried at 130°C to a water content of 11 ppm.

The chips were melted in a 3E4 extruder from Barmag, so that the temperature of the melt was 255°C. This melt had added to it various amounts of Plexiglas 7N polymethyl methacrylate from Röhm Gmb H/Germany as an extensibility enhancer which had beforehand been dried to a residual moisture content of less than 0.1%.

For this purpose, the additive polymer was melted in a melting extruder, fed using a gear wheel metering pump to the feed means and fed from there through an injection nozzle in the flow direction into the polyester component. The two melts were homogenized and finely dispersed in an SMX static mixer from Sulzer having 15 elements and an internal diameter of 15 mm.

The melt viscosity of the Plexiglas 7N was 810 Pa s (2.4 Hz, 255°C), as a result of which the ratio of additive and polyester melt viscosities was 2.5:1.

The transported amount of melt was 63 g/min coupled with a residence time of 6 min, and the amount metered from the spinning pump to the spinneret pack was adjusted so that the POY linear density was about 102 dtex. Various takeup speed settings were used. One element of an HD-CSE type static mixer from Fluitec having an internal diameter of 10 mm had been installed below the spinning pump, but above the point of entry into the spinneret pack. The secondary heating systems for the product line and the spin b lock, which

contained the pump and the spinneret pack, had been set to 255°C. The spinneret pack contained 350-500 µm steel sand 30 mm in height and also a 20 µm nonwoven filter and a 40 µm woven filter as filter media. The melt was extruded through an 80 mm diameter spinneret plate containing 34 holes 0.25 mm in diameter and 1.0 mm in length. The die pressure was about 120-140 bar.

The quench delay zone was 100 mm in length, made up of 30 mm in heated walling and 70 mm in insulation and unheated frame. The molten filaments were quenched with air flowing horizontally against the spinline over a length of 1500 mm. The quenching air had a flow rate of 0.35 m/sec, a temperature of 18°C and a relative humidity of 80%. The filaments became solid at about 800 mm below the spinneret.

A yarn oiler positioned at a distance of 1 050 mm from the spinneret was used to apply spin finish to the ends before converging. The oiler had a TriboFil surface and an inlet opening 1 mm in diameter. The amount of spin finish applied was 0.40%, based on fiber weight.

The converged spinline was then fed to the winding machine. The distance between the oiler and the first takeoff godet was 3.2 m. The conditioning time was between 105 and 140 ms. A pair of godets was S-wrapped by the yarn. Situated between the godets was a Temco entangling jet, which was operated using an air pressure of 1.5 bar. In line with the speed setting, the takeup speed of the Barmag SW6 winder was set in such a way that the takeup yarn tension was 5 cN. The room conditions were adjusted to 24°C and 60% relative humidity so that a temperature of about 34°C ensued in the environment of the yarn package.

A significant increase in productivity was obtained for

all amounts of additive added. The 10 kg b ob b ins produced were readily removab le from the winding mandrel. The POY yarns ob tained were notab le for good time constancy of the yarn properties over a storage period of 4 weeks under standard conditions as defined in DIN 53802. The b oiloff shrinkage directly after spinning and winding was found to be in the range of 51-54%. The texturability and the uniformity of dyeing achieved were found to be excellent. The draw ratio to be used was surprisingly high for the POY speeds used.

The other parameters and characteristic data are summarized in tables 1 to 4.

Table 1: Experimental parameters

Experimental parameters		**Example 1**	**Example 2**	**Example 3**
Additive concentration	[%]	0.5	0.7	1.0
Takeoff speed	[m/min]	3011	3520	4022
Takeup speed	[m/min]	3005	3500	4000
Spinline extension ratio		183	182	181
Yarn tensions				
above godets[1]	[cN]	13	15.5	16
between godets[1] max	[cN]	12	13	12.5
above godets[2]	[cN/dtex]	0.13	0.15	0.16
between godets[2] max	[cN/dtex]	0.11	0.13	0.12
Yarn tension[1]	[cN]	6.3	5.9	6.4
Yarn tension[2]	[cN/dtex]	0.062	0.058	0.062

[1]: absolute

[2]: based on linear density

Table 2: Material properties of PTMT POY filaments[1]

Material properties		**Example 1**	**Example 2**	**Example 3**
Linear density	[dtex]	102	102.5	103
Breaking strength	[cN/dtex]	20.2	21.8	22.3
Breaking extension	[%]	132.7	115.4	98.2
Normal Uster	[%]	0.80	0.90	0.94
Boiloff shrinkage	[%]	48	44	38

Birefringence•10^3	Δn	36	47	51
Density	[g/cm^3]	1.315	1.318	1.320
Breaking load CV	[%]	1.7	1.5	2.1
Breaking extension CV	[%]	1.9	1.9	3.3

CV: coefficient of variation

[1]: measured after 4 weeks of storage under normal conditions

Draw texturing

The PTMT filament bobbins were stored for 4 weeks under standard conditions as defined in DIN 53802 and then presented to a Barmag FK6-S-900 draw texturing machine. The experimental parameters for draw texturing to produce SET filaments are summarized in table 3 and the material properties of the resulting b ulky SET filaments in table 4.

Texturing defects were determined using Barmag's UNITENS system at the following limiting value settings: UP/LP = 3.0 cN, UM/LM = 6.0 cN.

Table 3: Experimental parameters of draw texturing

Experimental parameters		**Example 1**	**Example 2**	**Example 3**
Speed [m/min]	[m/min]	700	700	700
Draw ratio		1:1.70	1:1.60	1:1.44
D/Y ratio		2.1	2.1	2.1
Heater 1 temp.	[°C]	155	155	155
Heater 2 temp.	[°C]	160	160	160
Texturing defects	[n/10 km]	0	0	0
Yarn tension				
F^1, above assembly	[cN]	17	18	19
F^2, below assembly	[cN]	19	21	21
F^2-CV	[%]	0.78	0.93	0.89

F^2-CV: coefficient of variation of F^2

Table 4: Material properties of draw-textured filaments

Material properties		**Example 1**	**Example 2**	**Example 3**
Linear density	[dtex]	67	69	79
Breaking strength	[cN/tex]	26.9	29.6	28.2
Breaking extension	[%]	38.6	37.8	38.0
Inspection of dyeability		uniform	uniform	uniform
Crimp rigidity	[%]	84	85	79
Crimp contraction	[%]	25	24	23

What is claimed is:

1. A process for producing and winding POY polyester filaments not less than 90% by weight, based on the total weight of the polyester filament, polybutylene terephthalate (PBT) and/or polytrimethylene terephthalate (PTMT), preferably PTMT, characterized in that it comprises
   a) setting the spinline extension ratio in the range from 70 to 500,
   b) passing the filaments directly upon exit from the spinneret through a quench delay zone 30 mm to 200 mm in length,
   c) quenching the filaments to below the solidification temperature,
   d) converging the filaments at a distance between 500 mm and 2 500 mm from the underface of the spinneret,
   e) setting the yarn tension above and between the takeoff godets between 0.05 cN/dtex to 0.20 cN/dtex,
   f) taking the yarn up at a yarn tension between 0.025 cN/dtex to 0.15 cN/dtex,
   g) setting the takeup speed between 2 200 m/min and 6 000 m/min
   h) and using a polyester which contains 0.05% by weight to 2.5% by weight, based on the total weight of the filament, of additive polymer extensibility enhancer in admixture.

2. A process as claimed in claim 1, characterized in that PBT and/or PTMT having a limiting viscosity number in the range from 0.7 dl/g to 0.95 dl/g are used.

3. A process as claimed in claim 1 and/or 2, characterized in that the taking up is effected by setting a temperature ≤ 45°C in the ambience of

the yarn package.

4. A process as claimed in at least one of the preceding claims, characterized in that the POY bobbins are stored at 12-35°C and 40-85% relative humidity for at least 4 hours prior to further processing.

5. A process as claimed in at least one of the preceding claims, characterized in that the takeup speed is set between 2 500 m/min and 6 000 m/min.

6. POY polyester filaments obtainable by a process as claimed in at least one of the preceding claims, characterized in that it has
a) a breaking extension between 90% and 165%,
b) a boiloff shrinkage of at least 30%,
c) a normal Uster below 1.1%,
d) a birefringence between 0.030 and 0.058,
e) a density of less than 1.35 g/cm^3, preferably less than 1.33 g/cm^3,
f) a breaking load coefficient of variation ≤4.5% and
g) a breaking extension coefficient of variation ≤ 4.5%
after 4 weeks of storage under standard conditions as defined in DIN 53802.

7. A process for producing bulky polyester filaments, characterized in that filaments as claimed in claim 6 are processed in a draw-texturing machine at a speed of at least 500 m/min and a draw ratio of at least 1.35:1 into a bulky yarn.

8. Bulky polyester SET filaments obtainable by a process as claimed in claim 7, characterized in that their breaking strength is more than 26

cN/tex and the breaking extension more than 36%.

9. Bulky polyester HE filaments ob tainab le b y a process as claimed in claim 7, characterized in that their b reaking strength is more than 26 cN/dtex and the breaking extension more than 30%.

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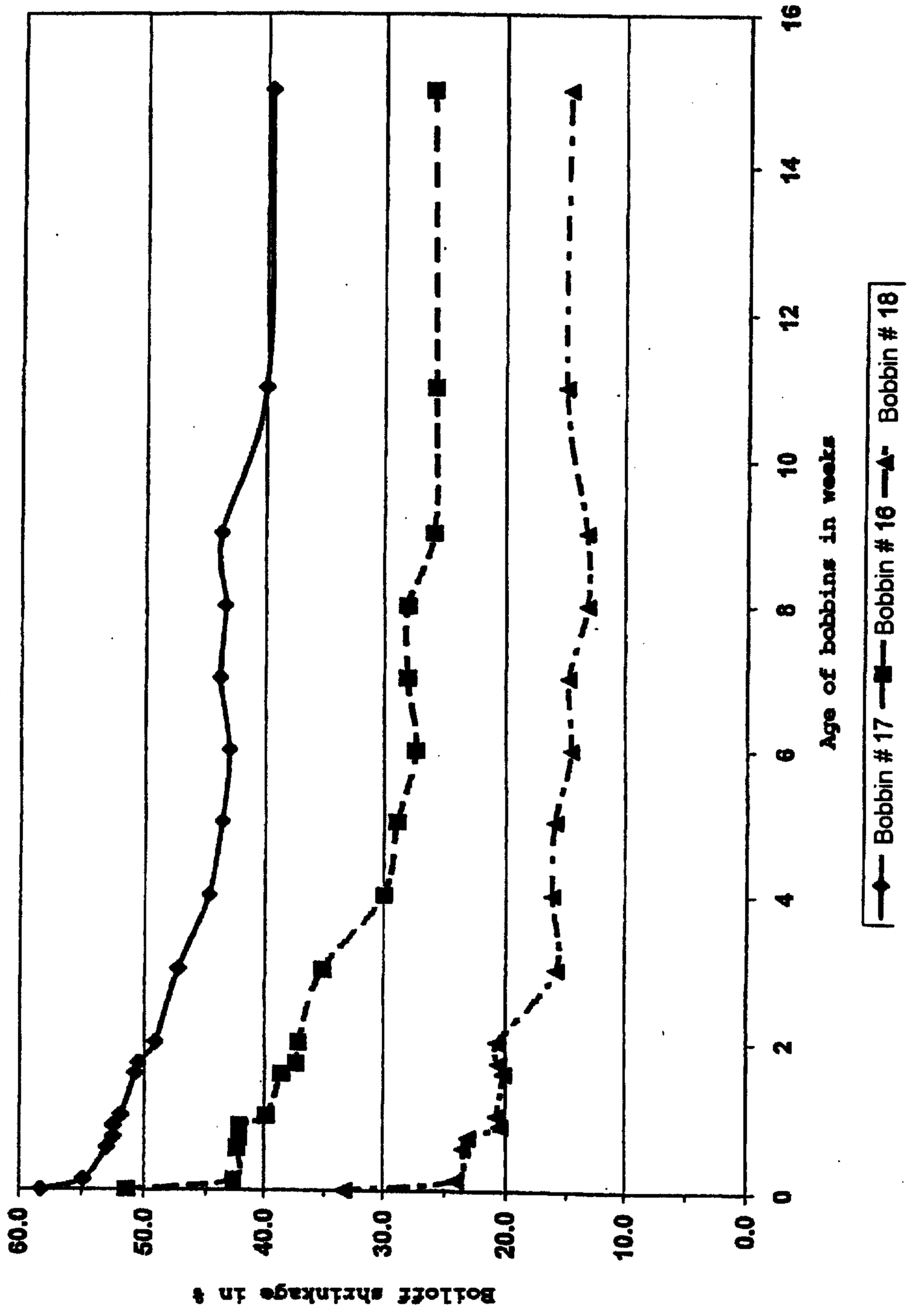
Bolloff shrinkage in %
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
2
4
6
8
10
12
14
16
Age of bobbins in weeks
Bobbin # 17
Bobbin # 16
Bobbin # 18

Figure 1