Oct. 31, 1967 A. L. BREEN 3,350,488

PROCESS FOR THE PRODUCTION OF SHARP-EDGE FIBERS

Original Filed May 27, 1958

2 Sheets-Sheet 1

Fig. 1.

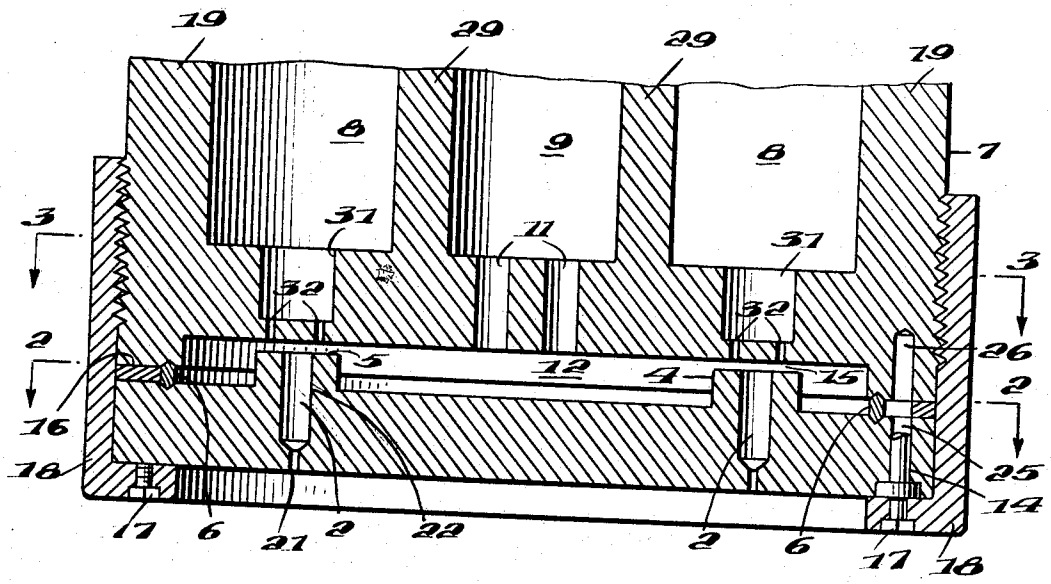


Fig. 2.

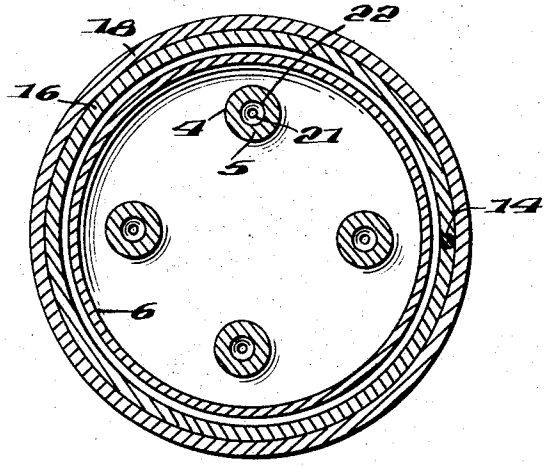
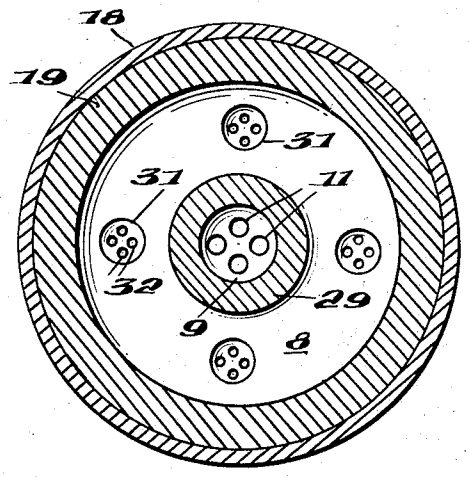


Fig. 3.



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Original Filed May 27, 1958

2 Sheets-Sheet 2

Fig. 4.

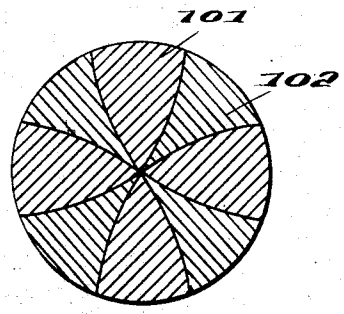


Fig. 5.

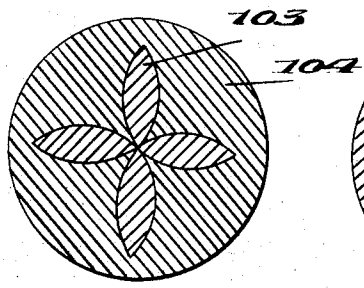


Fig. 6.

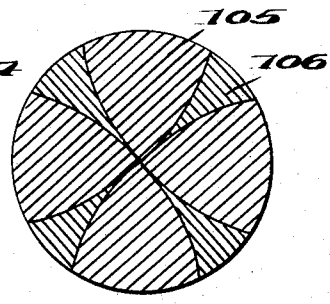


Fig. 7.

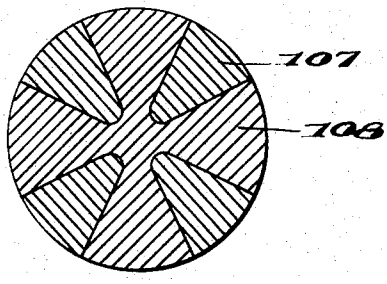


Fig. 8.

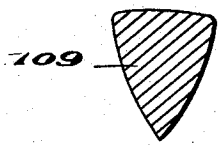


Fig. 9.

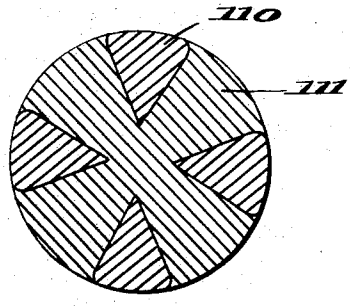
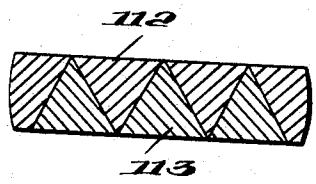


Fig. 10.



United States Patent Office 3,350,4
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3,350,488

PROCESS FOR THE PRODUCTION OF SHARP-EDGE FIBERS

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Original applications May 27, 1958, Ser. No. 738,166, and June 7, 1962, Ser. No. 200,758, now Patent No. 3,188,689, dated June 15, 1965. Divided and this application Mar. 3, 1965, Ser. No. 436,888
4 Claims. (Cl. 264—171)

This is a divisional application of application Ser. No. 200,758, filed June 7, 1962, which issued as U.S. Patent No. 3,188,689 on June 15, 1965, and of abandoned application Ser. No. 738,166, filed May 27, 1958.

This invention relates to the manufacture of filaments of a novel cross-section.

Much effort has been expended towards the preparation of fabrics that are silk-like from filaments of modern synthetic polymers. Although various proposed methods have duplicated one or more of the important characteristics of a silk fabric, a completely satisfactory substitute has not yet been found.

The desirability of producing textile filaments having one or more sharp longitudinal edges for obtaining silk-like fabrics has long been recognized. Despite numerous proposals mainly drawn to extrusion orifice designs, it has not been feasible to produce textile filaments having sharp edges in cross-section by the extrusion of melts or solutions of fiber-forming polymers. This is due to the fact that the surface of a filament formed by extrusion through an orifice tends to assume the cross-sectional contour of a circle, the smallest boundary for the given cross-sectional area.

One object of this invention is to produce filaments of synthetic linear polymers having one or more sharp longitudinal edges.

A further object is the preparation of a fabric that has the properties of silk. Other objects will become apparent in the course of the following discussion.

In accordance with one embodiment of the invention a composite filament of substantially uniform cross-section composed of segments of at least two dissimilar synthetic polymeric compositions, said cross-section having at least two segments of at least one of said polymers, any contact between such segments of the same polymer being substantially point contact is extruded. This may be accomplished through the use of the spinneret assembly more completely described below. After drawing, as desired, the filament may be separated into its component sections by mechanical action. Alternatively, all sections composed of one polymer composition are removed as for example by dissolution or chemical decomposition.

By the term "segment" is meant a portion of a filamentary cross-section having at least one sharp point formed by the intersection of two straight or curved lines. The segment may have: one sharp point such as in a tear-drop-shape as shown in **110** of FIGURE 9; 2 sharp points such as in a lens-shape as shown in **103** of FIGURE 5 or such as in the shape shown in **107** of FIGURE 7; 3 sharp points as in a plane or curvilinear triangle; or a multiplicity of sharp points such as in a figure formed by the joining of 2 or more simple segments as in a formee cross.

FIGURE 1 shows in axial section a spinneret assembly useful for this purpose. Front or bottom plate **1** with orifices **2** is recessed at the back about plateau-like projections **4**. The outer wall at the bottom of back or top plate **7** is sealed against and spaced from the front plate gasket **6** and shim **16**. Relatively unconstricted region between the two plates is interrupted at intervals by constricted regions **15** between the opposing face of the back plate and plateaous **5** of the protrusions from the front plate. The back plate is partitioned on top by outer wall **19** and inner wall **29** into annular chamber **8** and central chamber **9**. The annular chamber communicates with the constricted regions between the two plates through leak holes **31** and orifices **32**, and the central chamber communicates with the intervening relatively unconstricted region through holes **11**. The two plates are retained in place by cap **18** which is threaded onto the end of the back plate and is fixed to the front plate with set screw **17**. The upper part of the housing (not shown) receives suitable piping or other supply means for separate connection to the two chambers, which may constitute distribution or filtering spaces as desired. Pin **14** through cylindrical openings (opening **25** in the front plate and opening **26** in the back plate) near one edge of the plates ensures rotational positioning of the two plates.

FIGURE 2 shows a reduced view of the plan of the front plate. Appearing in this view are four plateaus, each concentric with an extrusion orifice and uniformly spaced about a circle inside the outer gasket. As shown in this view and in FIGURE 1, each orifice consists of capillary **21** at the exit end and larger counterbore **22** extending to the capillary from the plateau. Also visible, supported in a shallow annular groove, is gasket **6**, the opposing face of the back plate being similarly grooved to ensure a good seal between the two plates. FIGURE 3 shows a reduced view of the back plate sectioned as indicated on FIGURE 1. Visible are the concentric outer and inner walls, the capillaries and counterbores of four apertures spaced uniformly on a circle between the two walls, and four openings located within the central chamber defined by the inner wall. As shown in this view, the apertures in the top or back plate opposite the orifices of the bottom plate are each composed of four terminal capillaries **32** and introductory counterbore or lead hole **31**.

FIGURES 4, 5, 6, 7, 9, and 10 represent transverse cross-sectional views of representative composite filaments of the present invention before separation into their sharp-edged component filaments.

FIGURE 8 is a transverse cross-sectional view of one of the sharp-edged component filaments after separation from the composite filament.

Operation of the described apparatus in the practice of this invention is readily understood. Different polymer compositions are supplied to the inner and the outer chambers, respectively, of the back plate; the former flows through the openings into the relatively unconstricted space between back and front plates, through the relatively constricted regions between the plateaus and the opposing plate face, and through the extrusion orifices while the latter passes first through the apertures in the back plate and directly onto the top of the plateau and then through the aligned orifices in the front plate.

Suitable pairs of components for use in this invention can be found in all groups of synthetic fiber-forming materials. Where it is desired to separate the filament into its component sections by mechanical action, the components should have low adhesion to each other. Ob-

usly this is not necessary where one component of the r is to be removed by dissolution or chemical decom- ition. Because of their commercial availability, ease processing and excellent properties, the condensation ymers and copolymers, e.g., polyamides, polysul- amides and polyesters and particularly those that can readily melt spun are preferred for application in s method. Suitable polymers can be found for instance ong the fiber-forming polyamides and the polyesters ich are described in such patents as U.S. Patents 071,250, 2,071,253, 2,130,523, 2,130,948, 2,190,770 and 465,319. The preferred group of polyamides comprises ly(hexamethylene - adipamide), poly(hexamethylene bacamide), poly(epsilon-caproamide) and the co- lymers thereof. Suitable polyesters, besides poly(ethyl- e terephthalate), are the corresponding copolymers ntaining sebacic acid, adipic acid, isophthalic acid as ell as the polyesters containing recurring units derived om glycols with more than two carbons in the chain, .g., diethylene glycol, butylene glycol, decamethylene lycol and trans-bis-1,4-(hydroxy methyl)-cyclohexane.

Other groups of polymers useful as components in ilaments of the present invention can be found among he polyurethanes, the polyureas, cellulose esters and ellulose ethers as well as among the polyvinyl com- ounds including such polymers as polyethylene, poly- acrylonitrile, polyvinyl chloride, polyvinylidene chloride, polyvinyl alcohol, and copolymers containing the mono- mers of these polymers and similar polymers as disclosed in U.S. Patents 2,601,256, 2,527,300, 2,456,360 and 2,436,926.

When it is desired to remove all sections composed of one polymer composition by dissolution, a solvent for such polymer is selected that will not dissolve or have an adverse effect on sections composed of other polymer compositions. Thus in Example I which appears below, formic acid was used to remove the polyamide sections from the filament having both polyester and polyamide sections in its cross-section. The extent of dissolution of the soluble portion can be controlled as desired.

Similarly, all sections composed of one polymer composition can be removed by chemical decomposition. Thus polyester sections of a polysegmented filament having alternate polyester and polyamide sections, would be degraded by treatment with hot caustic as would the copolyamide or polyurea portions of polysegmented filaments having copolyamide-polyacrylonitrile or polyurea-polyacrylonitrile alternating sections by treatment with mineral acids.

In the examples, the relative viscosity (η_r) i.e., viscosity of a solution of polymer relative to that of the solvent is used as a measure of the molecular weight. The polyamide solutions contained 5.5 g. of polymer in 50 ml. of 90% formic acid and the viscosity was measured at 25° C. The polyester solutions contained 2.15 g. of the polymer in 20 ml. of 7/10 mixture of tetrachlorophenyl/phenol and the viscosity was measured at 30° C.

Example I

A spinneret similar to that shown in FIGURES 1 to 3 with 17 orifices was constructed. The plateau **4** was ⅛ in. in diameter and 1/16 in. high. The counterbore **22** was 40 mils in diameter and extended to within 48 mils of the face of the spinneret. The capillary **21** had a diameter of 12 mils. The lead hole **31** in the upper plate **7** was 3/32 in. in diameter was drilled to within 94 mils of the bottom of plate **7**. The upper orifices **32** were 9 mils in diameter and were drilled on a circle having a 39.5 mil radius the center of which was concentric with the upper lead hole and with the plateau in the orifice in the lower plate. The spinneret was assembled with a 3 mil thick shim **16**.

Poly(hexamethylene adipamide) of η_r 36 was fed to chamber **9** of the spinneret and extruded to form the triangular segments of the filament and poly(ethylene terephthalate) of η_r 33 containing 0.3% of TiO_2 was fed to annulus **8** and then through orifices **32** to form the formee cross segment of the filament's cross-section. The two molten polymers were extruded in the ratio of 9.5/10.0 by volume respectively at 290° C. and the yarn wound up at 1000 y.p.m. The yarn was drawn 4× (that is, to 400% of its original length) over an 88° C. pin and then passed over a 140° C. plate to reduce shrinkage. A cross-section of a typical filament is shown in FIGURE 7. The yarn had a tenacity of 3.9 g.p.d., an Mi (initial modulus, grams per denier) of 53, an ultimate elongation of 32% and a total denier of 50.

The yarn was knitted into a tubing which was quite lean in appearance and had poor visual covering power. The tubing was treated for 3 hours with 98% formic acid in a Soxhlet extractor, removed, rinsed with water and dried. Despite the loss of about 50% of the fiber weight by dissolution of the polyamide sections the visual covering power of the tubing was greatly increased. The extracted tubing had a soft silk-like handle and was scroopy. The cross-section of filaments remaining in the extracted fabric resembled a formee cross as shown in **108** of FIGURE 7.

Example II

Using the same spinneret as in Example I, poly(ethylene terephthalate) of η_r 26.9 containing 0.3% of TiO_2 was fed to chamber **8** of the spinneret and extruded as the segments of a composite filament designated **101** in FIGURE 4 while poly(hexamethylene adipamide) of η_r 36 was fed to chamber **9** and extruded as the segments of a composite filament designated **102** in FIGURE 4. The polymers were extruded at 290° C. and the yarn wound up at 400 y.p.m. The yarn was drawn 4.3× over a 98° C. pin. The resulting yarn had a tenacity of 4.1 grams per denier, and initial modulus of 56 and had a denier per filament of 8.3. A portion of the drawn yarn was wound on a perforated metal bobbin and immersed in cold 98% formic acid for 3 hours. After rinsing and drying the residual polyester yarn had a tenacity of 3.8 g.p.d., a Mi of 73, an ultimate elongation of 28% and a total denier of 80 for the 68 filaments then present. A typical cross-section of a filament is shown in **109** of FIGURE 8.

A portion of the original yarn was woven into a 2 x 2 twill fabric having 120 yarns per inch in the warp and 84 yarns per inch in the filling. The resulting fabric was immersed in 98% formic acid for 60 minutes until the poly(hexamethylene adipamide) sectors were dissolved from the composite filaments. The fabric possessed all of the properties of a silk fabric as liveliness and drape, the subtle scroop of silk, the handle, the low denier per filament, the high modulus and good recovery properties.

A repetition of the above spin with positions of the 2 polymers changed gives filaments which after treatment with formic acid leaves fillet-shaped sectors of the polyester similar to segment **102** of FIGURE 4.

Example III

The following example illustrates the different cross-sections obtained by varying the volume of polymers delivered to various sectors of the composite filaments Using the spinneret of Example I with a three mil shim, poly(ethylene terephthalate) of η_r 28.1 containing 2.0% of TiO_2 was fed to chamber **8** of the spinneret and poly-(ethylene terephthalate) of η_r 31 fed to chamber **9**. The polymers were extruded at 290° C. and the yarn wound up at 1000 y.p.m. The volume of the two polymers entering the composite filaments were varied by adjustment of their respective constant displacement pumps. In the first spin the volumes of the pigmented polymer to the non-pigment polymer was 1:1 and filaments were obtained having cross-sections similar to that shown in FIGURE 4. When the ratio of pigmented to bright polymer

was set at 4/16, filaments with cross-sections similar to FIGURE 5 were obtained. When the pumps were adjusted to give a ratio of 16/4. as above cross-sections similar to FIGURE 6 were obtained.

The above spins are repeated replacing the unpigmented polyester with the copolyester poly[ethylene/poly(ethylene oxide) glycol terephthalate] with a composition of 80/20 by weight, the poly(ethylene oxide) glycol units having a molecular weight of 6000. Upon immersing the yarns in a hot 5% solution of NaOH the segments corresponding to **102, 104, 106** in FIGURES 4, 5 and 6 are dissolved and residual cross-sections of poly(ethylene terephthalate) corresponding to **101, 103, 105** in FIGURES 4, 5, and 6 remain in the respective yarns.

Example IV

Using the apparatus and polymers of Example I the positions of the two polymers were reversed. The polyester and polyamide were extruded at 290° C. at a ratio of 12/16 by volume respectively and the composite filaments wound up at 500 y.p.m. The yarn was drawn 4.2× over a 100° C. pin. A typical cross-section of the drawn filament is shown in FIGURE 9. A portion of a yarn was wrapped on a perforated metal tube and immersed in acetone for 5 minutes. The dried yarn was pulled over the edge of a glass microscope slide under a tension of about 0.5 g.p.d. so that the yarn suffered a 90° change of direction in the process. The above process caused the filament to partially fragment longitudinally along the interfaces of the sectors. A total of three passages of the yarn over the sharp edge caused complete separation of the polyester and polyamide segments so that the yarn was composed of filaments which in cross-section resembled **110** and **111** of FIGURE 9.

The filaments in the above drawn yarn were completely separated into the polymeric components by one passage through an air jet as described in United States Pat. No. 2,783,609 at a feed rate of 50 y.p.m. and a windup rate of 48 y.p.m. using 2 cu. ft. of air per minute at 90 p.s.i. to operate the jet.

A portion of the above drawn yarn not exposed to acetone or fragmented was wound on a perforated metal tube and placed in 98% formic acid at the boiling point for 30 minutes. The tube and yarn was then placed in cold formic acid for an additional 15 minutes, rinsed with water and dried. The residual polyester filaments, which in cross-section resembled segment **110** of FIGURE 9, had a tenacity of 3.6 g.p.d., an ultimate elongation of 31%, a Mi of 68, and a denier per filament of 1.0. The yarn was used as a filling face in the weaving of a satin with yarn of round cross-section, poly(ethylene terephthalate), as a warp. The fabric had a dry, crisp, silk-like handle across the filling band but was less silk-like than the fabric of Example II.

Example V

Using the apparatus and polymers as in Example IV, composite filaments are extruded and the continuous filaments wound up at 5000 y.p.m. The yarn is dipped in acetone and then drawn 2× over a pin at 88° C. The segments of polyester break and split during the drawing so that a yarn somewhat resembling spun staple is obtained in which the cruciform-like filaments of polyamide in cross-section have short lengths of polyester microfibers attached to a surface which project beyond the original periphery of the filaments.

Example VI

Solutions of polyacrylonitrile and cellulose acetate both in dimethylformamide are dry spun from the spinneret of Example I. The resulting filaments in cross-section have alternate segments of the two polymers with a crenulated periphery. An acetate bath dissolves the cellulose

acetate portions of the filaments and leaves small d filaments of polyacrylonitrile of shape similar to URE 8.

The composite filaments have been produced in examples by the melt spinning technique. Obvio other spinning methods like "plasticized melt" spin dry spinning, wet spinning, can be employed successf In some instances, particularly when the melting beha or the solubility of the components in a combina would not permit spinning the components by sim methods, a combination of dissimilar methods can used. Thus, for instance, one component, can be spun a solution in a high boiling solvent or as a plasticized m while the other component is extruded as a mol polymer. In these instances, the solvents or plasticiz may be wholly or partially removed subsequently, pr erably by washing them out by the help of low boili solvents.

The composite filaments illustrated in this inventi have substantially round cross-sections before separati of the components. However, it will be apparent to tho in the art that by altering the shape of the orifice **21**, t final cross-section can be controlled to a certain exte Although square filaments cannot be extruded, filamen in cross-section which resemble a square with rounde corners can be obtained by the use of square or slotte orifices and these in turn would offer segments that ar plane triangles or a combination of plane and curvilinea triangles for example. Similarly, cross-sections in th shape of ellipses, cruciforms, etc. can be extruded an segments placed in such filaments as desired.

It will also be obvious to those skilled in the art tha other modifications of the composite filaments and henc of the shape of the residual filaments after dissolutio can be altered by changing the number and placemen of the upper orifices **32**. Other means of altering the configuration of the composite filaments will be by varying the diameters of the upper orifices used in relation to the size of the plateaus, and/or the rate at which polymers are extruded through the upper orifices **32** and over the plateau. Alteration of the viscosities of the component polymers affects the configuration obtained. A low viscosity polymer tends to be pushed inward more readily by the flow of a more viscous polymer and hesce alters the shape of the segment that it will make. The configuration in the component filaments is also affected by the interfacial tension and the individual tendencies of the polymers to wet the spinneret surfaces.

Although the spinneret used in the examples is a convenient apparatus for the preparation of the filaments of this invention it will be obvious to those skilled in the art that other spinnerets can be used. Other spinnerets permit the production of filaments or ribbons having alternating segments **112** and **113** as shown in FIGURE 10 which can be split or dissolved apart to give sharp-edged filaments.

The process of this invention affords a convenient means of obtaining filaments having one or more sharp points in cross-section and of a lower denier than can be otherwise attained. Thus, the invention permits the production of sharp-edged filaments having a denier of 0.1 to 10 or larger. Its greatest utility, however, is in the range of 0.1 to 5 denier per filament. The novel filaments can be used to obtain all manner of new and novel effects in fabric handle, scroop, appearance and covering power by proper selection of the polymer composition and filamentary cross-section.

The invention is to be limited only by the scope of the following claims.

I claim:

1. A method of making sharp-edged filaments which comprises spinning a composite filament of substantially uniform cross-section composed of segments of at least two dissimilar synthetic polymeric compositions, said cross-section having at least two segments of at least one

aid polymers, any contact between such segments of same polymer being substantially point contact and rating a sharp-edged filament therefrom.

. The process of claim 1 wherein said separation is mplished by physical means.

. The process of claim 1 wherein said separation is mplished by dissolution of all segments composed ne polymeric composition.

. The process of claim 1 wherein said separation is accomplished by chemical degradation of all segments composed of one polymer composition.

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