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(54) CONTINUOUS PROCESSES FOR MAKING COMPOSITE FIBERS

STETIGES VERFAHREN ZUR HERSTELLUNG VON VERBUNDFASERN

PROCEDES CONTINUS DE FABRICATION DE FIBRES COMPOSITES

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Description**BACKGROUND OF THE INVENTION****Field of the Invention**

[0001] This invention relates to continuous processes for making composite fibers. In particular, the invention relates to continuous processes for making composite fibers from aramid polymers.

Description of Related Art

[0002] U.S. Patent 5,135,687 discloses a batch process for making a poly(vinyl pyrrolidone) [PVP]/p-phenylene terephthalamide [PPD-T] composite fiber. The process includes agitating an anisotropic mixture of acid solvent, aramid, and PVP; heating the agitated mixture to above its melting point; extruding the mixture through a spinneret, into and through a non-coagulating layer; and passing the extruded mixture into and through an aqueous coagulating bath. In an exemplified embodiment, the mixture is heated with stirring for two hours at 85°C.

[0003] WO 96/30569 A1 relates to the field of making synthetic yarns, in particular to synthetic yarns spun from sulphuric acid anisotropic solutions of rigid chain aromatic polyamides blended with aliphatic polyamides.

[0004] Composite fibers made using aramids in conventional processes have desirable properties including tenacity, dyeability, thermal resistance and adhesion to elastomeric matrices. However, new and/or improved continuous processes for making composite fibers, particularly composite fibers made from aramid polymers, are desirable.

[0005] The present invention is directed to these and other important ends.

SUMMARY OF THE INVENTION

[0006] One aspect of the present invention is a process for making a composite fiber. The process includes: continuously combining an anisotropic polymer solution of an aramid polymer and an isotropic solution of a second polymer as defined claim 1 to form a combined polymer solution; passing the combined polymer solution through at least one static mixer to form a spin dope; and extruding the spin dope through a spinneret to form a composite fiber.

[0007] In some embodiments, the process further includes: passing the composite fiber through an air gap; contacting the composite dope fiber with a quench solution to form a coagulated composite fiber; contacting the coagulated composite fiber with a wash solution; contacting the washed composite fiber with a neutralization solution to form a neutralized and washed composite fiber; drying the neutralized and washed composite fiber; and winding up the dried composite fiber. The dried composite fiber can be wound onto a bobbin on a windup device.

BRIEF DESCRIPTION OF THE DRAWING(S)

[0008] The invention can be more fully understood from the following detailed description thereof in connection with accompanying drawings described as follows.

[0009] Figure 1 is a schematic illustration of one embodiment of a process for making composite yarn in accordance with the present invention.

DETAILED DESCRIPTION

[0010] The present invention provides composite fibers of aramids and processes for the preparation of such fibers. The invention further relates to yarns, cords, fabrics, and articles incorporating the fibers, and processes for making such yarns, cords, fabrics, and articles.

[0011] Herein, the term "fiber" is used herein interchangeably with "filament", and means a relatively flexible, macroscopically homogeneous body having a high ratio of length to width across its cross-sectional area perpendicular to its length. The fiber cross section can be any shape, but is often somewhat circular. Fiber spun onto a bobbin in a package is referred to as continuous fiber. Fiber can be cut into short lengths called staple fiber. Fiber can be cut into even smaller lengths called floc. Multifilament yarns can be combined to form cords. Yarn can be intertwined and/or twisted.

[0012] The term spin as used herein refers to the extrusion of a polymer solution through a spinneret.

[0013] The processes disclosed herein utilize anisotropic solutions of polymers. "Anisotropic", as used herein, means having microscopic regions that are birefringent; that is, a bulk sample of an anisotropic solution depolarizes plane polarized light because the light transmission properties of the microscopic regions of the solution (or polymer spin dope) vary with direction. Anisotropy in the polymer solutions and dopes used in the processes disclosed herein is due to the existence of at least part of the solution or dope in a liquid crystalline state.

[0014] By "aramid" is meant a polyamide wherein at least 85% of the amide (-CO-NH-) linkages are attached directly to two aromatic rings. Suitable aramid fibers are described in Man-Made Fibers-Science and Technology, Volume 2, Section titled Fiber-Forming Aromatic Polyamides, page 297, W. Black et al., Interscience Publishers, 1968. Aramid fibers are, also, disclosed in U.S. Patent Nos. 4,172,938; 3,869,429; 3,819,587; 3,673,143; 3,354,127; and 3,094,511.

[0015] Para-aramids are the primary polymers in aramid yarn fibers of this invention and poly(p-phenylene terephthalamide)(PPD-T) is the preferred para-aramid. By PPD-T is meant the homopolymer resulting from mole-for-mole polymerization of p-phenylene diamine and terephthaloyl chloride and, also, copolymers resulting from incorporation of small amounts of other diamines with the p-phenylene diamine and of small amounts of other diacid chlorides with the terephthaloyl chloride. As a general rule, other diamines and other diacid chlorides can be used in amounts up to as much as about 10 mole percent of the p-phenylene diamine or the terephthaloyl chloride, or perhaps slightly higher, provided only that the other diamines and diacid chlorides have no reactive groups which interfere with the polymerization reaction. PPD-T, also, means copolymers resulting from incorporation of other aromatic diamines and other aromatic diacid chlorides such as, for example, 2,6-naphthaloyl chloride or chloro- or dichloroterephthaloyl chloride or 3,4'-diaminodiphenylether. Preparation of PPD-T is described in U.S. Patent Nos. 3,869,429; 4,308,374; and 4,698,414.

[0016] The isotropic solution of flexible chain polymers comprises a polymer selected from poly(vinyl pyrrolidone) (PVP), poly(vinyl pyrrolidone)/vinyl acetate (PVP/VA), polyether ketone (PEKK), nylon 6, nylon 6,6, nylon 6,12, Poly(m-phenylene isophthalamide) (MPD-I), Poly(m-phenylene isophthalamide/terephthalamide) (MPD-I/T), and polyaniline. Preferred polymers are those that form isotropic solutions in mineral acids, including chlorosulfonic acid and fluorosulfonic acid, particularly sulfuric acid. A highly preferred polymer for use in the isotropic solution is PVP.

[0017] While it is not intended that the present invention be bound by any particular theory, it is believed that the formation, during mixing, of droplets of sufficiently small size, e.g., having average diameters of 10 percent or less the magnitude of the diameter desired in a filament formed from the combined polymers, aids in the mixing of the anisotropic aramid solution and the isotropic polymer solution. Preferably, the droplet size formed during mixing is less than 5 percent of the desired filament diameter, more preferably less than 2 percent of the desired filament diameter.

[0018] It has been found that when two polymers, such as PPD-T and PVP, are combined with acid in a continuous process to form a spin dope, the spin dope may not be able to be spun with satisfactory continuity and yarn quality may not be adequate. It is believed that this is due to phase separation of isotropic and anisotropic phases that can occur during a long hold-up time in transfer lines used in the process to transport the solution of combined polymers. In contrast, using the processes disclosed herein, a composite fiber can be prepared from PPD-T and a flexible chain polymer.

[0019] To form droplets of adequately small size, a static mixer is used. A static mixer comprises a pipe having therein an arrangement of elements (plates) at varying angles with respect to the wall of the pipe. The plates provide shear and mixing as a material flows through the pipe. The number of plates can vary. An example of a suitable static mixer is a Sulzer type SMX, manufactured by Sulzer Chemtech, Tulsa, OK. Conditions and parameters for mixing can be determined by one skilled in the art. For purposes of example only, the following is provided.

[0020] For miscible systems it is generally desirable to mix to a certain degree of homogeneity in the radial direction. Homogeneity can be measured by a compositional uniformity coefficient of variation (CoV) with values between 5% and 0.1 %. For simple blending 5% may be enough but for critical additions like colorants 0.1% is usually a better criterion. Lower CoVs are obtained by increasing mixer length.

[0021] An anisotropic solution of an aramid polymer and an isotropic solution, of a flexible chain polymer are not fully miscible and form a microscopically inhomogeneous system comprising small droplets that are generally not visible to the naked eye. For such systems a measurable parameter is drop size, also called particle size. The drop size can be set by a stress criterion and a strength parameter characteristic of the two polymer solutions, such as interfacial tension. An interfacial tension of about (10^{-5} N/cm) 10 dynes/cm is typical of organics in water, and can be used as a guideline or starting point. For example, the static mixer can be designed to produce dispersed droplets of the isotropic solution within the anisotropic solution with an average diameter that can be as small as about 1/60 of the diameter of the composite fiber or smaller. It is generally desirable to minimize drop size. An equilibrium drop size is preferably obtained by the time the combined polymers have passed through a length in the mixer that is less than 10 mixer diameters.

[0022] As will be recognized by those skilled in the art, the optimal dimensions of a static mixer can vary, depending upon the polymer solutions to be mixed, and in particular, on the viscosity and interfacial tension of the solutions. For example, for a pipe size of 0.815 inch (2.07 cm) inner diameter (ID) containing 18 mixer elements, a droplet size of about 0.45-1.3 microns has been estimated with a pressure drop of 407-141 psi (28.6-9.9 kg/cm²). With a second close-coupled mixer having a pipe ID of 0.612 inch (1.55 cm), containing 10 mixer elements, the droplet size is further reduced to an estimated diameter of 0.18-0.55 micron with a pressure drop of 594-205 psi (41.8-14.4 kg/cm²). As a general guideline, the pressure drop increases as the number of plates in the mixer increases, and as the diameter of the pipe decreases. One skilled in the art can adjust the diameter and/or number of plates to result in a desired droplet size at a given pressure drop.

[0023] Throughout this detailed description, similar reference characters refer to similar elements in all figures of the drawing. While a preferred embodiment is described hereinbelow with reference to Figure 1, it is not intended that the

invention be limited in scope to the embodiment shown in the figure. A person skilled in the art, armed with the present disclosure and the exemplary embodiments disclosed herein can select and configure appropriate equipment to carry out the present invention.

[0024] Referring to Figure 1, an aramid polymer 2 is dissolved in an acid solvent, such as sulfuric acid 4, to form an anisotropic solution of aramid polymer. The acid solvent is preferably concentrated, e.g., 98-101 %, typically 98-100.2%, sulfuric acid. Other acids that can be used include chlorosulfonic acid and fluorosulfonic acid. The anisotropic solution of aramid polymer preferably contains a high enough concentration of aramid polymer to form an acceptable filament after extrusion and coagulation. A practical minimum concentration of aramid polymer is that concentration that provides anisotropy in the solution. For example, a concentration of at least 18 weight percent can be used. Yarn properties such as tenacity and modulus are affected in part by the polymer concentration of the anisotropic solution of aramid polymer, and the concentration can be adjusted to enhance desired properties. Generally, lowering the concentration below 18 weight percent can cause a decrease in such properties.

[0025] The polymer and acid for the anisotropic solution are mixed in a mixer 6 such as a twin-screw mixer, while being heated to an elevated temperature, e.g., 80 to 100°C for p-phenylene terephthalamide (PPD-T), to provide a solution 58 of the polymer in acid. Using jacketed transfer lines throughout the system to maintain the solution 58 at a temperature above the crystal solvate melting temperature of the polymer, the polymer solution is then transferred via transfer pump 8, to vessel 10, where it is deaerated under reduced pressure. Deaeration can be facilitated, e.g., by agitation. The solution 58 is then transferred via pump 12 through filter 14.

[0026] Separately, an isotropic solution 60 is prepared by dissolving a flexible-chain polymer in acid, preferably the same acid as used in making the anisotropic solution. In some embodiments, e.g., for PVP solution, the isotropic solution preferably has a viscosity less than that of the anisotropic solution. Preferably, the acid is first cooled to a frozen slurry before the flexible chain polymer is added in powder form. Also preferably, the anisotropic solution and the isotropic solution are maintained at about the same temperature after they are made and before they are combined. For example, for an anisotropic solution of poly p-phenyleneterephthalamide combined with an isotropic solution of PVP, the solutions are preferably maintained at a temperature of about 80°C prior to mixing.

[0027] The isotropic solution 60 is prepared in mixer 18, then transferred using jacketed transfer lines via transfer pump 20 through filter 22 and into optional hold tank 24 and through stuffing pump 26 to injection pump 28 and injection valve 16. At injection valve 16, the isotropic solution 60 is injected into the anisotropic solution 58 to form a combined polymer solution 62. The combined polymer solution 62 is biphasic, due to immiscibility of the anisotropic solution of aramid polymer and the isotropic solution of flexible chain polymer. The combined polymer solution 62 is passed through at least one static mixer 30 and optional additional static mixer(s) 32 to form a mixed, combined polymer solution 64. In preferred embodiments, two mixers are used. Also preferably, the mixers are placed as to minimize the residence time (time between the initial combining of the solutions and extrusion from the spinneret) of the combined solutions. Typically, the residence time after mixing is less than 20 minutes, preferably less than 15 minutes, more preferably less than 10 minutes. More than two static mixers can be used, for example, to form smaller size droplets and/or to redisperse any agglomerated droplets. The mixed, combined polymer solution is then pumped via meter pump 34 to optional heat exchanger 36, to help control the temperature of the combined polymer solution.

[0028] The concentration of aramid polymer in the mixed, combined polymer solution 64 is preferably high enough to provide a liquid-crystalline dope, preferably at least about 15 weight percent, more preferably at least 16 weight percent. The maximum total concentration of polymers in the dope is limited primarily by practical factors, such as polymer solubility and dope viscosity. The concentration of total solids in the dope is preferably no more than 22 weight percent, and more preferably no more than 20 weight percent.

[0029] Each polymer solution and/or the combined stream can contain additives such as anti-oxidants, lubricants, ultra-violet screening agents, colorants and the like which are commonly incorporated.

[0030] The mixed, combined polymer solution 64 is extruded through spinneret 38 to form filament(s) 42. The spinneret 38 preferably contains a plurality of holes. The number of holes in the spinneret and their arrangement is not critical to the invention, but it is desirable to maximize the number of holes for economic reasons. The spinneret 38 can contain as many as 100 or 1000 or more, and they may be arranged in circles, grids, or in any other desired arrangement. The spinneret 38 can be constructed out of any materials that are resistant to the acids used in the process. Materials suitable for use in the spinnerets of the present process are disclosed, for example, in US Patents 4,137,032 and 4,054,468.

[0031] Filaments 42 exiting the spinneret 38 enter gap 40 between the spinneret 38 and a coagulation bath (quench bath) 44 containing water, where the acid solvent is removed. The gap 40 is typically called an "air gap" although it need not contain air. The gap 40 can contain any fluid that does not induce coagulation or react adversely with the stream, such as air, nitrogen, argon, helium or carbon dioxide.

[0032] Each filament 42 is drawn across the air gap 40, with stretching. The denier of the resulting filament is controlled in part by the "spin stretch factor". The spin stretch factor as used herein is the ratio of the velocity of a fiber as it exits the coagulating bath to the jet velocity. Jet velocity is the average velocity of the spinning dope exiting the spinneret as calculated from the volume of material passing through an orifice per unit time and the cross-sectional area of the orifice.

The minimum spin stretch factor for use with a particular composition and orifice is that which allows the formation of a filament of relatively uniform denier and having desired physical properties. The practical upper limit of the spin stretch factor is preferably that at which breakage is eliminated. As a general guideline, at a given jet velocity, increasing the spin stretch factor provides fibers with higher tenacities and moduli, lower elongations, and smaller denier. A person skilled in the art can readily determine an appropriate orifice diameter, jet velocity and spin stretch factor for a particular spinning dope, apparatus and application, to obtain a fiber of the desired denier and physical properties.

[0033] The filament then is passed via feed rolls 46 to wash station 48 and neutralization station 50. Then the filament is dried in dryer 52 to remove water. The temperature of the filament as it exits the dryer is typically 120°C to 220°C. The dryer residence time of the filament is typically several seconds. Finish is applied at finish applicator 54. The filament can be wound up onto a bobbin at windup device 56.

[0034] The coagulated filament is washed in one or more wash steps to remove acid solvent from the filament. The washing and neutralization of the filament is preferably carried out in a continuous process by running the filament through a series of baths and/or through one or more washing cabinets. Washing cabinets typically comprise an enclosed cabinet containing one or more rolls which the filament travels around a number of times, and across, prior to exiting the cabinet. As the filament travels around the roll, it is sprayed with a washing fluid. The washing fluid is continuously collected in the bottom of the cabinet and drained therefrom.

[0035] The temperature of the washing fluid(s) is preferably at least 5°C, and preferably no greater than 65°C, more preferably no greater than 50°C. In a continuous process, the duration of the entire washing process which includes the time in the coagulation bath and in the washing bath(s) and/or cabinet(s) is sufficient to remove acid solvent from the spun filaments.

[0036] The above embodiment is one preferred embodiment of a process according to the present invention. Other variations in the process can be determined by one skilled in the art and one or more steps can be adapted to accommodate process conditions and/or properties of the polymer(s) used. Extrusion processes suitable for use in making composite fibers within the scope of the present invention are disclosed in U.S. Patents 4,340,559, 4,298,565 and 4,965,033.

INDUSTRIAL APPLICABILITY

[0037] Fibers made according to the processes disclosed herein can be used in any applications where fibers are used. For example, composite fibers containing aramids made according to the processes disclosed herein can be used in yarn, cord (twisted yarn) or fabric form to provide reinforcement for a variety of matrices, including elastomers. The composite fibers, and yarns, cords and fabrics made therefrom, can be used in the manufacture of shaped articles, such as, for example, tires, belts and hoses.

TEST METHODS

[0038] The following test methods were used in the following Examples.

[0039] Temperature: All temperatures are measured in degrees Celsius (°C).

Denier

[0040] Denier is determined according to ASTM D 1577 and is the linear density of a yarn as expressed as weight in grams of 9000 meters of yarn. Denier times (10/9) is equal to decitex (dtex).

Tensile Properties

[0041] The fibers to be tested are conditioned and then tested based on the procedures described in ASTM D885-98. Tenacity (breaking tenacity), elongation to break, and modulus of elasticity are determined by breaking test yarns on an Instron tester. Tenacity is reported as breaking stress divided by linear density. Modulus is reported as the slope of the initial stress/strain curve converted to the same units as tenacity. Elongation is the percent increase in length at break. Both tenacity and modulus are first computed in g/denier units which, when multiplied by 0.8838, yield dN/tex units. Each reported measurement is the average of six breaks.

[0042] Tensile properties for yarns are measured at 24° C and 55% relative humidity after conditioning under the test conditions for a minimum of 14 hours. Before testing, each yarn is twisted to a 1.1 twist multiplier. Each twisted specimen has a test length of 50 cm and is elongated at a rate of 50%/minute in an Instron tester. The twist multiplier [TM] is computed from

$$TM = [\text{denier}]^{1/2} * [\text{tpc}]/30.3$$

5 where tpc=turns per cm

Heat Aged Strength Retention (HASR)

10 **[0043]** HASR is a test to determine how much of its initial strength a yarn retains after heat aging. HASR is reported in percent of the breaking strength retained after exposure to controlled heat.

[0044] To conduct the test, a fresh yarn sample is conditioned at 24°C and 55% relative humidity for 14 hours. A portion of the sample is subjected to dry heat at a temperature of 240°C for 3 hours and is then tested for tensile strength (Heat aged tenacity). A portion of the sample that has not been heated is also tested for tensile strength (As is tenacity).

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$$HASR = 100 \times [\text{Heat aged tenacity}]/[\text{As is tenacity}]$$

EXAMPLES

20 **[0045]** The following examples are given to illustrate the invention and should not be interpreted as limiting it in any way. All parts and percentages are by weight unless otherwise indicated. Examples prepared according to the process or processes of the current invention are indicated by numerical values. Control or Comparative Examples are indicated by letters.

25 **[0046]** An isotropic, 20 wt % solids solution of polyvinylpyrrolidone (PVP) in 100.1% sulfuric acid was injected, by means of a diaphragm pump, into an anisotropic, 19.5 wt % solids solution of poly(p-phenyleneterephthalamide) in 100.1 % sulfuric acid at a volumetric solution ratio (isotropic/anisotropic) of 'x', listed in Table 1. The resulting mixture was passed through a series of motionless Sulzer SMX mixers to intimately mix the two solutions. The first mixer had a pipe ID of 0.815 inch (2.07 cm) and was fitted with eighteen (18) removable mixing elements. The second mixer had an ID of 0.612 inch (1.55 cm) and was fitted with ten (10) mixing elements. Within about seven (7) minutes of passing through the second mixer, the resulting solution was extruded through a 667-hole spinneret having orifice diameters of 0.063 mm and spun using a conventional air gap-wet spinning process. The extruded solution was drawn through a 0.8 cm length air gap into an aqueous coagulating bath at 5 degrees C containing about 5% sulfuric acid. The resulting fiber was washed, neutralized and dried.

30 **[0047]** Fibers of varying PVP content were produced depending on the volumetric ratio (isotropic/anisotropic) 'x' of the two polymer solutions. Yarn tensile properties are shown for several compositions in Table 1 derived from isotropic and anisotropic polymer solutions with solids concentrations of 20% and 19.5%, respectively.

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TABLE 1								
EX	Ratio 'x'	PVP Wt %	dTex	Ten dN/tex	E, %	Modulus dN/tex	HASR %	
40 1	0.152	13	1090	19.6	3.5	516	92.7	
2	0.113	10	1085	19.3	3.6	525	92.9	
3	0.077	7	1097	20.8	3.5	566	84.4	
A	0	0	1104	23.2	3.4	626	73.7	

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[0048] Those skilled in the art, having the benefit of the teachings of the present invention as hereinabove set forth, can effect numerous modifications thereto. These modifications are to be construed as being encompassed within the scope of the present invention as set forth in the appended claims.

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Claims

1. A process for making a composite fiber, comprising continuously combining an anisotropic solution of an aramid polymer and an isotropic solution of a flexible chain polymer to form a combined polymer solution; mixing the combined polymer solution by passing the combined polymer solution through at least one static mixer to form a mixed combined polymer solution; and forming a composite fiber from the mixed combined polymer solution.

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2. The process of claim 1, wherein the anisotropic solution of an aramid polymer comprises para-aramid.
3. The process of claim 2, wherein the anisotropic solution of an aramid polymer comprises greater than 18 weight percent para-aramid, based on the total weight of the anisotropic solution.
- 5 4. The process of claim 1 or 3 wherein the anisotropic solution of an aramid polymer comprises, as a solvent, sulfuric acid. wherein the isotropic solution of a flexible chain polymer comprises a polymer selected from: poly(vinyl pyrrolidone) (PVP), poly(vinyl pyrrolidone)/vinyl acetate (PVP/VA), polyether ketone ketone (PEKK), nylon 6, nylon 6,6, nylon 6,12, Poly(*m*-phenylene isophthalamide) (MPD-I), Poly(*m*-phenylene isophthalamide/terephthalamide) (MPD- I/T), and polyaniline.
- 10 5. The process of claim 1 wherein the isotropic solution of a flexible chain polymer comprises PVP.
- 15 6. The process of claim 1 wherein the isotropic solution of a flexible chain polymer comprises 5 to 25 weight percent of polymer based on the total weight of the isotropic polymer solution.
7. The process of claim 1 wherein the solution comprises, as a solvent, an acid selected from H₂SO₄, ClSO₃H, and FSO₃H.
- 20 8. The process of claim 5 wherein the solution comprises, as a solvent, sulfuric acid.
9. The process of claim 8 wherein the solution is mixed to a degree sufficient to produce an average droplet size of 1.5 to 12 percent of the diameter of the composite fiber.
- 25 10. The process of claim 1 wherein the composite fiber comprises 80 to 99 weight percent aramid and 1 to 20 weight percent polymer from the isotropic solution of a flexible chain polymer based on the total weight of the composite fiber.

Patentansprüche

- 30 1. Verfahren zur Herstellung einer Verbundfaser, umfassend das kontinuierliche Kombinieren einer anisotropen Lösung eines Aramidpolymers und einer isotropen Lösung eines Polymers flexibler Kette, wobei die isotrope Lösung eines Polymers flexibler Kette ein Polymer umfasst ausgewählt unter: Poly(vinylpyrrolidon) (PVP), Poly(vinylpyrrolidon)/Vinylacetat (PVP/VA), Polyetherketonketon (PEKK), Nylon 6, Nylon 6,6, Nylon 6,12, Poly(*m*-phenylenisophthalamid) (MDP-I), Poly(*m*-phenylenisophthalamid/Terephthalamid) (MPD-I/T) und Polyanilin unter Bildung einer kombinierten Polymerlösung; das Mischen der kombinierten Polymerlösung durch Hindurchführen der kombinierten Polymerlösung durch mindestens einen statischen Mischer zur Bildung einer gemischten kombinierten Polymerlösung; und das Bilden einer Verbundfaser aus der gemischten kombinierten Polymerlösung.
- 35 2. Verfahren nach Anspruch 1, wobei die anisotrope Lösung eines Aramidpolymers Para-Aramid umfasst.
- 40 3. Verfahren nach Anspruch 2, wobei die anisotrope Lösung eines Aramidpolymers mehr als 18 Gewichtsprozent Para-Aramid, auf das Gesamtgewicht der anisotropen Lösung bezogen, umfasst.
- 45 4. Verfahren nach Anspruch 1 oder 3, wobei die anisotrope Lösung eines Aramidpolymers Schwefelsäure als Lösungsmittel umfasst.
5. Verfahren nach Anspruch 1, wobei die isotrope Lösung eines Polymers flexibler Kette PVP umfasst.
- 50 6. Verfahren nach Anspruch 1, wobei die isotrope Lösung eines Polymers flexibler Kette 5 bis 25 Gewichtsprozent Polymer, auf das Gesamtgewicht der isotropen Polymerlösung bezogen, umfasst.
7. Verfahren nach Anspruch 1, wobei die Lösung Säure als Lösungsmittel umfasst, die unter H₂SO₄, ClSO₃H und FSO₃H ausgewählt ist.
- 55 8. Verfahren nach Anspruch 5, wobei die Lösung Schwefelsäure als Lösungsmittel umfasst.
9. Verfahren nach Anspruch 8, wobei die Lösung bis zu einem Grad gemischt wird, der ausreicht, eine durchschnittliche

Tröpfchengröße von 1,5 bis 12 Prozent des Durchmessers der Verbundfaser herzustellen.

10. Verfahren nach Anspruch 1, wobei die Verbundfaser 80 bis 99 Gewichtsprozent Aramid und 1 bis 20 Gewichtsprozent Polymer aus der isotropen Lösung einer Polymers flexibler Kette, auf das Gesamtgewicht der Verbundfaser bezogen, umfasst.

Revendications

1. Procédé de fabrication d'une fibre composite, comprenant la combinaison de manière continue d'une solution anisotrope d'un polymère d'aramide et d'une solution isotrope d'un polymère à chaîne souple, la solution isotrope d'un polymère à chaîne souple comprenant un polymère choisi parmi la poly(vinyl pyrrolidone) (PVP), le poly(vinyl pyrrolidone)/acétate de vinyle (PVP/VA), la polyéther cétone cétone (PEKK), le nylon 6, le nylon 6,6, le nylon 6,12, le poly(isophthalamide de *m*-phénylène) (MPD-I), le poly(isophthalamide/téréphthalamide de *m*-phénylène) (MPD-I/T) et la polyaniline pour former une solution polymère combinée; le mélange de la solution polymère combinée en faisant passer la solution polymère combinée à travers au moins un mélangeur statique pour former une solution polymère combinée mixte; et la formation d'une fibre composite à partir de la solution polymère combinée mélangée.
2. Procédé selon la revendication 1, la solution anisotrope d'un polymère aramide comprenant du para-aramide.
3. Procédé selon la revendication 2, la solution anisotrope d'un polymère aramide comprenant plus de 18 pour cent en poids de para-aramide, sur la base du poids total de la solution anisotrope.
4. Procédé selon la revendication 1 ou 3, la solution anisotrope d'un polymère aramide comprenant, comme solvant, de l'acide sulfurique.
5. Procédé selon la revendication 1, la solution isotrope d'un polymère à chaîne souple comprenant de la PVP.
6. Procédé selon la revendication 1, la solution isotrope d'un polymère à chaîne souple comprenant 5 à 25 pour cent en poids du polymère sur la base du poids total de la solution polymère isotrope.
7. Procédé selon la revendication 1, la solution comprenant, comme solvant, un acide choisi parmi H_2SO_4 , $ClSO_3H$ et FSO_3H .
8. Procédé selon la revendication 5, la solution comprenant, comme solvant, de l'acide sulfurique.
9. Procédé selon la revendication 8, la solution étant mélangée à un degré suffisant pour produire une taille de gouttelette moyenne de 1,5 à 12 pour cent du diamètre de la fibre composite.
10. Procédé selon la revendication 1, la fibre composite comprenant 80 à 99 pour cent en poids d'aramide et 1 à 20 pour cent en poids de polymère provenant de la solution isotrope d'un polymère à chaîne souple sur la base du poids total de la fibre composite.

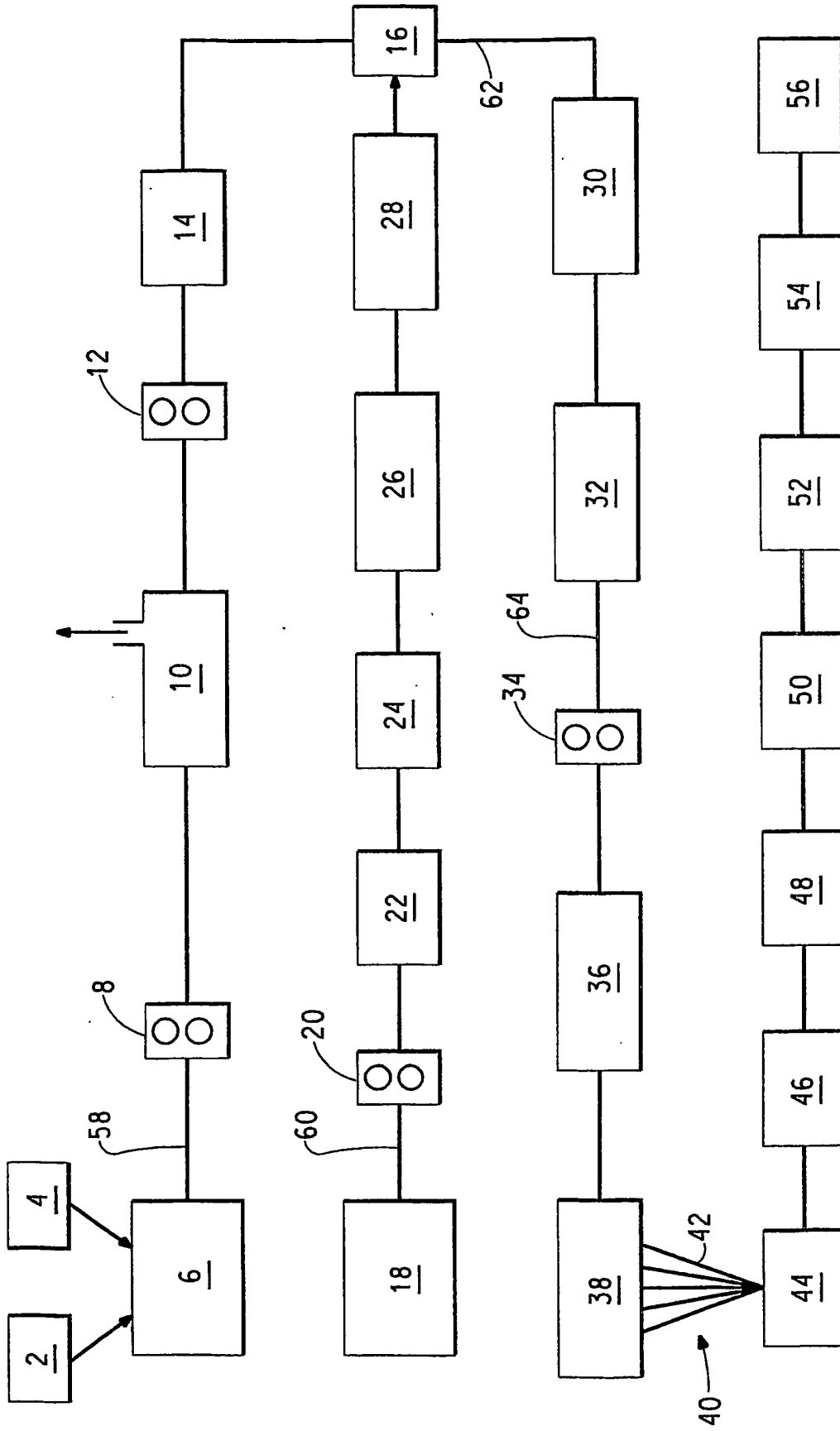


FIG. 1

REFERENCES CITED IN THE DESCRIPTION

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