

Sept. 25, 1945.

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2,385,358

METHOD OF MAKING FINE FIBERS

Filed Sept. 15, 1944

2 Sheets-Sheet 1

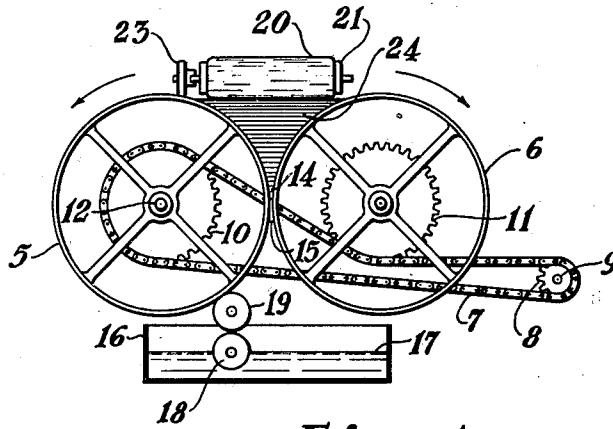


Fig. 1

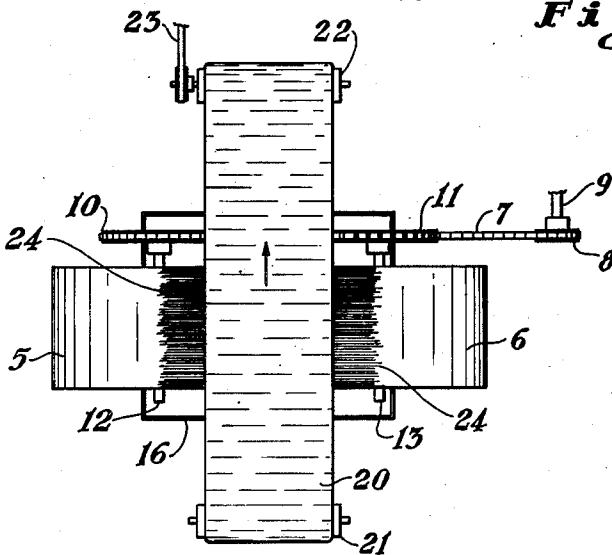


Fig. 2

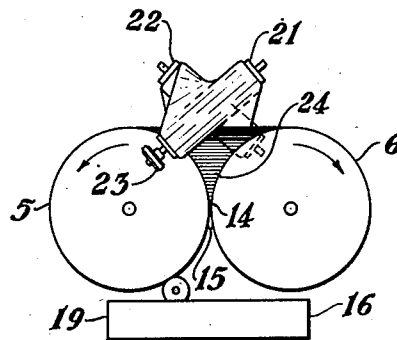


Fig. 3

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2 Sheets-Sheet 2

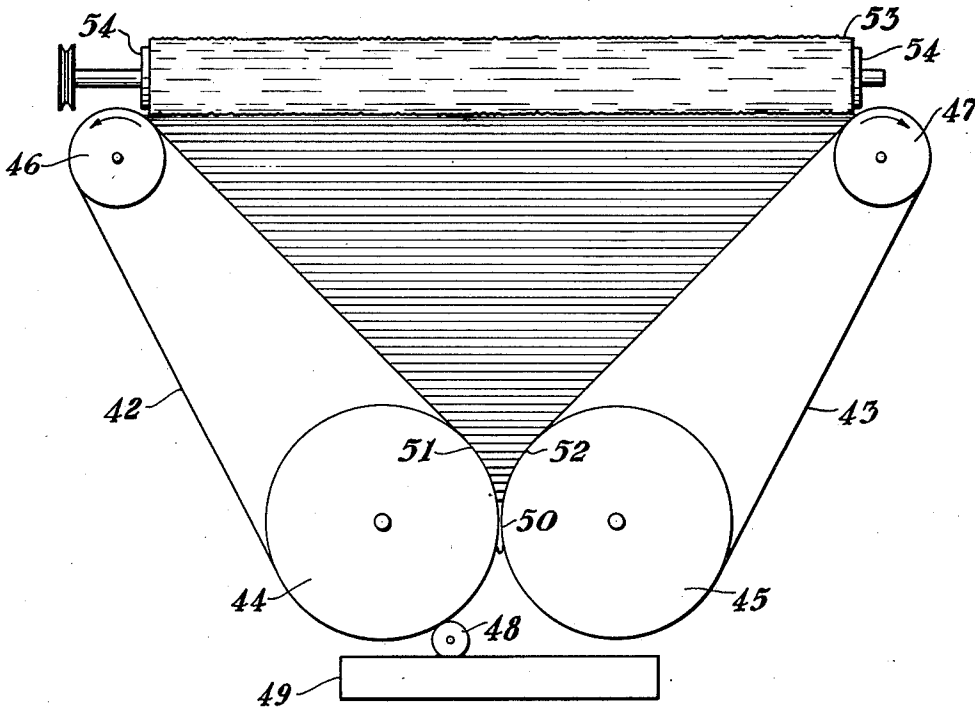


Fig. 5

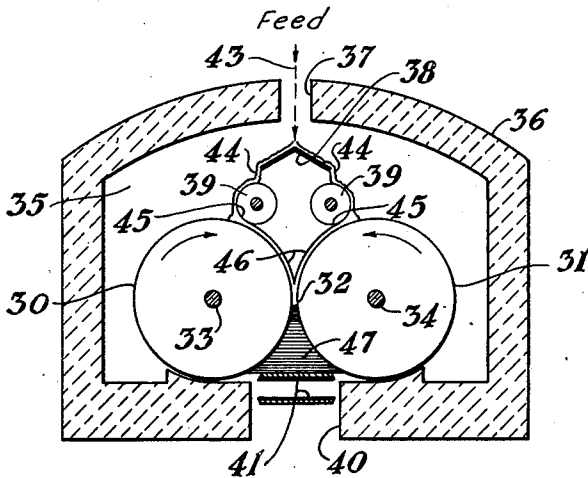


Fig. 4

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METHOD OF MAKING FINE FIBERS

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9 Claims. (Cl. 18—54)

The invention relates to a method of making fine fibers or filaments and is more particularly concerned with a method of producing such materials by a drawing operation from a fiber-forming material in a liquid or semi-liquid state. The method is regarded as having broad application to the production of fine fibers from organic and inorganic fiber-forming materials in solution, dispersion, or a melt, at such temperatures as permit viscous flow.

A principal object of the invention is to provide a method of drawing a multiplicity of fine fibers from a fiber-forming material in a liquid or semi-liquid state without the aid of the usual orifice or die means to impart the fiber shape to the material.

A particular object is to provide a method of simultaneously drawing a plurality of fine organic fibers to form a multiplicity thereof without the aid of the usual orifice, die, or spinneret means, said fibers being characterized by having a large proportion of fibers with a diameter of less than 0.5 micron and a ratio of length to diameter as great as 107.

According to the invention, oppositely acting tractive forces are applied to a thin layer or film of a fiber-forming material in the liquid or semi-liquid state in an elongated pressure zone defined by the surfaces of moving opposed members, at least one of said surfaces describing an arc or being curved through at least a portion of its travel in said zone. Such tractive forces cause the liquid confined laterally in said pressure zone to be split, divided, or pulled apart, and to promote the generation of a multiplicity of fine filaments or fibers. By the moving apart of the surfaces of the members defining the pressure zone, the film of liquid there between, which continues to adhere to both surfaces, yields to the tractive forces and is divided over much of its area. However, due to its cohesiveness, the material adhering to the opposing surfaces is not completely separated and, at numerous points, necks down into continuous threads or filaments stretching between the two surfaces. The mechanism involves the formation of opposed pairs of pools or fiber necks, on the two pressure members, each of the said pairs being connected by a single filament. As the pull on these filaments is continued at a continuously increasing rate, the filaments are lengthened, apparently by drawing fluid from each of said pairs of opposed necks or pools, and the filaments become extremely attenuated. The rate of drawing of the filaments should increase continuously during the early

stages of the operation but may be continued at a uniform or fixed rate thereafter. This latter step may be accomplished during the take-off operation or by suitable extension of the initially arcuate surface or surfaces upon which the fibers are first generated. During the drawing of the filaments, they are caused to set or harden, and means are provided for picking up and collecting the fibers after they have been formed. In the practice of the invention, fibers are consistently made having a submicroscopic diameter, e. g., as small as 0.02 micron, and a ratio of length to diameter as great as 107.

Fiber-forming materials exist having a great diversity of composition. Among them are natural and synthetic resins and polymers which are viscous and ductile or can be rendered so by heating or by treatment or compounding with appropriate agents, such as solvents or plasticizers. Examples of such materials are shellac, nylon, rubber, polystyrene, thermosetting resins in the B stage, cellulose acetate, copolymers of vinylidene chloride and vinyl cyanide, etc. Other examples are the glasses which, when molten, are ductile and can be drawn out into fine filaments or fibers which set as they cool. At appropriate temperatures, such materials are liquid or semi-liquid, generally viscous and tacky, and possess a high degree of adhesiveness as well as cohesiveness. When pulled apart in the manner herein described, such materials form extremely fine threads and, as the main bodies of the materials are separated, these threads increase in length, apparently by feeding from liquid pools or fiber necks, and extremely attenuated and highly oriented streamers or filaments result between the receding portions of the liquid.

The quality of adhesiveness permits solid surfaces to be used as a supporting and holding means for the liquid to be drawn into fibers so that by compressing or squeezing a quantity of the liquid in the nip between opposed arcuate supporting surfaces to cause the liquid to adhere to them, it is thereby strongly enough held so that on separation of the surfaces, the liquid divides within itself or is pulled out and necks down and does not become detached from the supporting surfaces. The liquid or semi-liquid filament is caused to set by treatment appropriate to the composition of fiber-forming material used. For example, the filaments of materials which require chemical treatment to cause setting or hardening may be treated by gaseous or liquid agents; those requiring thermal treatment may be cooled below the liquidus temperature, e. g. a glass, or

heated, e. g. a thermosetting resin; those requiring the removal of solvent, may be subjected to conditions favoring evaporation or extraction of the solvent, e. g. polystyrene dissolved or dispersed in ethylbenzene. In some instances, it may be desirable to carry out the setting or hardening treatment only partially before gathering or collecting the filaments or fibers and completing the setting or hardening after the fibers have been accumulated. This can be accomplished, for example, with fiber-forming compositions which are solutions in volatile solvents by partially evaporating the solvent from the drawn fibers or filaments before accumulating them and completing the drying or evaporation of the solvent from the fibers after they have been accumulated.

An example of a suitable synthetic resin with which to form fine fibers, according to this method, is that obtained by forming a viscid mixture of polystyrene and a volatile solvent therefor, such as ethylbenzene or isopropylbenzene. Such liquid, when pressed as between a pair of steel rotors and then pulled apart by revolving the rollers in opposite directions, is split or divided, and adheres to both liquid-supporting surfaces as a multiplicity of pairs of opposed fiber necks or pools each having a single filament therebetween. While being drawn out, the solvent evaporates from the fibers so that they harden or set. The operation of conveying the liquid through the pressure zone defined by the nip of the rollers and the drawing of fibers or filaments is repeated as the rotors revolve until the evaporation of the solvent from the liquid thickens it beyond the point where the fibers are formed. By controlling the concentration of the solvent to within a range suitable for fiber-drawing, for example, 25-60 per cent of polystyrene by weight, the drawing operation may be repeated continually so long as the supply of liquid is maintained, thus permitting the drawing of fibers to be carried out as a continuous process.

Apparatus which may be employed for carrying out the method and which is adapted to accumulate the fine fibers produced in various modes of operation is schematically illustrated in the accompanying drawings in which:

Fig. 1 is a side elevation of an apparatus including a pair of drawing rotors and a collector for use at ordinary temperatures, i. e. on fiber-forming materials which do not require to be especially heated;

Fig. 2 is a top plan of the same;

Fig. 3 is a side elevation of a portion of the apparatus of Figs. 1 and 2 showing alternate arrangements of the collector;

Fig. 4 shows a side elevation in partial section of an apparatus adapted for use with fiber-forming materials requiring elevated temperatures; and

Fig. 5 shows a side elevation of a modification of apparatus with which the invention may also be practiced, utilizing opposed endless belts travelling in arcuate paths immediately adjacent the point of origin of the filaments, and thence in divergent linear paths to the harvesting point.

Throughout the Figures 1, 2, and 3, like numerals designate like parts.

Referring to Figs. 1, 2, and 3, numerals 5 and 6 designate smooth faced revolving drums or rotors disposed face to face. The drums are driven by chain 7 engaging drive sprocket 8 on shaft 9, and sprockets 10 and 11 mounted on axles 12 and 13, respectively, of drums 5 and 6. At the

nip 14 between drums 5 and 6, the surfaces are juxtaposed at close clearance. A receptacle or pan 16 is provided below the drums for holding a body of fiber-forming liquid 17. In receptacle 16 is disposed a pair of applicator rolls 18 and 19 adapted to apply a film of liquid 17 to the face of rotor 5. Between the rotors 5 and 6 and approximately at a level just below the top of the faces thereof is a harvesting device or accumulator shown as a flexible belt 20 travelling over an idle pulley 21 and pulley 22 connected by belt 23 to a driving motor, not shown.

An alternative position of the pulleys 21 and 22 and consequently of the harvester or collector belt 20 which they support is shown in Fig. 3. Thus, pulleys 21 and 22 may be arranged either horizontally or at an inclination to the horizontal, or the pulleys may be inclined in opposite directions and the positions of the travelling surfaces constituted by the collector belt 20 between them will vary accordingly.

Referring to Fig. 4, numerals 25 and 26 designate smooth faced revolving drums juxtaposed at close clearance at the nip 27. They are supported on axles 28 and 29, respectively, driven in opposite directions (the bearings and driving means not being shown) in a heating zone 30 in a furnace setting 31, having a feed opening 32 in its roof. Below the feed opening 32 is a molten feed flow distributing means 33. Applicator rolls 34 are interposed between the feed distributor 33 and the faces of the rotors 25 and 26, respectively. An opening 35 is provided in the furnace setting to admit the fiber-collecting or harvesting means, which may be a horizontal flexible belt 36 below the rotors, the supports and driving means not being shown.

In Fig. 5, there is shown diagrammatically a modification of the apparatus illustrated in Figs. 1 and 2. Polished endless steel belts 42 and 43 of suitable width are carried by a pair of juxtaposed rotors 44 and 45 and idler rolls 46 and 47. A fiber-forming liquid is picked up by an applicator roll 48 from a tank 49 and applied as a film to the face of one of the belts 42. The film of liquid applied to the belt is carried into the nip 50 of the belts 42 and 43 as they pass between the driven rotors 44 and 45. The liquid is compressed in the nip or elongated pressure zone 50 and then formed into fibers by the separation of the belt surfaces at a continuously increasing rate as the belts 42 and 43 traverse the rotors 44 and 45 to the tangent points 51 and 52 at which the belts 42 and 43 leave the rotors 44 and 45 and travel to the idler rollers 46 and 47. The fibers are accumulated on a flexible belt 53 travelling over a driven roll 54 and an idler roll (not shown). This apparatus provides means to generate the fibers in a zone in which they are first drawn at a continuously increasing rate and then at a linear rate whereby extremely long fibers can be obtained. By providing suitably disposed pairs of drawing surfaces, the above-described two-step operation may be followed by still further changes in the drawing rate, as by traversing a second arcuate course and a second linear course prior to harvesting.

The advantage of the system illustrated in Fig. 5 is found in its ability to increase the yield of fibers in unit time by providing a long enough drawing interval to exhaust substantially all of the fiber-forming liquid from the fiber generating pools or necks disposed on the drawing surfaces.

Referring to Figures 1, 2, and 3, in operation

at ordinary temperatures a quantity of a fiber-forming liquid 17 is placed in pan 16 in amount sufficient to cover at least a portion of the roll 18. The rotors 5 and 6 are oppositely rotated at the same speed by the chain 7, the direction of rotation of the rotor faces being upward at the nip 14. The face of rotor 5 is in frictional engagement with the face of the applicator roll 19, and causes it to rotate and in turn causes the pick-up roll 18 to rotate in the body of liquid 17. As the rolls 18 and 19 are thus rotated, fiber-forming liquid is picked up on the face of roll 18 and transferred by it to roll 19, which in turn transfers the liquid to the face of rotor 5. It is to be understood that any of numerous other means may be used to apply the fiber-forming liquid to the drawing surfaces. The face of rotor 6 becomes coated with the fiber-forming liquid as it comes in contact with the coating on rotor 5 at the nip 14. The coating or layer of fiber-forming liquid on the faces of the rotors becomes evenly distributed by the squeezing action between the rotor faces. Surplus liquid which is unable to pass between the nip 14 may accumulate below it, as shown at 15, due to adhesion of the liquid to the rotor faces.

The rotation of the cylinders causes the fiber-forming liquid to be compressed between the rotor faces to film-like thickness, and then to be split and divided between portions adhering to each of the rotor faces above the nip 14. As this occurs, the liquid on each rotor face is pulled out and necks down at numerous points just above the nip 14. The necks at either end adhere to the liquid on the rotor faces and, as the motion of the rotors continues, are drawn apart and stretched into numerous more or less parallel fibers or filaments extending between the receding rotor faces, as indicated by the lines 24.

The numerousness of the fibers produced per unit area of divided or split liquid film on the rotors depends in part at least on the thickness of the film or the rotor clearance at the nip at the moment of division. This thickness may be adjusted during the operation of the rotors by altering the clearance between them until optimum results are obtained. Clearances in the order of from 0.005 to 0.025 inch have been used satisfactorily at ordinary temperatures.

As the fibers are thus being drawn, they are hardened or set. This may be caused by evaporation of solvent from the fiber, if formed from a solution in a volatile solvent, or by cooling the fiber, if formed from a material liquefied by heating, or by heating the fiber, if formed from thermosetting material, or by chemical treatment in appropriate cases.

The motion of the rotors carries the set or hardened fibers to the moving collector belt to which they adhere, thereby being broken loose from the rotors and carried away by the belt or other harvesting means. The fibers may be variously oriented on the belt, according to the plane and direction in which it travels. For example, as in Figures 1, 2, and 3, when the belt travels at right angle to the fibers and is horizontal, the fibers are laid parallel to each other crosswise of the belt. When the belt is inclined, the fibers lie parallel to one another but inclined to the length of the belt. When the pulleys are inclined in opposite directions, as shown in Fig. 3, the fiber axes lie on the belt at various inclinations to its length. By maintaining pulleys 21 and 22 in the same plane and rocking the plane of the pulleys 21 and 22 back and forth so that

the inclination of the belt 22 changes as the fibers are being collected, the fibers are caused to lie on the belt in a combination of different inclinations crossing each other.

The batt or web of fibers formed on the collector belt may be stripped from the belt continuously, or may be allowed to accumulate to greater thickness for a number of revolutions, and then stripped periodically. The web or batt so formed may be further processed by the usual drawing and spinning operations as employed with natural fibers.

As an example, of operating at ordinary temperatures, a pair of cylindrical rotors 4 feet in diameter with smooth faces 3 feet wide revolving at 4 R. P. M. in unconfined air at 22° C., and delivering drawn fibers from a viscid fiber-forming liquid comprising a 25 per cent solution of polymerized styrene in isopropylbenzene to a collector belt 18 inches wide and 13 feet in circumference traveling at 52 feet per minute in a plane parallel to the nip of the rotors and to the plane of the fiber and in a direction perpendicular to the fibers, forms a batt about 1/4 inch thick weighing about 0.13 pound in 10 minutes, containing about 3 per cent of residual solvent by weight and in which the fiber axes are generally parallel to one another and perpendicular to the length of the batt. In such operation, the solvent in the fibers largely evaporates as they are being drawn, leaving them sufficiently solvent-free so that they do not stick to one another as they are being accumulated even with solvent concentrations as high as 8 per cent. The residual solvent (3-8 per cent) in the accumulated fibers slowly evaporates under ordinary conditions. The drying rate can be increased by moderate heating. In operations of this type employing solvents, the fibers may be enclosed in a suitable hood during drawing and drying so that the evaporation of solvent may be controlled and solvent vapor recovered.

At elevated temperatures, the operation of the process is similar. Referring to Figure 4, a molten material 38 such as a glass, which in the molten state is adhesive and tacky so that it then can be drawn out into fibers, is fed onto the feed distributor 33 through the opening 32 and subjected to the fusing heat developed in the furnace heating zone 30. The feed flow regulator 33 divides the molten feed into two streams which fall onto the applicator rolls 34 which are revolved by the rotors 25 and 26. The temperature of the faces of the rotors is maintained, preferably above the liquidus temperature of the material by the heat developed within the heating zone 30 or within the rotors so that the molten feed material is rolled between the nips 40 as a liquid or semi-liquid, forming the molten films 41 on the rotor faces. These are rolled together at the nip 27 as the rotors revolve, as shown by the arrows, forming a single film of molten fiber-forming material having a thickness depending upon the nip clearance. The film adheres to the rolls and splits just beyond the nip 27 and as this occurs the molten material pulls out and necks down at a multiplicity of places. As the motion of the rotors continues each of the necked down portions of the material is drawn out into more or less parallel fine fibers or filaments stretching between the rotor faces as indicated by lines 42 and the split films are reunited when they again come together at the nip 27.

While the fibers are being thus drawn, they

are cooled and set in a zone, outside the heating zone, which is maintained below the liquidus temperature of the fiber material. The set fibers are harvested onto the surface of the collector belt 36 which travels in a direction normal to the fibers and horizontally below the nip 27 of the rotors and thus causes the fibers to lie parallel and crosswise of the length of the belt. Other collecting arrangements may be made and the fibers may be handled or treated, if desired, as in the operations described for ordinary temperatures.

The use of coating pairs of rotating drawing surfaces, of which the pairs of cylindrical rotors illustrated is one type, causes the respective ends of the fibers to be drawn apart at an increasing, i. e., an accelerated rate as the fibers are lengthened. This is desirable when using fiber-forming liquids, the drawn fibers of which harden as a result of evaporating solvent therefrom during the drawing operation. The process may be carried out with other pairs or multiples of coating surfaces of which the motion of separation is either uniform or variable throughout the duration of the drawing of the fiber. It is preferred that the initial drawing motion be the accelerated separation of corresponding points on coating cylindrical rotors. Such rotors are also the preferred means for drawing fibers according to the invention because they are readily adapted to carrying out continuously the sequence of steps of: feeding the fiber-forming liquid to the drawing surfaces; pressing the liquid between drawing surfaces to a desirable thickness; and separating the drawing surfaces so as to cause splitting and drawing out of the liquid in fine fibers therebetween, setting or hardening the fibers, and repeating the steps with or without supplying, or feeding, additional fiber-forming liquid to the drawing surfaces after each separation thereof. Such steps may be used also with various ways of collecting or harvesting the fibers in directions either parallel with or vertical to the direction of drawing, such ways, however, not constituting a part of the present invention.

It is to be understood that the coating rotors are preferably of the same diameter and that they are preferred to operate at the same angular rate of rotation, but that rotors of different sizes may be used, though less advantageously. It is also contemplated that the fiber drawing system may comprise a single rotor coating as a drawing means with a flat or other non-rotating surface.

Although in illustrating and describing the process particular reference has been made to the making of fine fibers from a liquid solution of polystyrene and from a molten glass, it is to be understood that other materials that are, or can be, rendered viscid and tacky can be similarly treated to draw them into groups of fine fibers. Also, it will be appreciated that the method can be applied to such fiber-forming materials as may be set by applying heat, as in the case of thermosetting materials, or by maintaining special atmospheres, either gaseous or liquid, about the fibers during or after drawing to bring about chemical changes in the fibers.

Webs or batts formed by the accumulation of fibers made in accordance with the invention are unique in that the individual fibers have a substantially uniform and very small diameter over substantially their whole length, averaging in the order of less than 2 or 3 microns in diameter, averages in the order of 1 micron being readily produced. In practice, fibers as fine as 0.02

micron have been obtained. The fibers may be arranged in parallel or inclined to one another or in a combination of parallel and inclined arrangements. Such webs or batts are excellent heat insulators and may be used advantageously in quilted forms.

This application is a continuation-in-part of my co-pending prior application, Serial No. 460,741, filed October 5, 1942.

I claim:

1. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, and drawing fibers from said necks at a continuously increasing rate.

2. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate, and collecting the fibers while continuing the drawing thereof until rupture occurs substantially at the necks.

3. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate, and collecting the fibers while continuing the drawing thereof in a direction at an angle to the line of the first said drawing until rupture occurs substantially at the necks.

4. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, and drawing fibers from said necks by moving pairs thereof along divergent arcuate paths.

5. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate through an initial period, and continuing the drawing at a substantially uniform rate.

6. The method of making fine fibers, which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity

of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks by moving pairs thereof through an initial period along divergent arcuate paths, and continuing the drawing by moving said pairs of necks along divergent linear paths.

7. The method of making fine artificial fibers, which includes: continuously conveying through an elongated pressure zone a fiber-forming substance dispersed in a volatile solvent capable of adhering to opposed pressure members defining said zone, releasing the pressure on said zone to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate, collecting the fibers while continuing the drawing thereof until rupture occurs substantially at the necks, and evaporating solvent from the fibers during the drawing operation.

8. The method of making fine artificial fibers, which includes: continuously conveying through an elongated pressure zone a fiber-forming material liquefied by heating and capable of adher-

ing to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate, collecting the fibers while continuing the drawing thereof until rupture occurs substantially at the necks, and cooling the fibers during the drawing operation to set them.

9. The method of making fine artificial fibers which includes: continuously conveying through an elongated pressure zone a liquid fiber-forming composition capable of adhering to opposed pressure members defining said zone, releasing the pressure on said liquid to produce a multiplicity of opposed pairs of fiber necks on said opposed pressure members, each of said pairs connected by a single filament, drawing fibers from said necks at a continuously increasing rate, partially hardening the fibers during the drawing operation, collecting the fibers while continuing the drawing thereof until rupture occurs substantially at the necks, and completing the hardening after the fibers have been collected.

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