

Feb. 6, 1973

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3,715,419

DRAG STABILIZED LOW VISCOSITY MELT SPINNING PROCESS

Filed Oct. 27, 1969

3 Sheets-Sheet 1

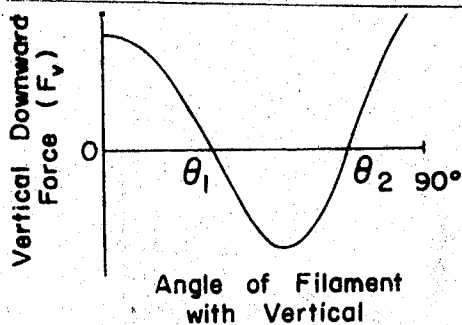


FIG. 1

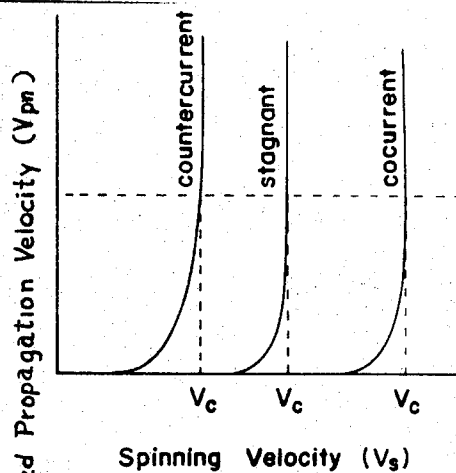


FIG. 2

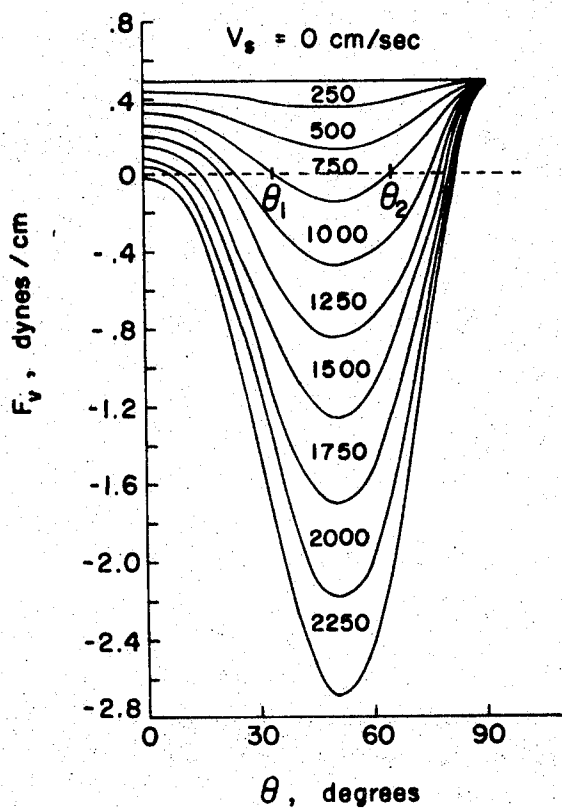


FIG. 3

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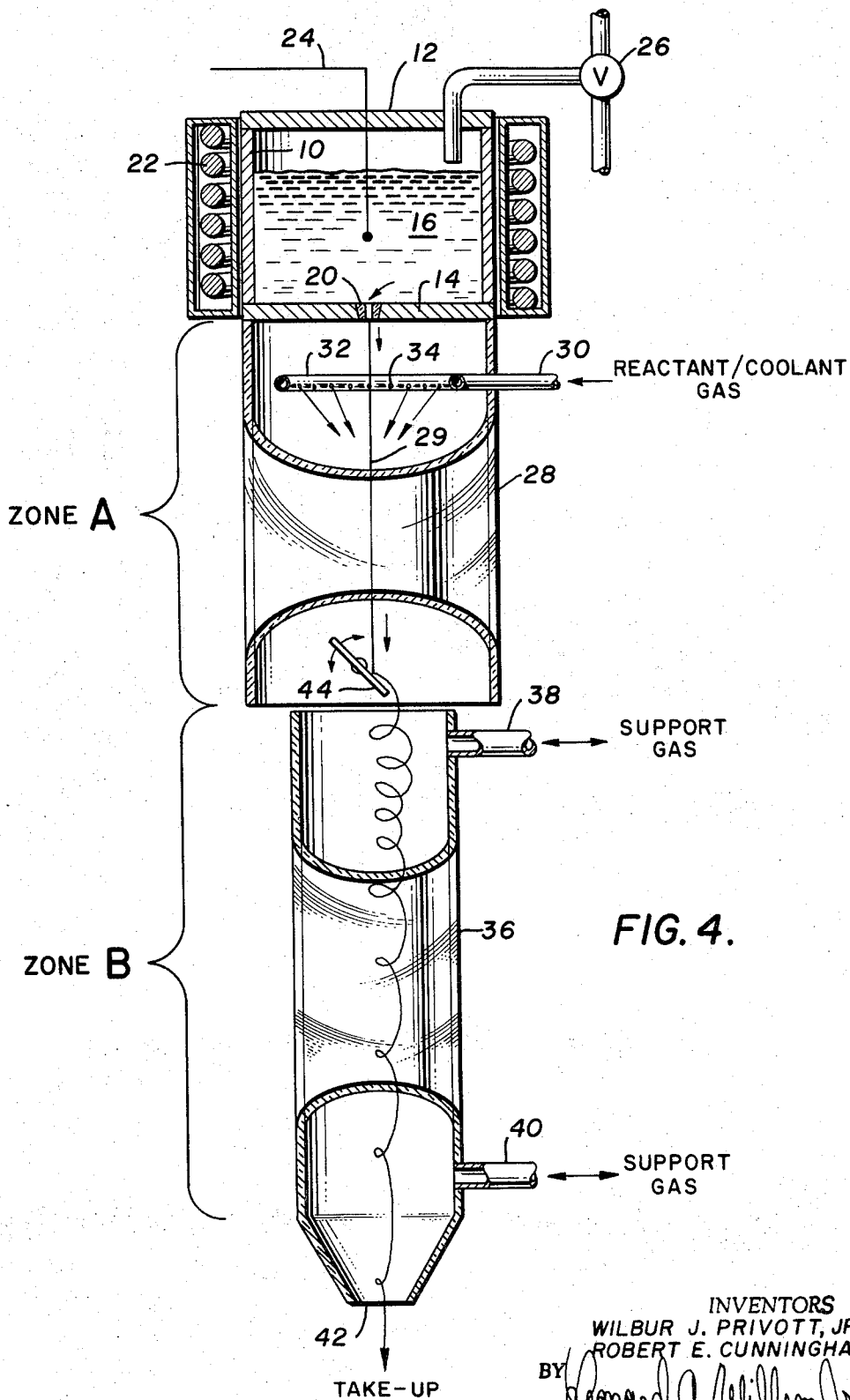
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3 Sheets-Sheet 3

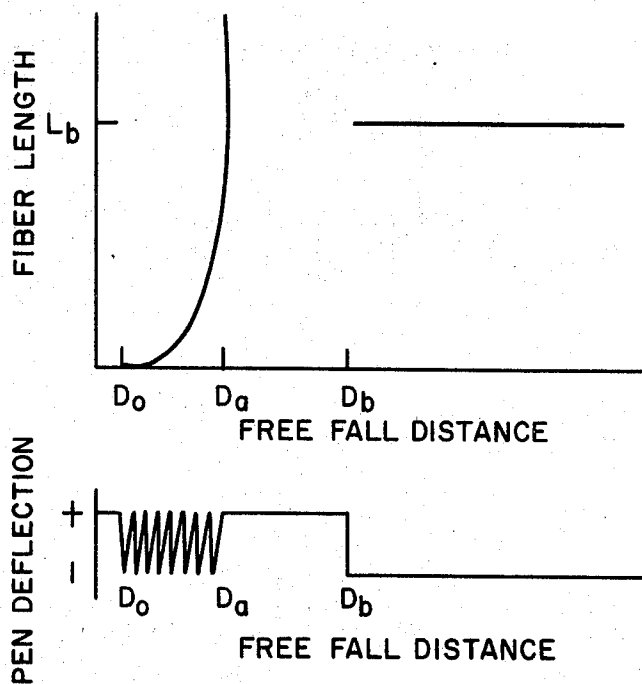


FIG. 5.

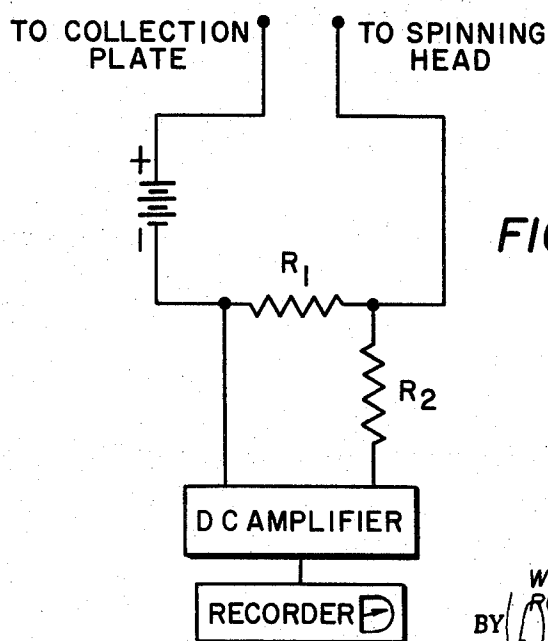


FIG. 6.

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DRAG STABILIZED LOW VISCOSITY MELT SPINNING PROCESS

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Continuation-in-part of application Ser. No. 680,898, Nov. 6, 1967. This application Oct. 27, 1969, Ser. No. 870,646

Int. Cl. B28b 3/20; B22d 11/00

U.S. Cl. 264—82

7 Claims

ABSTRACT OF THE DISCLOSURE

An improved stabilization process for forming filaments from streaming low viscosity melt materials by simultaneously preventing disruptions in the fragile, incompletely solidified portion of the stream due to the drag interaction between the stabilized stream and surrounding gaseous atmosphere and utilizing the drag to form a helical-like configuration in the solidified portion of the stream which permits applying sufficient tension thereto to enable filament take-up.

CROSS-REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of the copending and commonly assigned application Ser. No. 680,898, filed on Nov. 6, 1967, and now abandoned. Related copending and commonly assigned applications are Ser. No. 829,216, filed June 2, 1969; Ser. No. 863,311, filed Oct. 2, 1969; and Ser. No. 863,707, filed Oct. 3, 1969.

BACKGROUND OF THE INVENTION

Field of the invention

The present invention relates generally to the formation of filaments and fibers directly from low viscosity melt materials and, more particularly, to a process of decelerating and supporting a molten stream extruded from a low viscosity melt.

Description of the prior art

In attempting to form filaments or fibers directly from a streaming melt material of low viscosity, the initial problem is to stabilize the liquidous portion of the stream, pending its solidification, against an intrinsic tendency to break-up into shot due to surface tension. The U.S. Pat. 3,216,076, issued to Alber et al. and the above reference application Ser. No. 829,216 incorporated by way of reference herein disclose unique and practical approaches to solving the problem. In quite general terms, the approaches involve the concept of film-stabilized melt spinning wherein a "low viscosity" melt material is free-streamed under carefully controlled conditions through selected atmospheres which react or decompose upon exposure to the stream and form a film about the periphery thereof. The film serves to stabilize the stream against surface tension-induced breakup pending solidification by normal heat transfer phenomena.

The term "low viscosity" is used in this application to define viscosities of 10 poises or less. The materials generally employed to form filaments and fibers in accordance with low viscosity melt spinning techniques are those materials which are normally solid at 25° C. Among materials which have these characteristics are the metals, alloys thereof, intermetallic compounds, ceramics, metalloids, and various salts.

Examples of the metals which may be spun are beryllium, cobalt, aluminum, thorium, nickel, iron, copper, gold, uranium, zinc, manganese, magnesium, tin, and al-

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loys of such metals. Representative of the low viscosity ceramics are alumina, calcia, magnesia, zirconia, and mixtures of these and other oxides wherein the mixtures exhibit the above discussed characteristics. Metalloids, such as boron and silicon, salts such as potassium chloride and a variety of other normally solid inorganic materials with low melt viscosities are capable of being spun into filaments and fibers.

Subsequent to the advent of the low viscosity melt spinning techniques, the need to solve other related problems was soon recognized. For example, in addition to the inability of a low viscosity melt stream to withstand surface tension-induced disruptions while in the liquid state, it has also been noted that such a stream is susceptible to forces engendered by the impact or deceleration incident to taking up the streaming body after solidification. The forces generated during such deceleration often result in shearing rupture or deformation of the stream.

It was disclosed and claimed in the aforementioned application Ser. No. 863,311, filed Oct. 2, 1969 that there is a point (D_a), as measured from the stream origin, above which any given deceleration of the stream results in repeated disruptions; a point (D_b), as measured from the stream origin, above which the stream must be decelerated if repeated tensile breaking is to be avoided and that the decelerating of the stream intermediate the points (D_a) and (D_b) affords the obtaining of continuous filaments by obviating the stream disruptions and tensile breaks.

Still another problem was disclosed in application Ser. No. 863,707 incorporated by way of reference herein. Briefly, it was found that when the solidified portion of the stream deviated from its vertical path, the drag forces induced by the surrounding atmosphere caused the deviations to increase in amplitude and propagate up the stream into the liquidous portion. The drag-sustained deviations cause disruptions in the stream continuity.

Application Ser. No. 863,707 however, also discloses and claims a method, the practice of which advantageously avoids the effects of the deviations by maintaining the upstream velocity of the drag-sustained deviations less than the velocity of the stream so that the undesired deviations are caused to move harmlessly away from the fragile liquid portion of the stream.

In the continuous take-up of the solidified stream or filament, it may be necessary to apply tension (albeit slight) to the stream after it has solidified. The portion of the stream which has the least tenacity is the liquid portion at or near the extrusion orifice. When, however, the tension needed for wind-up is applied to the solidified portion of the stream, it is transferred to the liquid portion, causing the stream to be disrupted in the manner identical to that experienced when the stream, extends to a point (D_b) at which it breaks under its own weight.

Unexpectedly, however, it has been found that the effect of drag-sustained deviations may be advantageously employed to facilitate filament take-up while avoiding stream breaks or disruptions due to both tension and drag-sustained deviations. Generally, proper take-up conditions may be maintained in accordance with the present invention as follows: (1) controlling the conditions in a first zone which extends below the liquidous portion of the stream so as to cause the velocity of the drag-sustained deviations up the stream to be less than the velocity of the stream; and (2) controlling the conditions in a second zone immediately below the first zone so as to cause the velocity of the drag-sustained deviations up the stream to be greater than the velocity of the stream. As a consequence, the stream in the second region attains a helical-like configuration which cannot pass into the first region due to the conditions imposed therein. The presence of the helical configuration in the solidified portion of the stream, how-

ever, allows tension to be applied to that stream zone without transferring the tension to the fragile liquidous portion of the stream.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects, features, and advantages of the present invention will become apparent from the following, more detailed description and the accompanying drawings in which:

FIG. 1 graphically depicts the variation in vertical force (F_v) acting upon a stream as a function of the angle of stream deviation from the vertical;

FIG. 2 is a typical plot of the variation in the propagation velocity (V_p) of drag-sustained stream deviations as a function of the stream or spinning velocity (V_s), propagation velocity being normalized relative to spinning velocity;

FIG. 3 is a typical plot of the variation in vertical force (F_v) upon a stream with stream deviation angle (θ) for a series of stream velocities (V_s);

FIG. 4 is a simplified vertical, partially sectionalized view of a spinning apparatus which may be employed in the practice of the subject process;

FIG. 5 graphically depicts the interrelationship of fiber length (L) with stream free-fall distance for the case where the point of impact breakage deceleration (D_a) is maintained upstream of the point of tensile breakage deceleration (D_b), the condition which must obtain for indefinite length production; and

FIG. 6 is a schematic circuit diagram of conventional make-up which may be employed to electrically monitor and record stream continuity where issued from electrically conductive melts.

DESCRIPTION

The aforementioned application Ser. No. 829,216 discusses in detail the theory of low viscosity melt spinning and fundamental conditions necessary for proper stabilization of the molten stream pending solidification. For purposes of this disclosure, it is necessary to state only that the molten stream must be extruded at velocities and diameters which satisfy a range of values which are calculated from a dimensionless quantity known as the Rayleigh parameter (Ra). The Rayleigh parameter is defined as

$$Ra = V \sqrt{\frac{\rho D}{\gamma}}$$

where

V is the extrusion velocity, cm./sec.

ρ is the melt density, gms./cm.³

D is the stream diameter, cm.

γ is the stream surface tension, dynes/cm.

The range of values for the Rayleigh number is 1 to 50. For optimum spinning conditions, it is preferred that a value for the Rayleigh parameter be between 2 and 25. Thus, for example, given a melt density of 4 gms./cm.³, a surface tension of 1000 dynes/cm., and a stream diameter of 0.03 cm., it is necessary to have an extrusion velocity between 90 to 4500 cm./sec. to be within a Rayleigh parameter range of 1 to 50.

Although application Ser. No. 863,707 is incorporated herein by reference, it is thought that the following discussion on drag-sustained deviation will be helpful in more clearly pointing out the present invention. Initially, it will be observed that any deceleration of the stream must result in a deviation from its original vertically straight path. As distinguished from the general configuration that is characteristic of stream deviations within the liquid region, drag-sustained deviations are initiated in the relatively higher modulus, solid downstream region. The increased rigidity of the stream results in the formation of a three-dimensional, helical-like (as opposed to sinuous) con-

figuration which if allowed to will grow and move unimpeded.

Starting from literature studies of the viscous drag forces acting upon streaming bodies (see for example, Sakiadis, B. C., AICHEJ. 7, 467 (1961) and Knudsen, J. G. and D. L. Katz, Fluid Dynamics and Heat Transfer, McGraw-Hill, New York, 1958), a reasonably close approximation of the total downward force acting upon a streaming body has been derived as given by:

$$(1) F_v/\text{unit length} \approx [\pi r^2 g \rho_s] - 1.13 [\mu (V_s \cos \theta - V_g) \cos^2 \theta] - 7.07 [\mu r \rho_g (V_s \cos \theta - V_g)^3 \sin^5 \theta]^{1/4}$$

$F_v/\text{unit length}$ = the force acting upon the stream/unit length, dynes/cm.

r = stream radius, cms.

g = gravitational acceleration, 980 cm./sec.²

ρ_s = stream density, gm./cm.³

μ = spin gas viscosity, poise

V_s = stream velocity, cm./sec.

V_g = velocity of the gas into which the stream is extruded, cm./sec.

ρ_g = spin gas density, gm./cm.³

θ = angle of stream deviation between axis of the stream and the vertical degrees.

As may readily be appreciated, it is impossible, at least within the realm of practicality, to constrain a freely streaming body to move in a precisely straight path. Even though one takes elaborate precautions, it is virtually impossible to isolate the stream from at least initially minor disturbances engendered, for example, by controlled, though minor, fluctuations in extrusion conditions, eddy currents and the like due to such as minor orifice imperfections and external vibrations.

As shown by Equation 1 above, the total downward force per unit length on the stream varies with the angle of deviation of the stream from the vertical. FIG. 1 depicts a curve for a solution of Equation 1 as a function of the angle of stream deviation from the vertical. As there indicated, any deviation of the stream from the vertical less than θ_1 leaves the entire length of the stream in a positively tensioned state, with the result that there is no tendency for the stream to undergo further deviation or bending. That is, because a net tensional or downward force remains upon the stream at deviations of a magnitude less than the angle of θ_1 , the stream tends to return to its original vertically straight path even though initially deviated by some stream disturbing force. Once some portion of the filament, by whatever event, achieves an angle greater than θ_1 from the vertical, this portion will be decelerated in that the interaction between gravity and the increased drag force acting upon such deviated portion results in the imposition of a net upward, or compressive force upon the stream. Further, the force of on-coming upstream portions will tend to increase the angle of this portion of the stream and the filament will be urged to move to an angle θ_2 with the vertical. As shown by the typical curve of FIG. 1, the only two stable equilibrium configurations of the stream supplied at a rate V_s are a substantially straight, vertically falling stream with a constant speed V_s or a stream falling at an angle of deviation θ_2 with the vertical at a velocity of $V_s \cos \theta_2$. Any other magnitude of deviation will be found unstable, with the stream returning to either the configuration accompanying an angle of deviation of θ_2 or a substantially vertically straight path. Thus, it is seen that, for a given net tensional force acting upon a stream passing in a state of equilibrium along a straight, substantially vertically downward path, there exists an angle of deviation θ_1 , hereafter termed the angle of threshold deviation, below which the stream is, under the influence of gravity or other stream accelerating or tensioning forces, returned to the equilibrium state of its original, straight-line path, and above which a net upward, or negative (compressive) force comes to act upon the deviated portion urging it to

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an angle of deviation θ_2 , hereafter termed the angle of equilibrium deviation. Further, as the net tensional force acting upon a vertical, undeviated portion of the stream decreases with, for example, an increase in the force of viscous drag acting thereon, the angle of threshold deviation θ_1 decreases. Thus, the magnitude of stream disturbance necessary to initially deviate the stream to θ_1 decreases with decreasing stream tension.

In the case where a stream portion has come to occupy the angle of equilibrium deviation θ_2 , it is clear that oncoming upstream portions must be decelerated from a velocity V_s to a velocity $V_s \cos \theta_2$ as successive portions undergo deviation or helical bending to the angle of equilibrium configuration θ_2 . The effect of this successive bending of the stream is the propagation of the helically configured deviation along the stream. It is this understanding of the mechanism of drag-sustained stream deviations that has led to the discovery that, by manipulation of those spinning conditions affecting the magnitude of the viscous drag force, one can control the formation and streamwise propagation velocity of such deviations.

It has now been experimentally confirmed that a reasonable approximation of the propagation velocity, relative to the stream, of a drag-sustained stream bending or deviation is given by the relationship:

$$(2) \quad V_p \approx \frac{[0.038E(F_v/r)^2 \tan(\theta_2 - \theta_1)]^{1/3}}{\rho_s V_s (1 - \cos \theta_1)}$$

wherein:

V_p =velocity of propagation, relative to the stream, of a drag-sustained stream bending or deviation, cm./sec.

F_v =maximum upward or negative force/unit length acting upon the stream as given by Equation 1, dynes/cm.

E =stream modulus at the point of stream bending, dynes/cm.²

r =stream radius, cm.

ρ_s =stream density, gm./cm.³

V_s =velocity of the stream

θ_2 =angle of equilibrium stream deviation from the vertical degrees

θ_1 =threshold angle of stream deviation above which the stream undergoes further deviation to the angle θ_2 and below which such deviation is non-sustaining, degrees.

The magnitude and direction of such velocity of propagation relative to the orifice (V_{pn}) is obtained simply by subtracting the stream velocity from the value given by Equation 2. Thus,

$$(3) \quad V_{pn} = V_p - V_s$$

wherein

V_{pn} =the normalized velocity of the velocity of propagation relative to the orifice and being negative in value when propagating toward the orifice, cm./sec.

V_p =the velocity of propagation of the stream deviation relative to the stream as given by Equation 2, cm./sec.

V_s =the velocity of the stream, cm./sec.

There is depicted in FIG. 2 a typical plot of the propagation velocity or drag-sustained deviations as a function of the stream or spinning velocity wherein the propagation velocity is normalized relative to the orifice. For example, where the velocity of propagation matches spinning velocity, the deviation propagates at a velocity relative to the orifice or normalized velocity, of zero and the deviation would remain a fixed distance from the orifice. A plot is shown for the cases of counter-current, stagnant and co-current spin gas flow. As indicated, for spinning velocities greater than the critical value V_c , any disturbance which causes a stream deviation to an angle greater than θ_1 would propagate upstream to the fragile liquid region to result in either total breakup of the stream or a jointed appearance in the resulting filament. For stream velocities less than the critical spinning velocity V_c , any stream deviation would be swept harmlessly downstream.

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It should here be noted that the representation in FIG. 2 is made assuming the stream is of constant modulus along the length under consideration. In reality, the effective stream modulus may be found to decrease with increasing stream temperature encountered at progressive points upstream so that, where conditions are such that the disturbance propagates at a velocity sufficient to move upstream relative to the orifice, the length over which such stream bending acts becomes less and less until a point is reached where the propagation becomes arrested by a sharp bend, resulting in either broken lengths or filaments exhibiting knee-like joints.

To gain a better understanding of the interrelationship of the vertical force system acting upon a filamentary stream and the velocity at which stream deviations are caused to propagate, reference is now made to the showing of FIG. 3 where, employing Equation 1, the variation in vertical force per unit length with angle of stream deviation for a range of spinning velocities is shown. Forces acting in the down stream direction are positive. The FIG. 3 showing is for the case of an 86 micron diameter, 62 wt. percent lead/38 wt. percent tin stream passing through a stagnant atmosphere of 93 volume percent helium and 7 volume percent oxygen. With the fiber radius, density and modulus, and the gas viscosity, density and velocity thus established, the data necessary to calculate the vertical force at varying spin velocities and angles of stream deviation is provided.

As shown in FIG. 3, at a zero spin velocity (V_s), the vertical force per unit length acting upon the stream equals the stream weight. Up to a spinning velocity of about 625 cm./sec., the net force acting upon the fiber is always in the downward, or positive direction, regardless of the angle of the filament with the vertical, with the result that any incipient stream deviation is immediately overcome and the stream returned to its original path. Consequently, no drag-sustained propagation is possible. As the spinning velocity is increased, a point is reached at which, once the stream suffers a deviation greater than θ_1 , the resultant drag-sustained propagation due to the net upward, or negative, force acting upon the stream exactly equals the spinning rate, with the result that the deformation is stationary relative to the orifice as it rides the stream in treadmill fashion. According to Equation 2, a spinning velocity of approximately 2,050 cm./sec. is required to satisfy this condition. As previously discussed, it has been discovered that, under the conditions above specified, this constitutes the maximum allowable spinning velocity or critical spinning velocity V_c if non-jointed, continuous lengths are to be obtained. At higher spinning velocities, an upstream force is imposed upon the stream of such magnitude as to cause any stream deviation initially exceeding θ_1 to propagate at a velocity greater than the spinning velocity to move upstream relative to the orifice to a point where stream strength has decreased sufficiently to arrest such propagation by permanent distortion or breakage. At lower spinning velocities, any θ_1 deviation of the stream will, due to the reduced upward force, propagate at a velocity less than the spinning velocity to thereby pass harmlessly downstream. It is to be recognized that, in general, a precise balance between propagation and spinning velocities is not likely of observation since very minor variations in spinning conditions, particularly as regards spinning velocity or gas conditions, would result in movement of the deformation either upstream to a point of ultimate disruption or harmlessly downstream.

As will be understood, as the upstream-acting drag force increases with, for example, increasing spinning velocity, the threshold or minimum initial stream deviation θ_1 beyond which the drag force is sufficient to further deviate the stream to the stable θ_2 position decreases. So long as a stream is conveyed with such precision as to avoid any initial deviation greater than θ_1 , drag-sustained deviations and their propagation will not be sustained.

It has been observed, however, that particularly in the case of low density melts and/or small diameter streams, deviations greater than θ_1 are virtually impossible to avoid. Also, where it is found necessary to resort to counter-current gas flows in order to avoid stream breakage due to gravity (as may be the case with relatively large diameter streams of high density), the extent to which gravity, or stream weight, may be counteracted by, for example, a drag force is limited out of the same considerations. It will often be found desirable to impose drag forces of such magnitude as to result in a value for θ_1 so small that it will be found most difficult to isolate the stream from incidental θ_1 deviations. Again, once such a magnitude of initial deviation occurs, it is critical that the propagation of the resultant drag-sustained deviation be controlled not to exceed the spinning velocity if permanent distortions of the weak upstream region are to be avoided.

As determined by calculation from Equation 2, a still further increase in the spinning velocity above the critical spinning velocity of 2,050 cm./sec. (V_c) results in a propagation velocity of drag-sustained deviations greater than the spinning velocity, with the result that a deviation moves upstream to a point of ultimate stream disruption. For example, at a velocity of about 2,170 cm./sec., the gravitational and viscous drag forces are of equal magnitude at an angle of zero deviation and the net force of drag versus gravity acts upstream at any other angle. Under these conditions, even the most minor stream deviation is drag-sustained and propagated at a very rapid, theoretically infinite, rate.

It may readily be appreciated that, for other stream and gas compositions and other spinning conditions, a force diagram such as depicted in FIG. 3 may reflect considerably different values, but will generally conform to the pattern there indicated.

Among the spinning parameters which may readily be varied to accomplish the desired interrelationship between the propagation velocity of stream deviations and spinning velocity are those of stream radius, modulus and density and spin gas density, viscosity and velocity. In general, the critical spinning velocity (V_c) (i.e., that velocity above which, under a given set of conditions, propagational velocity is found to exceed stream velocity with concomitant migration of the deviation relative to the stream source) increases with increasing stream radius. Accordingly, with the increasing tensional force of larger and larger streams due to the influence of gravity, the greater the permissible drag force below which upstream migration is forestalled. Similarly, the critical spinning velocity increases with increasing melt density. Accordingly, in the case of very low density melts, it will normally be found necessary to resort to co-current spin gas flow or other modes of reducing stream decelerating force and/or increasing stream tension within those regions where migration of drag deviations is to be avoided. Contrary to what might be expected, it has been found that a fairly wide variation in stream modulus has little effect upon the critical velocity (V_c).

Spin gas density has been found to have a significant effect upon the critical spinning velocity, assuming that gas viscosity remains constant. This assumption is warranted for many of the gases, but not for all. For example, helium and oxygen have approximately the same viscosity, but it has been found that spinning into a primarily helium atmosphere can be executed without upstream migration at much higher velocities than into an atmosphere having the density of oxygen. Similarly, the effect of gas viscosity is also quite pronounced, especially in the cases of lower density streams and/or smaller diameter streams.

Spin gas velocity is another readily controlled spinning parameter having a significant effect upon the propagational velocity of drag-induced stream deviations and is found particularly convenient in that it may be controlled

independently of the factors of stream size and composition, as well as spin gas composition. Obviously, the higher the co-current gas velocity, or the lower the counter-current velocity, as the case may be, the less the viscous drag force and the greater the critical spinning velocity.

Aided by the foregoing observations regarding the propagation of stream deviations, it has now been discovered that, by imposing a particular differential or segmentation of the system of forces imposed along adjacent stream regions, such deviations may be localized to effect a stabilized elastic deformation of the stream within a selected region therealong to thereby decelerate and support same against tensile breakage while isolating the fragile upstream region from the disturbances attending such deceleration and accumulation. In further detailing this practice, reference is now made to FIG. 4 as symbolically depicting the basic interrelationship. Within the upstream region of the spinning column labeled Zone A, the tensional or accelerating and bending or decelerating forces acting upon the stream are controlled to maintain the velocity of propagation (V_p) of stream deviations less than the stream or spinning velocity (V_s). This is to say that the force system imposed upon the stream within Zone A is such that, if the angle of threshold stream deviation θ_1 is exceeded and the stream thus further deviated to the angle of equilibrium configuration θ_2 (wherein the up and downstream forces are in equilibrium), the propagation of this configuration along the stream would proceed at a rate less than the stream velocity. Thus, as previously explained, the deviation could not migrate upstream relative to the orifice, but would be passed downstream. In normal operation, it will be found preferable to maintain a force system within the upstream region of Zone A such that the stream remains under a net tensional force for any given angle of deviation, with the result that no propagation can occur and the deviation decays to allow the stream to return to its original path. Such a condition is graphically represented in FIG. 3 by that family of curves lying above or on the tensional side of the zero force line, which is to indicate that no amount of initial stream deflection will result in the imposition of that net upstream or bending force necessary to further deform the stream to the equilibrium configuration angle θ_2 , much less to cause such configuration to propagate at a velocity greater than that of the stream.

Within the downstream region labeled Zone B in FIG. 4, the system of tension or accelerating and bending or decelerating forces is controlled to maintain the velocity of propagation of stream deviations, once induced, greater than stream velocity to thereby cause an upstream migration of such deviations to the locus of demarcation between the stream and downstream regions. As previously set forth, when the force system imposed along the upstream region is so maintained that the bending rate is less than the stream or extrusion velocity (V_s), any deviation propagated with Zone B is prevented from passing into Zone A, and migrates only to the upstream extent of Zone B to occupy a stable position relative to the stream origin. Thus, below the fragile region with Zone A, the stream is smoothly decelerated and elastically contorted at a localized point to form an accumulation which serves to cushion the upper region against the disturbances attending collection.

The force system maintained along the downstream region of Zone B may be such that the angle of threshold deviation θ_1 is very small or essentially zero, in which case even a very small initial deflection of the stream results in a rapid upstream migration relative to the orifice. For example, under the conditions specified for the graphical portrayal of FIG. 3, it is seen that the gravitational and drag forces are of equal magnitude at an angle of zero stream deviation for a spinning velocity of approximately 2,170 cm./sec. and the net of drag vs. gravity acts upstream at any other angle. Thus, even the most minor stream deviation would be drag-sustained and rapid-

ly propagated upstream to the juncture with Zone A. Accordingly, where the force system imposed along Zone B is, concomitant with a close balance between stream accelerating and deceleration forces, characterized by an angle of threshold deviation θ_1 verging on zero, it is found that the stream is immediately deviated to the angle of equilibrium configuration θ_2 on passing into Zone B. This, of course, is due to the fact that, as a necessary incident to the disturbances attending normal spinning operations, the stream is constantly undergoing deflections which, though minute, are sufficient to exceed the small angle of threshold deviation θ_1 accompanying a close balance between stream accelerating and decelerating forces.

Where the force system imposed along Zone B is characterized by an angle of threshold deviation θ_1 too large to be reliably exceeded by incidental stream deflections, the stream must be purposely deflected through an angle greater than θ_1 . This is symbolically denoted in FIG. 4 by the use of a deflection plate located at the demarcation between Zones A and B, but the initial deflection of the stream beyond the angle of threshold deviation may be accomplished by a variety of techniques. For example, a lateral jet of gas or magnetic field may also be employed to initially deflect the stream.

The differentiation or segmentation of the force systems acting along Zones A and B necessary to attain stabilization of stream deviations is readily accomplished by any one or more of a variety of manipulations. No matter how such a differentiation in force systems is effected, it is always characterized by a decrease in stream tension and/or an increase in stream bending or decelerating forces in passing from Zone A to Zone B, the differentiation being sufficient to increase the bending rate from a value less than stream velocity in Zone A to one greater than stream velocity in Zone B. For example, the stream may be subjected to an increased viscous drag force on entering Zone B by virtue of passing through a supporting gas having a greater density and/or viscosity to that of Zone A. Similarly, the gas within Zone B may be maintained at a lower co-current or higher counter-current velocity, as the case may be, than that of Zone A. For example, extremely heavy or dense filaments may require a counter-current flow of supporting gas in Zone A to prevent tensile breaking from occurring in the molten portion of the stream. To provide the helical-like configuration in Zone B, it is necessary to increase the velocity of the counter-current flow in Zone B. As should now be apparent, the converse may be true for light filaments. As indicated in FIG. 4, the latter may be accomplished by introducing a counter-current flow of support gas within the lower region of Zone B. A decrease in counter-current gas flow on passing from Zone B to Zone A is readily accomplished by, for example, utilizing an enlarged spin chamber cross-section within Zone A to effect a reduction in counter-current flow on entering the larger zone with a consequent decrease in the force of viscous drag upon the streaming body. Numerous combinations of these techniques of force system differentiation between Zones A and B may be employed. Also, though it may not be feasible to segment those stream characteristics affecting the system of forces acting thereupon, such as spinning velocity, stream density and radius, it should now be recognized that, where a given differential in the force systems of Zones A and B is found insufficient to sustain a velocity of propagation within Zone B greater than the stream velocity, the spinning or stream velocity may be increased to a level where such a condition obtains within Zone B, subject of course, to the limitations on stream velocity imposed out of considerations attending stabilization of the liquid region and avoiding the generation of a drag force within Zone A sufficient to render deviation propagation greater than the stream or extrusion velocity. In other words, for a given differential in force systems, there exists a range of stream velocity wherein the velocity of propagation may be maintained less than the stream velocity

within Zone A and greater than the stream velocity within Zone B. If spinning within the prescribed spinning velocity range is found inexpedient, the differential in force systems is readily modified, as before indicated, to accommodate a more workable spinning velocity.

Though the foregoing discussion has largely been in terms of a differential in spin gas conditions between the upstream and downstream regions, it is to be recognized that the required segmentation of force systems may also be accomplished by establishing a differential in stream momentum between the two regions to thereby allow a differential stream bending rate or velocity of propagation even in the presence of identical gas conditions within the two regions. Segmentation of stream momentum between the two regions is readily established by imposing a change in the stream configuration and a consequent change in the force system acting therealong as between the two regions to result in a change in momentum such as to reduce the resistance to stream bending within the downstream region. The desired change in stream momentum concomitant with a change in configuration may be effected by imposing a sustained deviation of the stream with Zone B by an externally applied force. Such a deviation may be accomplished, for example, by a deflection plate, such as is symbolically indicated in FIG. 4. Even though conditions may be such that the propagation velocity of a deviation in a stream from the vertical to an equilibrium configuration θ_2 is less than the spinning velocity, the imposition of a sustained deflection, with the accompanying change in momentum, results in a force system along the continuously deflected portion of the stream such that the propagation velocity of a further deviation superimposed thereupon is greater there than along an undeviated portion. Thus, by proper control of the magnitude of the sustained stream deflection, the velocity of propagation (V_p) within Zone B can be caused to exceed the spinning velocity (V_s) to thereby form a stabilized helical deviation below Zone A under all conditions for which there exists a net upward force on the stream falling on some angle. Said differently, if there exists an angle of threshold deviation θ_1 and an angle of equilibrium configuration θ_2 for a given set of conditions, a stable deviation can be maintained by providing a sustained deviation of the stream below Zone A of sufficient magnitude that the propagation velocity of a further deviation beyond θ_1 exceeds spinning velocity. Again, this mode of operation can be used, but is not limited to situations where the gas conditions in the upstream and downstream zones are identical.

A further important aspect in the practice of the present invention relates particularly to the drag-sustained production of indefinite or continuous length articles extruded from low viscosity melts. This practice is generally characterized by control of the locus of demarcation between the differential in force systems imposed along the upstream and downstream portions (Zones A and B, respectively) relative to a particular interrelationship of the breaking characteristics of a given stream. As stated previously, application Ser. No. 863,311 describes and claims a process by which continuous filaments may be obtained. Because the teachings therein are highly relevant to the present invention, it is necessary to briefly discuss the breaking characteristics of a stream although for a more detailed explanation reference should be made to application Ser. No. 863,311 which is incorporated by way of reference herein. Low melt viscosity streams are characterized by a point (D_a), as measured from the stream origin and hereinafter termed the point of impact breakage deceleration, above which the application of a given deceleration results in repeated impact disruption of the stream. Further, there exists a point (D_b) hereinafter termed the point of tensile breakage deceleration, above which the stream must be decelerated if repeated tensile breaking is to be avoided. Thus, where stream continuity is to be preserved in the service of pro-

ducing articles of indefinite length, the present invention is embodied by the manipulative sequence of intercontrolling the force system imposed upon the stream relative to stream state to maintain the tensile breakage deceleration point (D_b) downstream of the impact breakage deceleration point (D_a) and maintaining the locus of demarcation between the differential in force systems imposed along the upstream and downstream portions at a position intermediate the points D_a and D_b . The control of the force system acting upon low viscosity streams such that the point D_a is maintained upstream of the point D_b may be effected by any one or more of a variety of modes, as typified in the application Ser. No. 863,311. The proper interrelationship of the impact and tensile breakage points, D_a and D_b , is diagrammatically indicated by the graph appearing in the upper portion of FIG. 5 wherein the free-fall distance of the stream is plotted against fiber length.

In the present discussion, the term free-fall distance shall be taken to denote that vertical distance between the stream origin or orifice and the point at which the stream undergoes initial deceleration. For example, in FIG. 4 the position of the deflection plate or collection surface constitutes the point of initial stream deceleration. However, it is to be emphasized that free-fall distance is not necessarily defined by a position of a solid collection surface such as depicted in FIG. 4. Such a surface is merely to be taken as symbolic of that point below the orifice at which a decelerating force is imposed upon the stream. Though this force may arise by the sudden impact of the stream upon a solid surface, a more gradual stream deceleration, such as may be effected by causing the stream to pass through a denser and/or counterflowing spin atmosphere may as well give rise to this force. Similarly, a more gradual stream deceleration may be achieved by the imposition of a suitable electrostatic field. It is only for the purpose of simplicity that the following discussion is made largely in terms of effecting initial stream deceleration by impingement upon a solid surface.

As shown in FIG. 5, four distinct regions appear on the fiber length vs. free-fall distance curve. For free-fall distances less than the distance D_0 , denoting the freeze point of the stream, the stream is yet molten and any attempt to collect the stream at lesser distances results in a molten mass. (The freeze point is taken in the pragmatic sense to denote that point along a solidifying stream at which discrete particles having an aspect ratio greater than unity are obtained upon intercepting the stream at that point; thus, interception at any upstream point by a chosen mode of deceleration results, by this pragmatic definition, in an indiscrete, as opposed to partialized, mass. For distances intermediate the points D_0 and D_a , fiber length is seen to increase in an exponential fashion with increasing free-fall distance. Although the stream is at least partially solid in this region, a disturbance sufficient to disrupt it is caused by the chosen deceleration, such as impingement upon a deflector plate or the like. At free-fall distances intermediate points D_a and D_b , indefinite lengths may be collected. When the stream is allowed to fall unhindered through distances greater than D_b , the tensile force due to increasing stream length (and, therefore, weight) is sufficient to cause stream breakage. Thus, at points below D_b , relatively uniform fiber lengths independent of free-fall distance may be obtained.

As will be illustrated, the relative and absolute positions of the points D_0 , D_a and D_b may be manipulated as desired by suitable variations of the process parameters. For example, the points D_a and D_b may occupy relative positions such that D_b occurs upstream of D_a ; in such case, there is no free-fall distance at which continuous length filaments can be collected. Such a circumstance may arise by numerous combinations of factors affecting the force system imposed upon the stream, but it is found to be a particular problem in the case of spinning high density

melts, especially in the larger diameter range. In such cases, the force system acting upon the stream must, if indefinite length production is to be obtained be modified in light of the present teachings such that the tensile break point D_b is caused to be shifted downstream relative to the point D_a and the demarcation between Zones A and B maintained at a position intermediate the deceleration break points D_a and D_b . Thus, the upstream portion denoted by Zone A must extend downstream of D_a and the downstream portion denoted by Zone B must extend upstream of D_b , with the result that a stabilized stream deviation is formed between the points D_a and D_b to thereby effect a smooth deceleration of the stream without breakage.

An effective aid in determining stream continuity relative to free-fall distance may take the form of a simple electrical continuity circuit tester, such as schematically diagrammed in FIG. 6. As there indicated, such a tester serves to electrically interconnect the collection surface with the melt crucible to thereby sense electrical continuity, or lack of it, between the collection surface and the spinning head through which an electrically conducting melt is being issued. Similar determinations are, of course, made by directly measuring the fiber lengths obtained at varying free-fall distances, but use of the continuity tester allows one to monitor stream continuity continuously and to modify spinning conditions accordingly.

As previously indicated, the fiber lengths obtained under various process conditions will vary in a characteristic manner with free-fall distance, as shown in FIG. 5. Indications of the mechanism by which stream breakup occurs in the regions above the impact breakage deceleration point D_a and below the point of tensile breakage deceleration D_b are obtained by relating fiber length to free-fall distance to indicate where and when stream breakage occurred, while examination of the ends of broken lengths serve to indicate stream state at the position of break as well as the rapidity of the break. In the lower portion of FIG. 5 is shown a recording of the continuity tester for the typical graph appearing thereabove. In relating this recording to the free-fall distance curve, it may be observed that the freeze point D_0 of the stream is the point below which fibrous shapes, as opposed to a molten mass, may be collected. Thus, when the stream is impinged upon an electrically conductive surface at distances less than D_0 , there is electrical continuity between the collection surface and the spinning head via the liquid stream, as indicated by the continuous positive deflection on the recorder curve. For free-fall distances between D_0 and D_a , the on-off shape of the continuity curve indicates that the impingement of the stream upon the collection surface initiates a disturbance sufficient to effect stream breakage at some point upstream of the collection surface. An examination of the fiber ends obtained by collection within this region indicates that stream breakage occurs very rapidly, but after solidification sufficient to retain a fibrous shape. Thus, the disturbances set up in the stream upon impingement on the collection surface at these free-fall distances are sufficient to physically disrupt the stream and only broken lengths can be obtained.

As indicated by the graph of FIG. 5, continuous fibers are obtained for free-fall distances between points D_a and D_b ; the continuity tester of course indicates electrical continuity within this region.

For distances greater than D_b , the stream undergoes breakage before impingement upon the collection surface. Because the stream accelerating force, normally due to gravity, has beyond this point become greater than the sum of stream strength and any upstream-acting drag force generated under the chosen spinning conditions, the stream is caused to break prior to contacting the collection surface. Thus, the recording indicates a continuously open circuit. The appearance of the ends on the broken

lengths obtained at free-fall distances greater than D_b indicate that they break in a very hot, solid region or in the liquid region; further, the lengths of the resultant fibers indicate that they break very near the freeze point.

As related in greater detail in the previously referenced copending application Ser. No. 863,311 the position of the point D_a and D_b may be shifted relative to the orifice and to one another. Any one or more of a number of spinning parameters are readily manipulated to attain the desired relationship. Among the more significant parameters, there may be mentioned those of reactant and coolant gas composition concentration and velocity in those cases where the liquid region of the stream is being stabilized by the formation of a film as it issues; also, the spinning or stream velocity and the spinning temperature may as well be varied to achieve similar results. For example, where the liquid region of the stream is film stabilized by a reaction between the stream and the spin gas, an increase in the reactant gas concentration may result in a stabilizing film of increased thickness and, consequently, strength, resulting in both an upstream shift of the impact break point D_a , as well as a downstream shift of the tensile break point D_b . Similarly, film thickness and strength may be increased by an increase in the spinning temperature to result in a more rapid rate of film formation; however, any gain in total stream strength may be compromised, if not more than offset, by the postponement of stream solidification due to the increased spinning temperature. A further mode of varying the spatial interrelationship of the points D_a and D_b is accomplished by varying the spinning or extrusion velocity (V_s). For example, with all other conditions fixed, an increase in spinning velocity is accompanied by an increase in both viscous drag and the rate of heat transfer to the spin gas; thus, by virtue of the buoying effect of increased drag, the tensile break point D_b is caused to shift downstream, while the increase in heat transfer is offset by the increased spinning velocity to normally result in a relatively very small down stream shift of the impact breakage point D_a to thereby enlarge the region within which indefinite lengths may be collected.

The foregoing are but limited illustrations of the great variety of manipulations which are readily accomplished to attain the desired arrangement of the points D_a and D_b . In all cases, the essential arrangement for continuous length production is one characterized by the point D_a occupying a position upstream of the point D_b with the demarcation between the differential force systems of Zones A and B being established therebetween.

In exemplifying the practice of the present invention, the simplified spinning assembly schematically depicted in FIG. 4 was employed. In the case of the lead/tin runs set out in Example I the assembly comprises a stainless steel melt crucible 10 enclosed by an upper header plate 12 and a lower orifice plate 14, each of which are in sealing engagement with crucible 10 to define a gas-tight melt chamber 16. Seated centrally of orifice plate 14 is a watch-sized jewel 18 formed of a material chemically compatible with the melt to be processed and drilled to provide a suitable spinning orifice 20. In the case of Example I a ruby jewel having an orifice diameter of 100 microns and a length/diameter ratio of unit was employed. Melting of the spin charge within chamber 16 was accomplished by means of electrical resistance heating elements 22 and charge temperature was monitored by thermocouple probe 24. Preferably, the spinning charge is melted under a vacuum just prior to effecting extrusion under an inert gas pressure, such as argon. This may readily be accomplished by the two-way valve arrangement indicated at 26, whereby chamber 16 may be alternately evacuated and pressurized to effect the desired rate of extrusion. In order to localize the various reactant and cooling gas mixtures employed, a Pyrex spin gas column 28 is arranged concentrically of stream 29 issuing from orifice 20. The spin gas mixture is supplied through conduit 30 to be gently

deployed over the spin gas column cross section by means of a gas distribution ring 32 provided with equi-spaced gas orifices 34; preferably, the orifices are oriented to discharge at a slightly downstream angle to assure effective spin gas circulation. The spin gas column 28 defining Zone A has an inside diameter of 6 inches and a length measured from orifice plate 14 of approximately 3 feet. Disposed immediately downstream of the spin gas column is the support gas column 36 provided with a pair of extending conduits 38 and 40 through which the support gas is introduced and extracted in a manner to effect the desired circulation, whether co-current, counter-current, or virtually stagnant. The direction and magnitude of the support gas velocity is of course determined according to the amount of viscous drag it is desired to impose upon the stream within Zone B. In the examples here reported, the spin gas column 36 had an inside diameter of 4 inches and length of approximately 4 feet and was of Pyrex construction. The lower extremity of spin gas column 36 tapers to a central outlet 42 through which the now solidified stream is withdrawn and taken up. The reactant/coolant gas mixture is normally introduced in the upper region of spin gas column 28 in a fashion such that the flow is a gentle sweeping one although, depending primarily upon melt density, stream diameter and spinning velocity, it may be found desirable to introduce the spin gas with a pronounced upstream or downstream velocity. Optionally, there may be provided a deflection plate 44 located at the demarcation between Zones A and B to invoke the desired initial deflection of the stream as it enters Zone B. In many operations, however, it will be found desirable to establish a sufficient differential in the force systems between Zones A and B such that the stream is readily contorted to the helical configuration when passing into Zone B without the aid of a deflection plate. As previously explained, this is accomplished by maintaining a drag force within Zone B sufficient to establish an angle of threshold deviation θ_1 small enough to be exceeded by incidental stream deviations.

As depicted in FIG. 4, the stream 29, on entering the higher drag Zone B becomes aerodynamically contorted to a substantially helical configuration to thereby define a drag-sustained accumulation within Zone B out of which the stream may be withdrawn without disruption. As before related, where it is desired to extract indefinite lengths, the demarcation between Zones A and B must be maintained at a point intermediate the points of impact and tensile breakage deceleration, it is to be emphasized that the above-described spinning assembly merely represents a simplified implementation of the present invention, the latter being in no way limited to the details of the apparatus. The length of the spin gas and support gas columns 28 and 36, respectively, defining Zones A and B may, by suitable variation of the process parameters as hereinbefore discussed, vary over wide ranges. Also, the depiction in FIG. 4 of a support gas column 36 of reduced diameter relative to the spin gas column 28 is but one technique for controlling viscous drag through a control of spin and support gas velocities. It will often be just as feasible to employ one continuous column length, the Zone B or lower region of which would contain, for example, a denser gas, or one moving at a lower co-current or higher counter-current velocity, as the case may be.

EXAMPLE I

The spinning assembly previously described with reference to FIG. 4 was employed and the spinning charge was in the form of a mixture of 62 weight percent lead/38 weight percent tin. The stream was film-stabilized by extrusion into an oxygen-containing atmosphere to form an oxide stabilizing film upon the stream as it was given issue. A reactant/cooling gas mixture of 7 vol. percent oxygen/93 vol. percent helium at room temperature was introduced through distribution ring 32 as a gentle sweep-

ing action at a flow rate of approximately 3.6 c.f.m. Atmospheric air was employed as the support gas within Zone B and was introduced as a counter-current flow of the magnitude set out in the accompanying table. Extrusion was through a 100 micron diameter orifice formed in a watch-sized ruby jewel, the orifice having an L/D ratio of unity to form a stream diameter of 85 microns. Preferably, heating to melt was accomplished under a vacuum of below 100 microns of mercury pressure and extrusion was commenced prior to introducing the spin gas into the spinning column. The gas distribution ring 32 was positioned approximately 30 cm. below the orifice. The helium flow rate, which functions as a coolant gas, was maintained constant at 3.3 c.f.m. to maintain a relatively constant heat transfer rate, the oxygen flow rate being varied to obtain the desired volume percent oxygen in the mixture.

The runs set out in the table below were made under the conditions specified wherein there was determined, for increasing extrusion or stream velocities, the minimum support gas velocity within Zone B necessary to sustain a helical deviation stabilized about the demarcation between Zones A and B.

SUPPORT GAS VELOCITY FOR HELIX STABILIZATION
85 μ Pb/Sn SUPPORTED BY AIR

Extrusion temperature (° C.)	Extrusion at—			Deflection plate	Support gas velocity ¹ (cm./sec.)
	Pressure (p.s.i.g.)	Rate (g./min.)	Velocity (cm./sec.)		
300.....	10	9.9	330	Yes.....	-335
300.....	10	9.9	330	No.....	-330
300.....	20	17.1	570	Yes.....	-270
300.....	20	17.1	570	No.....	-315
300.....	40	23.8	790	Yes.....	-254
300.....	40	23.8	790	No.....	-320
300.....	60	34.6	1,150	Yes.....	-200
300.....	60	34.6	1,150	No.....	-250
300.....	80	42.2	1,400	Yes.....	-110
300.....	80	42.2	1,400	No.....	-130

¹ The positive direction is downward or concurrent with the stream. Notice that for these experimental conditions, the minimum counter-current support gas velocity to maintain a helical configuration in Zone B, varies only slightly with the extrusion, or fiber, velocity. This is because of the changing momentum of the extruded stream and follows closely the theoretical requirements predicted by Equation 2. Notice also that the provision of a deflection plate intermediate Zones A and B slightly decreases the necessary relative gas/fiber velocities to maintain the fiber helical configuration as predicted by theory.

EXAMPLE II

The spinning assembly of Example I was employed with the following modifications: The crucible was entirely constructed of molybdenum, the orifice being formed in a molybdenum insert seated in the floor of the crucible and having a diameter of 4 mils and a length of 2 mils, to give an L/D ratio of 0.5; the support column 36 of FIG. 4 was removed, the stream being supported in stagnant room air on exiting the spin gas column 28; the crucible was inductively heated.

The melt composition was in the form of a 50 wt. percent aluminum oxide/50 wt. percent calcium oxide. In proceeding to spin, the melt was heated from room temperature to approximately 1,000° C. under a high vacuum of less than 10 microns; further heating from 1,000° C. through the melting point of approximately 1360° C. to a spinning temperature of approximately 1650° C. was under one atmosphere of argon; extrusion pressure was varied within the range of 30 to 100 p.s.i.g. Propane was employed as the film stabilization gas and introduced at a rate of 4 c.f.h. to form a carbon film upon the stream through pyrolysis of the propane, and; the cooling gas was helium supplied at a rate of 70 c.f.h. Again, the support gas was in the form of stagnant room air existing immediately below the spin gas column 28, to thereby define Zone B.

Proceeding under these conditions, it was found that when the spinning or stream velocity (V_s) was maintained within the range of from 600 to 680 cm./sec., the stream was observed to remain quite straight in passing through the helium/propane atmosphere within the spin

gas column 28. When the stream emerged from the helium/propane mixture into stagnant room air at approximately 3 ft. from the orifice, the resultant increase in viscous drag force caused the stream to assume a helical configuration, the upper extremity of which was stabilized about the lower extremity of the spin gas column. As before explained, this result was due to the fact that the drag force below the spin gas column was of such magnitude as to sustain a rate of helical propagation greater than the stream velocity, whereas the viscous drag within the spin gas column was inadequate to sustain a propagation rate exceeding stream velocity.

Upon further increasing stream velocity to a value greater than 680 cm./sec., the stream was observed to be deflected into a helical configuration with the helium/propane atmosphere of the spin gas column. Only by the utilization of a co-current spin gas velocity sufficient to maintain the relative velocity between the stream and the spin gas at a value less than 680 cm./sec. within Zone A, would the resultant stream remain undeviated due to viscous drag and fall in a straight path until exiting the spin gas column to thereafter assume the helical configuration.

The foregoing has disclosed an improved stabilization process by which filament take-up can be practiced as the filament forms. In a first region the drag-sustained deviations are prevented from moving up into the fragile liquid portion of the stream while simultaneously the drag interaction is utilized to form a helical-like configuration in a second region beneath the first region. Thus, a tension necessary for some take-up techniques can be applied to the stream in the second region without affecting the liquid portion of the stream.

While only certain specific embodiments and features of the present invention have been described, it will be understood by those skilled in the art, that various changes and modifications may be made without departing from the invention as set forth. Accordingly, all such modifications and changes as fall within the true spirit and scope of the present invention are intended to be covered by the appended claims.

We claim:

1. In a process for forming filaments from a normally solid inorganic melt having a viscosity not greater than 10 poises including the steps of

- extruding a free molten stream from the melt,
- exposing the extruded stream to a gaseous atmosphere to form a film about the periphery of the stream which film has sufficient strength to stabilize the stream against surface tension-induced breakup pending solidification,

the improvement which comprises passing the stream into a first gaseous zone which extends downstream below the point at which the stream becomes solidified so as to cause the velocity of the stream deviations resulting from the drag interaction between the solidified stream and said first zone gas to be less than the stream velocity in said first zone, and passing the stream into a second gaseous zone immediately below said first zone so as to cause the velocity of the stream deviations resulting from the drag interaction between the stream and said second zone gas to be greater than the stream velocity in said second zone thereby resulting in the stream in said second zone attaining a helical-like configuration and whereby tension, may be applied thereto without transferral of the tension to the molten portion of said stream.

2. The process of claim 1 wherein the boundary between the first gaseous zone and the second gaseous zone lies intermediate a first point (D_a) upstream of which stream deceleration causes disruptions in the stream continuity and a second point (D_b) downstream of which the net tensional force acting upon the stream causes

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disruptions in the stream continuut, D_a being upstream of D_b .

3. The process of claim 2 wherein the viscosity of the gas in the second gaseous zone is greater than the viscosity of the gas in the first gaseous zone.

4. The process of claim 2 wherein the density of the gas in the second gaseous zone is greater than the density of the gas in the first gaseous zone.

5. The process of claim 2 wherein the gas in the second gaseous zone has a counter-current flow.

6. The process of claim 2 wherein the gas in the first gaseous zone has a co-current flow and the velocity of the gas in the second gaseous zone is less than the velocity of the gas in the first gaseous zone.

7. The process of claim 2 wherein the stream is a metal or alloy thereof.

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