HIGH SPEED PRODUCTION OF FILAMENTS FROM LOW VISCOSITY MELTS

Inventor: Emerick J. Dobo, Cary, N.C.
Assignee: Monsanto Company, St. Louis, Mo.
Filed: Dec. 29, 1972
Appl. No.: 319,133

U.S. Cl. 65/1, 65/5, 65/16, 164/82, 264/DIG. 19, 264/176 F, 425/464
Int. Cl. C03b 37/02, D01d 11/00
Field of Search 65/1, 5, 16; 164/82, 86; 264/176 F, DIG. 19; 425/464

References Cited
UNITED STATES PATENTS
2,077,373 4/1937 Formhals 425/461 X
3,423,266 1/1969 Davies et al. 264/176 F X
3,613,158 10/1971 Mottern et al. 264/176 F X

ABSTRACT
Improvements in method and apparatus are provided for the fabrication of fibers and filaments from extremely low viscosity melts by continuous extrusion. Realizable production rates are increased up to threefold over existing capability. This is made possible by an improved orifice assembly which permits very high attenuation of the filamentary jet without an attending disturbance of the jet stream. In practice, the filamentary jet is brought into a zone of pressurized inert gas upon emergence from the extrusion orifice. The jet is then passed with the inert gas in concurrent flow through a supersonic nozzle and into a first zone occupied by a film-forming, gaseous atmosphere followed by passage through a converging orifice and into a second zone of film-forming gas.

10 Claims, 3 Drawing Figures
HIGH SPEED PRODUCTION OF FILAMENTS FROM LOW VISCOSITY MILTS

BACKGROUND OF THE INVENTION

This invention pertains to improvements in methods and apparatus for fabricating fibers and filaments by the continuous extrusion of very low viscosity melts. That is, the essentially inviscid melts of materials such as those of the various metals, metal alloys, metalloids and ceramics where poise measurements are less than 10 and often less than 1. In particular, process economy is greatly improved by a substantial increase in realizable production rates. Moreover, the resulting product exhibits superior structure characteristics in that porosity is greatly reduced.

U.S. Pat. No. 3,658,979 — incorporated as a part of this description by way of reference — sets forth the basic precepts by which fibers and filaments may be formed through an extrusion of essentially inviscid melts. In brief, a low viscosity melt is extruded at an appropriate velocity into a selective atmosphere. When the hot jet issuing from the extrusion orifice contacts the atmosphere a reaction occurs which results in the formation of a film or protective sheath about the jet surface. This film, called the stabilizing film, stabilizes the filamentary jet or stream against break-up from the forces of surface tension until sufficient heat can be removed to affect a phase change to the solid state. The stabilizing film must, of course, be formed very rapidly. Moreover, the film must be in the solid state at the high temperatures in which it is formed. The film must also be substantially insoluble in the molten jet at the extrusion temperatures to insure continuity of its function.

Since the introduction of this remarkable process, considerable effort has been expended towards improvement with particular emphasis being given to finding a means for increasing rates of productivity. Generally, filament attenuation is useful for this purpose. However, the nature of essentially inviscid materials precludes the attainment of attenuation by the conventional drawing technique as is practiced in the fabrication of fibers and filaments from polymeric materials.

U.S. Pat. No. 3,645,657 describes a means by which filamentary jets, formed by the extrusion of essentially inviscid melts, may now be attenuated. In accordance with the teachings of this patent, attenuation is accomplished by extruding the jet initially into a zone of pressurized inert gas followed by passage into the film-forming atmosphere. In practice, there is used a specialized orifice assembly having two concentric plates disposed in a stacked relationship, one above the other. Each plate contains a centrally disposed orifice with the orifice of one plate being in co-axial alignment with that of the other. The orifice of the uppermost or first plate is of straight bore and serves as the melt shaping die or extrusion orifice. The orifice of the second plate is larger in diameter than that of the first and has a straight or a tapered bore. The second plate, referred to as the "gas plate" is provided with gas inlet ports and gas distribution means in the form of a gap space, which defines an essentially enclosed chamber between the opposing faces of the two plates.

In operation, a quantity of inert gas is supplied under pressure through the inlet port of the gas plate and contacts the jet as it emerges from the extrusion orifice in a direction perpendicular to the jet path. The inert gas is then caused to change direction and flow countercurrently with the jet through the gas plate exit orifice and thence, into a reactive atmosphere. The resulting attenuation occurs in part as a result of the viscous drag interaction of the inert gas with the jet.

While the realizable benefits of the method and apparatus as set forth in U.S. Pat. No. 3,613,158 are clearly evident, under certain conditions of practice disturbances of the jet have been noted which cause an undesired sinuous effect on the stream. This has been observed particularly when employing conditions required for the attenuation of the jet diameter to less than half of the orifice diameter. Such large attenuations require a high pressure drag across the gas plate which frequently results in the continuity of the jet being disrupted to the point that only staple is formed rather than the desired continuous filament. When continuous filament does form, it is found to be sinuous in appearance and weakened by the stresses of solidification. Such disturbance is for the most part dependent upon the extent of the difference in relative velocity between the inert gas and the jet. It follows then that disturbances are more severe in larger attenuations which require the higher inert gas velocities.

This problem of disturbances developing in the jet stream when attenuating the same with a co-current flow has been recognized and dealt with in the U.S. Pat. No. 3,613,158. The solution provided therein is to strip the co-currently flowing inert gas from the molten stream a short distance downstream from the initial point of impingement against the jet. This is accomplished by applying suction to an enclosed chamber positioned beneath the chamber into which the inert gas is supplied.

The technique, as described in U.S. Pat. No. 3,613,158 has been found to be relatively effective, but it is not without disadvantages. One is that the pumping means necessary for creating the required suction adds to the complexity and cost of the operational equipment. Moreover, it has been found that effectiveness becomes greatly impaired when operating at higher production rates, i.e., in excess of 1,500 feet per minute of filament production.

Since the advent of the capability for jet attenuation, filament productivity rates have been raised to a level in the order of 1,300-1,400 feet per minute — the maximum attainable with known and existing equipment. Yet there has been a need for even higher rates of productivity in order to make over-all process economics commercially attractive. To accomplish this, not only must high attenuations be achieved, but this must be accomplished without an attending stream disruption.

Accordingly, the principal object of this invention is to provide process and apparatus improvements which permit productivity rates of up to 4,200 feet per minute when fabricating fibers and filaments from essentially inviscid melts — a three fold increase over that presently possible.

It is a further object of the invention to achieve large scale attenuation of low viscosity jets not heretofore attainable and without an attending disturbance or disruption of the filamentary jet. It is a still further object of the invention to improve the structural characteristics of the fila-
The above objects of this invention have been accomplished by an improved process which comprises the following steps in sequence: (1) extruding continuously an essentially inviscid melt through a shaping die to form a filamentary jet; (2) passing the filamentary jet immediately upon issue from the shaping die into a zone occupied by a pressurized inert gas; (3) forwarding the jet in co-current flow with the pressurized inert gas through a supersonic nozzle and into a first zone occupied by a gaseous atmosphere capable of causing a film to form about the jet surface by reaction therewith; whence (4) passing the filamentary jet through a converging passageway into a second zone occupied by the film forming atmosphere.

The novel apparatus by which this procedure may be carried out will be best understood by a description of the accompanying drawings in which:

FIG. 1 is a schematic vertical cross-section of a typical filament extrusion apparatus employing an orifice assembly in accordance with the present invention.

FIG. 2 is an enlarged, partial view of the orifice assembly of FIG. 1.

FIG. 3 is an enlarged partial view of the gas plate orifice in the orifice assembly of FIG. 1.

DESCRIPTION OF THE DRAWINGS

FIG. 1 depicts a crucible 10 enclosing a quantity of molten essentially inviscid material 11. Functionally as part of the base of crucible 10 is an orifice plate 12 having an extrusion orifice 13. Spaced beneath plate 12 is a gas plate 14 having a convergent-divergent shaped orifice 15 which is aligned substantially coaxial with orifice 13. Plates 12 and 14 define an essentially enclosed chamber 16, which can be referred to as an inert gas zone.

Beneath gas plate 14 is a third plate 17 hereinafter called a stream control plate. Stream control plate 17 has an orifice or throat 18 which is aligned substantially coaxial with throat 15 (and consequently with orifice 13). The walls of orifice 18 converge in the direction of its exit with the included angle of convergence being between 7° and 20°. Stream control plate 17 and gas plate 14 define a second substantially enclosed chamber 19, which can be referred to as a first reactive gas zone. Pedestal 20 supports the entire apparatus and also defines cavity 21, which can be referred to as the second reactive gas zone, since the molten jet further reacts therein with a film forming gas.

In operation, a positive pressure head is supplied to molten material 11, generally by means of an inert gas under pressure, such as for example, argon. The jet 22 is thus caused to issue from the extrusion orifice 13 into chamber 16. Chamber 16 is provided with a quantity of inert gas which is supplied under pressure through inert gas line 23. The inert gas is constrained to move laterally between orifice plate 12 and gas plate 14 and thus contacts the emerging jet 22 in a direction initially normal to the path of jet 22. This flow is in a large measure self-distributing towards symmetrical flow. The inert gas then flows co-currently with jet 22 through the gas plate throat 15 and into chamber 19. The nature of the inert gas is not critical so long as the gas is inert to the extruded materials, the orifice plate and other parts of the extrusion apparatus. Helium and argon have been used successfully with helium being particularly suitable.

Chamber 19 is provided with a quantity of gas reactive with jet 22 via gas line 24. The reactive film-stabilizing gas contacts jet 22 at the entrance of orifice 18 and is at a flow rate sufficient to penetrate the shroud of inert gas which has been caused to envelope the jet as it issues from gas plate orifice 15. A further quantity of reactive gas is supplied by gas line 25 into cavity 21 for contact with jet 22 proximate to the exit of orifice 18. The nature of the reactive gas is not critical so long as it is capable of forming a film about the surface of molten jet 22. In many instances oxidizing gases such as carbon monoxide and air have been successfully employed. For other suitable film-forming gases that may be used see U.S. Pat. No. 3,658,979.

FIG. 2 illustrates the general geometrical relationship between plates 12, 14 and 17 together with their respective orifices. Although the diameter of the throat section (most narrow section) of gas plate orifice 15 may be larger than the exit diameter of extrusion orifice 13, best results are obtained when it is of an equal or lesser diameter than that of the exit of orifice 13. Particularly good results may be obtained when the ratio of the exit diameter of orifice 13 to the throat diameter of orifice 15 lies in the range of from about 1.1:1.0 to 1.5:1.0. The length of orifice 15 is generally maintained at from about 5 to 100 times greater than the exit diameter of orifice 13. As noted, orifice 18 converges in the direction of its exit at an included angle of from about 7° to 20°. It is generally desirable although not critical that the entrance diameter of orifice 18 be from about 2 to 5 times larger than the throat diameter of gas plate orifice 15.

The gap distance of gap 31 between orifice plate 12 and gas plate 14 should be substantially equal to the diameter of gas plate throat 15. On the other hand, the dimensions of gap 32 between gas plate 14 and stream control plate 17 is not considered to be critical. However, enough space should be provided to accommodate a sufficient quantity of reactive gas to penetrate the inert gas which flows co-currently with jet stream 22. Generally, it has been found that a gap distance of from about 5 to 20 mils between gas plate 14 and stream control plate 17 in the vicinity of their respective orifices is satisfactory.

FIG. 3 illustrates gas plate 14 and its shaped orifice 15 schematically in an enlarged vertical section. The entry area or convergent section 28 is rounded gently to reduce friction. The extent of convergence is not critical, it being merely necessary that the orifice walls converge in some degree at the entry. The convergence terminates at throat section 29 from where the walls diverge to form divergent exit section 30. The included angle of divergence in this section should be between 4° and 12°, with from 6° to 8° being of preference for attenuation at the higher speeds. Best results are achieved when divergent section 30 is of greater length than convergent section 28, and particularly when the length is from 10 to 20 times greater. Arrows 26 and 27 illustrate the flow paths of the inert and reactive stabilization gases, respectively.

The following example illustrates the results achievable through practice of this invention.
EXAMPLE
An apparatus such as depicted in FIG. 1 was employed to form filaments by extruding the melt of steel alloyed with 1.0 percent by weight of aluminum at a realized production rate of 3500 feet per minute.

The orifice assembly used was of a design as typified by FIG. 2 of the drawings. The orifice plate 12 was 125 mils thick with the filament shaping orifice 13 in the plate measuring 8 mils in both length and diameter, i.e., having an aspect ratio L/D of 1. Gas plate 14 was also 125 mils thick with the throat of the supersonic nozzle 15 therein being 8 mils in diameter or equivalent to the diameter of filament shaping orifice 13. Stream control plate 17 was 62 mils thick and orifice 18 therein had an exit diameter approximately 4 times that of the throat diameter of convergent-divergent orifice 15. An included angle of 15° was formed by the converging walls of orifice 18.

In operation, an argon gas head pressure of 100 p.s.i.g. was used to force the melt through the orifice of extrusion plate 12 to form a filamentary jet emerging into gas space 16 between plates 12 and 14. Gap 16 was supplied with helium at a pressure of 76.8 p.s.i.g. and at a flow rate of 301 cm³/min (STP). The pressurized helium contacted the jet at an angle normal to its path of movement and then flowed co-currently with the jet through supersonic nozzle 15 in gas plate 14. Upon exit from nozzle 15, the filamentary jet entered gas space 19, which was supplied with carbon monoxide as the film-forming gas. The carbon monoxide flow rate into gas space 19 was 5080 cm³/min (STP). The jet then passed through stream control orifice 18 and into cavity 21 where additional carbon monoxide was supplied at a rate of 1630 cm³/min (STP). The film stabilized jet which solidified upon cooling was then taken up as a filamentary product. During the course of this high-speed extrusion, the molten jet remained continuous and did not deviate from a straight path. The steel wire produced without further treatment had an average tensile strength of 248,000 p.s.i. The porosity of the wire as measured by the Quantimet machine of Metals Research, Ltd. averaged 0.05 percent.

An eighteen fold increase in power is required to achieve a 3500 ft/min. production rate when compared with 1350 ft/min. — the optimum attainable by prior practice. By the method of this invention most of the power needed comes from the pressure drop across the gas plate. A large pressure drop occurs as a result of the flow of the pressurized inert gas through the supersonic or convergent-divergent nozzle orifice of the gas plate. In addition to exerting a piston-like pressure on the molten stream, expansion of the gas in the nozzle contributes substantially to the velocity of the stream or jet. That is; with a large pressure drop there is a corresponding reduction in the enthalpy of the gas. In all probability the energy released is initially transferred to the gas and then through viscous drag a part of it is in turn transferred to the molten stream where it acts to power a velocity increase. Calculations show only a small percentage (less than 5 percent) of the enthalpy released by the gas through pressure drop in the gas plate is converted to kinetic energy in the filamentary jet. Even with this poor conversion, approximately one-third to one-half of the power contained in the jet, when extruding at 3,500 ft/min., comes from this conversion of enthalpy. Because the thermodynamics of

As seen in Table I, there appears to be some deterioration of tensile strength as spinning speed increases from 3,000 to 3,700 rpm. Nevertheless, up to a spinning speed of 3,700 rpm, these figures are as good as 240,000 psi, the average value of the control.

The filamentary products produced in accordance with this invention have a myriad of practical uses. For example, small diameter copper and aluminum wire find wide use in various electrical devices. To mention but one other, filamentary steel is becoming even more widely used as a reinforcement element in the manufacture of modern automobile tires as well as tires for other vehicles.

Having thus described the invention using for illustrative purposes particular embodiments thereof, it will be apparent that modifications and variations are possible in the light of the teachings presented herein. Therefore, the invention is limited in scope only as defined by the following claims.

What is claimed is:
3,811,850

7

1. An orifice assembly for extruding a filamentary jet from an essentially inviscid melt to form fibers and filaments, which comprises in combination:
   a. a first plate;
   b. a second plate, said second plate being spaced beneath said first plate in a stacked relationship therewith;
   c. a third plate, said third plate being spaced beneath said second plate in a stacked relationship therewith and with said first plate;
   d. a first orifice, said first orifice being centrally disposed in said first plate;
   e. a second orifice, said second orifice being centrally disposed in said second plate in co-axial alignment with said first orifice, said second orifice having a nozzle configuration with a convergent entry section, an intermediate throat section and a divergent exit section, said exit section having an included angle of divergence of between 4° to 12°;
   f. a third orifice, said third orifice being centrally disposed in said third plate in co-axial alignment with said first and second orifices, said third orifice having walls which converge towards the exit thereof at an included angle of between about 7° to 20°;
   g. a first substantially enclosed chamber, said chamber being defined by a gap space between the opposing faces of said first and second plates, said gap space having a vertical distance which is substantially equal to the diameter of the throat section of said second plate orifice;
   h. a means for supplying an inert gas under pressure to said substantially enclosed chamber and into said second orifice;
   i. a second substantially enclosed chamber, said chamber being defined by a gap space between the opposing faces of said second and third plates;
   j. means for supplying a gas reactive with said filamentary jet to said second substantially enclosed chamber and into said third plate orifice.

2. The orifice assembly of claim 1, wherein the length of said second orifice is from about 5 to 100 times greater than the exit diameter of said first orifice.

3. The orifice assembly of claim 1, wherein the diameter of the throat section of said second orifice is substantially equal to the exit diameter of said first orifice.

4. The orifice assembly of claim 1, wherein the ratio of the exit diameter of said first orifice to the diameter of the throat section of said second orifice is from about 1.1:1.0 to 1.5:1.0.

5. The orifice assembly of claim 1, wherein the included angle of divergence in the divergent exit section of said second orifice is in the range of from about 6° to 8°.

6. The orifice assembly of claim 1 wherein the walls of the third plate orifice converge toward the orifice exit to form an included angle of from 10° to 15°.

7. In a process wherein fibers and filaments are formed by extruding a molten filamentary jet from an essentially inviscid melt and wherein the molten filamentary jet is stabilized against surface tension breakup by passage through a reactive film-forming atmosphere, an improvement which comprises:
   a. extruding the molten filamentary jet directly into a zone of pressurized inert gas;
   b. forwarding the jet together with the pressurized inert gas in co-current flow through a supersonic nozzle and into a first zone occupied by a film-forming gaseous atmosphere; and thence
   c. passing the filamentary jet through a converging passageway into a second zone of said film-forming atmosphere.

8. The improved process of claim 7, wherein said molten filamentary jet comprises a metal.

9. The improved process of claim 7, wherein said molten filamentary jet comprises a metal alloy.

10. The improved process of claim 9 wherein said metal alloy consists of steel and aluminum.

* * * *