

June 3, 1969

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3,448,185

MELT-SPINNING OF FILAMENTS

Filed March 28, 1967

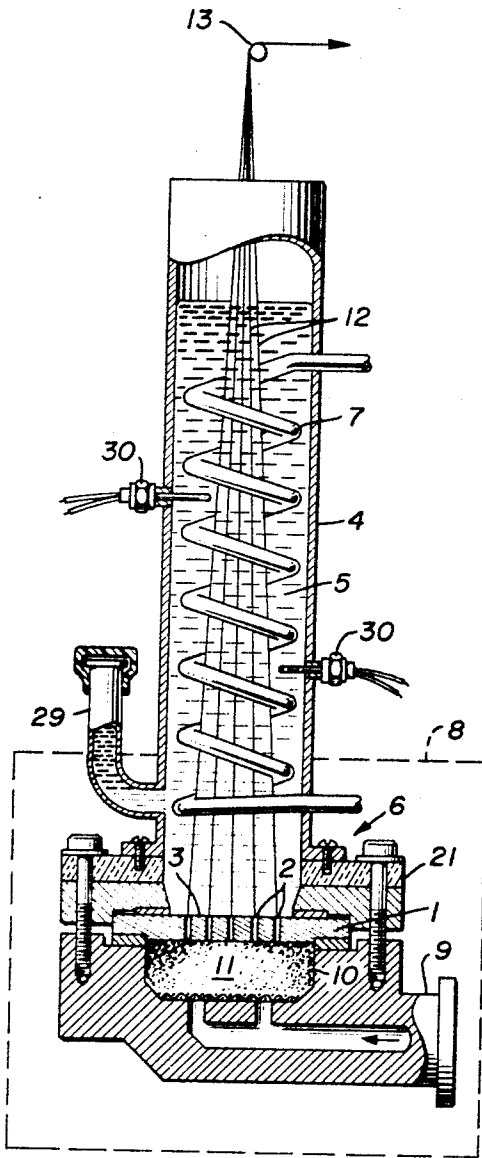


FIG. 1.

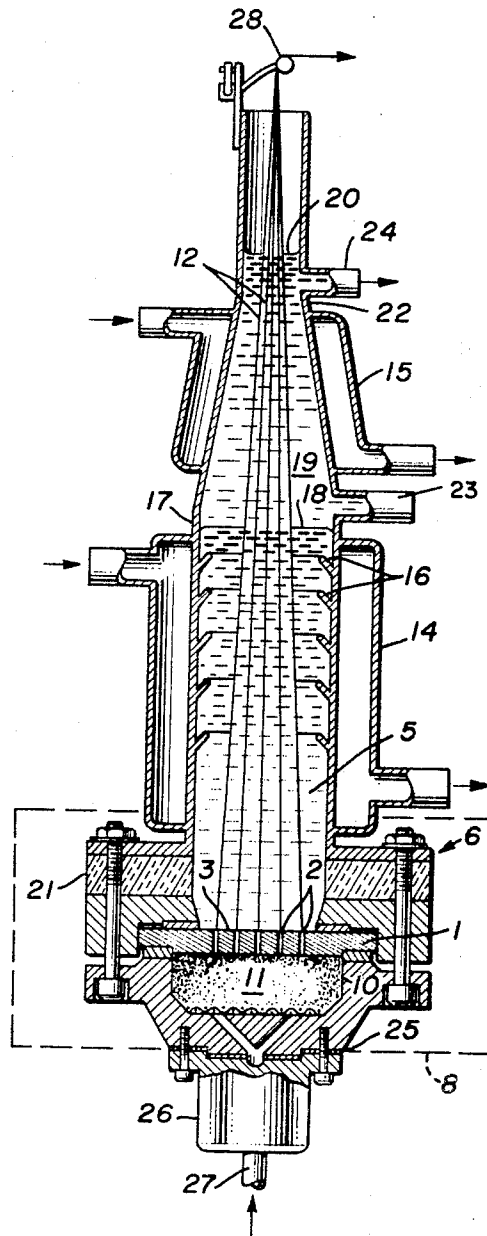


FIG. 2.

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MELT-SPINNING OF FILAMENTS

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Filed Mar. 28, 1967, Ser. No. 626,517

Int. Cl. D01d 5/08

U.S. Cl. 264—178

10 Claims

ABSTRACT OF THE DISCLOSURE

Melt-spun filaments, particularly of highly viscous materials, are produced by extruding molten fiber-forming polymeric material through a spinneret vertically upward through a layer of liquid metal that is in direct contact with the spinneret. A second or third stratum of other immiscible liquid may float on the liquid metal. Heat is transferred from the extruded streams of molten polymer to the liquid metal primarily by conduction to solidify the streams into textile filaments.

BACKGROUND OF THE INVENTION

The present invention relates to melt-spinning textile filaments. More particularly, it relates to a method and apparatus for melt-spinning textile filaments from fiber-forming material.

In the production of man-made filaments there are three basic procedures employed. These are known as wet spinning, dry spinning and melt-spinning. A basic technical difference between wet spinning or dry spinning and melt-spinning is that in the former two procedures filaments are solidified by the removal of material such as a solvent or the concentration of material in the filaments is changed, whereas in the latter only thermal energy is dissipated from the filaments.

Many factors can be varied independently that affect the wet or dry spun yarn properties, such factors as composition, concentration, diffusion environment, temperature, contraction, etc. In melt spinning the possible variable physical conditions are much more restrictive. Heat flows from the filaments under the potential of a temperature difference. A major problem is the establishment and maintenance of the requisite temperature difference. Heat energy within the filaments above certain temperature levels must pass through the external surface of the filament. Hence, dissipation of heat from the surface of filaments undergoing melt-spinning which is referred to as the quenching operation is the only factor that admits of variation from a practical standpoint.

Common quenching methods dissipate heat from the surfaces of the filaments by convection, a cool gas, such as air, being blown over the surfaces. In melt-spinning quench apparatus various geometrical and physical arrangements have been suggested, such as concurrent, counter-current or cross-current flow of the cooling gas relative to the moving filaments. While using a cooling gas is the most common practice, the heat capacity and thermal conductivity of all normal gases are relatively low so that certain real limitations exist for this general method of quenching.

A number of physical arrangements and methods have been devised for quenching of melt-spun filaments with a liquid, usually water or a combination of a cooling gas and liquid. Compared with common gases the thermal conductivity and heat capacity of liquids are many times greater. The thermal properties of liquids are, therefore, favorable for quenching of melt-spun filaments; but, there are other complicating factors. The molten filaments usually are passed down into a pool of liquid and are pulled out through the surface thereof at some other point. The

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specific gravity of many molten polymers is less than one so that the filaments tend to float as they enter the water or other commonly used liquids; furthermore, the surface tension of water is also relatively high. For these reasons considerable force is required to pull the molten filaments into and through the liquid just at the time the filament is least capable of sustaining tension; hence, the filaments tend to wander around, thereby causing irregularities to result in the yarn. Further, the frictional drag of liquids on moving filaments is an order of magnitude higher than the drag of a gas applied to the same filament area.

For the just-mentioned reasons liquid quenching of melt-spun filaments has never been practicable from a commercial standpoint with light denier textile yarns but has been useful only for yarns of 100 denier per filament or greater and only then if comparatively low speeds are employed.

It is an object of the present invention to provide liquid-quenching of melt-spun filaments useful in the production of both light and heavy denier yarns which exhibit new and improved physical properties not otherwise attainable.

Another object is to provide a novel method and apparatus for quenching melt-spun filaments that permit a wider range of quenching conditions than are normally possible and which maintain above-atmospheric pressure on the extruding filaments while excluding air or other degrading agents from directly contacting the molten filaments.

SUMMARY OF THE INVENTION

In accordance with the present invention a method is provided for producing melt-spun filaments wherein the solidification of the same is accomplished by liquid quenching. The method involves extruding at least one stream of molten fiber-forming polymer from a spinneret. A bath of liquid metal is maintained in direct contact with the extrusion face of the spinneret. The extruded streams are passed generally vertically upward through the liquid metal in heat exchanging relationship therewith to cause the filaments to become solidified by the transfer of heat from the streams to the liquid metal primarily by conduction. Thereafter, the resulting filaments are removed from the liquid metal. The method brings the nascent molten filaments into direct contact with the liquid quenching medium without intermediate contact with air or other gases.

Apparatus is provided for accomplishing the above objectives. The apparatus includes a source of molten fiber-forming polymeric material moving under proper extrusion pressure, a spinneret having at least one extrusion orifice providing passage for the molten material from the supply face thereof to the extrusion face thereof in an upward direction is employed between the source of material and an upright enclosure or vessel above the spinneret. The enclosure maintains the liquid metal in direct contact with the extrusion face of the spinneret. Means are employed for removing from the liquid metal the heat built up therein due to the quenching of the filaments. Means are also provided for removing the thus-formed filaments upwardly through the liquid metal bath and from the enclosure. The spinneret can form the bottom of the vessel containing the liquid. The liquid metal contacts the filaments moving vertically upward and serves as a heat transfer medium between the filaments and an auxiliary cooling fluid that maintains the liquid metal at the desired temperature.

DESCRIPTION OF THE DRAWING

In the drawing:

FIGURE 1 is a cross-sectional view of one embodiment

of the apparatus for melt-spinning thermoplastic material; and

FIGURE 2 is a cross-sectional view of a second embodiment of the apparatus.

With reference first to FIGURE 1, the apparatus includes a spinneret 1 having a plurality of orifices 2 permitting passage therethrough of spinnable thermoplastic material. The extrusion face 3 of the spinneret is preferably horizontally disposed so that vertically upward spinning can be accomplished. The face will ordinarily serve to form the bottom of the enclosure or vessel 4 in which liquid metal quench fluid 5 is contained. As shown, the enclosure is cylindrical in shape and is attached in sealed relation with melt-spinning block 6. Mounted concentrically with the container walls of the enclosure is a coolant coil 7 in the form of a circular helix. This assembly is supported by the spinning block which in turn is supported by framework (not shown). The spinning base and the lower portion of the vessel are surrounded by thermal insulation and conventional electrical heaters within the dotted boundary 8, details of which are not shown. The cylindrical vessel is fitted with a metal fluid 5 that is liquid under normal operating conditions. Molten thermoplastic material is pumped through conduit 9, this normally being accomplished through the use of a metering pump (not shown). The molten material moves through a spinning cavity 10 filled with inert bodies 11, usually sand, which exerts a filtering and shearing action on the material. The material extruded through the orifices of the spinneret is solidified into filaments 12 which are withdrawn from the metal-containing bath by any suitable yarn forwarding means. Normally, element 13 is a rotatable guide with the forwarding means in the path beyond it. However, element 13 can be driven and constructed to forward the yarn, if desired. Coolant flows through coil 7 and is used to remove heat from the quenching metal.

FIGURE 2 illustrates other useful features of the apparatus and method of the invention. In this second embodiment, coolant jackets 14 and 15 rather than a coil are used. Only two jacketed sections are shown but three or more are frequently desirable to give better control of the desired temperature gradient along the quench liquid-containing vessel. Fins 16 integral with the jacketed wall of the lower section 17 of the vessel increase the effective heat transfer area on the liquid metal side to provide better control and less abrupt changes in the temperature gradient. Above the free surface 18 of the liquid metal 5 is a second liquid 19 with upper free surface 20. Coolant through the upper jacket 15 dissipates heat principally from the upper stratum of liquid; and coolant in jacket 14 controls heat removal from the liquid metal stratum.

An insulating ring 21 made of transite or similarly strong material separates the metal wall of the quench vessel from the lower portion of the spinning block to avoid excessive heat loss therefrom. A ring thickness of $1\frac{1}{2}$ to 3 inches is ordinarily suitable.

Preferably, the upper section 22 of the vessel in accordance with the second embodiment converges in order to reduce the volume of liquid and to reduce circulation thereof induced by the drag of the vertical movement of the filaments. Inlet 23 and outlet 24 are provided so that the upper liquid 19 can be replenished to makeup liquid carried away by the threadline and for controlling the liquid surface height. Ordinarily, liquid 19 is circulated at a very low rate by a pump (not shown) with flow concurrent with the threadline movement, although countercurrent flow may often be preferred in order to change the temperature gradient and to aid in the removal of any solids accumulated at the interface between the two liquids.

The spinning block 6 in FIGURE 2 has a flat pump pad 25 to which a conventional gear type metering pump 26 is secured. Molten polymer is fed through conduit 27

to the pump which delivers the polymer through the sand pack 11 and the orifices 2 to form filaments 12. Upper threadline guide 28 is mounted on a pivotal arm so that it can be easily moved aside during startup of the spinning unit.

In operation, the spinning block 6 is heated to normal spinning temperature. Liquid metal is added. In FIGURE 1 tube 29 is used for accomplishing this; and addition of the liquid is continued until a layer about $\frac{1}{4}$ to $\frac{1}{2}$ inch deep covers the spinneret face. The metering pump is started delivering a stream of molten polymer that flows through conduit 10 into the filter cavity that is filled with sand or metallic particles that serve to filter and distribute polymer to the spinneret. Auxiliary distribution plates upstream of the spinneret may also be used. When the first flow of polymer collects on the surface of the liquid metal, the metering pump is immediately stopped. Then, liquid metal preheated to the spinneret operating temperature is added to raise the metal up to the desired level. The metering pump is started again; and liquid coolant is admitted slowly to the coolant coil flowing countercurrently to the extruding molten filaments that collect at the surface of the metal bath. The molten polymer is scooped up periodically as the coolant flow rate is gradually increased. When the filaments first appear solidified at the pool surface, the coolant flow rate is held constant. The solidified filamentary mass is pulled from the bath and taken up and away with a conventional air aspirator sucker gun. The filaments are brought upward and over the guide and forwarded for take-up in an orderly manner. Before take-up, the filaments may be treated such as by drawing, applying a finish or lubricant thereto, washing, etc. Heaters surrounding the spinning block and coolant flow rate are now adjusted until the temperature indicators at several points along the liquid metal bath, such as thermocouples 30, indicate that the desired temperature gradient is established. Once started, the apparatus may be operated almost indefinitely without shutdown. However, when shutdown is necessary, the metering pump is stopped and liquid metal is drained to the lowest level permitted by tube 29. The residual liquid metal can be sucked out by an aspirator to a suitable container. A plugged hole may be drilled in the retainer ring at the plane of the spinneret face so that the liquid metal can be drained directly.

Any suitable coolant can be used and the heat exchange requirement is not normally a problem. The heat capacity of the preferred coolants, water and lower polyhydric alcohols such as ethylene glycol and glycerine, is from 5 to 10 times greater than the heat capacity of most organic thermoplastic fiber-forming polymers. Consequently, only a low coolant mass flow rate is required to quench the filaments.

Many liquid metals are useful in the practice of the invention. The essential requirements are that the freezing point of the metal be substantially lower than the freezing point of the polymer being spun into filaments and that there be no adverse chemical interaction between the polymer and metal under the given operating conditions. Generally, it is desirable that the metal be chemically inert with respect to the molten polymer although very limited reaction at the polymer-metal interface can be tolerated.

For general applications it is highly desirable to use liquid metal whose freezing point is lower than the boiling point of water so that a water layer or aqueous emulsion may be applied above the liquid metal. Elemental gallium is highly suitable but is quite expensive. A wide range of liquid metal alloys are possible with low freezing points in which tin, lead, bismuth and cadmium are combined in various proportions, such as in Wood's metal, Lipowitz's metal and Rose's metal. (See, for example, Perry, Chemical Engineers Handbook, 3rd edition, p. 454, McGraw-Hill.) The vapor pressure of most

of the useful metal alloys is very low at spinning temperatures used for organic polymer, but it is desirable to always use at least a shallow stratum of a second liquid on the metal to reduce direct contact with air and to strip any possible toxic metallic fumes that might be evolved.

Elemental sodium-potassium alloys have very low freezing points, desirable high thermal conductivity and low viscosity but are very reactive. Certain polymers can be effectively spun through liquid sodium-potassium; for example, certain polyolefins. Sodium-potassium alloys containing about 40–78% potassium are very suitable; however, precautions must be taken at all times to exclude air, water vapor or compounds containing reactive hydrogen from contacting the sodium-potassium alloy. Potassium-mercury and sodium-mercury amalgams may also be used, but the high vapor pressure of mercury at most melt-spinning temperatures means that possible toxicity hazards should always be carefully evaluated.

Normally in melt-spinning operations, the filaments extrude downward and fall by gravity. Without applied tension, the filaments attain a "free fall velocity," the magnitude of which depends upon filament size, polymer viscosity, distance to first guide surface and quenching rate; the filaments must be withdrawn or woundup at a speed at least as great as the free fall velocity if the process is to be stable. A somewhat analogous situation exists in the method of the invention. Here one might refer to a "free float velocity." The windup speed must be at least twice as great as the "free float velocity" if the process is to be operable.

The specific gravity of liquid metals used in the method of the invention, except for sodium-potassium alloys, usually exceeds the specific gravity of the molten polymer by a factor of three or more. The net force is therefore vertically upward and is due to the buoyance of the liquid metal. The velocity attained due to the buoyant force acting on the filament is the "free float velocity" previously mentioned; its magnitude depends primarily upon filament size, temperature gradient, viscosity of the polymer and of the liquid metal, and the difference in specific gravity of the metal and the polymer. A useful estimate of the "free float velocity" can be made by stopping the polymer flow entirely and removing all polymer or filaments remaining in the liquids, then restarting the metering pump and timing with a stopwatch the first appearance of polymer at the surface of the liquid metal; the height of the liquid metal column divided by this time approximates the free float velocity. The buoyant force due to the upper layer of liquid is usually very small or even negative and can be ignored in estimating free float velocity. In practice, the magnitude of the free float velocity need not be known, since the extruding filaments unmistakably wander around or puddle at the liquid metal surface when windup or spinning speed drops below about twice the free float velocity.

Although the liquid metal stratum is primarily a conductive heat transfer medium that insures very low heat transfer resistance at the filament surface, the liquid metal is in effect an extension of the spinneret proper. The layer of liquid metal may be regarded as a labile spinneret whose capillaries are formed by the polymeric filaments.

The static head of the liquid exerts a positive pressure above atmospheric on the filament during the transition from melt to solid; even though this pressure head is relatively small, it effectively reduces the size of bubbles or voids that ordinarily form to weaken the filament. Because air is excluded from the molten filament, oxidation or degradation products do not accumulate at the spinneret face and "wiping" of the spinneret is never necessary until many days of operation.

When cylindrical liquid chimneys and circular spinnerets are used, it is desirable to have the spinneret holes in a circular array. That is, the holes are spaced about the circumferences of concentric circles. Center-to-center dis-

tance between neighboring spinneret holes should not be less than $\frac{1}{4}$ inch. Because of the relatively high interfacial tension between useful liquid metals and most organic polymers, the polymer tends to spread appreciably at the spinneret face when the extrusion commences; once the filament begins to float the polymer stream contracts but fusion of neighboring filaments together may occur at startup if hole spacing is much less than $\frac{1}{4}$ inch. This same minimum spacing of holes should also be observed with rectangular spinnerets in which holes preferably are arranged in parallel rows.

Polymers which can be converted into filaments by the use of the present method and apparatus include any suitable fiber-forming substance. As examples thereof, the following may be mentioned: polyethylene; polypropylene; polycarbonates; polyurethanes; polystyrene; copolymers of vinyl acetate and vinyl chloride; the copolymers of vinylidene chloride and a minor proportion of vinyl chloride; linear polyesters of aromatic dicarboxylic acids and dihydric compounds, such as polymeric ethylene terephthalate; linear polycarbonamides, such as polymeric hexamethylene adipamide, polymeric hexamethylene sebacamide, polymeric monoaminocarboxylic acids, such as polymeric 6-aminocaproic acid; and other fiber-forming thermoplastic polymers. Mixtures of polymers also can be used.

The following working examples illustrate the invention.

Example I

Apparatus is made as indicated in FIGURE 2. The liquid chimney is $3\frac{1}{2}$ inches inside diameter at the spinneret and is made of 20 gauge type 304 stainless steel sheet. The straight cylindrical portion immediately above the spinneret is 25 inches high and the upper portion tapers conically to a diameter of $1\frac{1}{4}$ inches at the top over a vertical distance of 20 inches, the total length of liquid chimney shell being 45 inches.

A concentric coolant jacket with a $\frac{1}{4}$ inch annular space and 10 inches long is welded around the lower section of the liquid chimney shell at a distance 6 inches above the bottom, the lower 6 inch portion and the upper 9 inches of the straight section being unjacketed. Standard $\frac{1}{4}$ inch pipe nipples welded to the jacket form the inlet and outlet nozzles for coolant. $\frac{1}{4}$ inch standard couplings are welded to the lower cylindrical shell, one at a position one inch above the bottom to serve for filling and draining of liquid metal; another similar coupling is welded to the shell one inch below the jacket to admit a sheathed copper-constantan thermocouple junction; another coupling one inch above the top of the jacket admits a second thermocouple into the liquid bath region. A $\frac{1}{4}$ inch nipple is welded to the shell 22 inches above the bottom end and serves to admit or drain the upper layer of liquid; a second nipple is located at the upper end of the conical portion to admit or remove the upper liquid when this liquid is circulated.

A conical jacket, also with $\frac{1}{4}$ inch annulus, is formed around the upper 15 inches of the conical portion, leaving an unjacketed section about 5 inches long in addition to the unjacketed portion of the straight cylindrical section. $\frac{1}{4}$ inch nipples are welded to the jacket to provide an inlet and an outlet for coolant in the jacket; a $\frac{1}{4}$ inch coupling is welded to the conical shell $\frac{1}{2}$ inch below the upper jacket to admit a thermocouple that may be bent through a right angle to extend the junction up into the conical portion that is actually surrounded by the jacket.

A flange $\frac{1}{4}$ inch thick by 6 inch O.D. is welded to the lower end of the cylindrical liquid chimney. Six $\frac{3}{8}$ inch holes on a 5 inch bolt circle of the flange mate with six shallow, tapped $\frac{5}{16}$ inch holes in the spinneret hold-down ring.

The spinning base is made of carbon steel with a spinneret ring to lock a circular spinneret disk of 4 inch diameter by $\frac{3}{8}$ inch thick. The filter cavity below the spinneret is $1\frac{1}{2}$ inch deep and the lapped pump pad receives

a standard single stream "Zenith" metering pump. The apparatus is assembled on an angle iron frame such that the top of the liquid chimney is 5 feet above floor level.

A stainless steel spinneret having 40 capillaries with 0.015 inch orifice diameter is made with 22 capillaries equally spaced on a circle $2\frac{3}{4}$ inch diameter and 18 capillaries equispaced on a concentric circle of 2 inch diameter. The filter cavity of the spinning base is filled with sand that passes a 50 mesh screen and is retained by an 80 mesh screen. A layer of 150 mesh screen is placed immediately below the sand stratum and immediately above the sand downstream of the spinneret. The spinneret retainer ring is bolted in place, and in turn the liquid chimney is bolted to the spinneret ring with short cap screws, an asbestos gasket $\frac{1}{8}$ inch thick separating the chimney flange and the spinneret ring.

A coolant supply of ethylene glycol is piped to the lower jacket by means of $\frac{3}{8}$ inch copper tubing. A $\frac{1}{4}$ inch needle valve and a solenoid valve are in series in the inlet line. The solenoid is actuated through a relay connected to an ordinary bimetallic temperature sensor in the outlet line of coolant; when coolant temperature drops below an arbitrarily set temperature the coolant flow is cut off to avoid the possibility of subcooling the spinning base and freezing the pump. Water coolant is similarly piped to the upper conical jacket.

A 500 watt strip heater is wrapped around the lower unjacketed portion of the liquid chimney. Two separate 1500 watt band heaters are bent for a snug fit around the spinning base after an ordinary single-stream Zenith metering pump is bolted in place. Each of the three heaters is connected to a variable voltage transformer. Ordinary iron-constantan thermocouples are placed with junctions inserted under the heaters and in reasonably good thermal contact with the surface of the spinning base; these temperature indication points are useful particularly during startup of the unit. For commercial operation, of course, automatic temperature indicator-controllers are used. The entire spinning base is insulated with an asbestos-coated cozy made of $2\frac{1}{2}$ inch thick magnesia heat insulation. A removable split cozy is made to fit snugly the entire liquid chimney, equivalent to about $1\frac{1}{2}$ inch thickness of 85% magnesia pipe covering. A small one-inch screw extruder with its own heating system and a short polymer transfer line is connected to the pump pad to supply polymer to the metering pump mounted on the spinning base.

With all components of the spinning apparatus assembled as described above, the three spinning base heaters are turned on at low voltage, and the screw extruder is started up. Pre-dried nylon-66 polymer containing 0.03% TiO_2 delusterant and having a standard relative viscosity of 45 measured in formic acid is charged to the hopper of the screw extruder. The polymer is allowed to flow out through a by-pass tap until the extruder is up to steady state operation.

The temperature of all temperature points at the spinning base is raised gradually up to 295°C . and voltage is adjusted to maintain this level initially. A metal alloy composed of approximately these proportions by weight is melted in a stainless steel beaker: lead 26%; bismuth 50%; tin 14%; and cadmium 10%. The freezing point of this alloy is about $58-60^\circ\text{C}$. The metering pump on the spinning base is started up. A small portion of the metal alloy is preheated to a temperature of 290°C . and is poured into the liquid chimney through the bottom inlet nipple until a layer of metal about $\frac{3}{4}$ inch deep covers the spinneret.

After a few minutes, the polymer flow to the metering pump is started and the surface of the metal pool is carefully observed with strong overhead lighting. When polymer melt first appears, the metering pump is stopped. Preheated liquid metal is slowly added until the level is about 24 inches above the face of the spinneret, then the metering pump is restarted. Molten polymer appears

at the surface of the liquid metal. The lower thermocouple indicates a temperature of 280°C . A very slow trickle of ethylene glycol coolant is started counter-current to movement of the filaments; concurrently voltage is increased to the heater at the bottom end of the liquid chimney. Voltage and coolant flow are increased until the lower thermocouple indicates 270°C . and solid polymer forms at the top of the liquid metal column. Polymer is cleared out with the aid of laboratory tongs, the metering pump being stopped periodically until solid filaments consistently appear. An air sucker-gun now takes up the filaments as coolant flow is further increased.

Water heated just below the boil is slowly added on top of the liquid metal while filaments continue to extrude to the air gun take-up. The water level is brought up to the top of the upper jacketed shell. Incipient boiling is observed at the water-liquid metal interface; a slow trickle of water coolant flow in the upper jacket is gradually increased until the temperature of the water at the top of the column is indicated to be $45-48^\circ\text{C}$.

The yarn composed of 40 filaments is carried over the upper convergence guide to a bobbin winder (type 959 Leesona) operating at windup speed of 350 yds./min. A small piece of cellulose sponge is mounted directly above the convergence guide to collect water thrown off by the threadline, the water dripping back into the top of the liquid chimney. Total denier of the spun yarn is 850.

Samples of the spun yarn are cold-drawn by hand about 500%. The stretched yarn has a quite unusual resilience and recovery, more elastomeric than in normally spun nylon-66.

Example II

The apparatus and operation described in Example I is continued with change-over to spinning of polypropylene.

A small quantity of nylon-6 (polymeric 6-aminocaproic acid) is added to the extruder to displace nylon-66 from the unit. Once nylon-6 is extruding freely, coolant to the upper jacket is increased and the water layer is drained out the side nipple, and the metering pump and extruder are stopped. The level of the liquid metal alloy is lowered about 4 inches and the coolant flow to the lower jacket is reduced. Residual traces of water evaporate from the liquid chimney.

More preheated liquid metal is now added until the level is up just below the level of the upper conical jacketed section. Coolant flow and voltages are adjusted until the surface thermocouples at the spinning base indicate a temperature of $295-298^\circ\text{C}$. The temperature at the top of the liquid metal is $165-170^\circ\text{C}$., and a 6 inch layer of high boiling white paraffin oil is added to the chimney, and a slow trickle of coolant water is admitted to the upper jacket flowing upward cocurrent with the direction of filament movement. The temperature of the thermocouple exposed to the oil indicates a temperature of $145-150^\circ\text{C}$.

Carefully pre-dried fiber grade polypropylene pellets (such as "Profax" supplied by Hercules) having a melt index of 1.6 (ASTM D-1238, 57T) is charged to the extruder. When a steady by-pass stream of polymer is flowing with an indicated temperature of 305°C . at the extruder outlet, the metering pump is restarted and polymer extrusion is resumed.

Polymer accumulation at the interface between the oil and metal is picked out periodically with long pincers. After 10 minutes, filaments are taken up in the pneumatic air gun as the filaments pass over a cellulose sponge that strips off excess oil. These filaments are rather weak and fibrillating, indicating that nylon-6 is still mixed with the polypropylene. After 30 minutes, filaments appear uniform and normal.

A sodium-potassium alloy composed of 45% sodium and 55% potassium is prepared in one pound lots by plac-

ing the elemental components together under a shallow layer of white paraffin oil heated to a temperature of 70° C. Caution in handling is exercised at all times.

A stainless steel funnel with long tube is used to add the sodium alloys carefully with minimal mixing to the top of the lead-tin-bismuth-cadmium metal which is withdrawn slowly at the bottom end of the column. By successive additions the first liquid metal is replaced by the sodium-potassium liquid metal alloy. The last portion of lower metal layer withdrawn is stored separately since some contamination with sodium-potassium may occur. The final level of sodium-potassium is 28 inches above the spinneret, and the oil layer is 12 inches deep.

The combined filaments are brought up over the upper guide and passed between two successive cellulose sponge oil-strippers and thence to the windup bobbin operating at 475 yds./min. Coolant rates and heater voltages are adjusted gradually until the lower temperature point in the liquid metal column is 260° C., an appreciably lower temperature than would ordinarily be operable for the spinneret in conventional spinning. The lower level of paraffin oil has a reasonably stable temperature of 75° C. A small amount of makeup oil is added occasionally to keep the level reasonably constant.

The spun denier of the polypropylene yarn is 675. Samples are drawn between godets after passing over a heated drag shoe at 135° C. The yarn is stretched about 850% without difficulty. The drawn yarn has very little residual stretch and some samples have a tenacity of 11-13 gms./denier.

At the conclusion of the test all heaters are turned off and the sodium-potassium alloy is drained carefully into a container while a supernatant layer of paraffin oil is maintained at all times. The last residual metal is removed when the chimney is removed from the spinneret retainer ring. The freezing point of the sodium-potassium alloy is about 12-15° C. The entire spinning assembly is then disassembled and cleaned.

There are many advantages of the present method and apparatus over prior practice. Both light and heavy denier yarns can be obtained satisfactorily using a liquid quench method. Above atmospheric pressure is maintained on the extruding filaments while air is excluded from directly contacting the molten filaments. Nylon-66 filaments have increased elastomeric properties which is one example of the improvements in the physical properties of yarn obtainable by the present invention. While the invention has been described in terms of preferred embodiments thereof, it is understood that variations from the details disclosed herein might be made without departing from the scope and spirit of the invention. Accordingly, the invention is to be limited only by the claims set forth hereinafter.

What is claimed is:

1. A process of producing melt-spun filaments comprising:

- (a) extruding at least one stream of molten fiber-forming polymer from a spinneret;
- (b) maintaining a bath of static liquid metal in direct contact with the extrusion face of the spinneret;
- (c) passing the extruded streams generally vertically upwardly through the liquid metal in heat exchanging relationship therewith to cause the streams to be cooled by the transfer of heat from the streams to the liquid metal; and
- (d) removing the resulting filamentary streams from the liquid metal.

2. The process of claim 1 wherein a second liquid immiscible with the liquid metal floats on the free surface of the liquid metal.

3. A process of producing melt-spun filaments comprising:

- (a) extruding at least one stream of molten fiber-forming polymer from a spinneret;
- (b) maintaining a bath of static liquid metal in direct contact with the extrusion face of the spinneret;
- (c) passing the extruded streams generally vertically upwardly through the liquid metal in heat exchanging relationship therewith to cause the filaments to become solidified by the transfer of heat from the streams to the liquid metal primarily by conduction; and
- (d) removing the resulting filaments from the liquid metal.

4. The process of claim 3 wherein the heat conductive liquid metal has a specific gravity greater than the specific gravity of the filament by a factor of three or more.

5. The process of claim 4 where the removal of the filaments from the liquid metal bath is accomplished at a speed of at least 2 times the free float velocity.

6. The process of claim 3 wherein the polymeric material is nylon-66, nylon-6, polypropylene or polyethylene.

7. An apparatus for melt-spinning textile filaments comprising:

- (a) a source of molten fiber-forming polymeric material;
- (b) a spinneret having at least one extrusion orifice providing passage for the molten material in an upward direction therethrough;
- (c) an upright enclosure above said spinneret for maintaining liquid metal in direct static contact with the top face of the spinneret;
- (d) means for removing the heat from the liquid metal supplied thereto; and
- (e) means for removing the filaments upwardly through the liquid metal and from the enclosure.

8. The apparatus of claim 7 wherein an upper portion of the enclosure converges to reduce the volume and circulation of liquid induced by the drag of the vertical movement of the filaments.

9. The apparatus of claim 7 wherein element (d) is a helical coil mounted within the enclosure in encircling relation with the filaments undergoing solidification.

10. The apparatus of claim 7 wherein an insulating member is associated therein between the spinneret and the enclosure to avoid heat loss directly from the spinneret to the enclosure.

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