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Washington, D.C.  
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NASA Case No. LEW-14921-1  
Print Figure 7

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(NASA-Case-LEW-14921-1) METHOD OF MAKING  
SINGLE CRYSTAL FIBERS Patent Application  
(NASA) 12 p CSCL 110

N91-13502

Unclas

G3/24 0320447

**AWARDS ABSTRACT**  
**METHOD OF MAKING SINGLE CRYSTAL FIBERS**

Single crystal fibers are made from miniature extruded ceramic feed rods. A decomposable binder is mixed with powders to form a slurry which is extruded into a small rod which may be sintered, either in air or in vacuum, or it may be used in the extruded and dried condition. A pair of laser beams focuses onto the tip of the rod to melt it thereby forming a liquid portion. A single crystal seed fiber of the same material as the feed rod contacts this liquid portion to establish a zone of liquid material between the feed rod and the single crystal seed fiber. The feed rod and the single crystal feed fiber are moved at a predetermined speed to solidify the molten zone onto the seed fiber while simultaneously melting additional feed rod. In this manner a single crystal fiber is formed from the liquid portion.

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FILING DATE: August 23, 1990

**METHOD OF MAKING SINGLE CRYSTAL FIBERS****Origin of the Invention**

The invention described herein was made by an employee of the United States Government and may be manufactured and used by or for the government for governmental purposes without the payment of any royalties thereon or  
5 therefore.

**Technical Field**

This invention is concerned with making single crystal fibers. The invention is particularly directed to forming small feed rods for use in the production of single crystal fibers.

10 It is customary in the art to produce feed rods from a hot pressed disk of the desired material. The disk is approximately four inches in diameter, and it is fully dense. The disk is then cut into small slices that are centerless ground to produce a round rod or surface ground into precise squares.

15 Each rod is about four inches or less in length depending on the size of the hot pressed disk. The cost of each rod is approximately \$200, and the time required to make a new material into these small rods is about four to five weeks.

20 One of the disadvantages of the prior art procedure is the high cost. Another disadvantage is the long lead time required to obtain a new material. Still another disadvantage is that feed rods produced in accordance with prior art procedures have short lengths.

25 It is, therefore, an object of the present invention to produce ceramic or metal feed rods quickly. Another object of the invention is to provide small diameter feed rods economically.

**Background Art**

U.S. patent No. 4,044,082 to Rosette is directed to the extrusion and re-extrusion of ionic salts which are transparent to various radiations, including laser beams. The invention deforms the salts by extrusion to  
30 fully densify them at a temperature below the melting point. The extruded billet is then extruded a second time to produce a shape without the loss of its transmission properties.

U.S. patent No. 4,163,140 to Bardet relates to sintering or melting ceramic or refractory products in a waveguide or sounding cavity wherein

the materials are subjected to the action of a microwave field. A close loop temperature measurement and control is utilized to improve the end product.

U.S. patent No. 4,725,391 to Bardhan et al teaches extruding ceramic  
5 powders, binders, and oxidizers to form ceramic tubes which are fired to produce coaxial capacitors. The disclosed process is a typical extrusion and is not concerned with making single crystals.

#### Disclosure of the Invention

The aforementioned objects are achieved by making small ceramic or metal  
10 feed rods having diameters between about 0.010 inch and about 0.050 inch from powder using a decomposable binder. The extruded rods may be sintered or used in the extruded and dried condition.

The feed rod is then utilized in a floating zone laser system which melts the tip of the feed rod. A single crystal seed rod or fiber of the  
15 same material is brought into contact with the molten tip which causes part of the single crystal to melt and maintain a stable liquid zone of the material between the feed rod and the single crystal seed rod. The feed rod and the single crystal seed rod are moved in controlled speeds to produce a single crystal fiber having the same orientation as the single  
20 crystal seed rod.

#### Brief Description of the Drawings

The foregoing, as well as other objects, features and advantages of the invention will become more apparent from the following detailed description when taken in conjunction with the appended figures in which

25 FIG. 1 is an axial cross section of a die used in a ram type extruder containing a ceramic powder slurry;

FIG. 2 is an axial cross section of a ram type extruder utilizing the die shown in FIG. 1 for extruding a ceramic feed rod from the powder slurry;

30 FIG. 3 is an axial cross section of the extruder shown in FIG. 2 used in conjunction with an oven illustrating an alternate embodiment of the invention;

FIGS. 4, 5, and 6 are schematic views illustrating the process of producing a single crystal fiber from the feed rod produced in a manner  
35 shown in FIGS. 1-3; and

FIG. 7 is an axial cross section of an extrusion die used to produce a continuous feed rod in the manner shown in FIGS. 1-3 that is used to produce a continuous single crystal fiber in the manner shown in FIGS. 4-6.

#### Best Mode for Carrying Out the Invention

5 Referring now to the drawings there is shown in FIG. 1 an extrusion die 10 that is used to produce small diameter feed rods in accordance with the invention. More particularly, ceramic powders are mixed with a suitable decomposable binder to form a slurry 12 that is placed in the die 10.

10 As shown in FIG. 2 an extrusion ram 14 forces the ceramic powder slurry 12 through an opening 16 to form an extruded ceramic rod 18. The diameter of the opening 16 is quite small, being on the order of 0.050 to 0.010 inch. By proper control of the slurry mixture, temperature and speed of the ram 14, the resulting feed rod 18 is straight and uniform in the as-extruded and dried condition. The feed rods 18 may be used in their as-  
15 extruded and dried condition.

The process for using the feed rod 18 is illustrated in FIGS. 4, 5 and 6. More particularly, the feed rod 18 extends through a suitable feeding mechanism in the form of a pull head 20 in a floating zone laser system. Two laser beams 22 and 24 shown in FIGS. 4-6 from lasers 26 and 28, respectively, are focused onto the feed rod tip. This causes a small portion of the feed rod 18 at the tip to melt, thereby forming a molten portion or liquid zone 30.

20 A single crystal 32 is in the form of a seed rod or fiber and supported by a rod 34 and pull head 36. This single crystal 32 is spaced from the molten tip of the feed rod 18 as shown in FIG. 4. This single crystal 32 is of the same material as the feed rod 18.

30 The single crystal 32 is brought into contact with the molten tip 30 of the feed rod 18 as shown in FIG. 5. This causes the end portion of the single crystal 32 to melt and become a part of a stable liquid zone 30 of the material between the feed rod 18 and the single crystal rod 32.

35 By moving both the feed rod 18 with the pull head 20 and the single crystal rod 32 with its pull head 36 at a controlled speed, a single crystal fiber 38 of the ceramic material is solidified from the liquid 30 and formed with the same orientation as the seed rod 32. At the same time the diameter of the single crystal fiber 38 is controlled by the speed of

the pull head 36 that is connected to the single crystal rod support 34 relative to the speed of the pull head 20.

The liquid pool formed by the molten portion 30 remains a constant volume and is fixed in space. The crystal 38 is grown out of the liquid pool, either up or down, while the feed rod 18 enriches the pool 30 causing its volume to remain constant. The liquid is always at the tip of the feed rod, and it is fixed in space by the focused laser beams.

By way of example, at the same speeds a 0.025-inch diameter 50% dense extruded feed rod 18 produces a 0.017 inch single crystal fiber 36. At twice the speed of the extruded feed rod 18, a single crystal fiber 36 having a diameter of 0.012 inches is formed.

#### Alternate Embodiment of the Invention

Referring now to FIG. 3 there is shown a ram extruder having the same construction as that shown in FIG. 2. However, the ceramic feed rod 18 is extruded into a furnace 40 comprising a hollow cylindrical wall 42 that contains electrical heating elements 44. The feed rod 18 is oven-dried and sintered at approximately 2600°F or higher in the furnace 40. This sintering may be done in air or inert gas in the furnace 40 or in a vacuum.

Still another embodiment of the invention is shown in FIG. 7 wherein the ram extruder shown in FIG. 2 extrudes a small diameter feed rod 18 in a continuous manner to the feeding mechanism 20 where the continuous single crystal fiber is formed in the same manner as discussed above with regards to FIGS. 4, 5 and 6.

From the aforementioned description it is apparent a major advantage of this invention is that a large number of small diameter feed rods of almost any size can be made in a short time. Also, single crystal fibers can be produced from these feed rods within a short time after the initial feed rod fabrication.

Another advantage is that the cost of the feed rods is essentially only the cost of the powder material. By way of example, five grams of powder produces 10 feed rods that are 6 inches long and 0.025 inches in diameter.

It is apparent that the as-extruded and dried feed rods shown in FIG. 2 are easily handled and support themselves so that the feed rod motor drive 20 can move the rod 18 smoothly into the laser beams 22 and 24. The extruded feed rod 18 becomes heated as it approaches laser beams 22 and 24 which cause the volatile binder to evaporate and the powders of the feed

rod 18 to sinter as the temperature increases. The final stage is for the small section of sintered feed rod 18 to begin to melt and form the molten zone 30 of the liquid material that is supported only by the sintered powders beneath the liquid.

5 Doped ceramics, complex ceramics, or metal alloys may be formed into feed rods 18 in a short time and into single crystal fibers in an equally short time by the use of this process. As shown in FIG. 7 the invention may be performed in a continuous fashion by extruding the feed rods slowly and directly into the focused laser beams 22 and 24. This makes possible  
10 the production of continuous single crystal fibers from powdered feed rod at low cost and with no contamination from any container.

The total cost savings utilized in this process is evident. Also, the possibility of continuous single fiber production cannot be underestimated.

15 It is further contemplated that the feed rod 18 may be extruded using a binder that is flexible which enables the feed rod to be dried and spooled onto a coil. The coiled feed rod material may be uncoiled and fed into the focus laser beams to produce a semicontinuous single crystal fiber that could be several thousand feet long.

20 While several embodiments of the invention have been disclosed and described, it will be apparent that various structural modifications may be made to the invention without departing from the spirit thereof or the scope of the subjoined claims.

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ABSTRACT OF THE DISCLOSURE  
METHOD OF MAKING SINGLE CRYSTAL FIBERS

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Single crystal fibers are made from miniature extruded ceramic feed rods. A decomposable binder is mixed with powders to form a slurry which is extruded into a small rod which may be sintered, either in air or in vacuum, or it may be used in the extruded and dried condition. A pair of laser beams focuses onto the tip of the rod to melt it thereby forming a liquid portion. A single crystal seed fiber of the same material as the feed rod contacts this liquid portion to establish a zone of liquid material between the feed rod and the single crystal seed fiber. The feed rod and the single crystal feed fiber are moved at a predetermined speed to solidify the molten zone onto the seed fiber while simultaneously melting additional feed rod. In this manner a single crystal fiber is formed from the liquid portion.



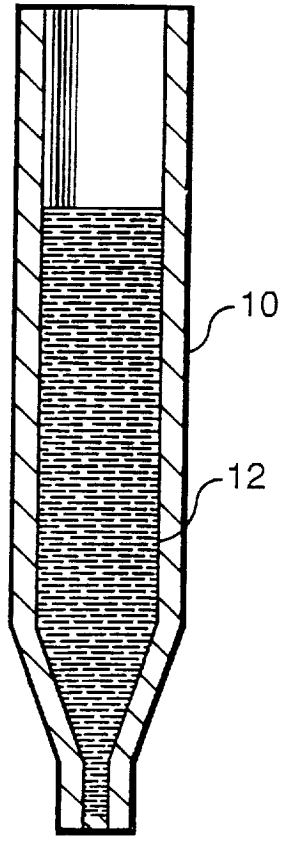


FIG. 1

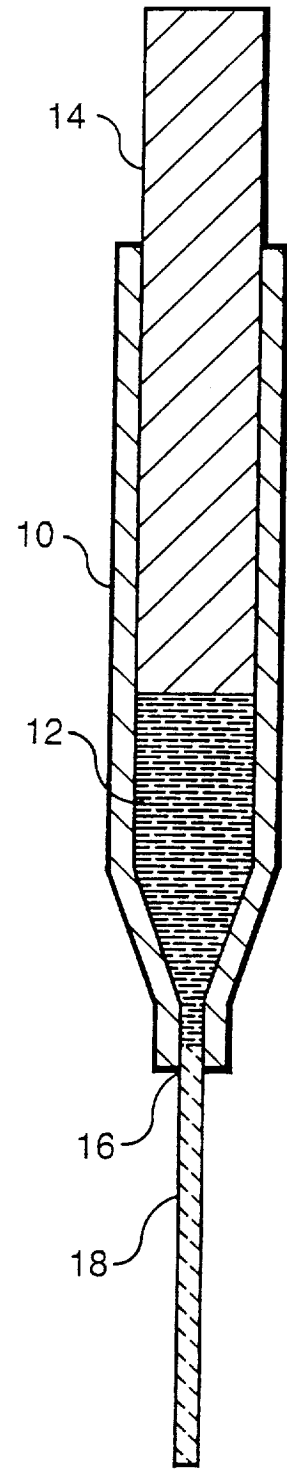


FIG. 2

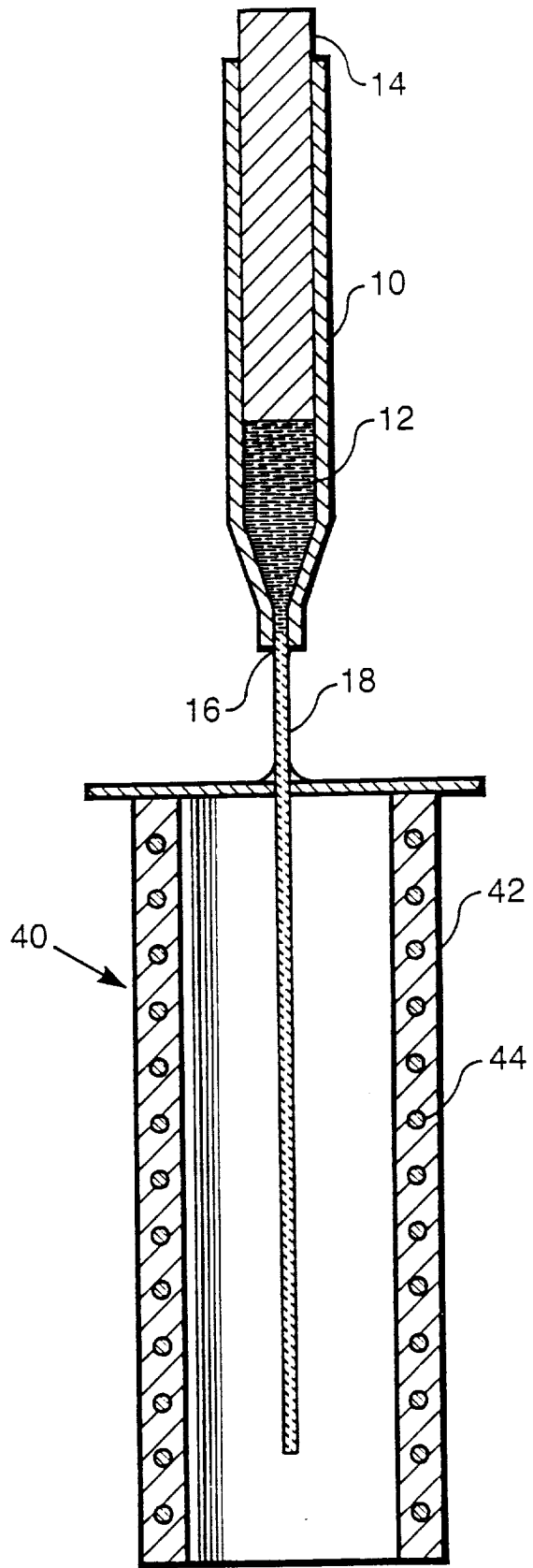


FIG. 3

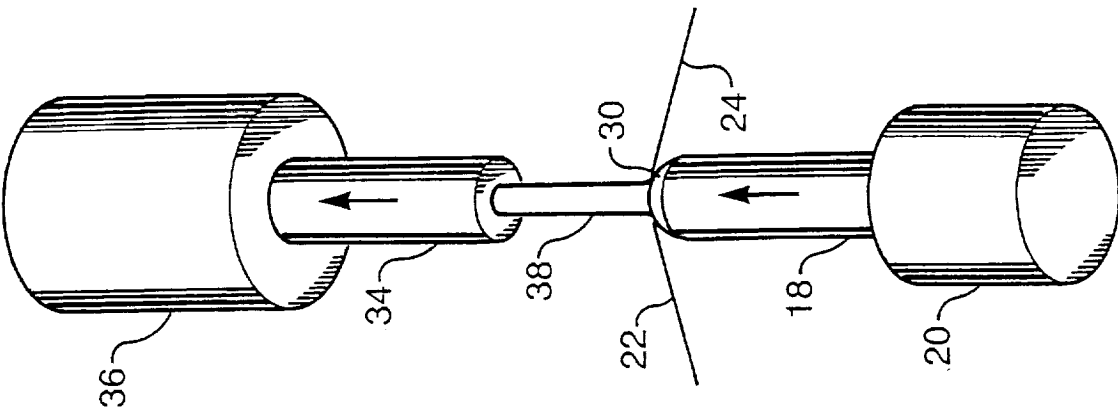


FIG. 4

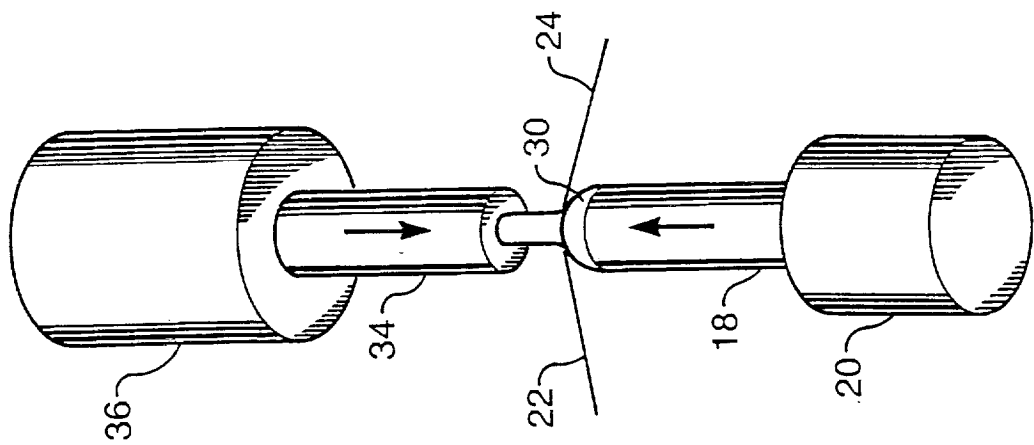


FIG. 5

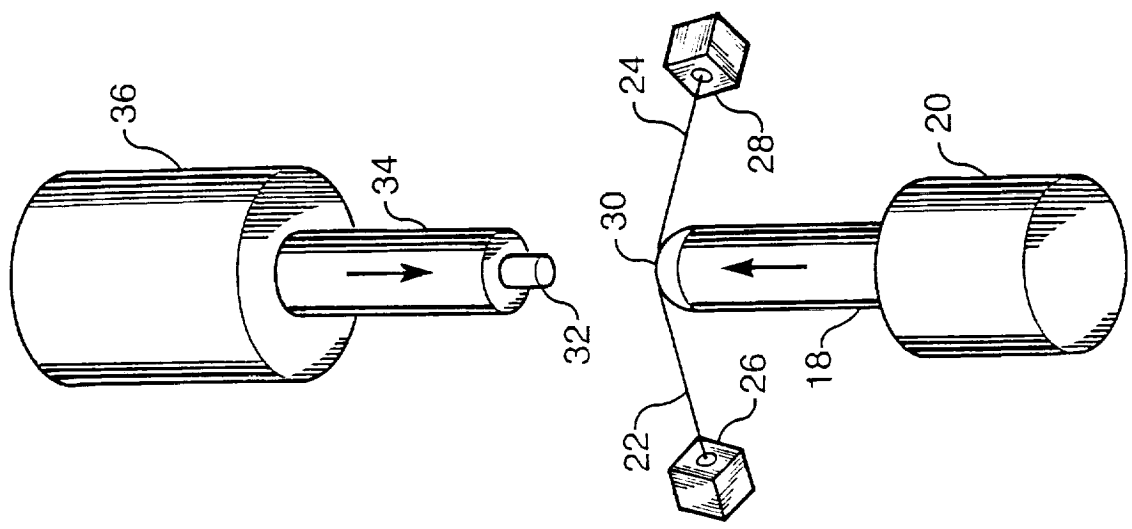


FIG. 6

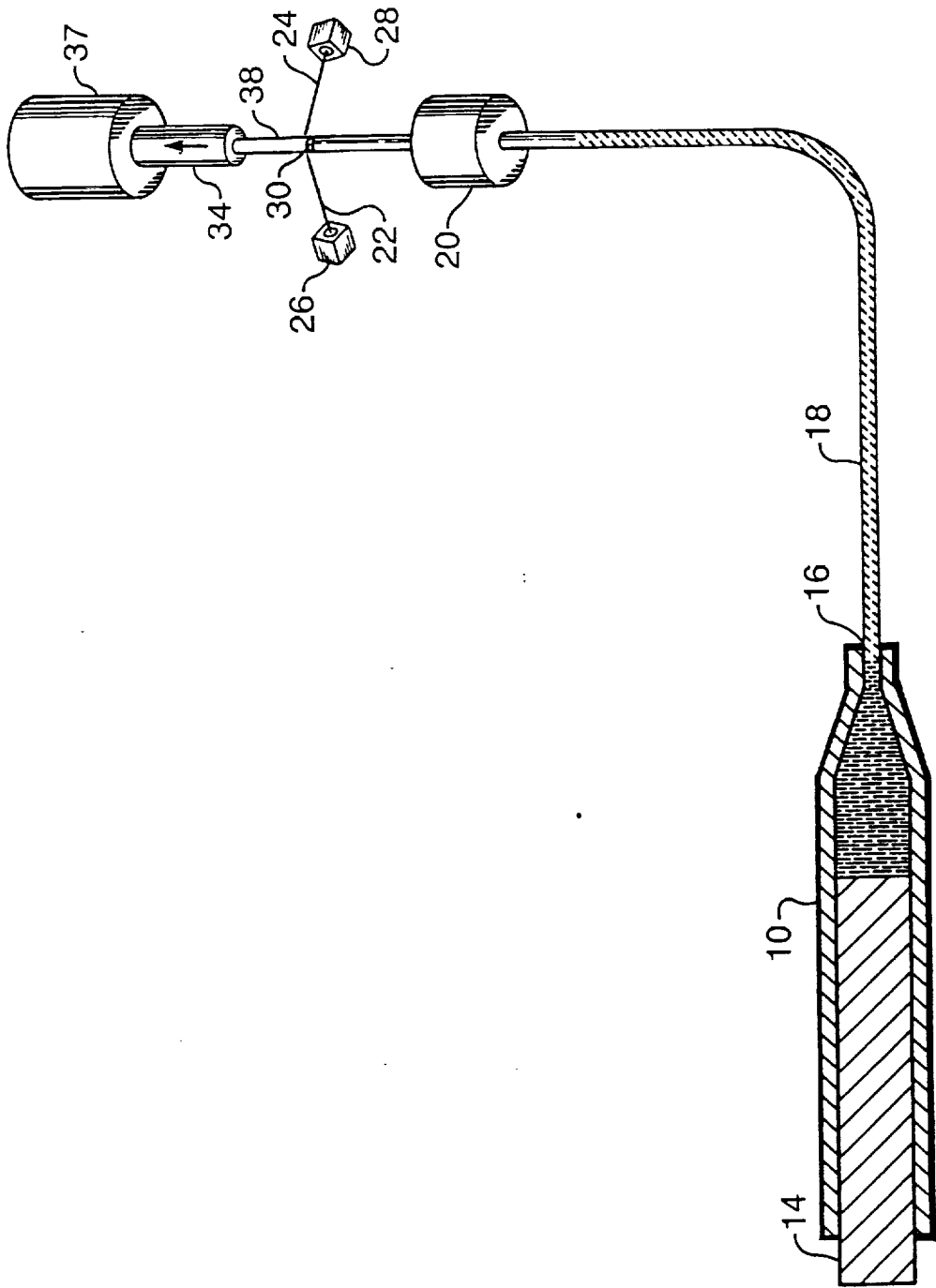


FIG. 7