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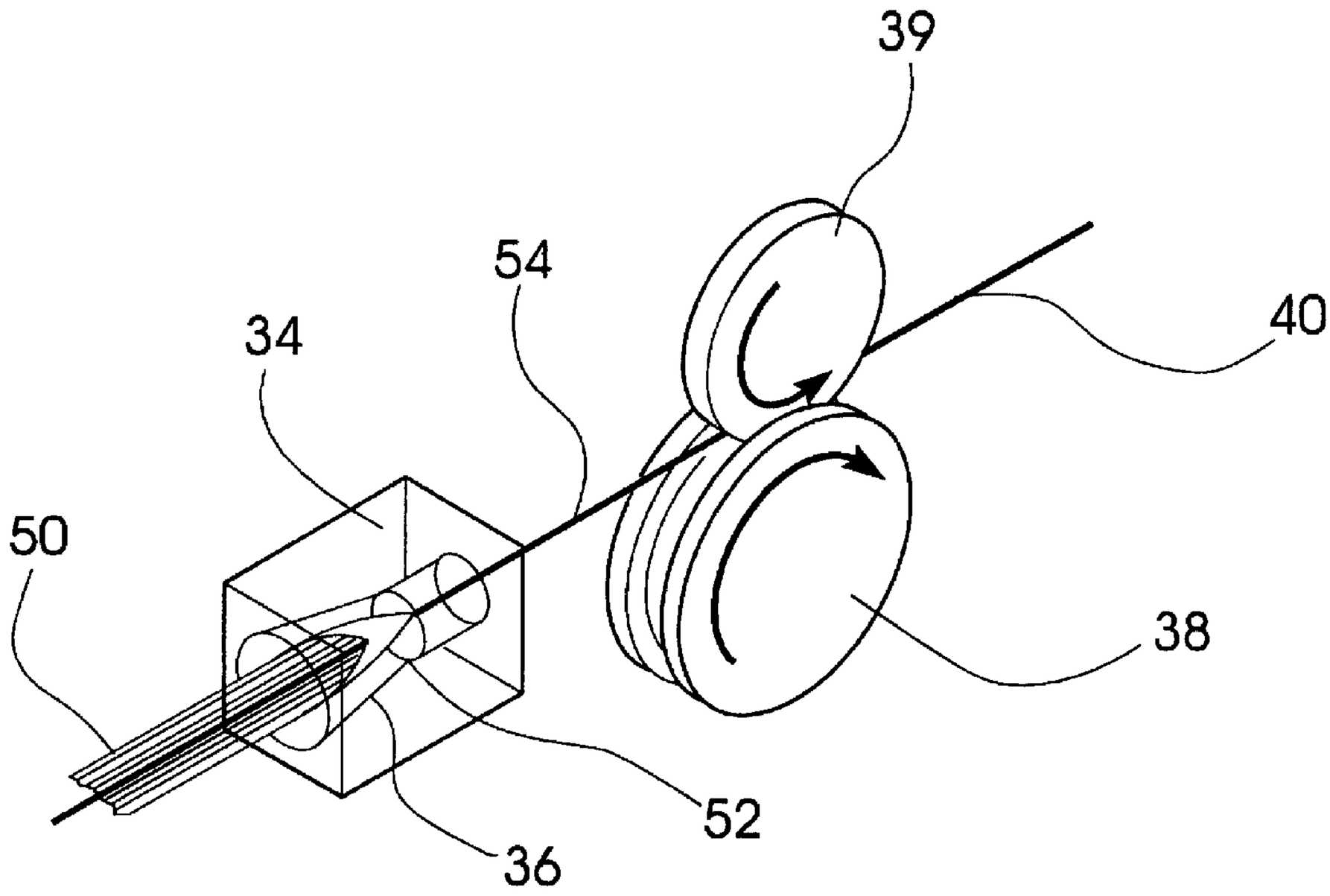
(54) **TRESSE DE GRAPHITE ET DE FEUILLE METALLIQUE, ET SA METHODE DE FABRICATION**

(54) **BRAIDED GRAPHITE-FOIL AND METHOD OF PRODUCTION**



(57) An improved method and apparatus for the production of a strand of reinforced graphite-foil yarn which is the basis of the braided graphite-foil yarns. To produce the reinforced graphite-foil strands what is needed is apparatus for a tapered, cone shaped circular die having a specific geometry suitable for curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving and other processing making it commercially useful wherever a dry, lubricious, strong, flexible material is desirable.

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ABSTRACT OF THE DISCLOSURE

An improved method and apparatus for the production of a strand of reinforced graphite-foil yarn which is the basis of the braided graphite-foil yarns. To produce the reinforced graphite-foil strands what is needed is apparatus for a tapered, cone shaped circular die having a specific geometry suitable for curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving and other processing making it commercially useful wherever a dry, lubricious, strong, flexible material is desirable.

SPECIFICATION

ROBERT A. CROSIER

BRAIDED GRAPHITE-FOIL AND METHOD OF PRODUCTION

FIELD OF THE INVENTION

The present invention relates to a reinforced vermiculated graphite textile yarn and a method for its production. For a number of years, it has been expected that braided graphite-foil yarns would become commercially viable and available. However, a method of manufacturing a low cost fiber reinforced, vermiculated natural flake graphite textile yarn suitable for braiding, weaving, twisting, and other common textile processing has been elusive, until now.

BACKGROUND OF THE INVENTION

For many years asbestos has played an important role as a mechanical packing material. After centuries of faithful service, asbestos now is shunned because of its toxicity and carcinogenic nature. In high temperature applications, it has been difficult to find a substitute that will work as well as asbestos and still be cost effective.

Cables of braided graphite yarns have shown promise as offering an effective substitute for asbestos. The best of these yarns, however, have not fulfilled their promise. The reason for this is that existing methods of production have compromised the structural integrity of the braided yarn in order to produce a reinforced graphite strand. Reinforced strands of graphite are the key to an effective braided graphite yarn.

Yarns intended for braiding, weaving and other textile processes which may be formed from the reinforcing fibers are jacketed with vermiculated natural flake graphite (vermiculated graphite was first described in Aylsworth Patent numbers: 1,137,373 of 4/27/1915 and 1,191,383 of 7/18/1916). Braiding of graphite yarns is discussed in Shane et al US Patent Numbers 3,404,382 and 3,494,382. Unfortunately, a practical, low cost method of fabricating these yarns from commercially available graphite sheet material had not become commercially practical. The wrapping method described in the above patents was not commercially implemented because it was destructive to the graphite material and therefore it never could be manufactured in the manner in which it was proposed in the patents.

DESCRIPTION OF RELATED ART

Applicant is aware of the following U. S. Patents concerning methods of producing long continuous articles employing dies and adhesives.

US Pat. No.	Issue Date	Inventor	Title
1,137,373	04-27-1915	Aylsworth	EXPANDED GRAPHITE AND COMPOSITION THEREOF
1,191,383	07-18-1916	Aylsworth	EXPANDED GRAPHITE
2,813,051	11-12-1957	MacHenry	METHOD OF PRODUCING AN ABSORBENT ELEMENT FOR FILTERS
3,404,061	04-01-1968	Shane	FLEXIBLE GRAPHITE MATERIAL OF EXPANDED PARTICLES COMPRESSED TOGETHER
3,494,382	03-10-1970	Shane	CHEMICAL PRODUCTS AND PROCESSES
3,723,157	03-27-1973	Druin	PRODUCTION OF RESIN IMPREGNATED

			FIBROUS GRAPHITE RIBBONS
4,151,031	04-24-1979	Goad	APPARATUS FOR CONTINUOUSLY FORMING COMPOSITE SHAPES
4,270,963	06-02-1981	Howat	WRAPPING ARTICLES OF INDEFINITE LENGTH
4,961,988	10-09-1990	Zhu	GENERAL PACKING OF EXPANDED GRAPHITE
5,134,030	07-28-1992	Ueda	PACKING MATERIAL AND PACKING MADE OF THE SAME

Aylsworth US Patent 1,137,373 teaches a novel form of graphite and a process for its production. This invention changes the physical orientation of flake graphite so that the flake graphite is greatly expanded in volume after treatment of the myriad of small leaves, which each flake is composed, opened up and separated like leaves of an open or partly open book. These leaves however, are not completely separated from one another. The separation is accomplished by the entrapment of gas or air between the leaves of the expanded flakes, resulting in a graphite of reduced specific gravity and vastly increased bulk or volume.

Aylsworth US Patent 1,191,383 teaches the use of expanded graphite from Aylsworth US Patent 1,137,373 compounded with a binder and other substances which can then be molded in various forms.

Shane US Patent 3,404,061 teaches a novel form of graphite and a process of producing the same. Shane further shows that threads or yarns can be woven, braided or otherwise worked into the thin, flat, flexible graphic sheet material.

Shane US Patent 3,494,382 teaches a tubular article comprising overlying layers of graphite material. The graphite is characterized by being composed of graphite particles which have been expanded at least 80 times and then recompressed into an integral mass. Shane also discloses that flexible graphite web materials can be produced and that it is desirable to reinforce the graphite sheets with filaments.

Druin US Patent 3,723,157 teaches the production of resin impregnated fibrous graphite ribbons. Uniform multi-filament bundles of a fibrous material are fed through a graphitization zone at elevated temperatures resulting in the conversion of the fibrous material to graphite carbon.

Goad US Patent 4,151,031 teaches an apparatus for continuously forming composite shapes. Goad's apparatus locally heats a fiber-reinforced thermoplastic tape, gradually forms the strip along lines parallel to the length of the strip, then cools the strip while maintaining the desired shape. Further Goad teaches the use of a resin/graphite composite. While the end product of Goad is an I beam, the teaching of joining composites together with adhesives may be important.

Howat US Patent 4,270,963 teaches a method of wrapping articles of indefinite length. A cable or optical fiber is wrapped in material by progressively pressing the wrapping material along a narrow width portion to the cable, then succeeding width portions are also turned around the article and pressed against it. Howat combines a tape, an adhesive and fibers.

Zhu US Patent 4,961,988 teaches a packing of expanded graphite and the process for production thereof. The packing mainly includes vermiform laminae of expanded graphite and auxiliary materials, which is characterized by embedment of the

auxiliary materials into the vermiform graphite laminae and the bond of the auxiliary materials to the vermiform graphite laminae with organic adhesives. In processing the graphite Zhu teaches the creation of a sheet of graphite with a resinous material which is subsequently reinforced with fibers. The problem here is that the resin volatilizes at high temperature and a significant volume of the mass turns to ash causing it to lose volume and cohesion. This means the material cannot be removed with standard extraction tools and must be picked out of the cavity.

Ueda US Patent 5,134,030 teaches packing material made from flexible graphite sheets. A cord-like structure is produced from flexible graphite sheets cut to a width of less than 5 mm which is then twisted or braided. The flexible graphite is laminated with at least one reinforcing fiber material or foil and the surface is covered with a fiber material to achieve a synergistic action of the fiber material, flexible graphite sheet and the reinforcing fiber or foil material. Ueda teaches the equivalent of putting graphite strips into a sock and compressing it. When this material is used as a gasket with volatile hydrocarbons, the hydrocarbon molecules have a porous path to follow, preventing effective sealing. Ueda teaches, in Column 1, lines 43 - 47, that flexible graphite cannot be braided or knitted.

SUMMARY OF THE INVENTION

While cables of braided graphite have theoretically been known for years, their execution has been disappointing because the effective reinforcement of vermiculated natural flake graphite has been elusive. Specifically, the actual method of joining a ribbon of graphite to reinforcing yarns while still maintaining its extreme service temperature capability has not been possible until now. Service temperatures of from -400°F up to 5400°F were not possible in earlier attempts at making these

yarns. It has been found that, by encapsulating the reinforcing fibers such as quartz, INCONEL™ wire, stainless steel wire, carbon fiber, graphite fiber and/or other high temperature reinforcements in a jacket of vermiculated graphite, the loss of the adhesive coating has no effect on the overall integrity of the yarns. The adhesive material, which carbonizes in the absence of oxygen (the core of the jacketed yarns are relatively oxygen free) at elevated temperatures is no longer required as an adhesive once it has been fully encapsulated and has been processed into a textile yarn. This permits the material to be used at extremely high temperatures without significant change in service capability, also the adhesive has no significant weight or volume loss effect because it has become carbon which is very suitable for the services intended.

This simple, inexpensive yarn is made by slitting a vermiculated graphite roll into ribbons having suitable widths for this purpose. The ribbons are mounted into a "protrusion" device, which is a combination extrusion/pultrusion die, wherein the workpiece is fed into the die by rollers and the product is removed by pulling from the exit end of the die. The end of the graphite ribbon is fed through a compression roller with a ribbon of two sided adhesive thin plastic film. One side is then bonded to the ribbon of graphite, the other side is intended for the reinforcing fiber. As the graphite ribbon leaves the compression rollers bonded to the adhesive strip, the protective paper or plastic is stripped, exposing the adhesive on the outside surface of the laminated ribbon to permit the reinforcing fibers to attach themselves to the adhesive surface of the laminated ribbon.

Fibrous strands are then threaded into the protrusion device to be laid up on the adhesive matrix and to become attached to the ribbon. The fiber reinforced laminated ribbon which still has exposed adhesive on the surface is then fed at a critical

angle through a tapered, cone shaped circular die having a specific geometry suitable for curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving and other processing making it commercially useful wherever a dry, lubricious, strong, flexible material is desirable.

The invention provides apparatus and a method for production of braided graphite-foil yarns. During normal operation, a ribbon of vermiculated graphite a ribbon of plastic adhesive film and a reinforcing fiber are brought together, and passed through the conical die which founds these elements onto themselves. Pressure is then applied to form a fiber reinforced composite strand of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving and other processing. The present invention is particularly useful for the production of braided graphite-foil yarns.

OBJECTS OF THE INVENTION

The principal object of the invention is to provide an improved method of producing a strand of reinforced graphite-foil yarn which is the basis of the braided graphite-foil yarns.

A further object of this invention is to provide a method of
curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake.

A further object of this invention is to provide a method of fully encapsulating the adhesive and reinforcing fibers, preventing these areas from becoming a leak path.

Another object of the invention is to provide a gasket made of braided graphite which can effectively replace asbestos gaskets and work effectively at temperatures at or above 2200° Fahrenheit, as well as at cryogenic temperatures.

A further object of this invention is to provide a method of fully encapsulating the adhesive protecting it from chemical exposure.

A further object of this invention is to provide a method of fully encapsulating the adhesive allowing carbonization of the adhesive when exposed to high temperatures.

Another object of the invention is to provide apparatus for a tapered, cone shaped circular die having a specific geometry suitable for curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving or other textile processing, making it commercially useful wherever a dry lubricious, strong, flexible material is desirable.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects will become more readily apparent by referring to the following detailed description and the appended drawings in which:

Figure 1 is an isometric view of a portion of the braided

graphite-foil yarn.

Figure 2 is an isometric view of a portion of the cone shaped curling die and the compression die which the reinforced graphite-foil strands are passed.

Figure 3 is a front view illustrating the production of a strand of reinforced graphite-foil yarn which is the basis of the braided graphite-foil yarns.

Figure 4 is an isometric view of a cone shaped curling die illustrating the geometry of its interior.

Figure 5 is a cross sectional view of the cone-shaped curling die of Figure 4.

Figure 6 is a front view illustrating an alternative embodiment for the production of a round strand of reinforced graphite-foil yarn which includes a second calendaring die.

Figure 7 is a cross section of an unprocessed portion of the reinforced graphite-foil strand.

Figure 8a is an isometric cross sectional view of an unprocessed portion of a reinforced graphite-foil strand.

Figure 8b is an isometric cross sectional view of a partially processed graphite-foil strand.

Figure 8c is an isometric cross sectional view of a compressed graphite-foil strand.

Figure 9 is a front view of a reinforced graphite-foil strand being passed through a die.

Figure 10 is an isometric view of a round reinforced graphite-foil strand.

Figure 11 is an isometric view of a gasket formed from braided graphite-foil yarn.

Figure 12 is an isometric view of an endless ring gasket formed from braided graphite-foil yarn.

Figure 13 is an isometric view of an alternative cone-shaped curling die illustrating the geometry of its interior.

Figure 14 is a cross sectional view of the alternative cone shaped curling die of Figure 13.

DETAILED DESCRIPTION

Referring now to the drawings, and particularly to Figure 3, a simple, inexpensive yarn is made by slitting a vermiculated graphite roll 20 into ribbons having suitable widths for this purpose. While a number of widths are possible, a preferred width of the vermiculated graphite ribbon 44 is 0.375 inches wide by 0.015 inches thick, while having a length of many feet. The ribbon of vermiculated graphite 44 and a ribbon of plastic adhesive film 46, such as polyester, cellophane, or acrylic, having a protective plastic or paper strip 26 are fed through compression rollers 22. The plastic adhesive is conveniently about 0.2 inches in width with a depth of about 0.00025 inches (one-quarter mil). The ribbon of vermiculated graphite 44 is laminated in the compressive protrusion rollers 22 with a ribbon of a suitably thin plastic film 46 so the exposed side is covered with protective plastic film 26. As the ribbon leaves the compression rollers, the protective plastic film 26 is stripped by a peeler 42, exposing the adhesive on the outside surface of the laminated ribbon 48, after which reinforcing fibers 30 are

placed on and attach themselves to the adhesive surface of the laminated ribbon. The reinforcing fiber may be made up of thousands of monofilaments. Alternatively the reinforcing fiber can be a single fiber or a bundle of fibers.

Fibrous strands are threaded into the protrusion device at roller 32, at which point the reinforcing fiber 30 is adhesively attached to the laminated ribbon 48. The fiber reinforced laminated ribbon 50 (also shown in Figure 8a) which still has exposed adhesive on the surface is then fed at a critical angle 64 of about 10° to 40°, but preferably from 20° to 30°, through a tapered, cone shaped, circular cross-section die 34 having a specific geometry suitable for curling the fiber and adhesive coated surface inward upon itself, as shown by reference numeral 52 in Figure 2, so that the curled fiber reinforced ribbon 54, also shown in Figure 8b, will adhere to itself. Interior geometry of the cone shaped circular die 34 is shown in Figure 5 where the angle 62 of the cone is from about 10° to 20°. The entrance to the cone shaped circular die 34 has a radius 56. On the interior of the die 34, the cone 74 extends about three-quarters of the way into the die, then radius surface 58 leads into a straight or tubular portion 76 of the die. Alternatively cone 74, as shown in Figure 13, can extend all the way through the die 78, so it terminates at the exit orifice 80 of the die. In this configuration it is preferable that the exit orifice 80 be provided with radius 82 to inhibit scoring or tearing of the ribbon 54 upon exiting the die.

The curled fiber reinforced ribbon 54 is fed into the final calendar protrusion die 38, 39, where it is compressed, as shown in Figure 9, forming a finished fiber reinforced composite strand 40 of expanded graphite flake having suitable tensile strength, flexibility, and toughness to permit braiding, weaving and other processing, making it commercially useful wherever a dry lubricous, strong, flexible material is desirable. The finished

fiber reinforced composite strand 40 of expanded graphite flake is then collected on a roller 41.

Fiber reinforced composite strands 40 of expanded graphite flake can then be braided together to form braided graphite-foil yarn 10 as shown in Figure 1.

ALTERNATIVE EMBODIMENTS

While a rectangular finished fiber reinforced composite strand 40 of expanded graphite flake can be produced by the apparatus depicted in Figure 3, there are applications where it is preferable to have a round finished fiber reinforced composite strand 72 of expanded graphite flake as shown in Figure 10. To produce such a strand, the apparatus of Figure 3 is modified as shown in Figure 6. The apparatus of Figure 6 is identical to that of Figure 3, but with the addition of protrusion die rolls 52, 53. The curled fiber reinforced ribbon 54 (Figure 6), is fed into the first calendar protrusion die 38, 39 where it is compressed as shown in Figure 9, forming a rectangular fiber reinforced composite strand 40 (Figure 8c) of expanded graphite flake. In order to make a round strand, the rectangular fiber reinforced composite strand 40 of Figure 6 is fed through a second calendar protrusion die 52, 53, where it is compressed, forming a round fiber reinforced composite strand 72, as in Figure 10, which can then be collected on the take up roller 41 of Figure 6. It is also possible to pull composite strand 40 through a second die, such as die 34 or 78, to yield a round strand 72.

Due to the improved method of producing a reinforced strand of vermiculated natural flake graphite, when the strands are braided into a yarn, applications that were not possible before are now possible. First, when braided or woven into suitable cross sections, the material can be used as an electrically

conductive joint sealant for electronic cabinetry for the purpose of providing electrical continuity for static electricity protection and as a jointing for protection for background radio interference in a Faraday Cage device. Second, in its braided, twisted or woven forms, reinforced vermiculated graphite yarn can be used as a jointing and sealant which can be placed into sealing cavities and compressed to form into a homogeneous mass conforming to exact geometry of the seal cavity providing a positive sealing medium. It is also capable of outstanding protection against destructive electrolytic or galvanic action on the metal surfaces by reducing the electric potential by sealing out electric current inducing electrolytes present in the sealed media, and further, (because it is highly conductive electrically) by short circuiting any possible electrochemically induced electrical current from attacking metal surfaces.

When used as a gasket as described above, the braided graphite strand can be used in two ways. First, an endless ring gasket can be formed from the braided graphite material as shown in Figure 12. Second, when not preformed, the braided graphite 10, as shown in Figure 1, can be used on site to form a gasket of the desired size. The braided graphite can be cut to the approximate length, then laid in the connection area with the ends 90, 92 overlapped. When compressed, a gasket is formed as shown in Figure 11. The overlapped area 70 has the same thickness as the non-overlapped areas 68. This gasket-forming process has three significant advantages: 1) each gasket can be produced as needed so that sized preformed gaskets need not be stocked; 2) the overlapped ends provide tabs which facilitate removal in tight areas; 3) gaskets of unusual size and shape can easily be configured.

Gaskets formed of the material made by the invented process can be used in pumps where the braided graphite-foil yarn acts as a heat sink in the sealing cavity allowing the pumps to run

leak- free without cooling liquid flushes at peripheral shaft speeds up to 4800 ft/minute.

It has also been found that braided graphite gaskets, and other braided graphite items, stand up under extremes of temperature. They operate successfully at temperatures as low as -400°F, and at temperatures as high as 5400°F. Graphite sublimes (changes from a solid state to a gaseous state) between 6605°F and 6687°F. (Note that while carbon is amorphous, graphite is crystalline). The preferred upper operating limit is about 2300°F.

SUMMARY OF THE ACHIEVEMENT OF THE OBJECTS OF THE INVENTION

From the foregoing, it is readily apparent that I have invented an improved method of producing a strand of reinforced graphite-foil yarn which is the basis of the braided graphite-foil yarns. Also encompassed is a method of curling the fiber and adhesive coated surface inward upon itself, so that the curled ribbon will adhere to itself when compressed into the final protrusion die forming a finished fiber reinforced composite strand of expanded graphite flake. This method ensures the full encapsulation of adhesive and reinforcing fibers, preventing these areas from becoming a leak path when the material is used as a seal. Full encapsulation also protects the adhesive and reinforcing fibers from chemical exposure and insures structural integrity at high temperatures. Utilizing this method braided graphite-foil yarns can be produced faster and more economically than heretofore has been possible.

It is to be understood that the foregoing description and specific embodiments are merely illustrative of the best mode of the invention and the principles thereof, and that various modifications and additions may be made to the apparatus by those

skilled in the art, without departing from the spirit and scope of this invention, which is therefore understood to be limited only by the scope of the appended claims.

What is claimed is:

1. Apparatus for forming a laminated vermiculated graphite ribbon having sufficient tensile strength, flexibility, and toughness suitable for braiding, weaving, and compressing, comprising:

a source of vermiculated graphite ribbon of predetermined width;

means for laminating said ribbon of vermiculated graphite with a ribbon of plastic adhesive film;

means for adhesively laying a reinforcing fiber on one side of said laminated vermiculated graphite ribbon;

means for curling said laminated vermiculated graphite ribbon inward upon itself about said reinforcing fiber to form a curled fiber reinforced ribbon; and

means for compressing said curled fiber reinforced ribbon into a finished fiber reinforced composite strand.

2. Apparatus according to claim 1 wherein said means for curling said laminated vermiculated graphite ribbon inwardly is a tapered, cone shaped circular die of a specific geometry.

3. Apparatus according to claim 2 wherein the taper angle of said cone shaped circular die from its central axis is from 5 to 60 degrees.

4. Apparatus according to claim 3 wherein the angle of said cone shaped circular die is from 10 to 20 degrees.

5. Apparatus according to claim 2, further comprising means for feeding the reinforcing fiber, adhesive film and

vermiculated graphite ribbon into said die at an angle from about 10 to 40 degrees.

6. Apparatus according to claim 7 wherein the reinforcing fiber, adhesive strip and vermiculated graphite laminate ribbon is fed into the die at an angle of from 20 to 30 degrees.

7. A method of producing a strand of reinforced graphite-foil comprising:

slitting a vermiculated graphite roll into a ribbon of vermiculated graphite;

laminating said ribbon of vermiculated graphite with a ribbon of plastic adhesive film having a protective film thereon, forming a laminated vermiculated graphite ribbon;

stripping said protective film away from laminated vermiculated graphite ribbon;

laying a reinforcing fiber on one side of said laminated vermiculated graphite ribbon;

curling said laminated vermiculated graphite ribbon inward upon itself forming a curled fiber reinforced ribbon; and

compressing said curled fiber reinforced ribbon into a finished fiber reinforced composite strand.

8. A method of making braided graphite-foil yarn comprising

preparing a plurality of fiber reinforced composite strands in accordance with claim 7, followed by braiding multiple strands into a finished product.

9. A method of making an electrically conductive joint sealant, comprising:

preparing a plurality of fiber reinforced composite strands in accordance with claim 7, followed by braiding multiple strands into an electrically conductive joint sealant.

10. A method of making an electrically conductive joint sealant for a cavity in accordance with claim 9, further comprising:

compressing said braided graphite-foil yarn to form a homogeneous mass conforming to the exact geometry of the seal cavity.

11. A method of making a liquid pump sealant comprising:

preparing a plurality of fiber reinforced composite strands in accordance with claim 7, followed by braiding multiple strands into an endless ring gasket.

12. A method of making a liquid pump sealant comprising:

preparing a plurality of fiber reinforced composite strands in accordance with claim 7, followed by braiding multiple strands into an elongated braided strand of approximate predetermined length, then overlapping the ends of the braided strand, and compressing the strand with the ends overlapped, thereby forming a gasket;

wherein the braided graphite-foil yarn acts as a heat sink in the sealing cavity allowing a pump to run leak-free without cooling liquid flushes at peripheral shaft speeds up to 4800 ft/minute.

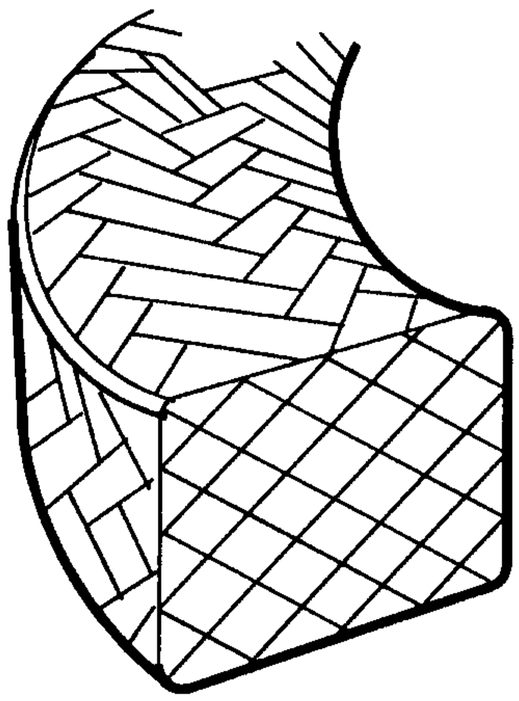


Fig.1

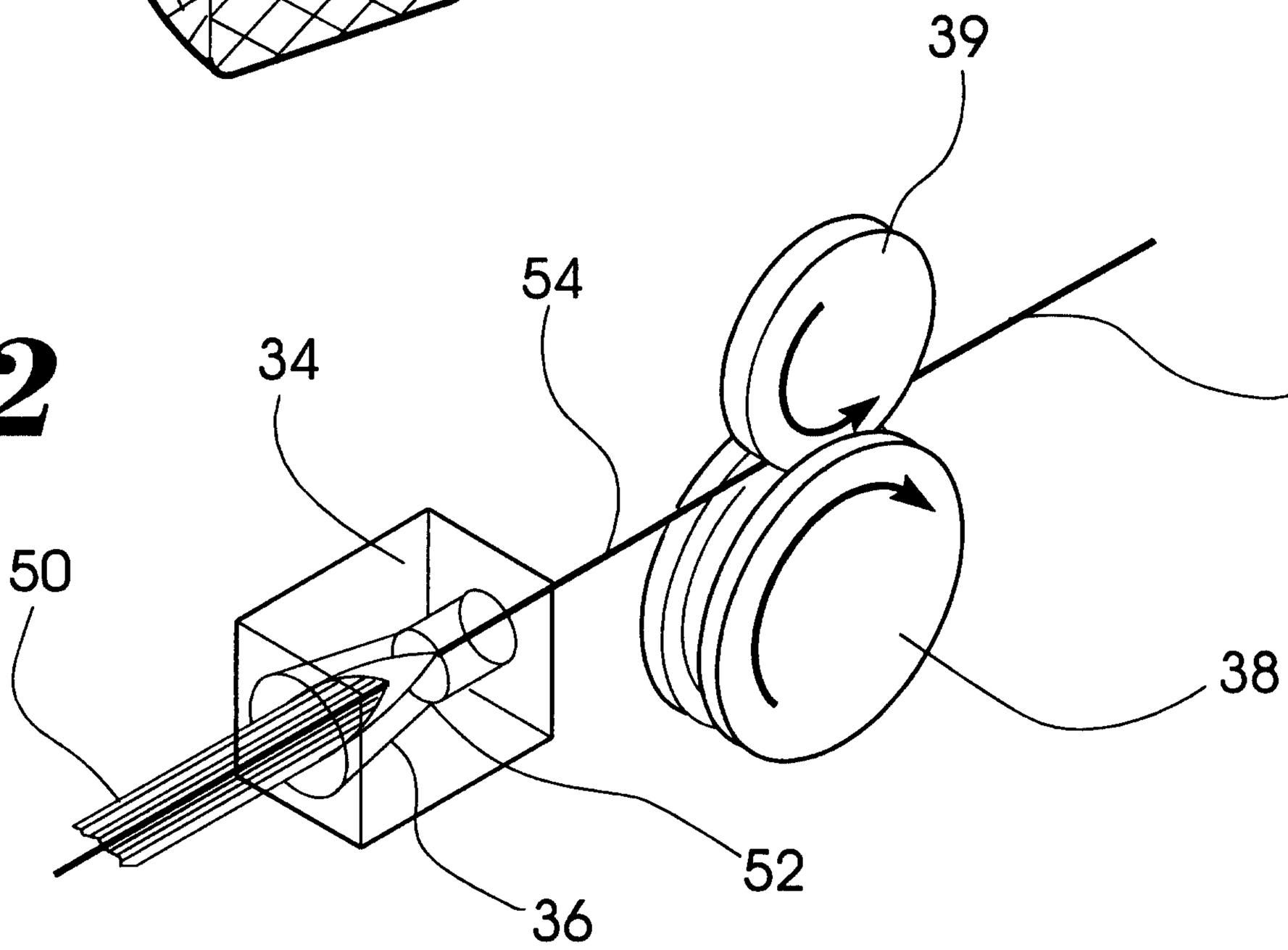


Fig.2

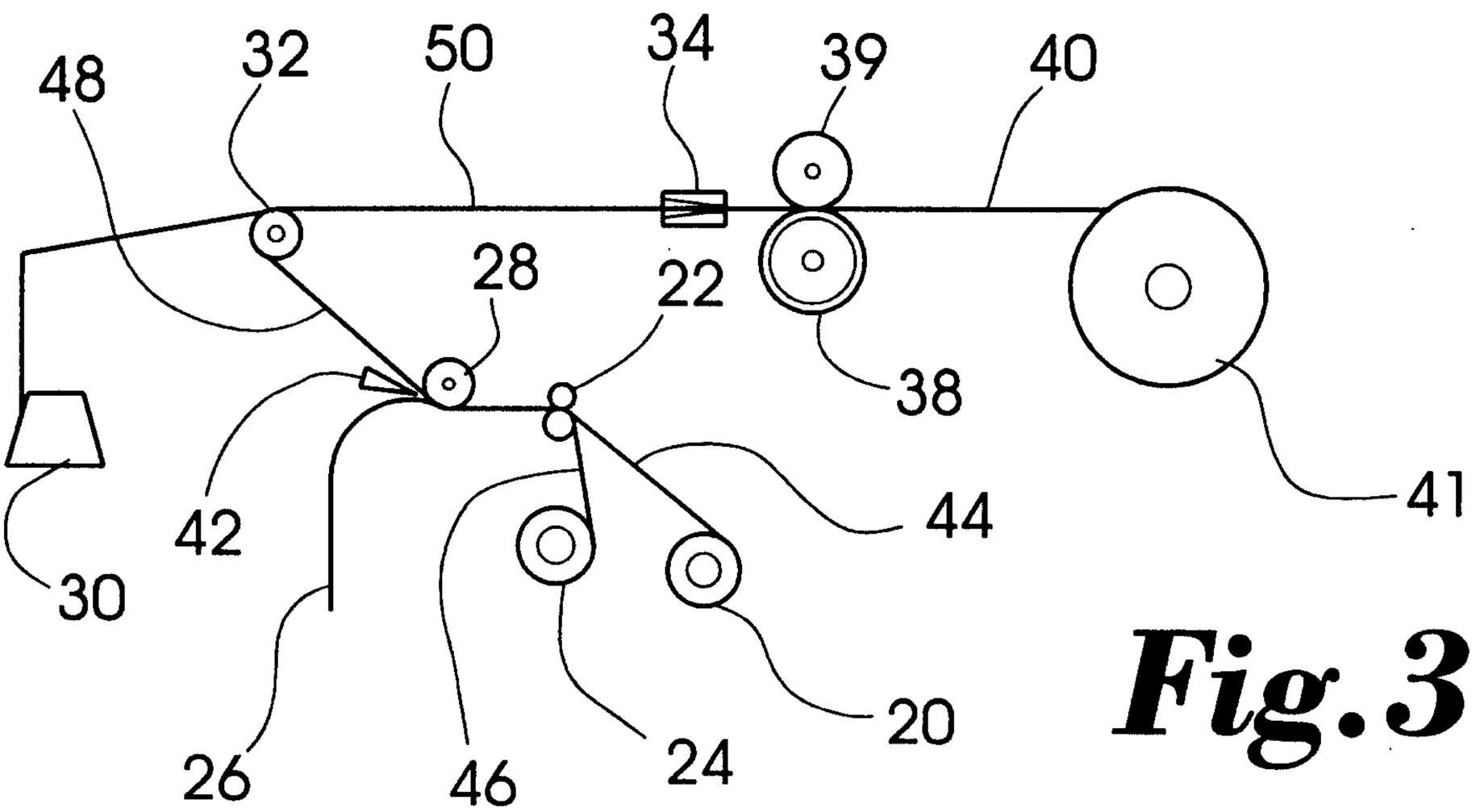


Fig.3

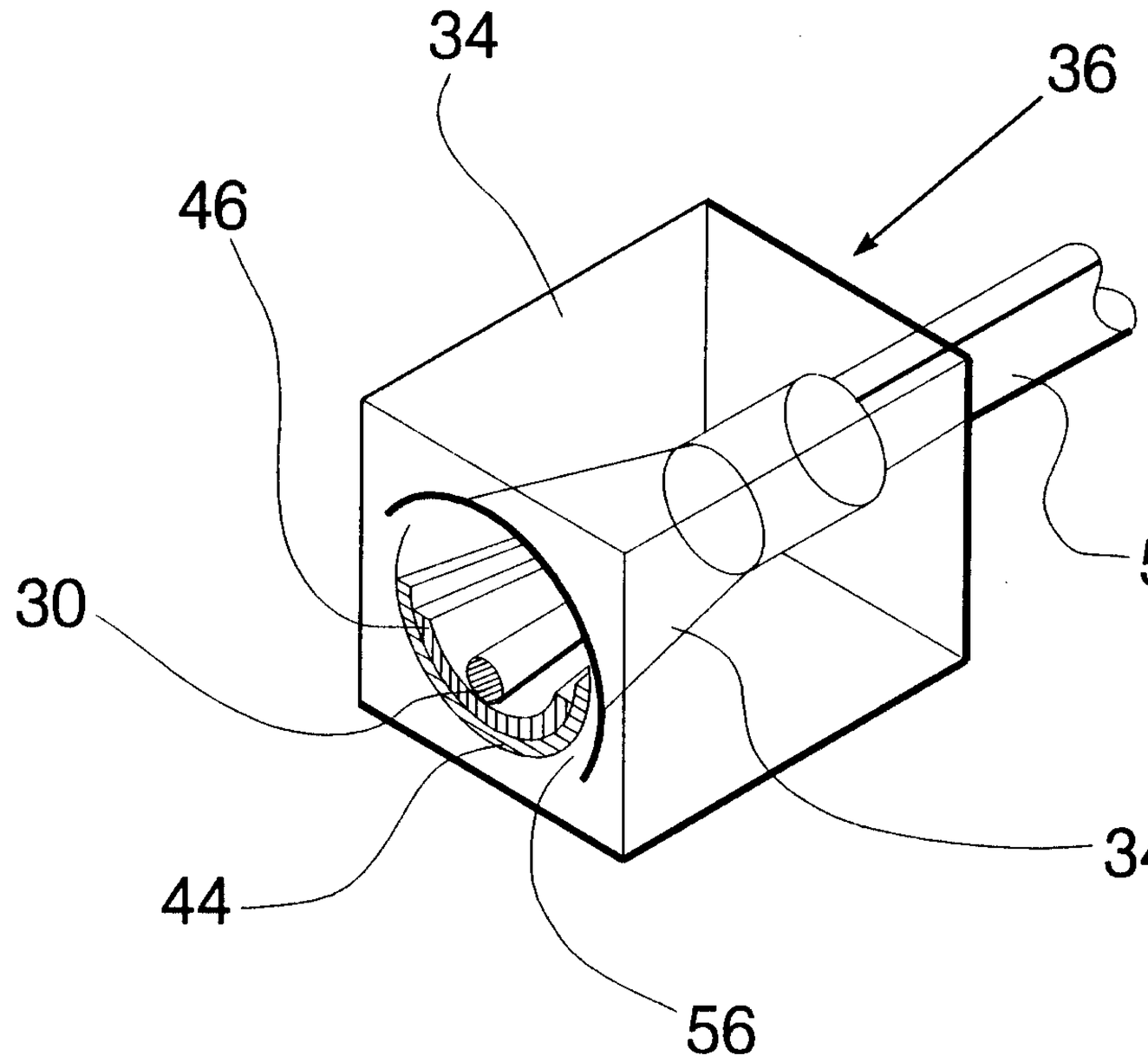


Fig.4

Fig.5

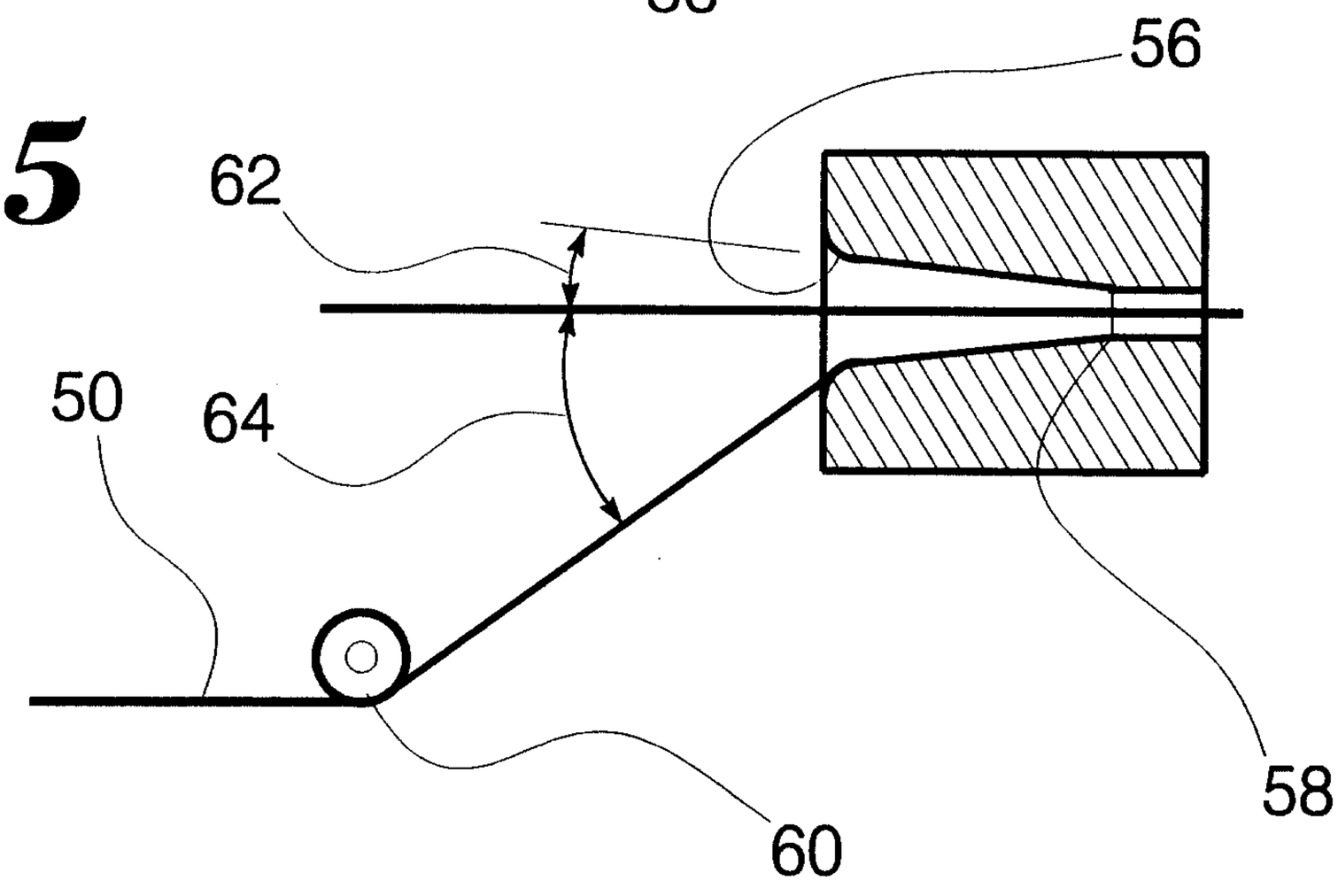
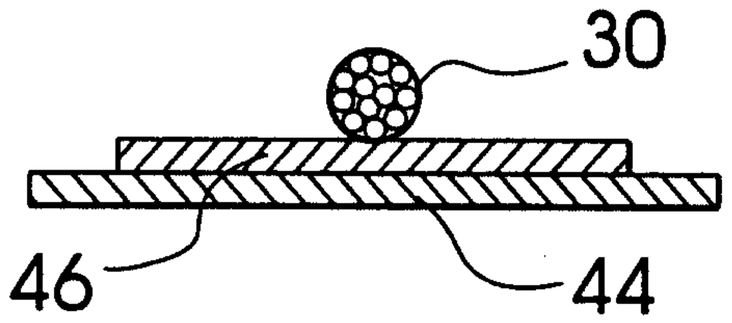


Fig.6

Gowling, Strathy & Henderson

Fig.7



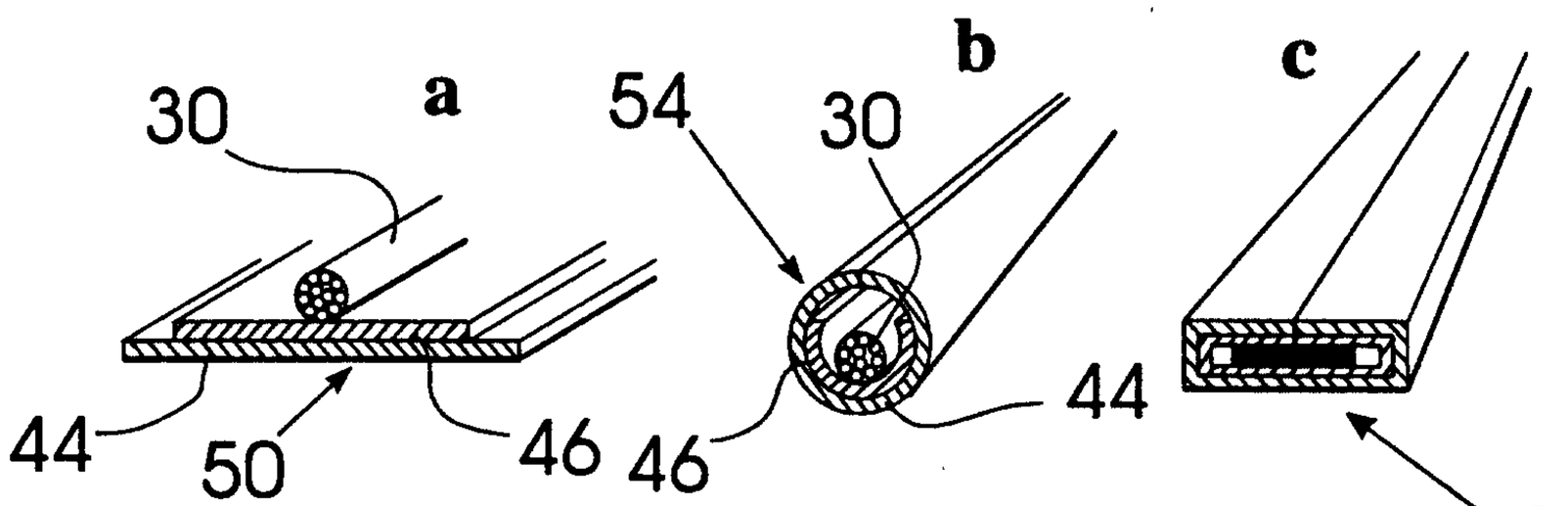


Fig.8

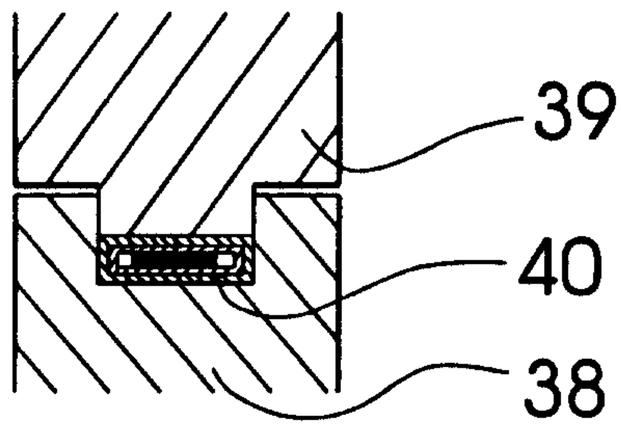


Fig.9

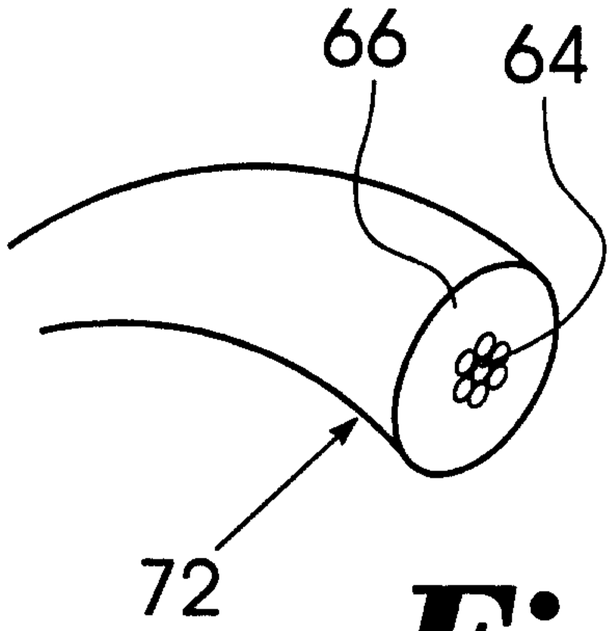


Fig.10

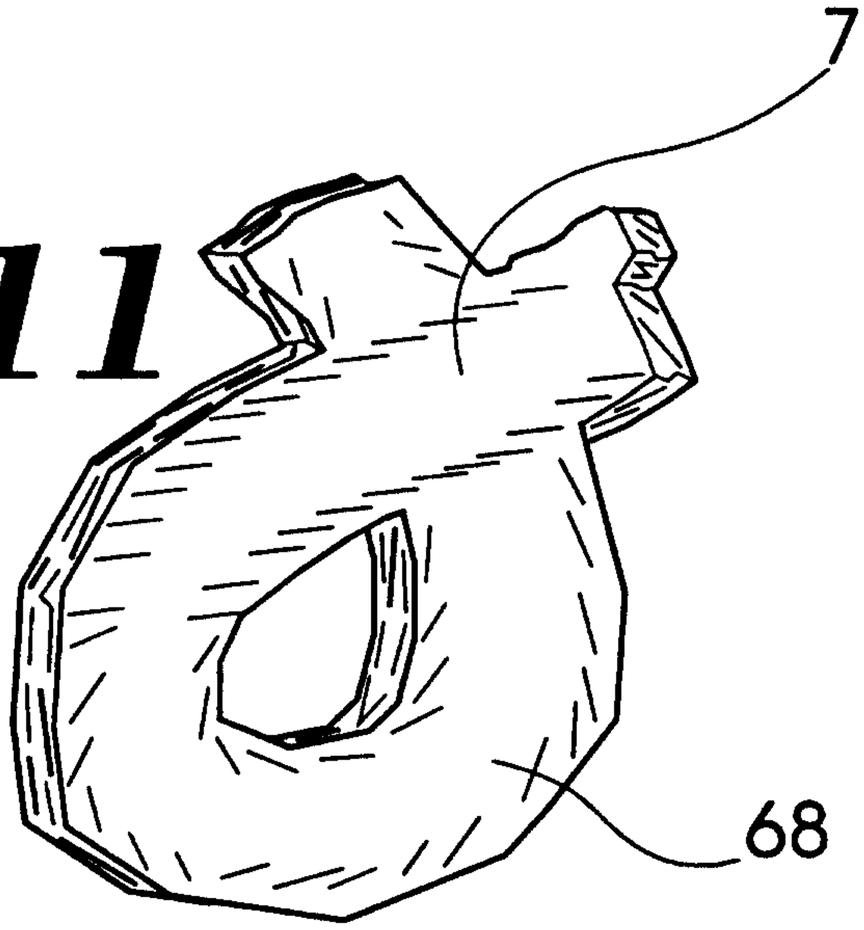


Fig.11

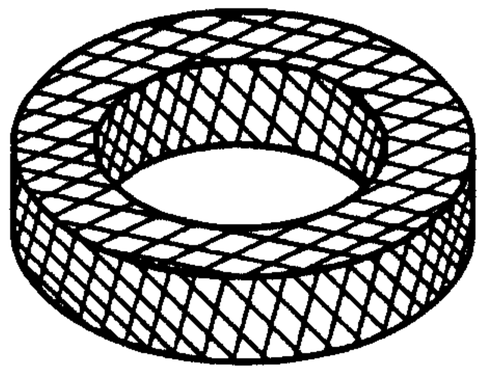


Fig.12

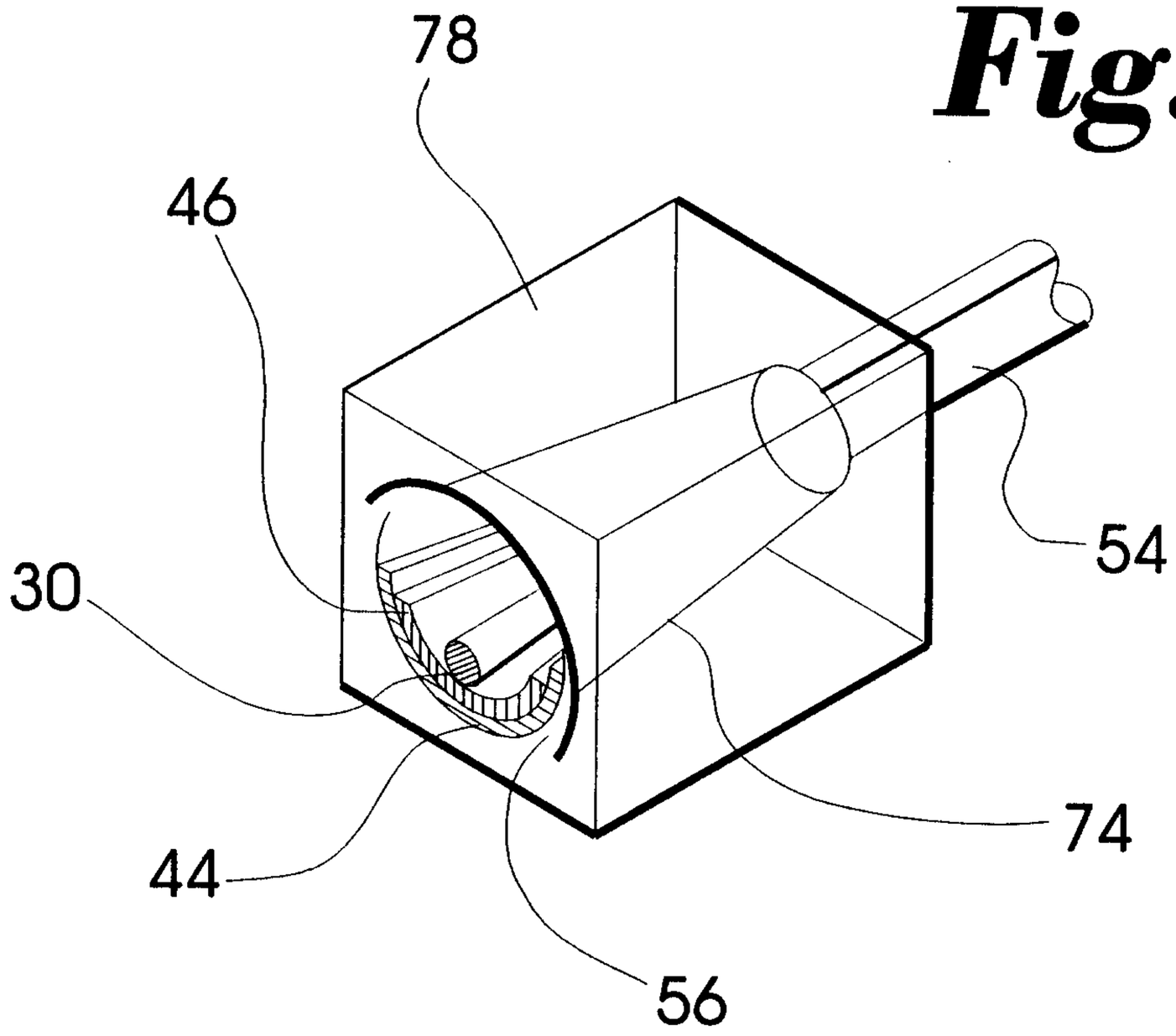


Fig.13

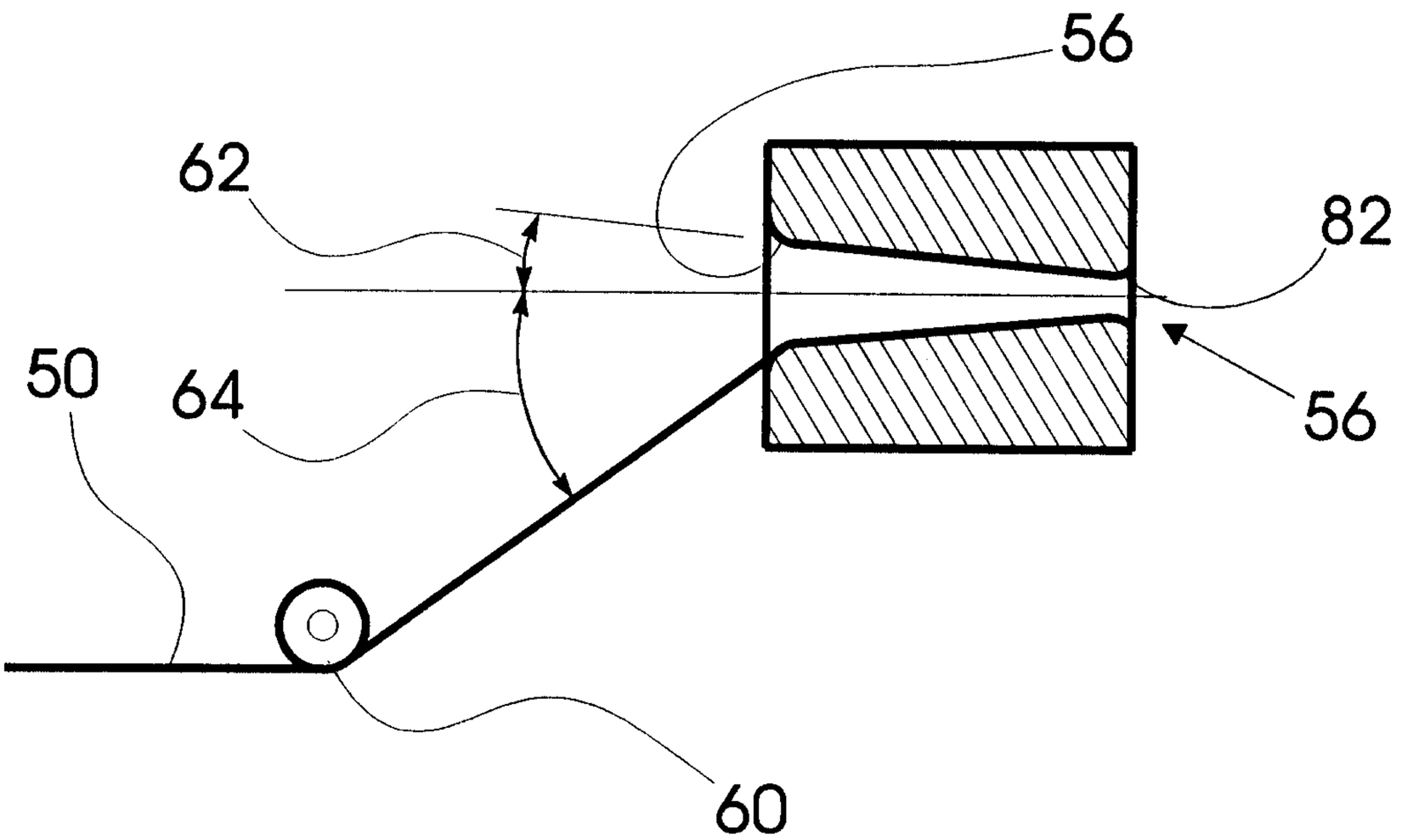
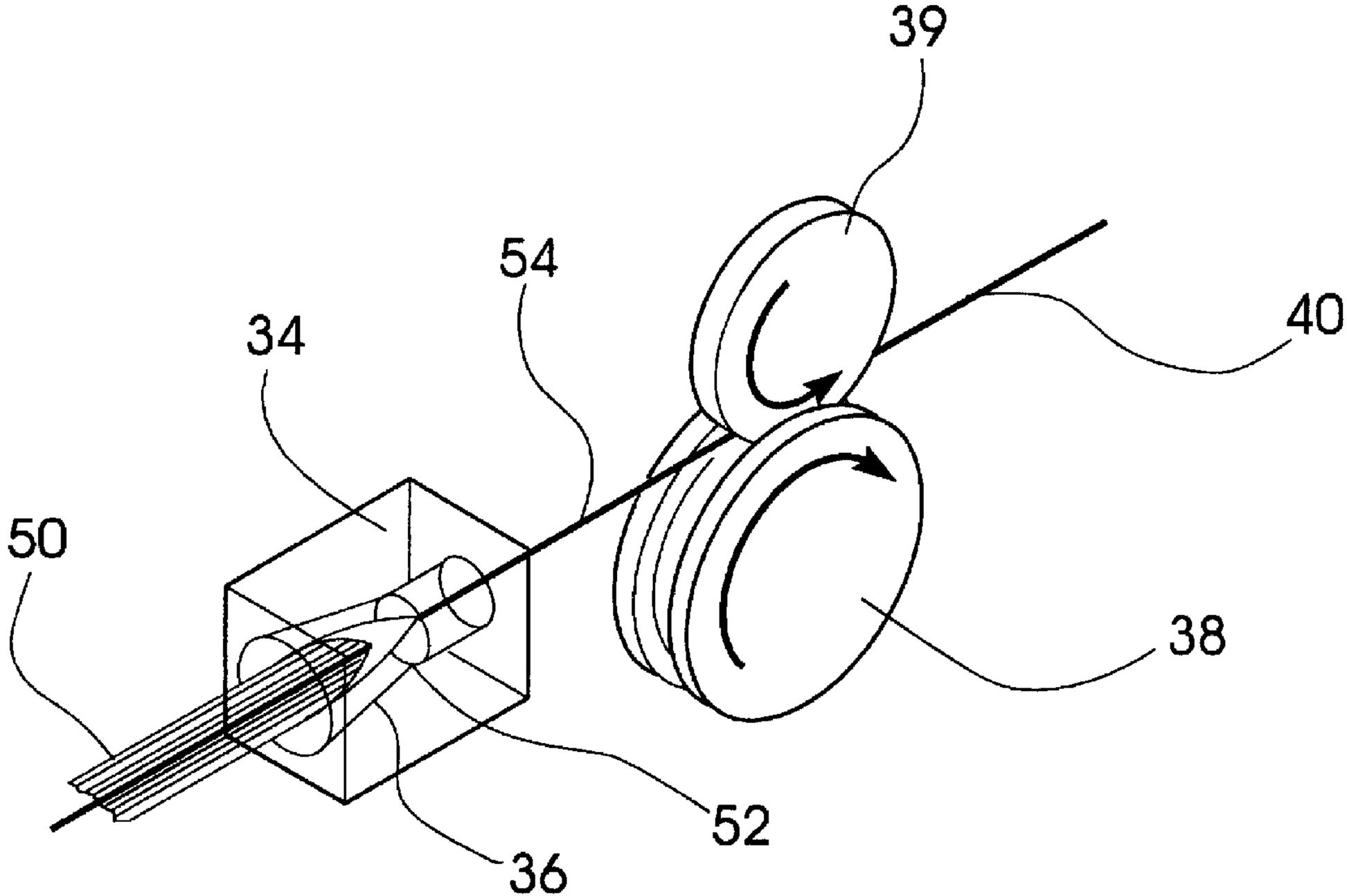


Fig.14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