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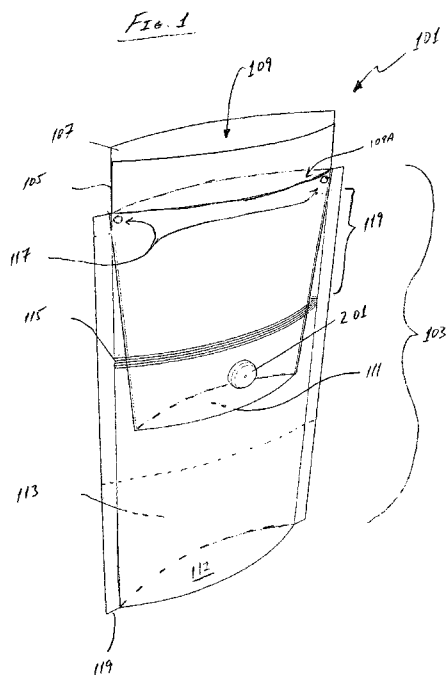
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(54) Title: SAMPLING BAG AND FUNNEL FOR COLLECTION OF SOILS, MUDS, OR OTHER SOLIDS OR LIQUIDS
FOR SUBSEQUENT ANALYSIS OF HEADSPACE GASES AND/OR OTHER CONTENTS VIA STABLE ISOTOPE ANAL-
YSIS (C,H,N,O,S) OR OTHER ANALYTICAL PROCEDURES



(57) Abstract: A collapsible bag or pouch for sample collecting having an internal mechanical seal and a heat sealable area near the opening that is protected during sample loading, preferably by a funnel detachable from the pouch. After loading and prior to heat sealing the pouch is secured from the outside by mechanical interlocking seal. The bag also has an air-tight port for sampling the gaseous headspace after the bag is sealed.

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Sampling bag and funnel for collection of soils, muds, or other solids or liquids for subsequent analysis of headspace gases and/or other contents via stable isotope analysis (C, H, N, O, S) or other analytical procedures

Related Applications

This application is based on provisional application 61/217944, filed 5 June 2009, the disclosure of which is incorporated herein by reference.

Background

Historically, isotope analysis originated as a major tool for the field of geochemistry. However, as mass spectrometers and other equipment for taking isotopic measurements improve and advance, so does the utility of isotopic data in other areas. These have come to include oil and gas exploration, and gas identification and differentiation. In addition, isotope data has also become a useful tool in other fields, such as ecology, forensics, food adulteration, and others where isotope data can often provide a means of differentiating two otherwise chemically indistinguishable or undifferentiated substances.

As our world's energy supplies diminish, increasing attention is being given to recovering sources of oil and gas that were previously considered uneconomical and/or unconventional. These sources may be identified through "geochemical prospecting" of surface soils. A bag for containing a sample of soil and its associated gases, without altering its isotopic "signature" would be extremely valuable in helping to identify these sources. However, isotopic differences can arise in a sample during storage. Accordingly, it would be beneficial to control for mechanisms that might alter the isotopic signature of a sample substance. Geological sampling often requires specialized sampling containers and equipment, as described in various of the following published US applications: 20100083771 (Gas sampling apparatus); 20090260416 (Low dead-volume core-degassing apparatus); 20080282814 (Gas sampling apparatus); 20070056394 (Pressurized gas sampling container); 20040123679 (Gas sampling apparatus); the disclosures of which are incorporated herein by reference. Such devices can be used to contain a substance without altering or "fractionating" the sample's isotopic signature.

Traditional means of collecting soil samples utilize jars or other rigid containers which are quite bulky and relatively expensive to ship, especially to remote regions of the world where such prospecting often occurs. A rigid container (such as a jar) is always contaminated and/or its contents "diluted" by the atmospheric gases that occupy the container, and which constitute the headspace after the sample is introduced and the container sealed. This gaseous contamination (including atmospherically-borne contaminants) makes subsequent isotopic analysis difficult if not impossible. Additionally, techniques have been developed for sourcing groundwater from core samples using stable isotope analysis. In order to use such techniques, however, it is essential that samples be stored in such a way that prevents isotopic fractionation of the water vapor inside (*i.e.*, no leakage on a molecular level) .

Summary of the Invention

In light of the foregoing, this invention provides a sample collection pouch having a sample collection bag in the form of a pouch having an opening to an interior portion and a closed end, the pouch comprised of opposing facing films sealed along opposing edges, a portion of the film defining the interior portion and adjacent said opening comprising a heat sealable material, corresponding protrusions formed in said facing films to create a closure for sealing the bag, a self-sealing port disposed in one of said films between said closure and said closed end, and a removable funnel disposed in and extending from said opening to and beyond said closure.

More generally, this invention provides a collapsible bag or pouch for sample collecting having an internal mechanical seal and a heat sealable area near the opening that is protected during sample loading, preferably by a funnel detachable from the pouch. After loading and prior to heat sealing the pouch is secured from the outside by mechanical interlocking seal. The bag also has an air-tight port for sampling the gaseous headspace after the bag is sealed.

The invention also provides a clip for sealing the pouch, comprising a channel formed of a relatively rigid but flexible material, a softer, compressible material within the channel mostly surrounding a rigid rod inserted into and removable from the channel.

In use, the portion of the pouch adjacent the opening is heat sealable and is shielded from contamination by the funnel, which is removed after the sample is introduced. Thereafter, the

closure is sealed, the clip is then secured between the closure and the opening, and one or more heat seals are formed from the facing films of the pouch between the claim and the opening to produce a sample contained within a pouch having two mechanical seals and at least one heat seal.

This invention advantageously provides a compressible container, a bag or pouch, that addresses the need to reduce dilution of a sample substrate that outgases and/or desorbs gases over time. The ability to squeeze out excess air prior to sealing means the headspace will have in a higher concentration of gases targeted for analysis (*e.g.*, isotopic analysis). This is important because some testing (such as isotopic testing) requires a sufficient concentration of the gaseous compound to provide data other than its mere presence. The compressible wall of the inventive container allows removal of air from the air head space, which reduces both air contamination and dilution of the sample gases.

Brief Description of the Figures

Fig. 1 is a perspective view of a sample collection pouch according to the invention.

Figs. 2A and 2B are, respectively, cross-sectional and perspective views of the self-sealing port.

Figs. 3A and 3B are, respectively, perspective views of the clip components (shown partially exploded) and of the clip sealed onto the pouch.

Fig. 4 is a perspective view of a sample collection pouch with sample and sealed.

Detailed Description of Particular Embodiments

As shown in Fig. 1, the invention preferably includes a wide-mouth, pleated sample bag 101 produced of a material that will not alter the isotopic "signature" of the material which it contains. The bag is generally made of plastic and/or foil films; transparent or translucent plastic is most preferred; metallized, mostly transparent films are acceptable if non-reactive with the sample contents. Different film materials can also be utilized to create customized pouches specifically designed or chosen to contain the particular "species" of interest.

The pouch or gas bag portion 103 has an open end in which a funnel 105 is positioned. The funnel includes a wider mouth portion 107 defining an opening 109 that is essentially coextensive with the opening of the pouch, and continues to a narrower end opening 111 disposed within the pouch. The base 112 of the pouch defines the closed end, and the adjacent portion of the pouch defines an interior portion 113 in which a sample is retained.

Between the sample-retaining portion of the interior and the opening of the pouch is a mechanical seal closure 115. Preferably, this closure is a profile extrusion in the opposing films to provide an interlocking closure, such as described in US 4,561,109 and Re. 28,969 (*e.g.*, a re-sealable zip type closure), the disclosures of which are incorporated herein by reference.

The funnel is preferably tacked to the pouch (*e.g.*, glued, heat-sealed, or tack-welded) at small areas, 117, adjacent the opposing seams where the films meet, the areas being sufficient to keep the funnel in place during transport, but small enough to allow the funnel to be torn out without significant (and preferably without any) damage to the pouch. The funnel then can be tacked to the pouch only at the portion near the opening. By virtue of this structure, the user tears the funnel away from the bag.

The pouch is preferably made from opposing films. The films are preferably multilayer films to provide both strength and durability to the pouch and to protect the sample from isotopic signature changes. In addition, the use of films allows the pouch to be collapsed and the air forced out prior to sealing, in contrast to the rigid containers used presently. Still further, the portion of the films defining the interior space are preferably heat-sealable in the area between the closure 115 and the bag opening 109A. As described, the funnel spans the same extent, thereby protecting preferably both the heat-sealable material and the mechanical closure, and the port, from contamination by sample materials, which are directed by the funnel to the sample retaining portion (113) of the pouch.

The portions of the films defining the heat-sealable area 119 preferably comprise a polyolefin that is heat sealable. Commercial and home-use heat-sealing systems for storing food are well-known. In this invention, polyethylene (PE), especially low density, is preferred. Depending on the sample material collected, one or more layers may be treated, and/or layers may be provided, to secure the chemical signature of the sample contents remains unchanged.

For example, an ethylene vinyl alcohol (EVOH) copolymer can be used as the heat sealable material to reduce oxygen penetration through the bag to the sample. For samples containing carbon dioxide, a higher density (HDPE) and/or a more crystalline polyolefin, and/or a metal foil, can be used to provide a hermetically sealed interior. The outer layer is preferably relatively tough, such as a polyamide (*e.g.*, a nylon), a polyethylene terephthalate (PET), or polypropylene, or combinations thereof. Various ways of making multilayer film materials are well-known, including co-extrusion, lamination, and combinations thereof. Exemplary multilayer films include a two layer film (outside to inside) with nylon and EVOH/PE, and a five layer film with PET, HDPE, foil, regular density PE, and low density PE. Such films are commercial available as, for example, a 5mil clear biaxially oriented nylon and EVOH/LLDPE laminated barrier film, and a 4mil white laminated PET/PE/foil/PE/LLDPE film. The collected samples will produce gasses, termed off gasses. The films are substantially impermeable to off gasses and thus the gasses are retained. The films are also substantially impermeable to ambient atmosphere and gasses generally existing in the environment to prevent dilution of off gasses within the bag. Further more the films are non-reactive with the gathered sample and the sample off gases to preserve the isotopic fractions of the off gasses.

Disposed in one of the films is a self-sealing port 201, which is shown in more detail in Figs. 2A and 2B. The port is preferably designed to be snapped in place over the film. The port has an upper cap 203 that will be on the outside of the pouch and, as shown, preferably has a tapered bore 205 and a depending side wall having exteriorly a circumferential foot 207, and interiorly a rib 209 defining an annulus. The rib engages a circumferential recess 211 formed in the bottom portion 213 of the port when the top and bottom snap together (*i.e.*, the rib of the cap snaps into the recess of the bottom).

The side wall of the pouch 103 is disposed between the cap and bottom portion when the two are snapped together. The bottom also has a flange 215 extending about the perimeter which forms a surface on which the foot of the cap lies flush, and for added sealing includes a groove 217 in which is disposed a seal (*e.g.*, an O-ring or synthetic washer) 219 secured between the cap foot and the flange. Axially, the bottom has a bore 221 that aligns with the tapered hole in the cap and opens into a center cavity 223 in the bottom portion. The cavity accepts a penetrable,

self-sealing plug or septum that blocks the bore and is positioned between the tapered hole in the cap and the bore. When assembled, a hypodermic needle can be inserted through the port (the first insertion thus penetrating the film) to remove material for analysis without compromising the chemical signature of the sample contents, or to add material (*e.g.*, a liquid or gaseous reagent, or an inert gas or liquid). As shown, the upper or outermost portion of the cap preferably is separated from the foot by a groove to provide improved tactile sensation while grasping the cap. This can be an important consideration for avoiding puncturing the opposing film of the pouch when the plug is penetrated by the hypodermic needle. The groove also provides a means by which the pouch can be suspended and the head space sampled through the port.

In field use, for example, a hand spade or garden trowel is used to collect soil and then inserted through the funnel to place a sample of the material collected into the closed end portion of the sample container. Preferably, the outside of the pouch in the area in which the sample is retained (113) includes printed indicia with desired information such as, for example, a fill line, instructions for use, a form for indicating sample information (*e.g.*, date, location, sample identification (such as a unique sample number or name), location from which the sample was taken, by whom the sample was taken, sampling conditions), or other notes or comments. After the sample is introduced, the bag is held vertically, the funnel, is removed, the bag compressed to remove the head space air and the re-sealable zip closure is secured, to retain the sample in the bag during the remaining manipulation.

It is preferred then to place a clip onto the bag because the closure does not form a gas tight seal, so leakage or reaction with incoming gas would fractionate or otherwise change the sample. The clip is secured onto the outside of the pouch. A preferred clip 301 is shown in Figs. 3A and 3B as having a channel 303 in which is positioned a filling 305. The channel includes opposing cantilevers 307 to define an opening 309 of the channel. The channel material is preferably plastic that is relatively rigid, such as polyvinyl chloride, and also flexible. The filling is a soft material, preferably plastic, such as a vinylidene polymer, and may be highly elasticized. The clip also includes a rigid rod 311 which is inserted into the channel and retained by a combination of a close fit with the filling and the rigidity of the opposing cantilever support

walls; the walls allow sufficient expansion for the rod to fit into a bore 313 defined by the filling and then reform the bore around the rod. The rod can be metal or plastic, or even ceramic, so long as it is durable in the field and during transport, and acts cooperatively with the filled channel to produce a seal. Preferably, the channel and filling are co-extruded. As shown in Fig. 3B, the heat-sealable portion of the pouch is secured by the clip.

The clip is capable of creating a vacuum tight seal. By virtue of the difference in rigidity of the two parts of the clip and the conformable nature of the soft inner portion, soft inner section of the clip conforms to irregularities in the films and can 'surround' these inconsistencies, providing consistent pressure along the length of the clip, and therefore providing a reliable means of sealing gases inside the pouch. Without these properties, the pressure on the inner rod tends to be inconsistent and incapable of providing a vacuum tight seal.

As noted, the closure is capable of being opened. Accordingly, it is preferable to heat-seal the sample pouch, and multiple seals are more preferred. As noted above, the funnel protects the heat-sealable material during introduction of the sample. After the funnel is removed, a commercially-available heat sealing machine can be used to add additional, permanent seals. It may be impractical, or impossible, to have such a device at the point of sampling, or even on location. Accordingly, the aforementioned clip is used to secure the pouch until the pouch can be heat-sealed. As shown in Fig. 4, a fully sealed pouch containing a sample 401 is secured by the closure 115, the clip 301, and one or more heat seals 403. After heat sealing, the clip may be removed.

The foregoing description is meant to be illustrative and not limiting. Various changes, modifications, and additions may become apparent to the skilled artisan upon a perusal of this specification, and such are meant to be within the scope and spirit of the invention as defined by the claims.

What is claimed is:

1. A sample collection bag, comprising:
 - a. a pouch having an opening to an interior portion and a closed end, said pouch comprised of opposing facing films sealed along opposing edges, a portion of said films defining the interior portion and adjacent said opening comprising a heat sealable material, corresponding engageable protrusions formed in said facing films to create a closure for sealing the bag;
 - b. a self-sealing port disposed in one of said films between said closure and said closed end; and
 - c. a removable funnel disposed in and extending from said opening to said closure.
2. The bag of claim 1, wherein said films defining the interior portion comprise heat sealable polyethylene.
3. The bag of claim 1, wherein said films are multilayer films.
4. The bag of claim 3, wherein said films comprise a first polyolefin layer and one side thereof, on or more layers selected from the group consisting of foils, a second polyolefin, a polyamide, a polyalkylene terephthalate, compatible combinations thereof, and compatible mixtures thereof.
5. The bag of claim 3, wherein said first polyolefin comprises a mixture of polyethylene and ethylene vinylalcohol.
6. The bag of claim 3 wherein said films are substantially impermeable to sample off gasses.

7. The bag of claim 3 wherein said films are substantially impermeable to ambient air and gases.
8. The bag of claim 3 wherein said films are non-reactive with said sample and sample off gasses.
9. A clip, comprising: a channel of a relatively rigid material filled with a relatively softer and more elastic material compressively holding within the channel a rigid rod.
10. The bag of claim 1, further comprising the clip of claim 9 disposed and sealing a portion of the pouch between the closure and the opening.
11. The bag of claim 1, further comprising at least one heat seal between said facing films disposed between the closure and the opening.
12. The bag of claim 7, further comprising at least one heat seal between said facing films disposed between the clip and the opening.
13. A kit, comprising, in combination, the bag of claim 1 and the clip of claim 6.
14. The bag of claim 1, further comprising indicia printed thereon.

15. A method for securing a sample for testing, comprising:
 - a. providing a collapsible pouch having
 - i. a sample-containing interior portion at the closed end of the pouch and defined by a material non-reactive with the retained sample,
 - ii. a first interlocking mechanical seal disposed between the sample-containing portion and the open end; and
 - iii. a heat-sealable interior portion adjacent the opening of the pouch;
 - b. protecting the heat-sealable interior portion and the interlocking mechanical seal from contamination;
 - c. introducing a sample into the sample-containing interior portion of the pouch;
 - d. compressing the collapsible pouch allowing headspace air to be removed.
 - e. removing the protection applied in step b.;
 - f. securing the first interlocking mechanical seal;
 - g. sealing the pouch from the outside with a second interlocking mechanical seal;
 - h. abutting and heat sealing said heat sealable interior portion adjacent the opening.

16. The method of claim 13, further comprising a plurality of heat seals.

Fig. 1

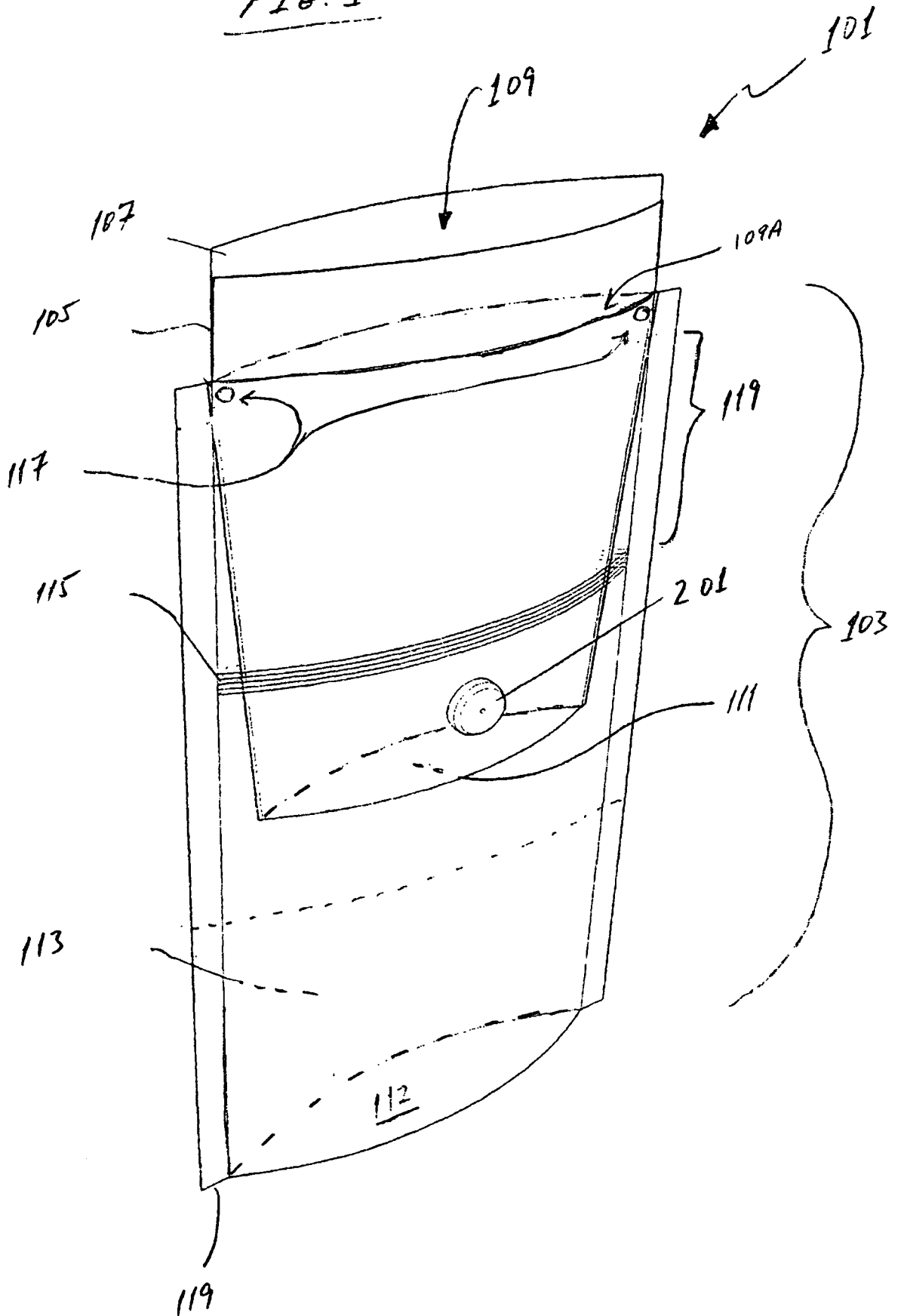


FIG. 2A

FIG. 2B

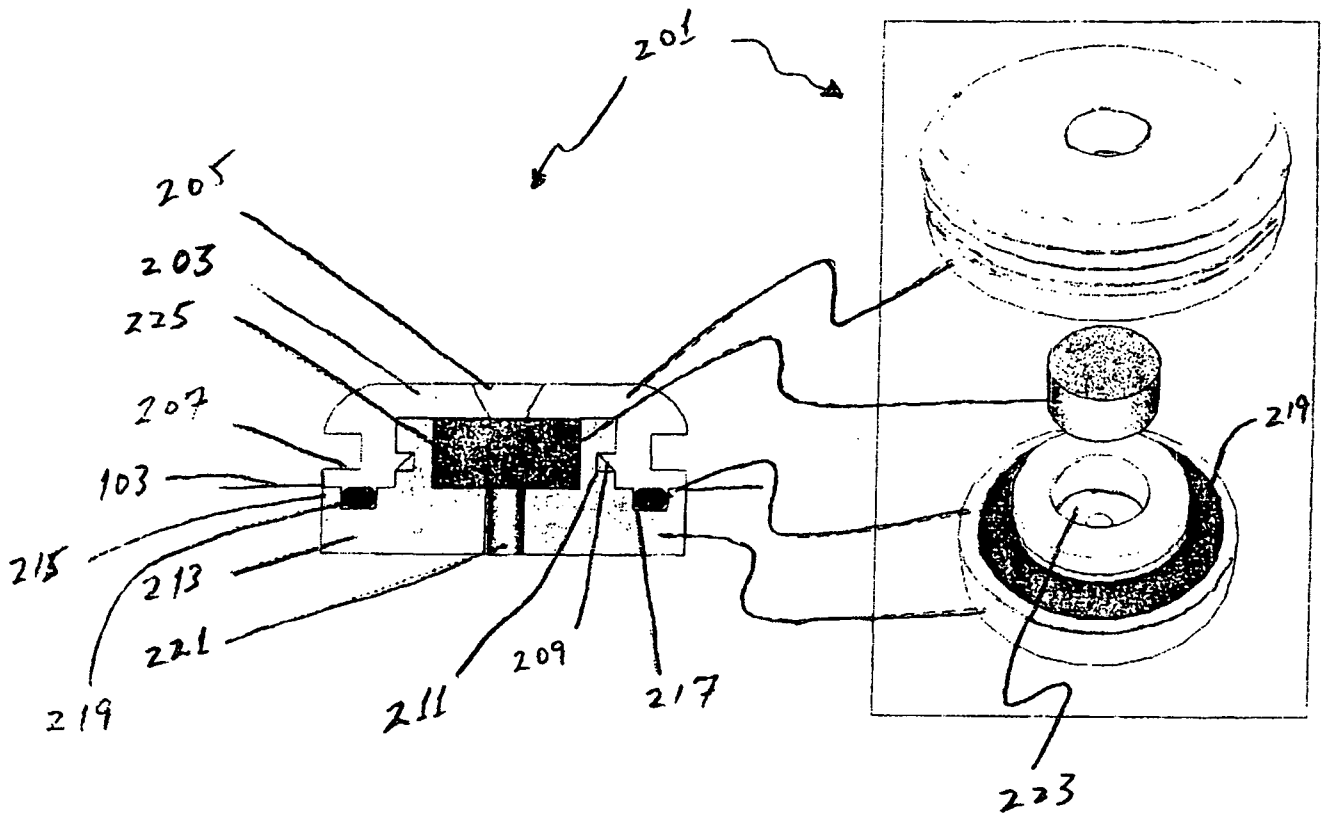


FIG. 4

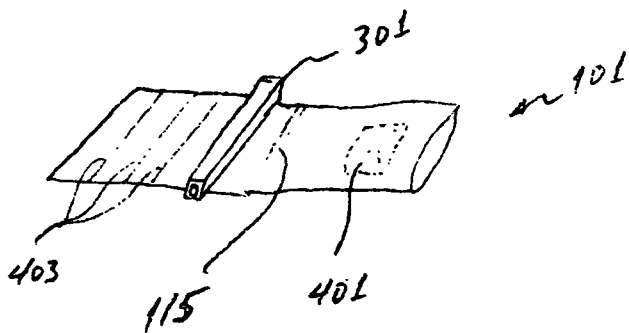


FIG. 3A

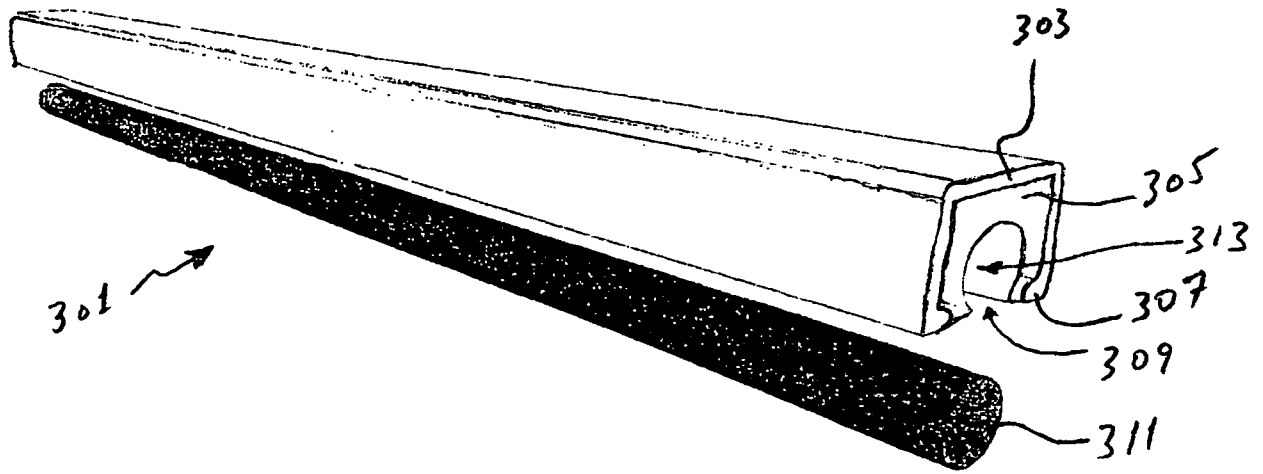


FIG. 3B

