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(54) **REFRACTORY PLUG OR BRICK FOR INJECTING GAS INTO MOLTEN METAL**

FEUERFESTER BAUKÖRPER ODER STEIN ZUM EINBLASEN VON GAS IN GESCHMOLZENE
METALLE

BRIQUE OU BOUCHON REFRACTAIRE POUR L'INJECTION DE GAZ DANS DU METAL EN FUSION

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Description

[0001] The present invention relates to a refractory plug or brick for vessel lining for injecting gas into molten metal and to the manufacture of a refractory plug or brick for injecting gas into molten metal.

[0002] Gases are often injected into molten metal in vessels such as ladles, crucibles or tundishes for diverse purposes. For instance, a gas may be introduced into the bottom part of a vessel to clear the relatively cool bottom area of solidification products, e.g. to remove them from the vicinity of a bottom pour outlet where the vessel has such an outlet. In steel making for example, the use of slow injection of a fine curtain of gas bubbles in the tundish assists in inclusion removal; the inclusions being attracted to the fine gas bubbles and rising upwards through the melt to the surface where they are conventionally captured by the tundish cover powder or flux. Gas may also be introduced for rinsing or to homogenise the melt thermally or compositionally, or to assist in dispersing alloying additions throughout the melt.

[0003] Usually, an inert gas is used but reactive gases may also be employed, e.g. reducing or oxidising gases, when the melt compositions or components thereof needs modifying. For example, it is customary to inject gases such as nitrogen, chlorine, freon, sulphur hexafluoride, argon, and the like into molten metal, for example molten aluminium or aluminium alloys, in order to remove undesirable constituents such as hydrogen gas, non-metallic inclusions and alkali metals. The reactive gases added to the molten metal chemically react with the undesired constituents to convert them into a form such as a precipitate, a dross or an insoluble gas compound that can be readily separated from the remainder of the melt. These gases (or others) might also be used for example with steel, copper, iron, magnesium or alloys thereof.

[0004] In order to efficiently carry out a gas injection operation, it is desirable that the gas be introduced into the molten metal, preferably from the bottom of the recipient, in the form of a very large number of extremely small bubbles. As the size of gas bubbles decreases, the number of bubbles per unit volume increases. An increase in the number of bubbles and their surface area per unit volume increases the probability of the injected gas being utilised effectively to perform the expected operation.

[0005] Previous gas injection proposals have included the installation of a solid porous refractory plug or brick in the refractory lining of the vessel, generally on the floor but also in the walls. In use, the plugs or bricks introduce a flow of gas in the form of bubbles.

[0006] For example, a known technique (GB-A-2 096 290) for introducing gas into molten metal consists of lining a portion of a molten metal-containing vessel (preferably the floor of the vessel) with a porous ceramic body. The gas is introduced into the porous body at a location remote from the metal-contacting surface of the body. During its passage through the body, the gas follows a number of small, tortuous paths such that a large number of bubbles will be issued into the molten metal.

[0007] Generally a metal casing that acts as a manifold to introduce gas into the body supports the porous ceramic body. Typically, the casing is made of mild steel (for use with inert or slightly reactive gas such as argon or nitrogen) or inconel (for use with highly reactive chlorine or freon). The assembled body/casing is surrounded and supported on all sides except its upper surface by refractory material such as low-cement alumina castable or bricks. When castable is used this can either be cast in situ around the porous body or formed from pre-cast components fixed in place during the hot metal container lining installation. The lining material will "butt" up against the porous body construction.

[0008] A problem with the foregoing construction is that it is difficult to maintain an effective gas seal between the casing and the body, and between the casing and the support castable/bricks. One difficulty arises in part because the coefficients of thermal expansion of the metal casing and the refractory materials are considerably different; also, the metal casing is subject to attack if chlorine is the gas being used. If a crack should develop (as used herein, the term "crack" refers to any defect in the gas dispersing apparatus that permits undesired gas leakage), gas will leak through the crack, and hereafter frequently will migrate through the next brick and the refractory support to the ambient atmosphere. Gas migration through 50 cm or more of refractory material is possible. The problem is undesirable as the effect of gas leakage upon the flow of gas through the designed gas bubbling surface can be seriously reduced and effectiveness of the bubbling block diminished. In some cases gas flow by means of fine gas bubbles will cease and be replaced with uncontrolled direction of gas flow by means of large ineffective gas bubbles. If argon is being used the relatively great expense must be considered. The problem is particularly acute in the case of chlorine due to the harmful effects of chlorine upon release into the atmosphere. Regardless of the type of purifying gas being used, it is important that cracks be prevented so that gas leakage will be prevented.

[0009] Desirably, a technique would be available for injecting gas into molten metal that would accomplish the objectives of dispersing a large number of exceedingly small bubbles into the molten metal while, at the same time, avoiding cracks in the gas dispersing apparatus that results in gas leakage.

[0010] It also would be desirable for any such apparatus to be capable of being manufactured easily, at reasonable expense and having smaller dimensions than the existing apparatus. Further, it would be desirable that any such gas injection apparatus be usable with existing equipment such as a tundish, ladle, melting vessel, and the like, with no

modification, or with only minor modification, of the existing equipment.

[0011] Further, in order to insert this apparatus into the existing refractory lining of the molten metal vessel, it would be desirable that any such gas injection apparatus be compatible with the surrounding refractory materials to prevent any adverse chemical reactions of thermal expansion miss-matches.

[0012] Further, it would be desirable to provide an apparatus that could be adjusted to a very broad range of bubbling conditions (bubble size, volume, pressure, etc.) by only minor adjustments during the manufacturing process so that the apparatus can suit specific customer requirements.

[0013] The invention relates thus to a solid porous refractory plug or brick for vessel lining for injecting gas into molten metal through a molten metal-contacting surface comprising

- i) a porous refractory body substantially surrounded by a substantially non-porous body except at the molten-metal contact surface; and
- ii) gas conducting means for conveying the gas from a gas source to the porous body.

The plug or brick according to the invention is characterized in claim 1. The process for manufacturing it is set out in claim 7.

[0014] US-A-4 791 978 and 4 836 508 relating to a stopper rod and a pouring nozzle, respectively, do disclose copressed refractory bodies which are dense and porous, respectively, but do not refer to the problem of improving gas tightness of a non-metal permeable plug or brick forming part of a vessel lining and having the sole purpose of injecting gas for treating the molten metal in the vessel.

[0015] In the scope of the present specification a plug or brick for vessel lining for injecting gas can be a plug, brick, block, dam, tile, bar, and the like. As discussed above, the plug or brick of the invention can be used to inject any gas (whether reactive or inert) into any molten metal or alloy thereof. The plug or brick has at least a molten-metal contacting surface through which the gas is injected. The plug or brick comprises a porous refractory body substantially surrounded by (for example encased by or embedded in) a non-porous body except, of course, at the molten-metal contacting surface. It can be included or form part of the lining of the molten metal vessel.

[0016] The porous body can be made of any porous refractory material. As a matter of fact, the nature of the material used is not essential as far as said material has the required porosity. Generally, it will be considered that a material having an apparent porosity higher than 20 % is porous. Suitable materials typically comprise alumina, alumina spinel, magnesia or magnesia spinel, or combinations of any of the above.

[0017] The plug or brick also comprises gas conducting means for conveying the gas from a gas source, to the porous body. The gas conducting means generally comprises a conduit extending through the side wall of the non-porous body. This conduit can be made from metal or a refractory material for example. The conduit can be fixed into place by means of a conventional refractory sealing material (mortar or cement) or it can be pressed within the non-porous body.

[0018] Conventional gas conducting means can be utilised. However, since the tightness of the connection is to be particularly monitored, special arrangements such as for example these disclosed in WO-AI-01/83138 are particularly preferred.

[0019] It is also advantageous that the gas conducting means comprises a plenum chamber through which the gas contacts a surface of the porous body at least substantially equivalent to the molten metal contacting surface so that the gas is perfectly homogeneously distributed into the porous body and, consequently, will bubble into the molten metal through substantially the whole molten-metal contacting surface.

[0020] This type of plug or brick of injecting gas into molten metal is known for example from USP 5,054,749, 5,423,521 or 5,219,514. However, none of them satisfies the above identified requirements.

[0021] The plug or brick of the present invention is characterised by the fact that the non-porous body is made of refractory material and that the porous and non-porous bodies have been co-pressed. All the above identified requirements are fulfilled with such a plug or brick.

[0022] Again, the nature of the non-porous material is not essential as long as it is a refractory material and has the required porosity. Generally, it will be considered that a material having an apparent porosity lower than 20 % is non-porous.

[0023] The non-porous body and the porous body are preferably constituted of refractory materials with similar coefficients of thermal expansion. This serves to prevent the formation of cracks upon thermal cycling.

[0024] By use of the present invention, the granulometry and permeability of the inner porous body can be carefully and consistently controlled to provide an even fine pore structure so that small evenly distributed gas bubbles flow from the molten metal contacting surface of the body. This permeability can be readily adjusted via formulation granulometry changes and a plug or brick according to the invention can be manufactured to suit specific individual customer requirements.

[0025] This process route is further advantageous in that high magnesia content refractories can be used for the

formulations such as magnesia spinel. Such formulations are more compatible with the composition of steel plant tundish linings which are usually basic (magnesia) based. Chemical and thermal characteristics are therefore very similar. Advantageously the porous and non-porous refractory bodies have thus a high magnesia content, more than 50 %, preferably more than 80 % and even more preferably more than 90 % by weight of the composition.

[0026] Accordingly, similar materials but with different granulometries can be used for the porous and non-porous bodies. Thus, it is possible to manufacture a plug or brick according to the invention from high magnesia content materials having different granulometries.

[0027] By virtue of the co-pressing of the two refractory materials, the natural low permeability of the non-porous body prevent gas leakage without having to resort to other gas leakage restriction techniques. Another advantage of the co-pressing is that a plug or brick for injecting gas with lower overall dimensions, achieving the required degree of gas bubbling can be used. This assists in handling of these plug or brick during transport and installation in the vessel; in particular in the lining.

[0028] The co-pressing concept is not restricted to oblong, square, round or oval shapes but can also be used to produce any refractory cross section suitable for co-pressing. For example a ring co-pressed component can be envisaged which would be located to surround the exit ports of a tundish thereby forming a surrounding stream of gentle rising bubbles through which the hot liquid metal would have to pass prior to entry into the continuous casting moulds.

[0029] According to another of its aspect, the invention concerns a process for the manufacture of a plug or brick for injecting gas into molten metal. According to the invention, this process comprises the steps of:

- 1) introducing into a mould of appropriate amounts of the refractory materials constituting the porous and non-porous bodies while respecting the desired limits for these bodies;
- 2) simultaneous co-pressing of both refractory materials;
- 3) providing gas conducting means;
- 4) heat treating the co-pressed materials.

[0030] Preferably, a delimiter, for example made from a thin (but rigid) plastic or metal foil is placed into the mould prior to introducing the refractory material. The delimiter can be shaped as a cylinder (with circular or oval base), or a parallelepiped, without upper and lower surfaces. The refractory material which will form the porous body is then introduced in the central portion formed by the delimiter and the refractory material which will form the non-porous body is introduced between the delimiter and the mould wall. The delimiter is then carefully removed and another amount of the material forming the non-porous body is introduced into the mould to form the surface opposite to the molten metal contacting surface.

[0031] The step of providing the gas conducting means can be carried out before or after the co-pressing step or both before and after. In a preferred embodiment of the invention, a plenum chamber is formed by introducing a strip of consumable material into the mould at the junction between the base of the porous body forming material and the adjacent surface of the non-porous body forming material.

Alternatively or in addition, a hole can be drilled or a conduit placed through the non-porous body after or before co-pressing the materials to connect the porous body (whether or not through the plenum chamber) to an external gas source.

[0032] The co-pressing step can be carried out according to any known pressing method, for example in an hydraulic press.

[0033] The heat treating step should be carried out at a temperature sufficient to develop a ceramic bond between the porous and non-porous bodies so that the integrity of the plug or brick and its gas tightness are enhanced. The consumable material (if used) placed to generate the plenum chamber will advantageously be removed during the heat treating step. This consumable material can burn (cardboard, paper) or melt (wax, alloy) at the temperature used. Typically, the heat treating step consists in firing the co-pressed material for a temperature comprised between 800 and 1800°C for 2 to 12 hours.

[0034] The invention will now be better described with reference to the enclosed drawings which are only provided for the purpose of illustrating the invention and not to limit its scope. Fig. 1 and 2 show cross-sectional views of embodiments of the invention.

[0035] Both figures show a plug or brick (1) for injecting gas into molten metal through a molten metal-contacting surface (11) comprising a porous refractory body (2) substantially surrounded by a substantially non-porous body (9) except at the molten metal-contacting surface (11). Also visible on figures 1 and 2 are the gas conducting means comprised of a metallic or refractory conduit (4) extending through a wall (6) of the plug or brick and connecting to the plenum chamber (3). The conduit (4) is typically fixed into place by means of a conventional sealing cement or mortar (5).

[0036] Advantageously, a gradual tapered section (7) is created towards the molten metal-contacting surface (4) during the pressing step as depicted on figure 1. This taper effect is created during the pressing action by the porous body deforming into the non porous medium at the vertical sides of the pressing mould. This tapered shape further

protects the porous body (2) by forming a key from major spalling effect.

[0037] According to an example of the invention, the materials used are the following: (% given by weight):

	Non-porous body	Porous body
Silica	0.1	0.13
Alumina	3.3 (< 45µm)	0.06
Iron oxide	0.2	0.48
Lime	0.4	0.69
Magnesia	96.0*	98.5**

Granulometry: *: >1mm 30%
<45µm 30%

** : >1mm 0%
<45µm 5%

[0038] After being introduced into the mould, the materials have been mechanically pressed at a rate to ensure the best possible compaction and integration of the co pressed materials . The heat treating step was carried out by slowly heating the co-pressed material at a rate to avoid thermal fractures/cracking within the pressed body until 1600°C, leaving the plug or brick at this temperature for 4 hours and allowing it to cool gently.

The following properties have been measured:

	Apparent porosity (%)	Bulk density (g/cm ³)	Cold modulus of rupture (MPa)	Cold crushing strength (N/mm ²)	Average pore diameter (µm)
Non-porous body	15.4	2.99	7.11	90.15	6.861
Porous body	24.9	2.59	7.12	52.15	44.762

[0039] In use the plug or brick has injected fine bubbles reliably and constantly.

Claims

1. Solid porous refractory plug or brick (1) for vessel lining for injecting gas into molten metal through a molten metal-contacting surface (11) comprising

i) a porous refractory body (2) substantially encased in a substantially non-porous body (9) except at the molten metal-contacting surface (11); and

ii) gas conducting means (3,5) for conveying the gas from a gas source to the porous body (2),

characterized in that the non-porous body (11) is made of refractory material and **in that** the porous and non-porous bodies (2,11) have been co-pressed.

2. Plug or brick according to claim 1 **characterized in that** the gas conducting means (3,5) comprises a plenum chamber (3).

3. Plug or brick according to claim 1 or 2, **characterized in that** the porous and non-porous bodies are constituted

of refractory materials with similar coefficients of thermal expansion.

4. Plug or brick according to any one of claims 1 to 3, **characterized in that** the porous and non-porous bodies are constituted of more than 50 weight % of magnesia, magnesia spinel, alumina or alumina spinel, preferably more than 80 weight %.

5. Plug or brick according to any one of claims 1 to 4, **characterized in that** the non-porous body comprises more than 50 weight %, preferably more than 80 weight %, of magnesia, magnesia spinel, alumina or alumina spinel.

6. Plug or brick according to any one of claims 1 to 5, **characterized in that** the porous body (2) has a tapered section (7) towards its molten metal-contacting surface.

7. Process for the manufacture of a solid refractory porous plug or brick for vessel lining for injecting gas into molten metal through a molten metal-contacting surface comprising the steps of:

- 1) introducing into a mould of appropriate amounts of the refractory materials constituting the porous and non-porous bodies while respecting the desired limits for these bodies;
- 2) simultaneous co-pressing of both refractory materials;
- 3) providing gas conducting means;
- 4) heat treating the co-pressed materials.

8. Process according to claim 7, wherein the gas conducting means comprises a plenum chamber.

9. Process according to claim 8, **characterized in that** the step (3) of providing the gas conducting means comprises a step of introducing a strip of consumable material into the mould at the junction between the porous and non-porous bodies.

10. Process according to claim 9, **characterized in that** the consumable material comprises wax.

Patentansprüche

1. Fester poröser Feuerfeststopfen oder -ziegel (1) für eine Behälterauskleidung zum Injizieren von Gas in geschmolzenes Metall durch eine Berührungsfläche (11) zum geschmolzenen Metall, umfassend

- i) einen porösen Feuerfestkörper (2), der im Wesentlichen außer an der Berührungsfläche (11) zum geschmolzenen Metall in einen im Wesentlichen nicht-porösen Körper (9) eingeschlossen ist; und
- ii) eine Gasleiteinrichtung (3, 5) zum Fördern des Gases aus einer Gasquelle zu dem porösen Körper (2),

dadurch gekennzeichnet, dass der nicht-poröse Körper (11) aus Feuerfestmaterial besteht und **dass** der poröse und der nicht-poröse Körper (2, 11) gemeinsam gepresst worden sind.

2. Stopfen oder Ziegel nach Anspruch 1, **dadurch gekennzeichnet, dass** die Gasleiteinrichtung (3, 5) eine Verteilerkammer (3) umfasst.

3. Stopfen oder Ziegel nach Anspruch 1 oder 2, **dadurch gekennzeichnet, dass** der poröse und der nicht-poröse Körper von Feuerfestmaterialien mit ähnlichen Wärmedehnungskoeffizienten gebildet werden.

4. Stopfen oder Ziegel nach einem der Ansprüche 1 bis 3, **dadurch gekennzeichnet, dass** der poröse und der nicht-poröse Körper von mehr als 50 Gewichts-% Magnesiumoxid, Magnesiumoxid-Spinell, Aluminiumoxid oder Aluminiumoxid-Spinell gebildet werden, vorzugsweise von mehr als 80 Gewichts-%.

5. Stopfen oder Ziegel nach einem der Ansprüche 1 bis 4, **dadurch gekennzeichnet, dass** der nicht-poröse Körper mehr als 50 Gewichts-%, vorzugsweise mehr als 80 Gewichts-% Magnesiumoxid, Magnesiumoxid-Spinell, Aluminiumoxid oder Aluminiumoxid-Spinell umfasst.

6. Stopfen oder Ziegel nach einem der Ansprüche 1 bis 5,

dadurch gekennzeichnet, dass der poröse Körper (2) in Richtung seiner Berührungsfläche zum geschmolzenen Metall hin einen verjüngten Querschnitt (7) aufweist.

7. Verfahren zur Herstellung eines festen porösen Feuerfeststopfens oder -ziegels für eine Behälterauskleidung zum Injizieren von Gas in geschmolzenes Metall durch eine Berührungsfläche zum geschmolzenen Metall, umfassend die Schritte:

- 1) Zuführen von geeigneten Mengen der den porösen und den nicht-porösen Körper bildenden Feuerfestmaterialien in eine Form unter Beachtung der gewünschten Grenzen für diese Körper;
- 2) Gleichzeitiges gemeinsames Pressen beider Feuerfestmaterialien;
- 3) Bereitstellen einer Gasleiteinrichtung;
- 4) Wärmebehandeln der gemeinsam gepressten Materialien.

8. Verfahren nach Anspruch 7, bei dem die Gasleiteinrichtung eine Verteilerkammer umfasst.

9. Verfahren nach Anspruch 8, **dadurch gekennzeichnet, dass** der Schritt (3) eines Bereitstellens der Gasleiteinrichtung einen Schritt eines Einbringens eines Streifens von verzehrbarem Material in die Form an der Stoßstelle zwischen dem porösen und dem nicht-porösen Körper umfasst.

10. Verfahren nach Anspruch 9, **dadurch gekennzeichnet, dass** das verzehrbare Material Wachs umfasst.

Revendications

1. Bloc ou brique (1) réfractaire poreux solide pour un revêtement de récipient pour injecter un gaz dans un métal fondu à travers une surface (11) de contact d'un métal fondu comprenant:

- i) un corps (2) réfractaire poreux substantiellement entouré par un corps (9) substantiellement non-poreux, sauf à la surface de contact (11) du métal fondu; et
- ii) des moyens d'amenée du gaz (3,5) pour conduire le gaz depuis une source de gaz vers le corps poreux (2),

caractérisé en ce que le corps non-poreux (11) est formé d'un matériau réfractaire et **en ce que** les corps poreux et non poreux (2,11) ont été copressés.

2. Bloc ou brique selon la revendication 1 **caractérisé en ce que** les moyens d'amenée du gaz (3,5) comprennent une chambre (3).

3. Bloc ou brique selon la revendication 1 ou 2, **caractérisé en ce que** les corps poreux et non-poreux sont constitués de matériaux réfractaires avec des coefficients de dilatation similaires.

4. Bloc ou brique selon l'une quelconque des revendications 1 à 3, **caractérisé en ce que** les corps poreux et non-poreux sont constitués de plus de 50% en poids de magnésie, de magnésie spinelle, d'alumine ou d'alumine spinelle, de préférence de plus de 80% en poids.

5. Bloc ou brique selon l'une quelconque des revendications 1 à 4, **caractérisé en ce que** le corps non-poreux comprend plus de 50% en poids, de préférence plus de 80% en poids, de magnésie, de magnésie spinelle, d'alumine ou d'alumine spinelle.

6. Bloc ou brique selon l'une quelconque des revendications 1 à 5, **caractérisé en ce que** le corps poreux (2) possède une section (7) effilée vers la surface de contact du métal fondu.

7. Procédé pour la fabrication d'un bloc ou brique poreux réfractaire solide pour un revêtement de récipient pour injecter du gaz dans un métal fondu à travers une surface de contact du métal fondu les étapes de :

- 1) introduction de quantités appropriées de matériaux réfractaires constituant les corps poreux et non-poreux tout en respectant les limites désirées pour ces corps;
- 2) copressage simultané des deux matériaux réfractaires;
- 3) fourniture des moyens d'amenée de gaz;

4) traitement thermique des matériaux copressés.

8. Procédé selon la revendication 7, dans lequel les moyens d'amenée de gaz comprennent une chambre.

5 9. Procédé selon la revendication 8, **caractérisé en ce que** l'étape (3) de fourniture des moyens d'amenée de gaz comprend une étape d'introduction d'une bandelette d'un matériau consommable dans le moule à la jonction entre les corps poreux et non-poreux.

10 10. Procédé selon la revendication 9, **caractérisé en ce que** le matériau consommable comprend de la cire.

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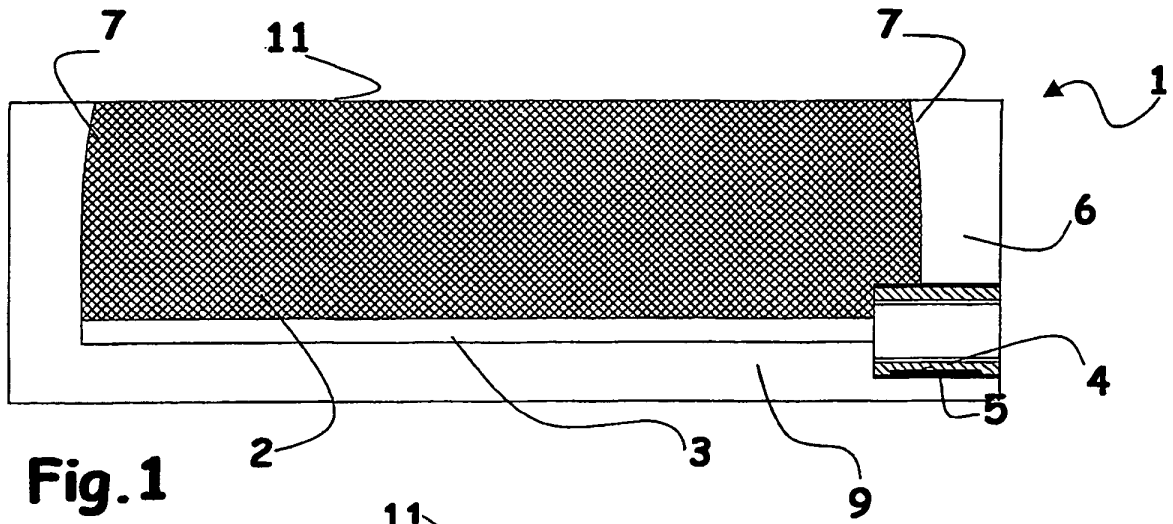


Fig. 1

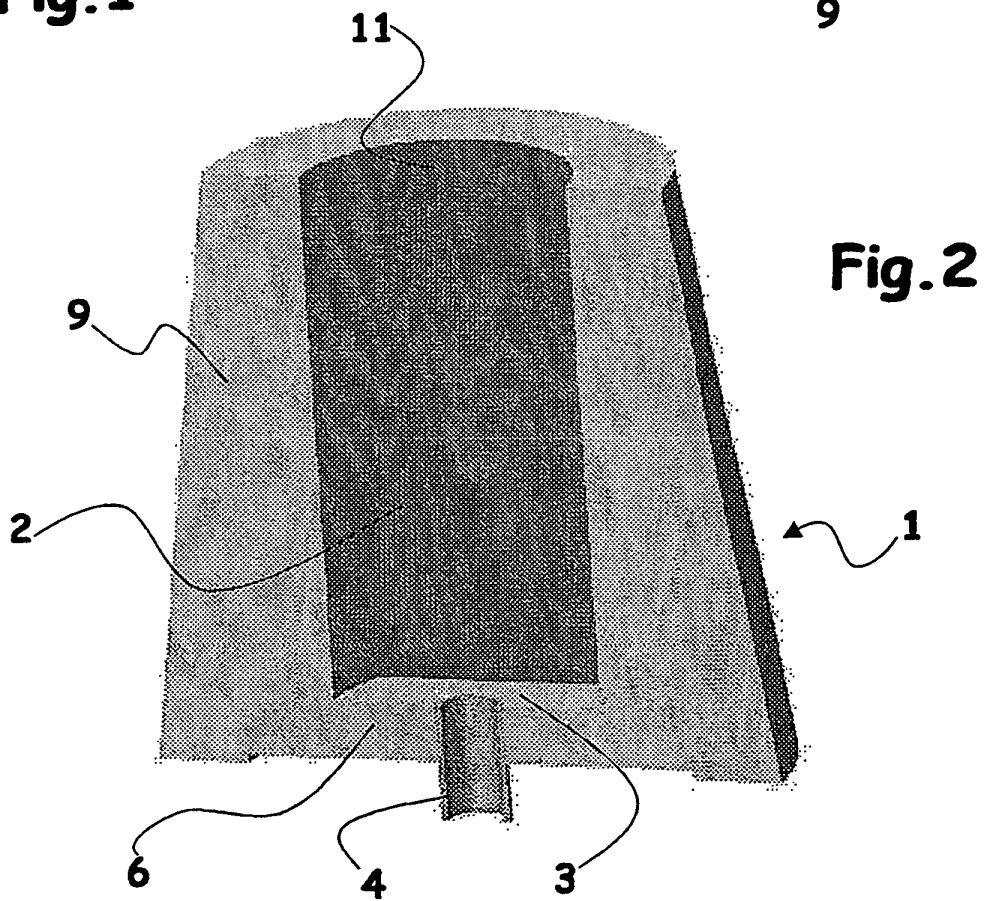


Fig. 2