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(54) MOUNTED X-RAY WINDOW

(57) A mounted x-ray window can be strong and transmissive to x-rays, can have a hermetic seal, and can withstand high temperatures. The mounted x-ray window can include a film located on an inner-side of a flange of a housing and can be attached to the flange by a ring of elastic adhesive. The film can comprise silicon nitride.

A method of mounting an x-ray window can include placing a ring of elastic adhesive on an inner-side of a flange of a housing, placing a film on the ring of elastic adhesive, and baking the housing, the ring of elastic adhesive, and the film.

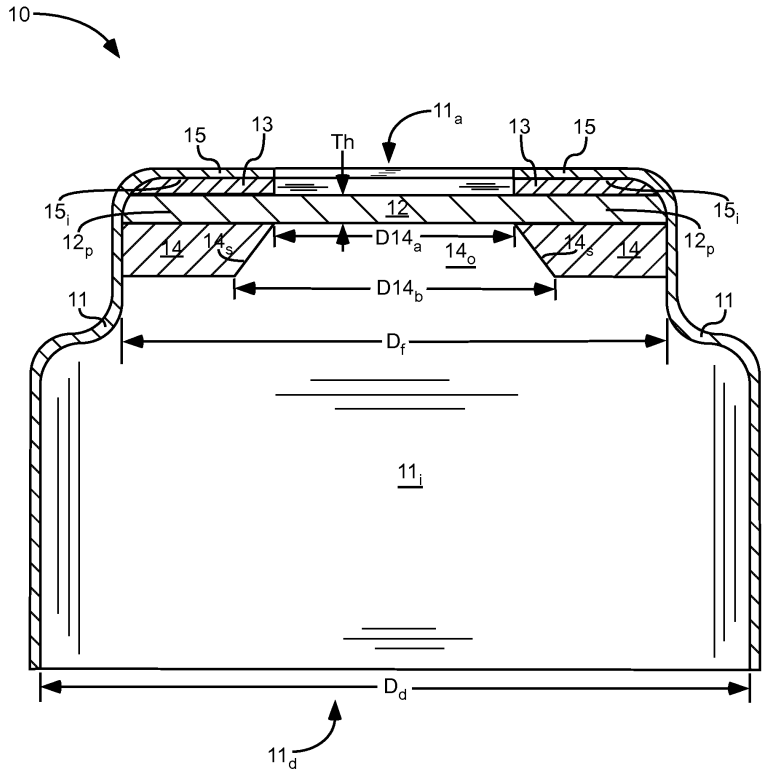

Fig. 1

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Description

FIELD OF THE INVENTION

[0001] The present application is related generally to x-ray windows.

BACKGROUND

[0002] X-ray windows are designed to allow high transmission of x-rays, even low-energy x-rays. For some applications, it can be important for x-ray windows to block visible and infrared light transmission, in order to avoid creating undesirable noise in sensitive instruments.

[0003] It can be important for x-ray windows to be strong, because the x-ray window may need to withstand a differential pressure of about 1 atm. X-ray windows need sufficient thickness for strength, but not a thickness that will cause excessive attenuation of x-rays. A vacuum on one side and air on an opposite side can cause the x-ray window to bow or deflect, damaging the x-ray window, and also possibly causing a short circuit by creating an unintended electrical-current path. Thus, it can be important to minimize deflection.

[0004] It can be important for x-ray windows to be substantially impervious to gases. It can be important for the x-ray window to form a hermetic seal.

SUMMARY

[0005] It has been recognized that it would be advantageous to provide a strong x-ray window that is substantially opaque to visible and infrared light, transmissive to x-rays, substantially impervious to gases, and able to form a hermetic seal. The present invention is directed to various embodiments of mounted x-ray windows, and methods of mounting x-ray windows, that satisfy these needs. Each embodiment may satisfy one, some, or all of these needs.

[0006] The mounted x-ray window can comprise a housing with an aperture and a flange encircling the aperture. The flange can have an inner-side that faces an interior of the housing. A film can be located on the inner-side of the flange and can extend across the aperture. A ring or sheet of elastic adhesive can be sandwiched between the film and the inner-side of the flange and can form a hermetic-seal between the film and the housing.

[0007] The method can comprise: (1) providing a housing with an aperture, a flange encircling the aperture, the flange having an inner-side that faces an interior of the housing; (2) placing a ring of elastic adhesive on the inner-side of the flange; (3) placing a film on the ring of elastic adhesive on the inner-side of the flange, extending across and covering the aperture, with the ring of elastic adhesive sandwiched between the film and the inner-side of the flange; (4) and baking the housing, the ring of elastic adhesive, and the film.

BRIEF DESCRIPTION OF THE DRAWINGS (drawings might not be drawn to scale)

[0008]

FIG. 1 is a schematic, cross-sectional side-view of a mounted x-ray window 10, including a film 12 sealed to a housing 11 by a ring of elastic adhesive 13, the ring of elastic adhesive 13 immediately adjoining the housing 11 and the film 12, in accordance with an embodiment of the present invention.

FIG. 2 is a schematic top-view of the mounted x-ray window 10 of FIG. 1, in accordance with an embodiment of the present invention.

FIG. 3 is a schematic, cross-sectional side-view of a mounted x-ray window 30, similar to mounted x-ray window 10, but also showing that the film 12 can include a first layer 31 and a second layer 32, with the second layer 32 sandwiched between the first layer 31 and the ring of elastic adhesive 13, in accordance with an embodiment of the present invention.

FIG. 4 is a schematic, cross-sectional side-view of a mounted x-ray window 40, similar to mounted x-ray window 10, but also showing that the film 12 can include a first layer 31 and a second layer 32, with the first layer 31 sandwiched between the second layer 31 and the ring of elastic adhesive 13, in accordance with an embodiment of the present invention.

FIG. 5 is a schematic, cross-sectional side-view of a mounted x-ray window 50, similar to mounted x-ray window 10, but also showing that the film 12 can include a first layer 31, a second layer 32, and a third layer 33, with the second layer 32 sandwiched between the first layer 31 and the third layer 33, in accordance with an embodiment of the present invention.

FIG. 6 is a first step in mounting an x-ray window, providing a housing 11 with an aperture 11a, and a flange 15 encircling the aperture 11a, the flange 15 having an inner-side 15_i that faces an interior 11_i of the housing 11, in accordance with an embodiment of the present invention.

FIG. 7 is a second step in mounting an x-ray window, placing a ring of elastic adhesive 13 on the inner-side 15_i of the flange 15, in accordance with an embodiment of the present invention.

FIG. 8 is a third step in mounting an x-ray window, placing a film 12 on the ring of elastic adhesive 13 on the inner-side 15_i of the flange 15, the film 12 extending across and covering the aperture 11a, with the ring of elastic adhesive 13 sandwiched between the film 12 and the inner-side 15_i of the flange 15, in accordance with an embodiment of the present invention.

FIG. 9 is a fourth step in mounting an x-ray window, applying a force to the film 12 and baking the housing

11, the ring of elastic adhesive 13, and the film 12, in accordance with an embodiment of the present invention.

FIG. 10 is a schematic, cross-sectional side-view of a mounted x-ray window 10, including a film 12 sealed to a housing 11 by a sheet of elastic adhesive 103, the sheet of elastic adhesive 103 immediately adjoining the housing 11 and the film 12, in accordance with an embodiment of the present invention.

DETAILED DESCRIPTION

[0009] As illustrated in FIGs. 1 & 2, a mounted x-ray window 10 is shown comprising a housing 11 with an aperture 11_a and a film 12. An adhesive, which can comprise or consist of a ring of elastic adhesive 13, can be sandwiched between the aperture 11_a and the film 12. As will be described below and also illustrated in FIG. 10, the adhesive can include or consist of a sheet of elastic adhesive 103.

[0010] The film 12 can extend across the aperture 11_a. The ring of elastic adhesive 13 can have an opening aligned with the aperture 11_a. The opening of the ring of elastic adhesive 13 can be about the same size as, or larger than, the aperture 11_a. The ring of elastic adhesive 13 can encircle the aperture 11_a of the housing 11 and can be sandwiched between the film 12 and the housing 11. The ring of elastic adhesive 13 can form a hermetic-seal to the housing. The film 12 can be attached or sealed to the housing 11 by the ring of elastic adhesive 13, forming the hermetic-seal between the film 12 and the housing 11. Thus, the film 12 can immediately adjoin the ring of elastic adhesive 13. Alternatively, another component can be sandwiched between the film 12 and the ring of elastic adhesive 13. A support structure 14, described below, can be sandwiched between the film 12 and the ring of elastic adhesive 13.

[0011] The housing 11 can be metallic. The housing can include nickel or a nickel alloy.

[0012] The housing 11 can include a flange 15 encircling the aperture 11_a. The flange 15 can have an inner-side 15_i that faces an interior 11_i of the housing 11. The ring of elastic adhesive 13 can be sandwiched between the film 12 and the inner-side 15_i of the flange 15 and can attach or seal the film 12 to the inner-side 15_i of the flange 15, forming a hermetic-seal between the film 12 and the flange 15. Damage to the film 12 can be avoided by mounting the film 12 on the inner-side 15_i of the flange 15-if the film 12 is mounted on the opposite, outer side of the flange 15, air pressure can press the film 12 against an edge of the flange 15, which can damage the film.

[0013] The film 12 can include some or all of the properties (e.g. low deflection, high x-ray transmissivity, low visible and infrared light transmissivity) of the x-ray window described in U.S. Patent Application Serial Number 14/597,955, filed on January 15, 2015, which is incorporated herein by reference in its entirety.

[0014] For example, the film 12 can include one or more of the following: silicon (e.g. silicon nitride), a polymer (e.g. polyimide), beryllium, carbon nanotubes, graphene, hexamethyldisilazane, amorphous carbon, diamond, diamond-like carbon, boron hydride, and aluminum. Some of these materials can be used for strength, some for blocking visible and/or infrared light, some for gas-tightness, and some for corrosion-resistance.

[0015] If the film 12 includes silicon nitride, examples of its material composition include at least 90% silicon nitride in one aspect, at least 95% silicon nitride in another aspect, or at least 99% silicon nitride in another aspect. The film 12, including silicon nitride, can be made by depositing nitrogen into surface(s) of a silicon wafer, then etching a center region to a desired thickness.

[0016] An outer portion of the silicon wafer can be an annular-support 14 attached to a perimeter 12_p of the film 12. The annular-support 14 can provide structural support for the film 12. The annular-support 14 can be made of or can include silicon. The annular-support 14 can have an opening 140, which can be formed during etch of the silicon wafer. The opening 140 of the annular-support 14 can align with the aperture 11_a of the housing 11. The film 12 can extend across the opening 140 of the annular-support 14. Although not shown in the figures, there can be a pair of annular-supports 14 sandwiching the film 12.

[0017] The etch of the silicon wafer can extend not only vertically into the film, but also horizontally into the annular-support 14. Thus, the annular-support 14 can include a sloped-face 14_s at the opening 140 and the annular-support 14 can have a smaller inner diameter $D14_a$ adjacent to the film 12 sloping to a larger inner diameter $D14_b$ farther from the film 12.

[0018] It can be important to locate the annular-support 14 on an opposite side of the film 12 from the ring of elastic adhesive 13 because by doing so the sloped-face 14_s can allow x-rays to expand into the interior 11_i of the housing 11. Also, locating the annular-support 14 on an opposite side of the film 12 from the ring of elastic adhesive 13 can increase surface area of attachment between the film 14 and the housing and can avoid air pressure separating the film 12 from the annular-support 14.

[0019] Some films 12 (e.g. silicon nitride) can be brittle due to internal stress. A mounting process, the annular-support 14, and the ring of elastic adhesive 13, can release at least some of this stress, resulting in a more robust film 12. For example, the film 12 and annular-support 14 can be mounted to the housing 11 with the ring of elastic adhesive 13 by baking. The film 12 can have a very different coefficient of thermal expansion than the housing 11. If the annular-support 14 has a similar coefficient of thermal expansion to the film 12, and thus also very different from the housing 11, then stresses in the film 12 can be transferred to the annular-support 14 during the baking process. The annular-support 14 can be substantially thicker than the film 12, and thus withstand more stress than the film 12, because the annular-support 14 can be located where it won't block desired x-

rays (e.g. outside of the aperture 11_a of the housing 11).

[0020] Examples of differences between the coefficients of thermal expansion of the housing 11 and the annular-support 14 include at least 5 μm/(m*K) in one aspect, at least 7 μm/(m*K) in another aspect, or at least 9 μm/(m*K) in another aspect. Examples of similarities between the coefficients of thermal expansion of the film 12 and the annular-support 14 include less than 1.5 μm/(m*K) in one aspect, less than 1 μm/(m*K) in another aspect, less than 0.7 μm/(m*K) in another aspect, less than 0.5 μm/(m*K) in another aspect, or less than 0.3 μm/(m*K) in another aspect.

[0021] Use of a ring of elastic adhesive 13 can also aid in reducing stress in the film 12. Elastic generally means a material able to resume its normal shape spontaneously after stretching or distortion. As used herein, elastic materials have a relatively low modulus of elasticity, such as for example less than 50 GPa in one aspect, less than 20 GPa in another aspect, less than 10 GPa in another aspect, or less than 5 GPa in another aspect. Elastic materials include many polymers, including polyimide (modulus of elasticity ~ 3 GPa). Thus, the ring of elastic adhesive 13 can be or can include a polymer, and can be or can include polyimide.

[0022] The housing 11 can be shaped for easier placement of the film 12 and the ring of elastic adhesive 13. The housing 11 can have two openings - the aperture 11_a and a distal-opening 11_d, opposite of each other and located at opposite ends of the housing 11. The distal-opening 11_d can have a larger diameter D_d than a diameter D_f of the flange 15 (measured inside of the housing 11). This can allow easy placement of the film 12 and the ring of elastic adhesive 13 because they can naturally center themselves when placed in the housing 11, and can allow a larger area for a detector at the distal-opening 11_d.

[0023] It can be important that the mounted x-ray window 10, and particularly the ring of elastic adhesive 13, is capable of withstanding high temperatures without substantial degradation or failure of the hermetic-seal, because the overall device can be improved by high temperatures during manufacturing. For example, the mounted x-ray window 10 can be used with an x-ray detector. It can be important that an interior 11_i of the housing 11 has a very low pressure, for proper cooling of the detector, and to avoid interference of x-rays by air molecules. In order to obtain and maintain this low pressure, the housing 11 can be baked at a high temperature (e.g. ~ 400 °C) to drive gas molecules out of the housing. The detector can then be sealed to the housing at a high temperature (e.g. ~ 400 °C) to activate getters, which can continuously remove gas molecules.

[0024] The ring of elastic adhesive 13 can be a material capable of withstanding a temperature of at least 250 °C in one aspect, a temperature of at least 300 °C in another aspect, or a temperature of at least 400 °C in another aspect, without substantial degradation or failure of the hermetic-seal. Polyimide meets this requirement.

[0025] By proper selection of the ring of elastic adhesive 13, and a proper method of manufacture (e.g. baking), the housing 11 can be hermetically-sealed to the film 12 by the ring of elastic adhesive 13 with a relatively low internal pressure (e.g. < 5 pascals, < 2 pascals, or < 1 pascal) and the mounted x-ray window can have a relatively low leak-rate (e.g. < $1x10^{-14}$ bar*L/s or < $2x10^{-15}$ bar*L/s).

[0026] The film 12 can have various thicknesses Th, depending on material of construction, span-width, and use. For example, the film 12 can have a thickness Th of < 10 μm in one aspect, < 1 μm in another aspect, or < 600 nm in another aspect; and > 400 nm in one aspect, > 50 nm in another aspect, or > 5 nm in another aspect,

[0027] Mounted x-ray windows 30, 40, and 50, shown in FIGs. 3-5, can be similar to mounted x-ray window 10, except that in mounted x-ray windows 30, 40, and 50 the film 12 can include a first layer 31 and a second layer 32 (and also a third layer 33 in FIG. 5). In mounted x-ray window 30, the second layer 32 is sandwiched between the first layer 31 and the ring of elastic adhesive 13. In mounted x-ray window 40, the first layer 31 is sandwiched between the second layer 31 and the ring of elastic adhesive 13. In mounted x-ray window 50, the second layer 32 is sandwiched between the first layer 31 and the third layer 33. Examples of materials of the first layer 31, the second layer 32, and the third layer 33 include silicon nitride, a polymer (e.g. polyimide), beryllium, carbon nanotubes, graphene, hexamethyldisilazane, amorphous carbon, diamond, diamond-like carbon, boron hydride, and aluminum. Each of the first layer 31, the second layer 32, and the third layer 33 can include one or more of these materials. One, two, or all three of the first layer 31, the second layer 32, and the third layer 33 of the film 12 can extend across the aperture 11_a.

[0028] As shown in FIG. 10, a sheet of elastic adhesive 103 can be used instead of a ring of elastic adhesive 13. The sheet of elastic adhesive 103 can extend across the aperture 11_a of the housing 11. The term "ring of elastic adhesive" may be replaced with "sheet of elastic adhesive" anywhere in this detailed description. Possible advantages of using a sheet of elastic adhesive 103 instead of a ring of elastic adhesive 13 include providing structural-support to the film 12 and improved gas-diffusion barrier. Possible disadvantages of using a sheet of elastic adhesive 103 instead of a ring of elastic adhesive 13 include increased attenuation of x-rays, spectral contamination, increased outgassing, increased stress in the film due to coefficient of thermal expansion mismatch, and manufacturing difficulties. The advantages and disadvantages can be weighed for each application in order to decide whether to use a sheet of elastic adhesive 103 or a ring of elastic adhesive 13.

[0029] A method of mounting an x-ray window can comprise some or all of the following steps, which can be performed in the following order. There may be additional steps not described below. These additional steps may be before, between, or after those described.

1. Providing a housing 11 with an aperture 11_a. The housing 11 can also include a flange 15 encircling the aperture 11_a, the flange 15 having an inner-side 15_i that faces an interior 11_i of the housing 11. See FIG. 5.
2. Locating (by hand, with a hand tool, by machine, pouring a liquid, etc.) a ring of elastic adhesive 13 on the housing (e.g. on the inner-side 15_i of the flange 15). See FIG. 6.
3. Locating a film 12 over the ring of elastic adhesive 13 (e.g. on the inner-side 15i of the flange 15). The film 12 can extend across and can cover the aperture 11_a, with the ring of elastic adhesive 13 sandwiched between the film 12 and the housing 11 (e.g. the inner-side 15_i of the flange 15). See FIG. 7.
4. Baking the housing 11, the ring of elastic adhesive 13, and the film 12 at a temperature of at least 300 °C in one aspect or at least 400 °C in another aspect. The bake can extend for a sufficient time to relieve stress in the film 13, to soften the ring of elastic adhesive 13, and to degas the housing 11 (e.g. at least 1 hour in one aspect or at least 3 hours in another aspect). A force can be applied to the film 12 while baking, in order to improve the hermetic seal. For example, the force can be at least one newton in one aspect, at least 5 newtons in another aspect, or at least 10 newtons in another aspect. See FIG. 8.

[0030] The housing 11, the ring of elastic adhesive 13, and the film 12 can have properties as described above.

Claims

1. A method of mounting an x-ray window, the method comprising the following steps in the following order:

providing a housing with an aperture, a flange encircling the aperture, the flange having an inner-side that faces an interior of the housing;
locating a ring of elastic adhesive on the inner-side of the flange;
locating a film over the ring of elastic adhesive on the inner-side of the flange, extending across and covering the aperture, with the ring of elastic adhesive sandwiched between the film and the inner-side of the flange; and
baking the housing, the ring of elastic adhesive, and the film at a temperature of at least 300 °C.

2. The method of claim 1, wherein the film has a material composition comprising silicon nitride.

3. The method of claim 1 or 2, wherein the ring of elastic adhesive includes polyimide.

4. The method of claim 1, 2 or 3, wherein baking further comprises applying at least one newton of force to the film while baking the housing, the ring of elastic adhesive, and the film.

5. A mounted x-ray window comprising:

a housing including an aperture and a flange encircling the aperture, the flange having an inner-side that faces an interior of the housing;
a film located on the inner-side of the flange and extending across the aperture; and
an adhesive sandwiched between the film and the inner-side of the flange, the adhesive including a ring of elastic adhesive, a sheet of elastic adhesive, or both.

6. The mounted x-ray window of claim 5, wherein the adhesive is a ring of elastic adhesive encircling the aperture of the housing.

7. The mounted x-ray window of claim 5 or 6, wherein the adhesive includes a sheet of elastic adhesive extending across the aperture of the housing.

8. The mounted x-ray window of claim 5, 6 or 7, wherein the film includes graphene, polymer, or combinations thereof.

9. The mounted x-ray window of any of claims 5 to 8, wherein the film includes silicon nitride.

10. The mounted x-ray window of any of the claim 5 to 9, wherein the film includes a silicon nitride thin film sandwiched between two aluminum thin films.

11. The mounted x-ray window of any of claim 5 to 10, wherein:

the adhesive forms a hermetic-seal between the film and the housing;
the adhesive is capable of withstanding a temperature of at least 300 °C without failure of the hermetic-seal; and
the adhesive has a modulus of elasticity of less than 5 GPa.

12. The mounted x-ray window of any of claims 5 to 11, wherein:

the housing is hermetically-sealed by the adhesive to the film;
the housing has an internal pressure of less than 5 pascals; and
the mounted x-ray window has a leak-rate of less than $1x10^{-14}$ bar*L/s.

13. The mounted x-ray window of any of claims 5 to 12, wherein:

the housing has two openings, the aperture and a distal-opening, opposite of each other and located at opposite ends of the housing; and
the distal-opening is larger than a diameter of the flange, measured inside of the housing.

14. The mounted x-ray window of any of claims 5 to 13, further comprising an annular-support, wherein:

the annular-support is attached to a perimeter of the film;
the annular-support has an opening;
the film extends across the opening of the annular-support;
the annular-support is located on an opposite side of the film from the ring of elastic adhesive;
a difference between a coefficient of thermal expansion of the housing and a coefficient of thermal expansion the annular-support is at least 7 μm/(m*K); and
a difference between a coefficient of thermal expansion of the film and a coefficient of thermal expansion the annular-support is less than 0.7 μm/(m*K).

15. The mounted x-ray window of any of claim 5 to 14, further comprising an annular-support, wherein:

the annular-support is attached to a perimeter of the film;
the annular-support has an opening;
the film extends across the opening of the annular-support;
the annular-support is located on an opposite side of the film from the ring of elastic adhesive; and
the annular-support includes a sloped-face at the opening and a smaller inner diameter adjacent to the film sloping to a larger inner diameter farther from the film.

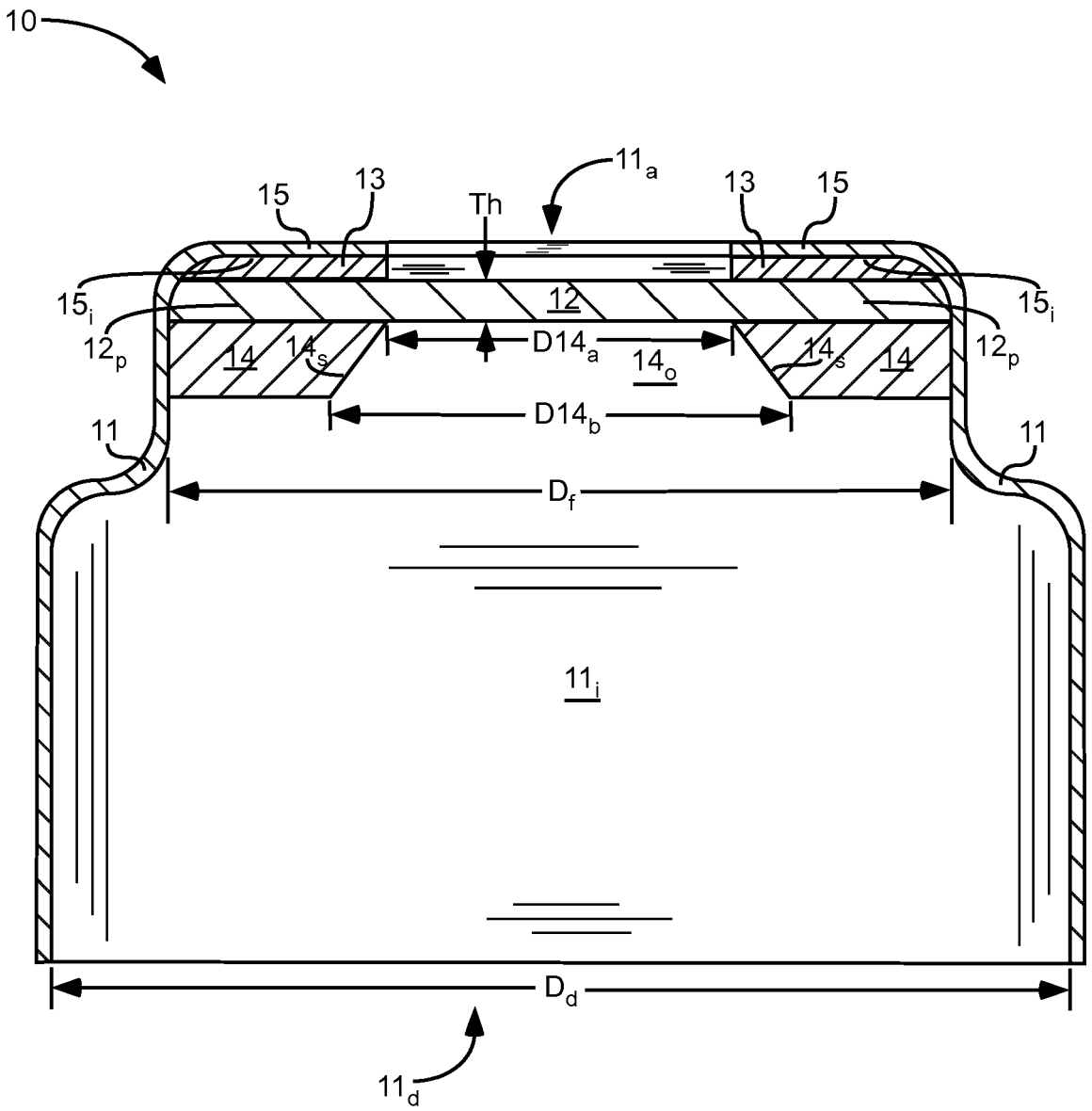
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Fig. 1

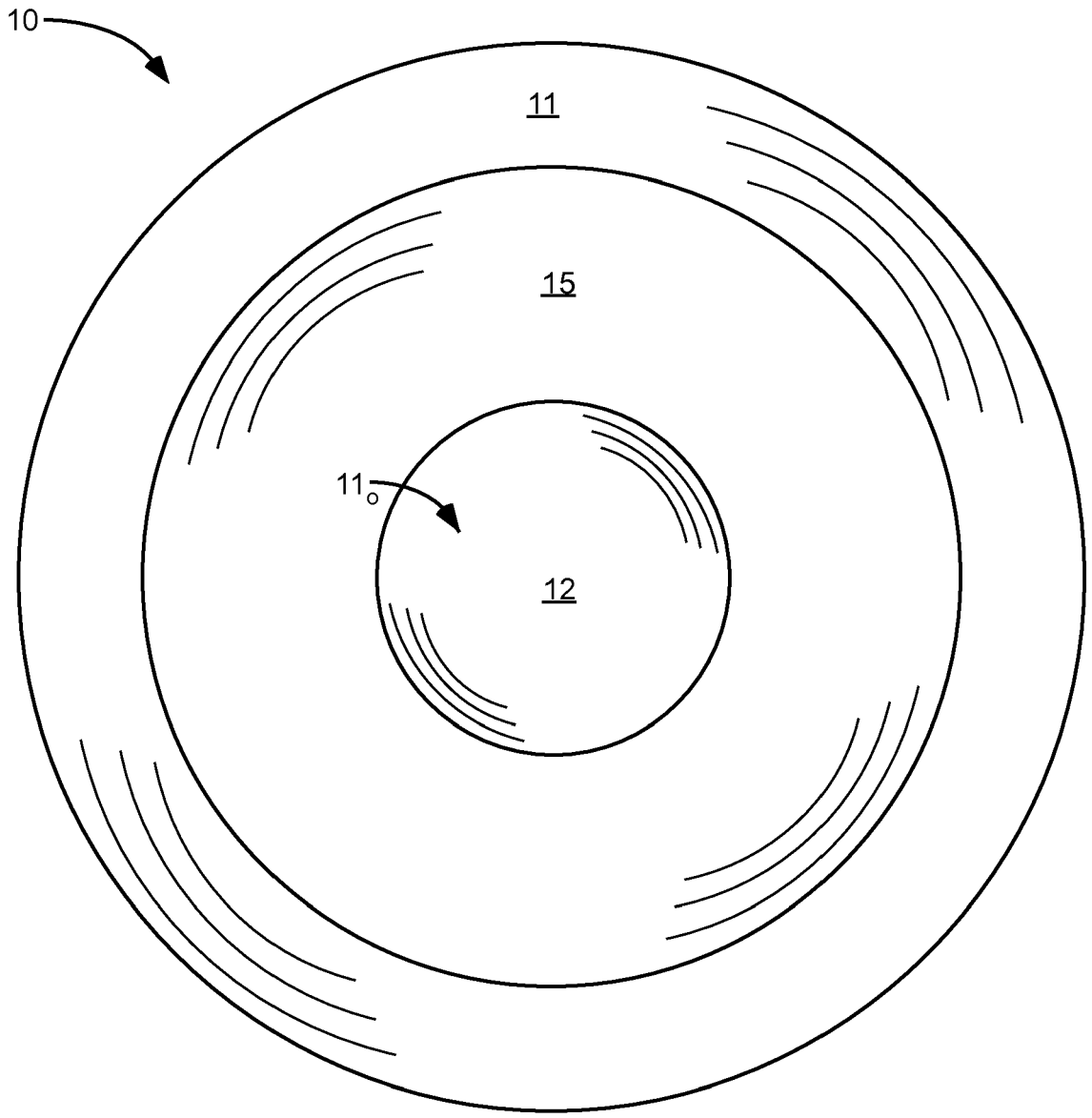

Fig. 2

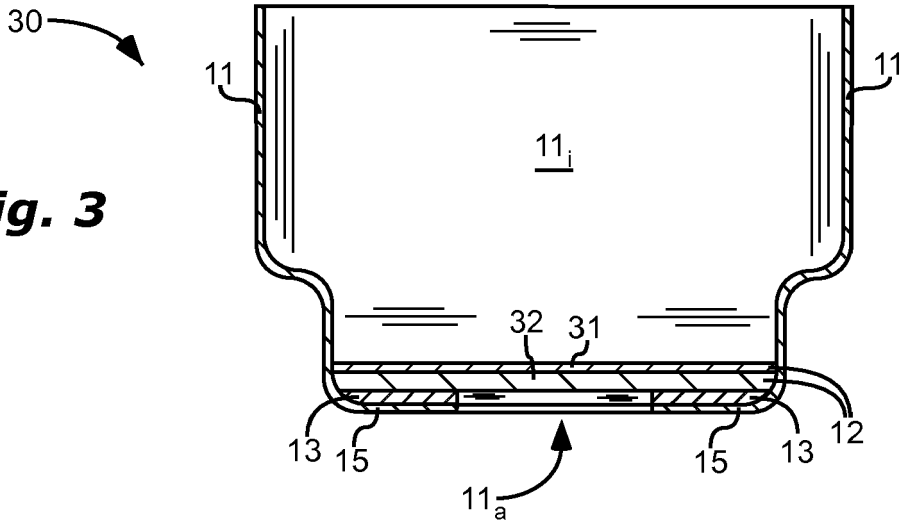

Fig. 3

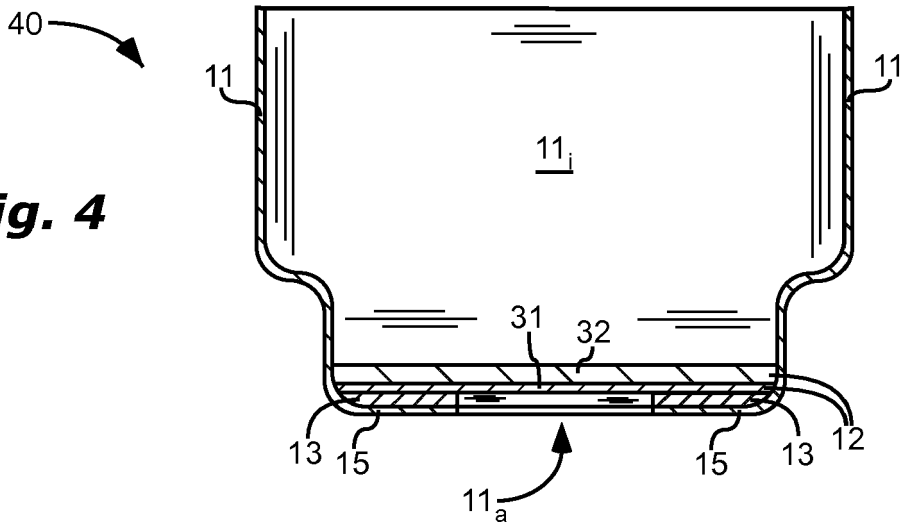

Fig. 4

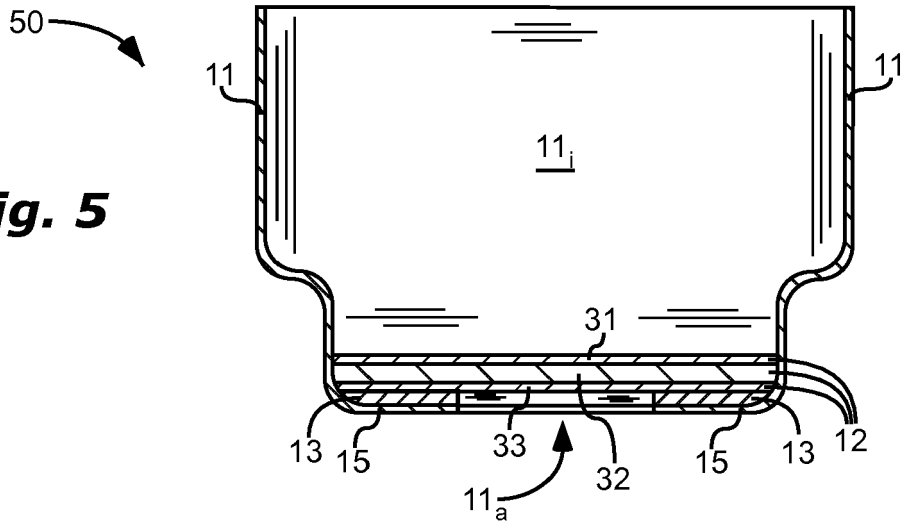

Fig. 5

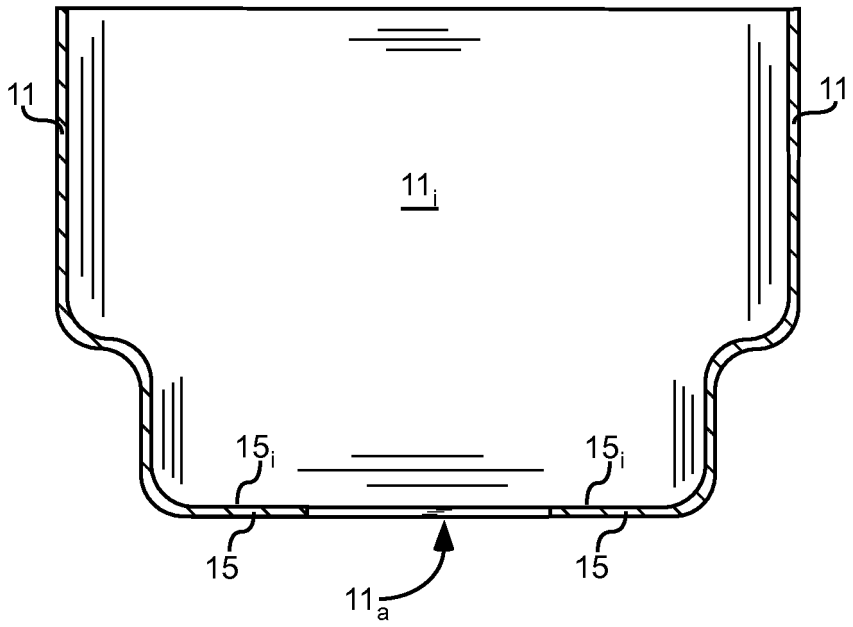

Fig. 6

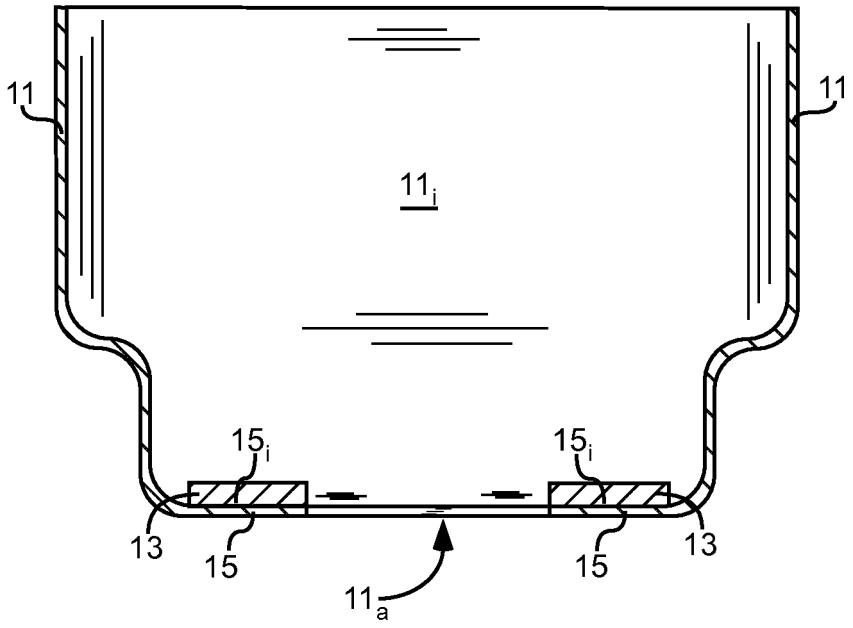

Fig. 7

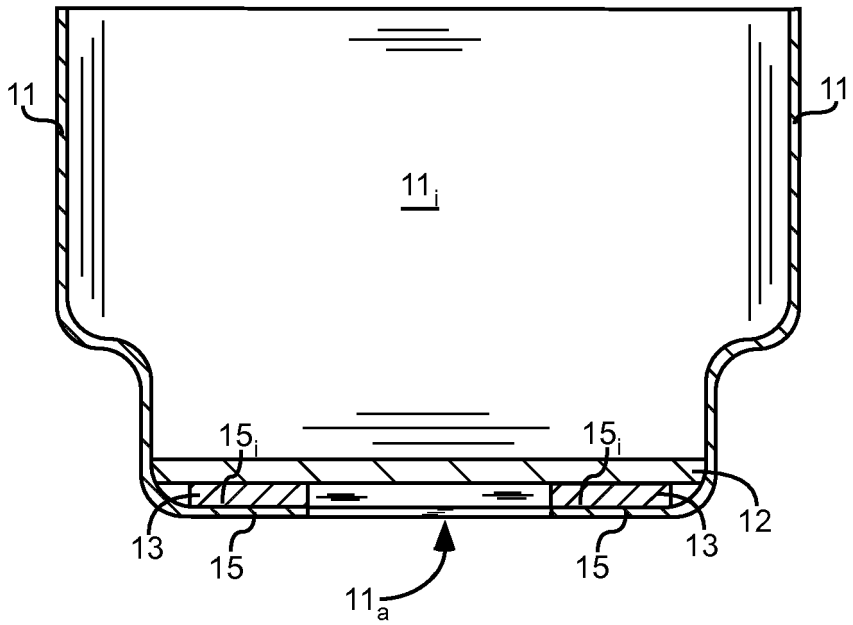
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Fig. 8

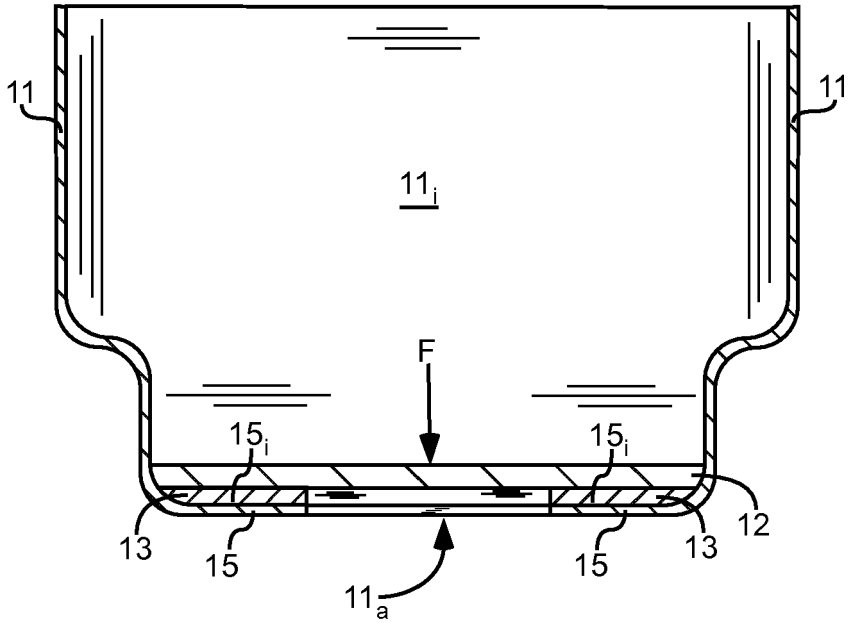
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Fig. 9

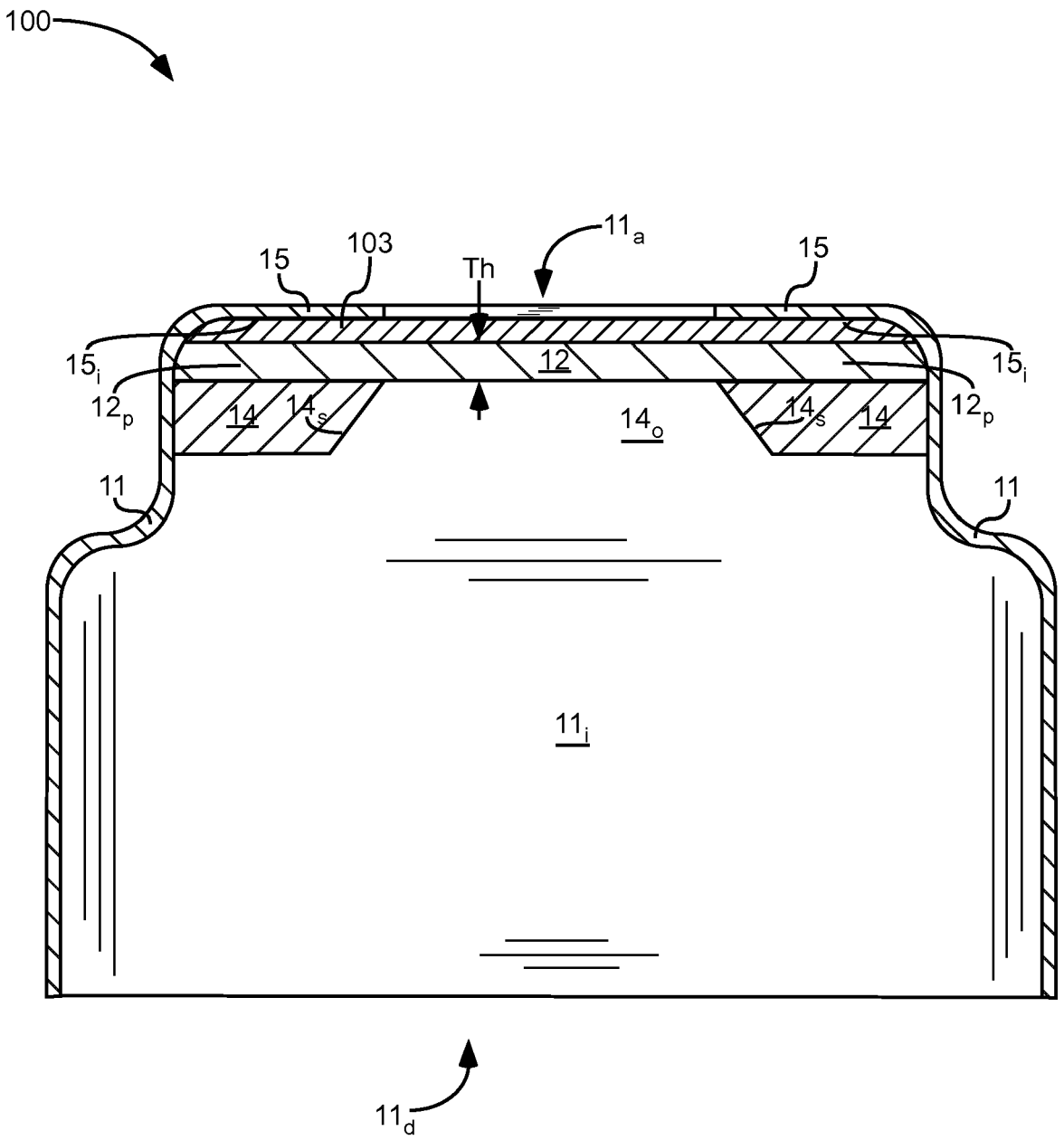
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Fig. 10

Europäisches Patentamt
European Patent Office
Office européen des brevets

EUROPEAN SEARCH REPORT

Application Number
EP 18 18 6189

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X A	US 4 617 465 A (YOSHIDA YUZO [JP]) 14 October 1986 (1986-10-14) * column 3, line 37 - column 4, last line; figures 4,5 * * column 5, line 37 - column 6, line 28; figures 6,7 * -----	5,6,14, 15 1,3,4,11	INV. H01J5/18 G01T1/00
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~~The present search report has been drawn up for all claims~~

Place of search	Date of completion of the search	Examiner
Munich	14 May 2019	Krauss, Jan

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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Application Number
EP 18 18 6189

CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing claims for which payment was due.

☐ Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due and for those claims for which claims fees have been paid, namely claim(s):

☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet B

☐ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.

☐ As all searchable claims could be searched without effort justifying an additional fee, the Search Division did not invite payment of any additional fee.

☒ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:

1-6, 11-15

☐ None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

☐ The present supplementary European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims (Rule 164 (1) EPC).

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LACK OF UNITY OF INVENTION
SHEET B

Application Number
EP 18 18 6189

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

1. claims: 1-6, 8-11, 13-15

   dedicated to a method including baking the housing, etc. at a temperature of at least 300°C as defined in present independent claim 1

   ---

2. claim: 7

   dedicated to a mounted x-ray window with the sheet of elastic adhesive extending across the aperture as defined in present claim 7

   ---

3. claim: 12

   dedicated to a mounted x-ray window the hermetic sealing at the specific pressure and leak rate as defined in claim 12

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 18 18 6189

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14-05-2019

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