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(54) Title: METAL TO GLASS SEAL

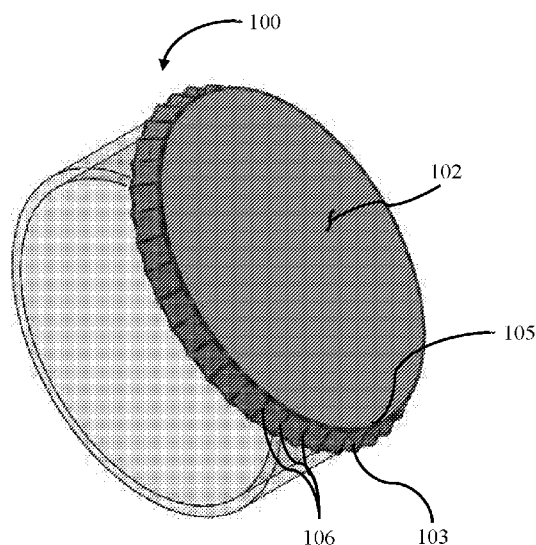


FIG. 1

(57) Abstract: A metal to glass seal comprising a metal cap having a depth D and comprising a base and a crown, a glass tube inserted into the metal cap to a depth less than D, and a low melting point glass (LMPG) material between the glass tube and the crown, such that when the LMPG material is heated to or above a melting temperature and then cooled, the LMPG material seals the metal cap to the glass. Methods of sealing metal to glass comprise, inserting a glass tube into a metal cap, the metal cap having a depth D and comprising a base and a crown, filling a space between the glass tube and the metal cap with a LMPG material, heating the LMPG material to or above a melting temperature, and cooling the LMPG material at least until it solidifies and seals the metal cap to the glass tube.



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— *of inventorship (Rule 4.17(iv))*

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METAL TO GLASS SEAL

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority under 35 U.S.C. § 119(e) to U.S. provisional application Serial No. 62/799,406, filed January 31, 2019, which application is specifically incorporated herein, in its entirety, by reference.

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GOVERNMENT LICENSE RIGHTS

[0002] This invention was made with government support under Award No. DE-EE0008399 awarded by the Department of Energy. The government has certain rights in the invention.

FIELD OF THE INVENTION

10 [0003] The present invention relates to the field of metal to glass seals. More specifically, the invention relates to novel ways of sealing metal to glass utilizing low melting point glass (LMPG).

DISCUSSION OF THE BACKGROUND

15 [0004] Metal to glass seals are airtight (hermetic) assemblies of glass and metal that are used in the construction of various components. For example, vacuum tubes, electric discharge tubes, incandescent light bulbs, and glass encapsulated semiconductor diodes all require metal to glass seals. Additionally, metal to glass seals may be useful in certain solar collector applications (see e.g., Winston, WO 2018/191757 A1, published Oct. 18, 2018).

20 [0005] Many conventional methods of sealing metal to glass require the thermal expansion of the glass and the metal be closely matched because different rates of thermal expansion between the glass and the metal will cause mechanical stresses during the heating and cooling processes, leading to weakening of the seal and/or glass breakage. For example, if the coefficient of thermal expansion of the metal is larger than the coefficient of thermal expansion of the glass, the metal will shrink more than the glass upon cooling,
25 putting additional stresses on the glass, and in some cases, causing the glass to break. Use of closely matched coefficients of thermal expansion precludes the use of certain metals

(e.g. aluminum and aluminum alloys) that have a much higher coefficient of thermal expansion than that of glass. Such matching of coefficients may also preclude the use of certain types of glass that have a particularly low coefficient of thermal expansion (e.g. PYREX[®]).

5 **[0006]** Other conventional methods of forming a metal to glass seal utilize a “compression seal.” The compression seal is designed to exert concentric compressive stress on the glass within a certain temperature range. Such metal to glass compression seals typically use a steel or stainless steel housing, iron seals or Alloy 42 (KOVAR[®]). KOVAR is a nickel-iron alloy that has a low, and normally constant, coefficient of thermal
10 expansion up to 570 °F (300 °C). Compression seals, however, may have less integrity than other conventional seals and also preclude the use of certain metals such as aluminum in order to exert the proper compressive force on the glass. Because of its light weight, low cost and ease of fabrication, it is desirable to use aluminum in metal to glass seals.

15 **[0007]** Therefore, it is desirable to provide an unconventional metal to glass seal that allows for the use of different types of metal and glass having significantly different coefficients of thermal expansion.

SUMMARY OF THE INVENTION

[0008] The present invention advantageously provides novel metal to glass seals using a low melting point glass (LMPG) as the sealant, and a metal cap shaped to absorb the differential thermal expansion rate between the metal cap and a glass tube. The shape of the metal cap also accounts for manufacturing tolerances in both the glass tube and the cap.

[0009] An embodiment of the invention comprises a metal cap having a shape similar to a bottle cap with a crown sloped to accommodate naturally occurring manufacturing tolerances in both the cap and the glass tube. The metal cap is fitted to the glass tube such that the glass tube does not “bottom out” on the cap (i.e., the base of the cap does not contact a flat end of the glass tube).

[0010] In typical embodiments, a metal to glass seal comprises: a metal cap having a depth D, the metal cap comprising a base and a crown; a glass tube inserted into the metal cap to a depth less than D; and a LMPG material between the glass tube and the crown. When the LMPG material is heated to or above a melting point and then cooled, the LMPG material hermetically seals the metal cap to the glass tube. In typical embodiments, the metal cap comprises corrugations (ridges), similar to a bottle cap, to absorb the thermal expansion and contraction of the metal cap as it is heated and cooled.

[0011] It is therefore an object of the invention to provide an improved metal to glass seal utilizing nonconventional metals for the cap (e.g., aluminum and aluminum alloys).

[0012] It is also an object of the invention to provide an improved metal to glass seal that reduces stresses on the glass.

[0013] It is also an object of the invention to provide an improved metal to glass seal wherein the integrity of the seal remains intact over a wide range of temperatures.

[0014] It is also an object of the invention to provide a metal to glass seal utilizing a low melting point glass material as the sealant.

[0015] It is also an object of the invention to provide a novel method for creating a metal to glass seal.

[0016] It is to be understood that both the foregoing general description and the following detailed description are exemplary, but not restrictive, of the invention. A more complete understanding of the improved metal to glass seal and the methods disclosed herein will be afforded to those skilled in the art.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] FIG. 1 is a perspective view of a metal to glass seal according to an embodiment of the instant invention.

5 [0018] FIG. 2 is a side view of the metal to glass seal of FIG. 1 showing a depth D of the metal cap and an angle θ between the base and the crown of the metal cap.

[0019] FIG. 3 is a top view of the metal to glass seal of FIG. 1.

[0020] FIG. 4 is a section view of the metal to glass seal of FIG. 3 showing the space between the metal cap and the glass tube filled with a low melting point glass material.
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[0021] FIG. 5 is a flow diagram for a method of forming a metal to glass seal.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0022] Reference will now be made in detail to the preferred embodiments of the invention, examples of which are illustrated in the accompanying drawings. While the invention will be described in conjunction with the preferred embodiments, it will be understood that they are not intended to limit the invention to these embodiments. On the contrary, the invention is intended to cover alternatives, modifications, and equivalents that may be included within the spirit and scope of the invention. Furthermore, in the following detailed description of the present invention, numerous specific details are set forth in order to provide a thorough understanding of the present invention. However, it will readily be apparent to one skilled in the art that the present invention may be practiced without these specific details.

[0023] In other instances, well-known methods, procedures and components have not been described in detail so as not to unnecessarily obscure aspects of the present invention. These conventions are intended to make this document more easily understood by those practicing or improving on the inventions, and it should be appreciated that the level of detail provided should not be interpreted as an indication as to whether such instances, methods, procedures or components are known in the art, novel, or obvious.

[0024] As shown in FIGS. 1-4, a metal to glass seal 100, comprises: (i) a metal cap 101 having a depth D, the metal cap comprising a base 102 and a crown 103 at angle θ to the base; (ii) a glass tube 110 inserted into the metal cap 101 to a depth less than D; and (iii) a low melting point glass material (LMPG) 120 between the glass tube 110 and the crown 103. When the LMPG material 120 is heated to or above a melting point and then cooled (typically to room temperature; e.g., between 20 °C and 27 °C) the low melting point glass material 120 seals the metal cap 101 to the glass tube 110.

[0025] Most typically, the glass tube will comprise borosilicate and/or soda lime glass. Borosilicate glass (also called PYREX[®]) is a low iron glass with a high transparency and low thermal expansion rate. Because of these properties, borosilicate glass may be used in preferred embodiments.

[0026] Although the glass tube 110 is shown in FIGS. 1-4 to have a circular cross section, other cross sections may be possible as well (e.g., semi-circular, oval, square,

rectangular, etc.). For glass tubes having a cross-sectional shape that is not circular, the metal cap would have a corresponding shape (e.g., if the cross-sectional shape is oval, the metal cap would be oval; if the cross-sectional shape is square, the metal cap would be square, etc.).

5 **[0027]** As best shown in FIGS. 1 and 2, the crown 103 generally comprises a free edge 104 and a shoulder 105, the shoulder 105 is defined by the intersection of the base 102 with the crown 103. In a typical embodiment where the glass tube 110 has a circular cross section, the diameter of the crown 103 at the shoulder 105 is smaller than the outer diameter of the glass tube 110, and the diameter of the free edge 104 of the crown 103 is
10 larger than the outer diameter of the glass tube 110. As a result, when the glass tube 110 is inserted into the metal cap 101, a flat end 112 (see FIG. 4) of the glass tube is at a depth less than the depth D of the cap 101. In preferred embodiments, the flat end 112 of the glass tube 110 is inserted to a depth approximately one half the depth D (0.5D) of the cap 101. In other embodiments, the glass tube 110 may be inserted into the cap 101 at a depth
15 ranging from about 0.2D to 0.8D.

[0028] Regardless of the cross-sectional shape of the glass tube and corresponding cap, the glass tube is inserted into the metal cap at a depth less than the depth D of the cap, such that the flat end of the glass tube does not bottom out or contact the base of the cap. Because the glass tube does not contact the base of the cap, tension on the glass tube is
20 reduced or eliminated.

[0029] As best shown in FIGS. 1 and 3, in typical embodiments the crown 103 comprises corrugations (ridges) 106 to absorb the thermal expansion and contraction during the heating and cooling processes such that the differing thermal expansions between the glass tube 110, the LMPG material 120 and the metal cap do not put unwanted
25 stresses on or break the glass tube 110.

[0030] Typically, the glass tube 110 will have a coefficient of thermal expansion of between 3 and 12 ppm/°C. The LMPG material 120 will typically have a coefficient of thermal expansion between 3 and 15 ppm/°C. The metal cap 101 will typically have a coefficient of thermal expansion between 20 and 28 ppm/°C, particularly when it
30 comprises aluminum, and/or one or more alloys of aluminum (e.g., aluminum with one or more of copper, magnesium, manganese, silicon, tin, zinc, etc.). Consequently, in typical

embodiments wherein the crown 103 contains the corrugations 106, the corrugations 106 reduce and/or eliminate stresses on the glass tube 110 resulting from the differing thermal properties of the glass tube 110, LMPG material 120 and the metal cap 101.

[0031] In some embodiments, the length of the corrugations 106 may be substantially equal to a length of the crown tangentially along its slope. In other embodiments, the length of the corrugations 106 may be less than the length of the crown tangentially along its slope (e.g., 20%, 33%, 50%, 60%, 75%, etc. of the length of the crown). In such embodiments, the corrugations may begin at the shoulder 105 and terminate prior to the free edge 104, or may begin at the free edge 104 and terminate prior to the shoulder 105.

[0032] The low melting point glass (LMPG) material 120 may be a powder or a paste, may be lead-free and may have a melting point between 200 °C and 780 °C. Preferably, the melting point of the LMPG material 120 will be between 300 °C and 600 °C. In some embodiments, the melting point is 550 °C. Typically, when the flat end 112 of the glass tube 110 is inserted into the cap 101, the LMPG material 120 fills the corrugations 106 to a depth less than the depth D of the cap 101.

[0033] The angle θ of the crown 103 is configured to accommodate naturally occurring manufacturing tolerances of the glass tube 110. For example, if the glass tube 110 has an outer diameter of 90 mm, plus or minus 1 mm, then a diameter of the metal cap 101 may be 92 mm at the free edge 104 of the crown 103 and 88 mm at the shoulder 105 of the crown 103. Thus, a first diameter at the base 102 of the cap 101 is less than an outer diameter of the glass tube 110, and a second diameter at the free edge 104 of the cap 101 is larger than the outer diameter of the glass tube 110. This is just one example of possible dimensions of the glass tube and the cap, and these dimensions should not be construed to be limiting.

[0034] In typical embodiments, the outer diameter of the glass tube may range from about 25 mm to about 125 mm. The corresponding range of the first diameter at the base 102 of the cap 101 will be somewhat smaller than the outer diameter of the glass tube 110 (e.g., from about 22 mm to about 124 mm), and the corresponding range of the diameter at the free edge 104 will be somewhat larger than the outer diameter of the glass tube 110 (e.g., from about 26 mm to about 130 mm).

[0035] Similarly, depending on the diameter of the glass tube 110 and the depth D of the cap 101, an angle θ between the base 102 and the crown 103 (see FIG. 2) may range from about 5° to about 85°. Most typically, θ will range from about 10° to about 60°. The depth D of the cap 101 may range from about 2.5 mm to about 40 mm.

5

Method of Forming a Metal to Glass Seal

[0036] Referring now to FIG. 5, a method 500 of sealing metal to glass, typically comprises: at step 501, inserting a glass tube into a metal cap, the metal cap having a depth D and comprising a base and a crown; at step 502, filling a space between the glass tube and the metal cap with a low melting point glass material; at step 503, heating the low melting point glass material to or above its melting point; and at step 504 cooling the low melting point glass material until the low melting point glass material solidifies and seals the metal cap to the glass tube. Typically, the crown is at an angle θ to the base, wherein θ ranges between 10° and 60°. In preferred embodiments, the melting point of the LMPG material is between 300 °C and 600 °C.

[0037] Typically, the depth the glass tube is inserted into the metal cap is approximately one half D (0.5D). In some embodiments, the crown comprises corrugations configured to absorb the thermal expansion and contraction of the metal cap during the heating and the cooling processes. Typically, a first diameter of a base of the metal cap is less than an outer diameter of the glass tube, and a second diameter at an edge of the metal cap is greater than the outer diameter of the glass tube. Preferably, the metal cap comprises aluminum.

[0038] The foregoing descriptions of specific embodiments of the present invention have been presented for purposes of illustration and description. They are not intended to be exhaustive or to limit the invention to the precise forms disclosed. Obviously, many modifications and variations are possible in light of the above teaching. The embodiments were chosen and described in order to best explain the principles of the invention and its practical application, to thereby enable others skilled in the art to best utilize the invention and the various embodiments and modifications as are suited to the particular use contemplated. It is intended that the scope of the invention be defined by the components and elements described herein and their equivalents.

CLAIMS

What is claimed is:

1. A metal to glass seal, comprising:
a metal cap having a depth D , the metal cap comprising:
a base; and
a crown at an angle θ to the base;
a glass tube inserted into the metal cap to a depth less than D ;
a low melting point glass material located between the glass tube and the crown;
wherein, when the low melting point glass material is heated to or above a melting point and then cooled, the low melting point glass material seals the metal cap to the glass tube.
2. The metal to glass seal of claim 1, wherein the crown comprises corrugations that absorb thermal expansion and contraction of the metal cap during heating and cooling.
3. The metal to glass seal of claim 1, wherein the depth the glass tube is inserted into the metal cap is approximately one half D .
4. The metal to glass seal of claim 1, wherein the melting point of the low melting point glass material is between about 300 °C and about 600 °C.
5. The metal to glass seal of claim 1, wherein the angle θ ranges from about 10° to about 60°.
6. The metal to glass seal of claim 1, wherein the metal cap comprises aluminum.
7. The metal to glass seal of claim 1, wherein a first diameter at the base of the metal cap is less than an outer diameter of the glass tube.
8. The metal to glass seal of claim 7, wherein a second diameter at a free edge of the metal cap is greater than the outer diameter of the glass tube.

9. A metal to glass seal, comprising:
a metal cap having a depth D, the metal cap comprising:
a base; and
a crown having corrugations, the crown at an angle θ to the base;
a glass tube inserted into the metal cap at a depth approximately equal to one half D;
a low melting point glass material between the glass tube and the crown;
wherein, when the low melting point glass material is heated to or above a melting point and then cooled, the low melting point glass material seals the metal cap to the glass tube.
10. The metal to glass seal of claim 9, wherein a first diameter of the base is less than an outer diameter of the glass tube.
11. The metal to glass seal of claim 10, wherein a second diameter at a free edge of the crown is greater than the outer diameter of the glass tube.
12. The metal to glass seal of claim 9, wherein the melting point of the low melting point glass material is between about 300 °C and 600 °C.
13. The metal to glass seal of claim 9, wherein the metal cap comprises aluminum.
14. The metal to glass seal of claim 9, wherein the angle θ ranges from about 10° to about 60°.
15. A method of sealing metal to glass, comprising:
inserting a glass tube into a metal cap, the metal cap having a depth D and comprising a base, and a crown, the glass tube inserted into the metal cap to a depth less than D;
filling a space between the glass tube and the metal cap with a low melting point glass material;
heating the low melting point glass material to or above a melting point;
cooling the low melting point glass material at least until the low melting

point glass material solidifies and seals the metal cap to the glass tube.

16. The method of claim 15, wherein the crown comprises corrugations that absorb thermal expansion and contraction of the metal cap during the heating and the cooling.

17. The method of claim 15, wherein the depth the glass tube is inserted into the metal cap is approximately one half D.

18. The method of claim 15, wherein a first diameter of a base of the metal cap is less than an outer diameter of the glass tube.

19. The method of claim 18, wherein a second diameter at a free edge of the metal cap is greater than the outer diameter of the glass tube.

20. The method of claim 15, wherein the metal cap comprises aluminum.

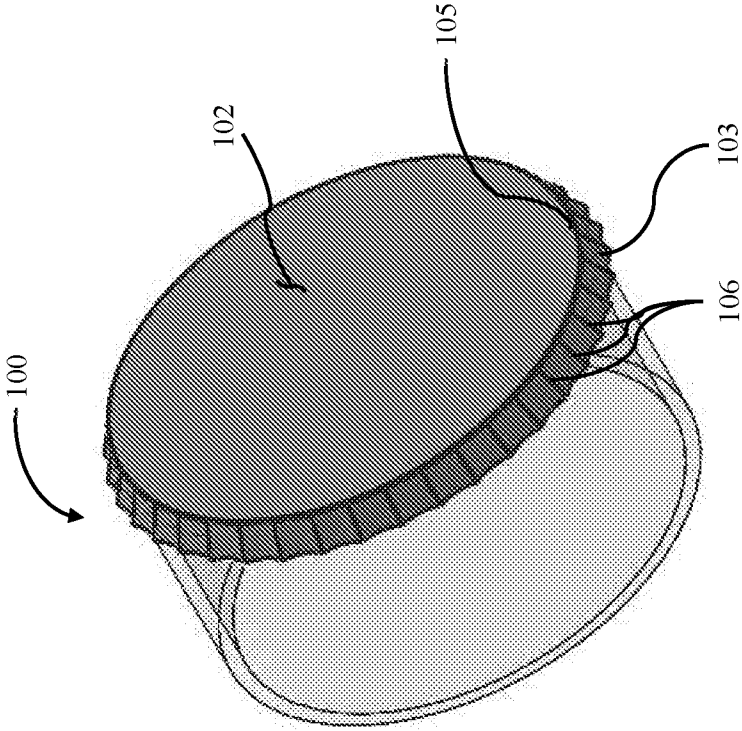


FIG. 1

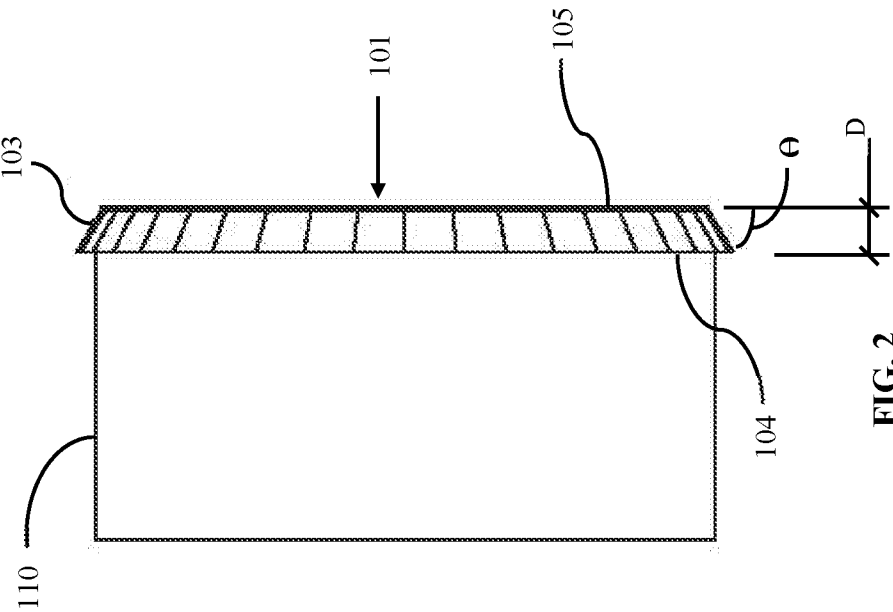


FIG. 2

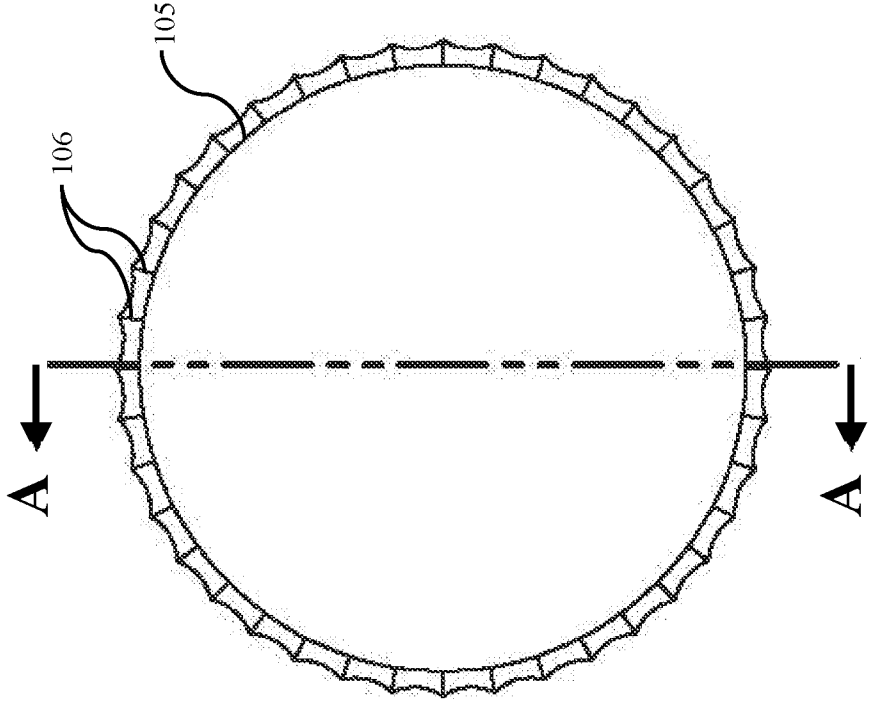
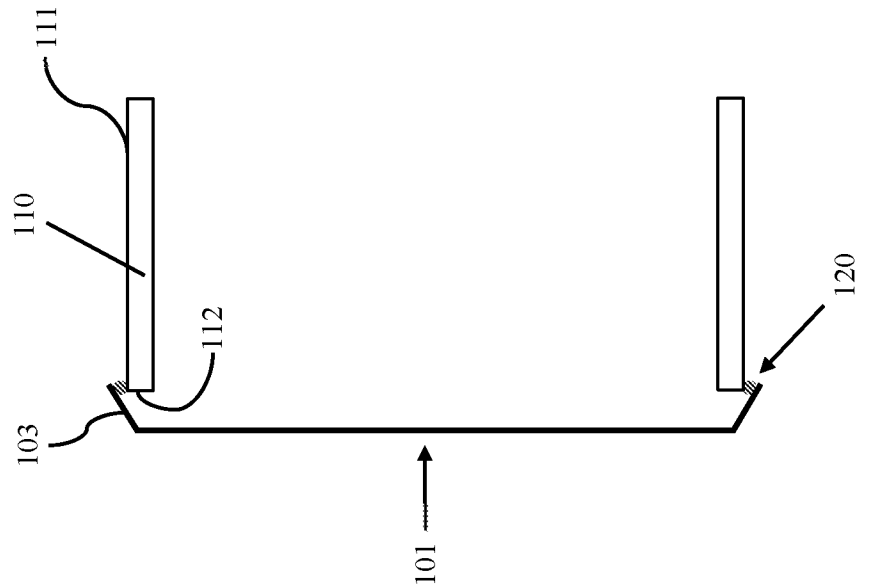
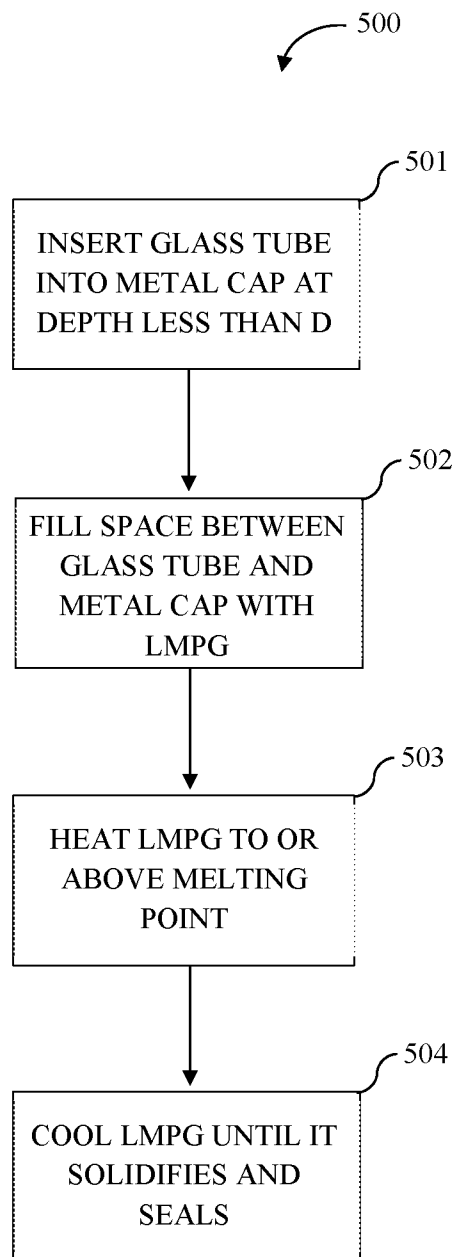


FIG. 3



SECTION A-A
FIG. 4

**FIG. 5**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 19/63959

A. CLASSIFICATION OF SUBJECT MATTER

IPC - B65D 41/12; B67B 3/00; C03C 8/24; H01L 23/10 (2020.01)

CPC - B32B 2439/70; B65D 41/12; B67B 3/023; C03C 8/24; H01L 23/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

See Search History document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JPH08-59295A (Nippon Electric Glass Co.) 05 March 1996 (05.03.1996); entire document, but especially: para [0001], para [0002]	1-20
A	US 2014/0299256 A1 (Sridharan et al.) 09 October 2014 (09.10.2014); entire document, but especially: para [0002], para [0008], para [0010], para [0056]	1-20
A	US 2017/0081087 A1 (Hillmann et al.) 23 March 2017 (23.03.2017); entire document, but especially: para [0006], para [0007], para [0015], para [0017], para [0021], para [0064], para [0080], para [0083], fig. 1A, fig. 1B	1-20
A	US 2004/0195580 A1 (Nishizawa et al.) 07 October 2004 (07.10.2004); entire document	1-20
A	US 5,106,886 A (Hofeldt et al.) 21 April 1992 (21.04.1992); entire document	1-20

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

27 January 2020

Date of mailing of the international search report

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