Feb. 17, 1953 J. A. PASK ET AL 2,629,093

MULTISEAL ENVELOPE AND THE METHOD OF MAKING

Filed March 8, 1949

Fig. 1.

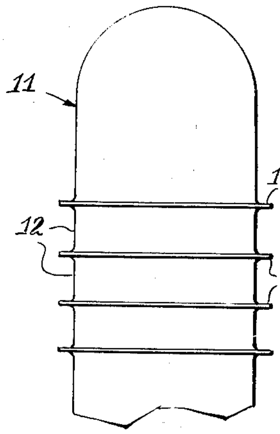



Fig. 2.

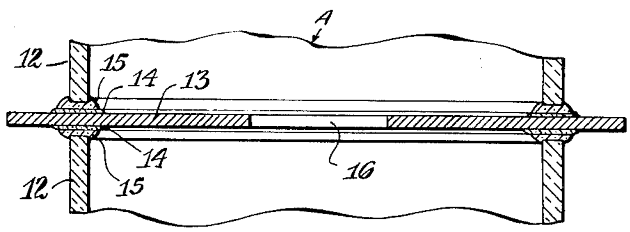



Fig. 3.

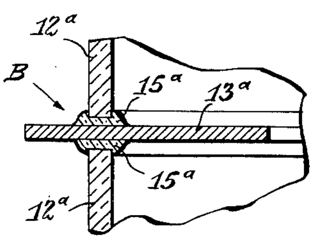
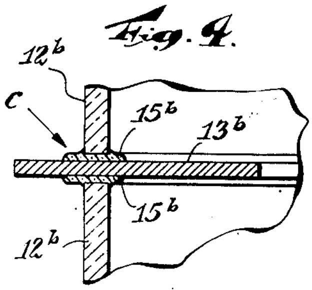



Fig. 5.

A
B
C
TREATED METAL DISCS COATED WITH GLASS OF PROPER EXPANS.
METAL DISCS TREATED
METAL DISCS TO BE COATED WITH WELDING GLASS POWDER
APPLY WELDING GLASS WITH THERMAL EXPANS. EQUIVALENT TO THAT OF METAL
FIRE COATING TO FORM ENAMEL.
WHOLE ASSEMBLY SUPPORTED BY JIGS, INSERTED IN OVEN & TEMP. RAISED ABOVE SOFTENING POINT OF WELDING GLASS BUT STILL BELOW THAT OF GLASS ENVELOPE
ANNEAL

Fig. 4.

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2,629,093

## MULTISEAL ENVELOPE AND THE METHOD OF MAKING

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8 Claims. (Cl. 220—2.3)

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This invention relates to the manufacture of multiseal envelopes or tubes and more particularly to the simultaneous making of the seals in such tubes.

The principal object of our invention, generally considered, is to connect the parts of a multi-seal tube together as one operation, thereby saving time and reducing costs.

Another object of our invention is to manufacture multi-seal tubes involving butt seals between "Kovar" or other metal parts and envelope portions of glass, vitreous or insulative material having a corresponding coefficient of expansion, with welding glass of lower softening point and a narrower viscosity working range, but with an approximately corresponding coefficient of expansion, applied between said metal parts and envelope portions.

Other objects and advantages of the invention will become apparent as the description proceeds.

Referring to the drawing:

Figure 1 is an elevational view of a portion of a vacuum tube or envelope embodying our invention and adapted for making a discharge device of the general type illustrated in the Machlett et al. Patent No. 2,376,439.

Figure 2 is an axial sectional view of a portion of the tube illustrated in Figure 1, on an enlarged scale.

Figure 3 is a fragmentary view corresponding to Figure 2, but showing a modification.

Figure 4 is a fragmentary view corresponding to Figure 3, but showing another modification.

Figure 5 is a flow diagram showing how our invention may be practiced.

Prior to our invention, in the manufacture of seals in a multi-section tube, a portion of one form of which is illustrated in Figure 1, a glass blower made each seal individually. Over-oxidization during the sealing was prevented by first powder-glassing or glass-enameling the seal metal. Successful assemblies were thus achieved but they were time-consuming.

In accordance with the present invention, we propose to effect the simultaneous sealing of the parts of a complete multi-section tube 11, a portion of which is illustrated in Figure 1, within an electric or other oven (not shown), thereby simultaneously exposing the whole assembly for heating to the same elevated temperature. Figure 2 illustrates a smaller portion of such a tube, comprising a plurality of hollow cylindrical vitreous, glass or insulative envelope sections 12, butt-sealed to an annular metal disk 13 therebetween, and hav-

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ing a coefficient of expansion adapting it for such sealing. The metal disks 13 in the present embodiment are assumed to be made from flat plates of "Kovar," which is an alloy of nickel, cobalt, and iron, as described in the Lempert et al. Patent No. 2,279,831, dated April 14, 1942, each cut to circular form and provided with a central aperture 16. However, we contemplate the employment of disks of other metal, such as nickel-iron alloy, nickel, iron, and copper.

The metal disks 13, of the embodiment of Figures 1 and 2, are first powder-glassed on the sealing areas with rings 14 of glass having a coefficient of expansion corresponding with that of the metal to which applied. If the disks 13 are of "Kovar" or molybdenum, we desirably employ glass which we arbitrarily designate No. 6 or that which we arbitrarily designate No. 3, the properties of which and four others, respectively designated Nos. 1, 2, 4 and 5, are disclosed later. It will be seen that No. 2 has the highest softening point and a coefficient of expansion close to that of No. 5.

However, if the metal of the disks 13 is not "Kovar," the glass applied is of a composition corresponding with the coefficient of expansion of the metal used. Glass No. 5 may, for example, be employed with tungsten disks. The glass 14, may, in any event, be applied as disclosed in the Pask et al. application, Serial No. 591,998, filed May 4, 1945, now Patent No. 2,560,593, dated July 17, 1951, as by roughening the metal in accordance with one or more of the methods selected from the group consisting of controlled pre-oxidization, sand blasting, and chemical treatment such as etching with acid. After such metal treatment, the glass is applied to the metal in any desired manner, as by spraying, painting, or otherwise applying, for example, as a suspension, dried and fired to form an enamel on the metal.

On these enameled areas 14 are then applied coatings 15 of glass, which may be designated "welding glass" in that, while having a coefficient of thermal expansion equivalent to, or corresponding with, that of the glass to which applied, it has a considerably lower softening point and a narrower viscosity working range. It is applied in any desired manner as by spraying, painting, or otherwise applying, for example, a suspension of the glass as powder. If the sections 12 are of hard glass, we may use an experimental sealing, welding, or brazing glass 15, of the kind designated No. 4, and employ it for connecting "Kovar" parts enameled with No. 6

glass to parts made of Corning No. 6 glass, for example.

If it were desired to use sealing glass to connect metal parts 13 of corresponding expansivity, that is, agreeing near enough in coefficient of thermal expansion to allow satisfactory sealing thereto, with sections 12 made of soft glass, such as No. 1 lime glass, then a soft glass composition with a softening point of not higher than 695° C. and with a coefficient of expansion between 0° and 300° C. of from 88 to $94 \times 10^{-7}$, is suitable for the purpose.

A suitable sealing or welding glass may be manufactured by using a large proportion of lead oxide, PbO, relatively smaller proportions of silica and alumina, and still smaller proportions of alkali and boric oxide, in order to make a composition having the desired coefficient of expansion and the proper attributes, insofar as softening and melting points are concerned.

After forming the metal disks with fired coating 14 and unfired coating 15, as illustrated in Figure 2, they are assembled with a glass section 12 between each pair of metal disks, supported by proper jigs, and inserted in an oven. All parts of the whole assembly are thus simultaneously exposed for heating to the same, or substantially the same, temperature above the softening point of the welding glass, but still below that of the glass envelope sections, thereby causing said welding glass to soften and seal the parts together. After the seals are made, the assembly is annealed within the same oven or transferred to a lehr for mass annealing. All of the foregoing operations are outlined by part "A" of Figure 5.

Figure 3 shows another form of our invention in which metal disks 13ª, like the disks 13 of the preceding embodiment, are connected to envelope sections 12ª, like the sections 12, in which the metal sections are not first glassed or enameled. These sections 13ª are rather treated for sealing, as by a controlled pre-oxidizing, sand blasting, and/or chemical treatment, as previously referred to, and then have a suitable welding glass that will develop adherence to the metal directly applied. This is possible, since the temperature for softening the welding glass during sealing just as in glass-enameling is not sufficient to cause excessive oxidization of the metal in the time required for making the seals.

The rest of the procedure is the same as that outlined in connection with the first embodiment, and as represented by the part "B" of Figure 5. That is, the welding glass 15ª is applied like the welding glass 15, only in this case to the metal, rather than to the enameled surface of the metal. The whole assembly is supported by jigs, inserted in an oven, and the temperature raised above the softening point of the welding glass, but still below that of the glass sections 12ª, whereby said welding glass melts and effects a sealing of the parts. Annealing is then effected as in the preceding embodiment. Both procedures are suggested since over-oxidization of the metal would probably occur if multi-sealing is attempted without any powder-glassing of the metal with the proper glass as indicated in either Figure 2 or 3.

Figure 4 shows still another modification wherein the preferably "Kovar" disk 13ᵇ is treated for sealing, as in the preceding embodiments, and then powder-glassed with appropriate welding glass that will develop adherence to the metal, such as that corresponding in expansion with glass No. 6, 3, or 5. This powder-glassing is indicated at 15ᵇ, and the application and treatment to form an enamel may be like that of the coating 14 of Figure 2. The parts are then assembled as in the preceding embodiments, that is, the enameled disks 13ᵇ are alternately stacked with and between vitreous envelope sections 12ᵇ. The whole assembly is supported by jigs, inserted in an oven, and the temperature raised above the softening point of the welding glass enamel, but still below that of the envelope sections 12ᵇ, whereby the enamel softens or melts and the parts become sealed together. Annealing may be effected as in the preceding embodiments. The chief advantage of this modification, outlined by part "C" of Figure 5, is that placement of unfired powdered welding glass in the jig assembly as indicated in the first two examples is eliminated, allowing much simpler and rougher handling of the parts.



It should be understood that the above methods can be applied with the use of ceramic rather than glass envelope portions 12, 12ª, and 12ᵇ, provided metals and welding glass are employed whose thermal expansions correspond, so the term insulative is used as generic to both.

Some of the advantages of the proposed methods are:

1. Rapid mass production may take place under controlled conditions, resulting in reduced cost.
2. The assemblies may be condensed with clearances between the metal parts that would be impossible for a glass blower to achieve in the ordinary manner.
3. Multi-section assemblies may be manufactured safely with low shrinkage.
4. Safe production may be accomplished, particularly with assemblies employing ceramic envelope sections, since normal glass blowing methods would cause defects due to rapid thermal changes and corresponding thermal shocks.

The properties of the glasses heretofore referred to, are as follows:

Glass No. 1 is a clear lime bulb glass. It has a softening point at 696±5° C., an annealing point at 510° C., a strain point at 475° C., a coefficient of expansion of $92 \pm 2 \times 10^{-7}$ between 0 and 300° C., a working point at approximately 1000° C., and a density of 2.47. The $\log_{10}$ of its resistivity at 350° C. is 5.17. Its power factor is .009 (expressed as a decimal—not as per cent, its dielectric constant 7.2, and its loss factor .0652 (expressed as a decimal—not as per cent).

Glass No. 2, is designated as an ignition tube glass. It has a softening point at 915° C., an annealing point at 712° C., a strain point at 672° C., a coefficient of expansion of $41 \times 10^{-7}$ between 0 and 300° C., a working point at approximately 1198° C., and a density of 2.53. The $\log_{10}$ of its resistivity at 350° C. is 9.90. Its power factor is .0038 (expressed as a decimal—not as per cent), its dielectric constant 7.2, and its loss factor .0273 (expressed as a decimal—not as per cent).

Glass No. 3 is a potash-borosilicate glass containing a small percentage of alumina. It is sold for radio special sealing. It has a softening point at 702±7° C., an annealing point at 484° C., a strain point at 450° C., a coefficient of expansion of $47.5 + 1.5 \times 10^{-7}$ between 0 and 300° C., a working point at approximately 1080° C., and a density of 2.24. The $\log_{10}$ of its resistivity at 350° C. is 7.95. Its power factor is .0018 (expressed as a decimal—not as per cent), its dielectric constant 4.8, and its loss factor .0086 (expressed as a decimal—not as per cent).

Glass No. 4 is sealing, welding, or brazing glass.

It has a softening point at 600° C., an annealing point at 455° C., a strain point at 432° C., and a coefficient of expansion of $49\times10^{-7}$ between 0 and 300° C.

Glass No. 5 is a soda-aluminum-borosilicate glass. It is designated a clear sealing glass. It has a softening point at 704° C., an annealing point at 467° C., a strain point at 431° C., a coefficient of expansion of $40.5\pm1.5\times10^{-7}$, between 0 and 300° C., and a density of 2.19.

Glass No. 6 is a potash-barium-borosilicate glass sold for sealing purposes. It has a softening point at $708\pm5°$ C., an annealing point at 480° C., a strain point at 442° C., a coefficient of expansion of $46\pm1.5\times10^{-7}$ between 0 and 300° C., a working point at approximately 1115° C. and a density of 2.29. The $\log_{10}$ of its resistivity at 350° C. is 7.40. Its power factor is .0026 (expressed as a decimal—not as per cent) its dielectric constant 5.1, and its loss factor .0133 (expressed as a decimal—not as per cent). The chemical analyses of the foregoing glasses, in percentages, are indicated by the following table:

| | Glasses by Nos. previously assigned | | | | | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| $SiO_2$ | 73.3 | 56.3 | 68.0 | 40 | 68.1 | 65.2 |
| $B_2O_3$ | | 5 | 22.5 | 20 | 25.9 | 15.3 |
| $R_2O_3$ | 1.4 | 20.8 | 2.5 | | 1.3 | 8.9 |
| CaO | 5.4 | 4.9 | | | Trace | 0.2 |
| BaO | | | | | | 2.8 |
| $Na_2O$ | 15.6 | 1 | 3.5 | | 3.7 | 2.5 |
| $K_2O$ | 0.5 | | 3.5 | | .5 | 3.6 |
| $Li_2O$ | | | | | .2 | 1.1 |
| F | | | | | .2 | 0.2 |
| $As_2O_5$ | | | Trace | | .1 | |
| $Sb_2O_5$ | | | | | | |
| PbO | | | | 35 | | |
| MgO | 3.8 | 12.1 | | | | |
| $Al_2O_3$ | | | | 5 | | |
| Alkali oxide | | | | $<1$ | | |

Although preferred embodiments of our invention have been disclosed, it will be understood that modifications may be made within the spirit and scope of the appended claims.

We claim:

1. The method of making multi-seal envelopes for evacuated electron-discharge devices, comprising treating a plurality of annular metal members for the application of vitreous material, as means for connecting thereto a plurality of hollow-insulative enevlope sections alternating with said annular metal members, applying vitreous material to said annular metal members in the form of at least one annular layer on each side thereof, said layers being smaller in radial dimension than the annular metal members, so as to terminate short of the inner and outer edges of said members, having a softening point considerably lower than that of the envelope sections, but a coefficient of expansion approximately corresponding with that of said metal members, a mean diameter approximately corresponding with, and a radial width exceeding the thickness of, the envelope sections, assembling a plurality of said hollow insulative envelope sections alternating with said annular metal members, the adjacent edges of said envelope sections engaging the annular layers of said lower-softening-point material, supporting said assembly, simultaneously exposing the whole assembly for heating to a temperature, which is the same for all parts thereof, above the point of fusion of said lower softening point vitreous material but still below the softening point of said envelope sections, whereby said lower softening point vitreous materials fuses, allowing the temperature of said assembly to be reduced to cause the vitreous material to simultaneously seal the parts together, and annealing the formed envelope.

2. The method of making multi-seal envelopes for evacuated electron-discharge devices, comprising treating a plurality of annular metal members for the application of glass, as a means for connecting thereto a plurality of hollow insulative envelope sections alternating with said annular metal members, coating the surface portions of said metal members with glass having a coefficient of expansion approximately corresponding with that of said metal, applying to the coated portions a coating of a welding glass having a coefficient of expansion approximately corresponding with, but a softening point considerably lower than, that of said first-mentioned coating, said coatings being all in the form of annular layers of smaller radical dimensions than the annular metal members so as to terminate short of the inner and outer edges of said members, assembling a plurality of said hollow insulative envelope sections alternating with said annular metal members, the adjacent edges of said envelope sections engaging the annular layers of said welding glass, supporting and simultaneously exposing the whole assembly for heating to a temperature, which is the same for all parts thereof, above the point of fusion of said welding glass, but still below that of said glass envelope sections, whereby said welding glass fuses, allowing the temperature of said assembly to be reduced to cause the welding glass to simultaneously seal the parts together, and annealing the formed envelope.

3. The method of making multi-seal envelopes for evacuated electron-discharge devices, comprising treating a plurality of annular metal members for the application of glass as a means for connecting thereto a plurality of hollow insulative envelope sections alternating with said annular metal members, coating the surface portions of said metal members with a welding glass having a coefficient of expansion approximately corresponding with, but a softening point considerably lower than, that of said envelope sections, said coatings being all in the form of annular layers of smaller radial dimension than the annular metal members, so as to terminate short of the inner and outer edges of said members, assembling a plurality of said hollow insulative envelope sections alternating with said annular metal members, the adjacent edges of said envelope sections engaging the annular layers of said welding glass, supporting and simultaneously exposing the whole assembly for heating to a temperature, which is the same for all parts thereof, above the point of fusion of said welding glass, but still below that of said envelope sections, whereby said welding glass fuses, allowing the temperature of said assembly to be reduced to cause the welding glass to simultaneously seal the parts together, and annealing the formed envelope.

4. The method of making multi-seal envelopes for evacuated electron-discharge devices, comprising treating a plurality of annular metal members for the application of glass, as a means for connecting thereto a plurality of hollow insulative envelope sections alternating with said annular metal members, coating the surface portions of said metal members with a welding glass powder having a coefficient of expansion approximately corresponding with, but a softening point

considerably lower than, that of said envelope sections, firing said coating to form an enamel on said members, said coatings being all in the form of annular layers of smaller radial dimension than the annular metal members, so as to terminate short of the inner and outer edges of said members, assembling a plurality of said hollow insulative envelope sections alternating with said annular metal members, the adjacent edges of said envelope sections engaging the annular layers of said welding glass, supporting and simultaneously exposing the whole assembly for heating to a temperature, which is the same for all parts thereof, above the point of fusion of said welding glass, but still below that of said envelope sections, whereby said welding glass fuses, allowing the temperature of said assembly to be reduced to cause the welding glass to simultaneously seal the parts together, and annealing the formed envelope.

5. A multi-seal envelope for evacuated electron-discharge devices comprising a plurality of hollow insulative envelope sections, adjacent ones of which are united by a mating annular metal disk of larger outside diameter, with vitreous material having a softening point considerably lower than that of the envelope sections, but a coefficient of expansion corresponding approximately with that of said metal members, as a thin layer terminating short of the inner and outer edges of said metal members, between each envelope section and the adjacent metal members and bonding said sections and members together.

6. A multi-seal envelope for evacuated electron-discharge devices comprising a plurality of hollow insulative envelope sections, adjacent ones of which are united by a mating annular disk of larger outside diameter and formed of nickel-cobalt-iron alloy, with coatings on the surface portions of said disk, adjacent the envelope sections to which they are united, of glass selected from the group consisting of Nos. 3 and 6, and glass No. 4, having a softening point considerably lower than that of the envelope sections, between each envelope section and the glass coatings on the adjacent metal members, terminating short of the inner and outer edges, and bonding said sections and disks together.

7. A multi-seal envelope for evacuated electron-discharge devices comprising a plurality of hollow insulative envelope sections, adjacent ones of which are united by a mating annular disk of larger outside diameter and formed of nickel cobalt-iron alloy, with a thin layer of glass No. 4, terminating short of the inner and outer edges of said disk, between each envelope section and the adjacent metal member and bonding said sections and members together.

8. A composite envelope comprising a plurality of hollow insulative sections, adjacent ones of which are united by a mating annular disk of larger outside diameter and composed of a nickel-cobalt-iron alloy, the surfaces of which disk have a roughening layer of oxide thereon, and carry a thin layer of glass terminating short of the inner and outer edges, of approximately the same coefficient of expansion as that of said alloy, fused onto said roughened surfaces and in which the layer of oxide is absorbed, the edges of said envelope sections abutting the glass-coated surfaces of said rings and sealed thereto by the glass coatings, whereby metal edge portions project from said envelope.

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