SOLDERABLE STAINLESS STEEL

Original Filed Feb. 14, 1968

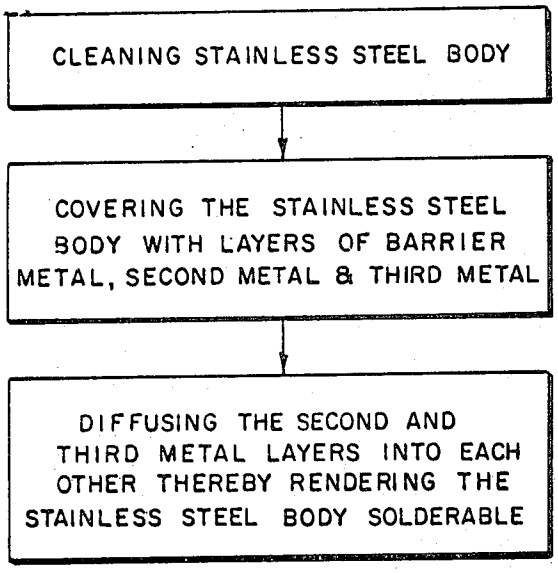


FIG. 1

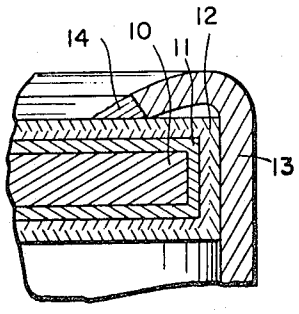


FIG. 2

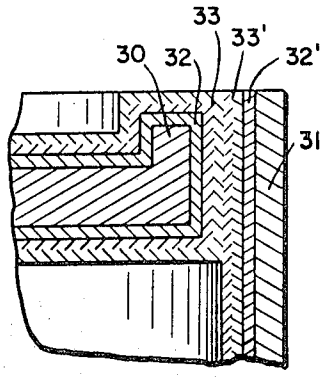


FIG. 3

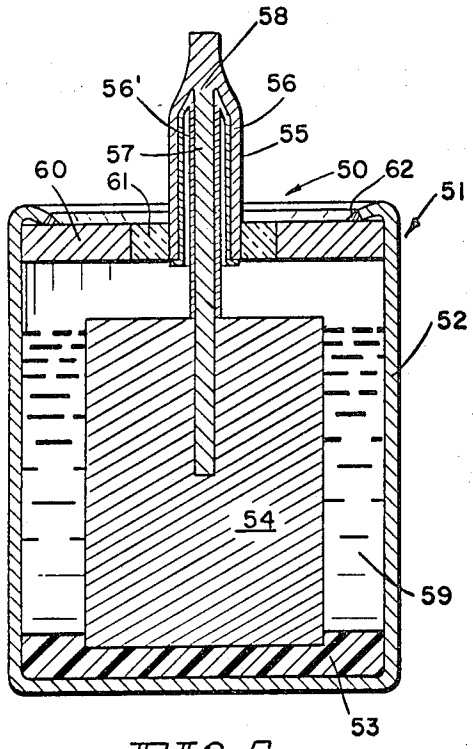


FIG. 5

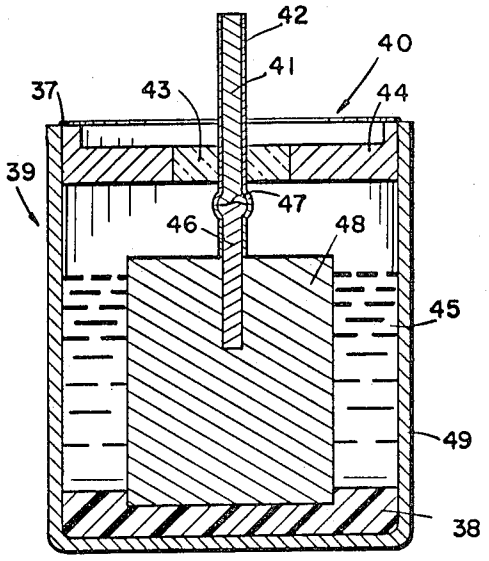


FIG. 4

United States Patent Office 3,698,880 Patented Oct. 17, 1972

3,698,880

SOLDERABLE STAINLESS STEEL

Edwin Russell Koons, Whiteland, Ralph G. Parker, Jr., Shelbyville, and Jeffery P. Rupley, Lawrence, Ind., assignors to P. R. Mallory & Co., Inc., Indianapolis, Ind.

Application Nov. 5, 1969, Ser. No. 871,344, now Patent No. 3,634,048, which is a division of application Ser. No. 705,372, Feb. 14, 1968, now Patent No. 3,515,950. Divided and this application Apr. 30, 1971, Ser. No. 139,205

Int. Cl. B32b *15/04*

U.S. Cl. 29—195 5 Claims

ABSTRACT OF THE DISCLOSURE

Means and method for rendering solderable a stainless steel body resistant to corrosive attack from common electrolytes. The method includes the steps of covering at least one surface of a stainless steel body with layers of metal including a first metal layer selected from the group consisting of nickel, cobalt and chromium which serves as a barrier layer to the diffusion of a subsequent metal layer or layers therethrough to the stainless steel body. The subsequent metal layer is resistant to corrosive attack from common electrolytes and is solderable. The subsequent metal layer consists of silver alloyed with a noble metal selected from the group consisting of gold, palladium, platinum, rhenium and osmium. The alloy layer may be formed by covering the barrier layer with a layer of silver and then a layer of gold or one of the other metals. The layered stainless steel body is heated to a temperature below the melting point temperature of the metal layer having the lowest melting point so that the layers, with the exception of the barrier layer, diffuse into one another, thereby forming the alloy layer overlying the barrier layer which is resistant to corrosive attack from common electrolytes and is solderable. The stainless steel body is rendered solderable.

This is a division of application Ser. No. 871,344, filed Nov. 5, 1969, now Pat. 3,634,048 dated Jan. 11, 1971 which in turn is a divisional of Ser. No. 705,372, filed Feb. 14, 1968, now U.S. Pat. 3,515,950.

The present invention relates to a means and method for rendering solderable a stainless steel body useful for, inter alia, the metal portion of a glass-to-metal seal in a wet electrolytic capacitor resistant to corrosive attack from common electrolytes.

Stainless steels include additions of nickel and chromium which improve the corrosion resistance of the major constituent iron. Due to the improved resistance of iron to corrosive attack from common electrolytes when about 11% by weight or more of free chromium and nickel and other alloying metals are added to the iron, it is thought that the use of such stainless steel as an end seal for capacitors using an electrolyte having a concentration of about 39% or more of sulfuric acid would be advantageous. Other electrolytes are hydrochloric acid, perchloric acid, phosphoric acid, acetic acid, formic acid, oxalic acid, propanoic acid, a combination of selenous acid-selenic acid-sulfuric acid and the like.

Stainless steels fall into various categories in accordance with the amount of chromium and nickel mixed with iron. The stainless steel body may also include minor additions of other metals such as columbium, tantalum, manganese, phosphorous, silicon, copper and the like to improve selected properties of the stainless steel such as corrosion resistance and the like. However, the mechanism which protects these alloys from environmental and chemical attack also prevents the effective cleaning of the surfaces by fluxes so that chemical preparation of

the surface of stainless steel for wetting is difficult even under conditions where the stainless steel is not subjected to an extraordinary treatment such as that required for preparing a glass-to-metal seal generally used with a hermetically sealed wet electrolyte capacitor.

A glass-to-metal hermetic seal is usually formed by selecting a ring of suitable metal and filling the center of the metal ring with a suitable glass frit. A metal wire extends through the glass frit and is used as a lead wire for a capacitor body. The metal ring, the glass frit and the lead wire are heated in situ at a sufficiently high temperature to melt the glass and render the viscosity of the glass such as to cause the molten glass to flow. In the case of compression glass-to-metal seals, the temperature to which the components of the seal are heated is usually about 930° C. or above in order to render the glass molten. The glass and metal members of both compressive and noncompressive seals are so constrained that the glass flows only radially to fill the entire space between the lead wire projecting through the glass and the metal ring. A glass-to-metal seal suitable for use as a hermetic seal is provided upon cooling of the molten glass.

It was found that when stainless steel resistant to corrosive attack from common electrolytes such as sulfuric acid is subjected to the high temperature necessary to render the glass frit molten, a thermal oxide film or coating is formed thereon which renders joining of the stainless steel to a solderable body more difficult than the usual difficulty encountered with the soldering of stainless steel to a solderable body. The means and method of the present invention permit the use of stainless steel in glass-to-metal hermetic seals yet the seal exhibits excellent solderability characteristics.

Therefore, it is an object of the present invention to provide a solderable stainless steel body including a body of stainless steel coated with materials so as to substantially prevent thermal oxidation of the stainless steel body during the formation of a glass-to-hermetic seal thereby improving the solderability characteristics of the resultant glass-to-stainless steel seal.

Another object of the present invention is to provide a glass-to-metal seal wherein the metal portion thereof is predominantly stainless steel covered with a coating which undergoes no detrimental thermal oxidation when subjected to elevated temperatures and which can be easily soldered with a solder.

A further object of the present invention is to provide a strong mechanical and corrosion resistant joint between a stainless steel body and an open ended container of solderable metal so that the open end of the container is closed by the stainless steel body.

Another object of the present invention is to provide a method for making a glass-to-stainless steel hermetic seal which is solderable.

A further object of the present invention is to provide a hermetically sealed wet electrolytic capacitor comprising a solderable can having an open end and a closed end, the open end thereof closed by a glass-to-stainless steel hermetic seal, the can and the hermetic seal being resistant to chemical attack by the electrolytes of the capacitor.

Another object of the present invention is to provide a hermetically sealed, liquid electrolyte capacitor.

Yet another object of the present invention is to provide an improved hermetically sealed electronic device wherein the housing components thereof are substantially directly and substantially permanently bonded together to resist cracking therebetween even when subjected to widely fluctuating temperatures.

Still another object of the present invention is to provide a process for joining at least two pieces or parts of metal of which piece or part one is stainless steel so as to provide a strong mechanical joint and provide a hermetic seal between the metallic pieces or parts.

Yet another object of the present invention is to provide a method wherein nickel, silver and gold respectively are plated on a stainless steel piece to thereby render the stainless steel piece solderable.

A further object of the present invention is to provide a method whereby a nickel layer is electro-plated over a stainless steel body so as to prevent diffusion of a silver-noble metal alloy over the nickel layer through the nickel layer to the stainless steel body, the silver-noble metal layer being resistant to chemical attack and being solderable.

With the aforementioned objects enumerated, other objects will be apparent to those persons possessing ordinary skill in the art. Other objects will appear in the following description, appended claims and appended drawings. The invention relates to the novel construction, combination, arrangement and cooperation of elements as hereinafter described and more particularly as defined in the appended claims.

The appended drawings illustrate an embodiment of the present invention constructed to function in the most advantageous modes devised for the practical application of the basic principles involved in the hereinafter described invention.

In the drawings:

FIG. 1 illustrates the several steps of a process for rendering solderable stainless steel used in a glass-to-metal seal;

FIG. 2 is a diagrammatic side view of two parts, one of which is stainless steel having the surfaces thereof prepared in accordance with the method of the present invention and joined to a solderable material;

FIG. 3 shows an embodiment of the present invention wherein two stainless steel parts prepared in accordance with the method of the present invention and joined to each other by diffusion;

FIG. 4 is a sectional view of a capacitor using a liquid electrolyte and having a glass-to-stainless steel hermetic seal closing the open end of the housing for the capacitor; and

FIG. 5 is a sectional view of a capacitor including a liquid electrolyte and an embodiment of the glass-to-stainless steel hermetic seal illustrated in FIG. 4.

Generally speaking the present invention relates to a corrosion resistant stainless steel part rendered solderable by covering the part with at least two metal layers. The initial layer of metal substantially prevents the metal layer or other metal layers from diffusing therethrough to the stainless steel. The other metal layer or layers are solderable and resistant to chemical attack from common types of electrolytes.

It was found that during the fabrication of the glass-to-metal seal, a stainless steel resistant to corrosion attack from a sulfuric acid electrolyte formed a thermal oxide which rendered the stainless steel unsolderable using conventional stainless steel soldering techniques. The temperature used to form the glass-to-metal seal is about 930° C. to 950° C. The corrosive resistant stainless steel consists essentially of about 2% by weight manganese, about 1% by weight silicon, 19–21% by weight chromium, about 30–38% by weight nickel, about 2–3% by weight molybdenum, about 3–4% by weight copper with minor additions of columbium, tantalum, phosphorus and sulfur, and the remainder iron. The stainless steel fabricated from the above listed constituents is resistant to chemical attack from hot sulfuric acid electrolytes having a concentration of up to 40%.

Referring now to the figures of the drawing, FIG. 1 illustrates the several steps of the method involved in the present invention. A stainless steel body resistant to

corrosive attack from sulfuric acid electrolyte is thoroughly cleaned by any suitable electro cleaning method or the like. The stainless steel body may have a ring-like configuration if the stainless steel is to be used as the metal portion of a glass-to-metal seal in a hermetically sealed capacitor. The stainless steel body is electroplated with a barrier layer having a sufficient metal thickness to prevent diffusion therethrough of a subsequent metal layer or layers. The barrier layer is selected from the group consisting of nickel, cobalt and chromium. Of the several metals, nickel is most preferred. If nickel is used, the thickness of the nickel layer is about 0.0002 to 0.0004 inch thick. A nickel layer having a thickness of about 0.0003 inch thick is preferred. Electro-plating a metallic barrier layer of nickel less than 0.0002 inch thick seriously subtracts from the ability of the nickel barrier layer to prevent a subsequent metallic layer or layers from diffusing therethrough to the stainless steel body. The upper limit of the thickness of the nickel barrier layer may be exceeded, however, no tangible benefit appears to result therefrom.

The barrier layer metal is electro-plated with a silver layer having a thickness of about 0.0008 to 0.002 inch thick. A layer of silver having a thickness of about 0.001 inch thick is preferred. Electro-plating a silver layer over the barrier layer metal having a thickness of less than 0.0008 inch undermines the ability of the silver layer to prevent the subsequent metal layer or layers from diffusing therethrough. Exceeding a silver layer thickness of about .002 inch does not appear to result in any tangible benefits nor does exceeding the upper limit thickness result in a condition detrimental to the system. Copper may be substituted for silver in situations where the stainless steel is used in an environment where sulfuric acid is not present.

The silver layer is electro-plated with a noble metal selected from the group consisting of gold, palladium, platinum, rhenium and osmium. Of the several metals used gold is preferred, and 24 k. gold is most preferred. The thickness of the gold layer is about 0.00005 to 0.0002 inch thick. A gold layer having a thickness of about 0.0001 is preferred. A 24 k. gold layer having a thickness of less than about 0.00005 inch thick results in detrimental oxidization of the underlying silver layer during treating of the plated stainless steel body to cause the silver and gold to diffuse into one another. If the silver oxidizes to an appreciable extent, the solderability of the stainless steel is seriously undermined. Exceeding a gold thickness of about 0.0002 inch does not appear to result in any tangible benefit nor does the increased thickness of the gold layer result in a condition detrimental to the system.

The plated stainless steel body is heated to a temperature of about 800°–950° C. for approximately 15 to 30 minutes and allowed to cool slowly. A temperature of about 930–950° C. for about 15 to 20 minutes is preferred for the diffusion step and glass melting step may be conveniently performed simultaneously. During the heating of the plated stainless steel part to a temperature of about 800–950° C. there is effected a diffusion of the noble metal layer or film into the silver layer or film underneath the noble metal film so as to provide a layer of silver-noble metal alloy. Diffusion of the silver layer and the gold layer into each other appears to result in a silver-gold alloy having a thickness greater than the sum total of the thicknesses of the individual silver layer and the gold layer. For example, a silver layer having a thickness of about 0.00108 inch and a gold layer having a thickness of about 0.00010 inch form a swollen silver-gold alloy having a thickness of about 0.00132 inch. In another case, a silver layer having a thickness of about 0.00111 inch and a gold layer having a thickness of about 0.00010 inch form a swollen silver-gold alloy having a thickness of about 0.0123 inch. In the first example, the alloy has an increased thickness of about 16%

greater than the sum total of the thicknesses of the silver layer and the gold layer. In the second example, the alloy has an increased thickness of about 1% greater than the sum total of the thicknesses of the silver layer and the gold layer. It is thought that the interaction of the metals during the diffusion step causes random imperfections in the alloy. However, the imperfections do not extend through the alloy layer.

The preferred alloy of silver-gold contains about 80–98% by weight silver, the remainder essentially gold with minor amounts of impurities. The most preferred composition of the silver-gold alloy is about 88% by weight silver, the remainder essentially gold with minor amounts of impurities. It should be recognized that the percent by weight of the gold content of the alloy could be increased significantly above 20% by weight, but the fact that no additional benefits are realized does not appear to justify the increased cost associated therewith. The apparent limitation of the gold content of the alloy is that a minimum thickness of silver is required to be electro-plated over the barrier layer metal to substantially prevent diffusion of the gold to the barrier layer metal.

The silver and noble metal layers prevent thermal oxidization of the stainless steel during subjection of the stainless steel to the elevated temperatures required for forming the glass-to-metal seal. The silver-noble metal alloy is resistant to chemical corrosion from the sulfuric acid electrolyte, does not form a thermal oxide when subjected to the elevated temperatures required for the formation of the glass-to-metal seal and is easily solderable to any material solderable under normal conditions such as zinc, iron, cadmium, indium, nickel, tin, lead, antimony, bismuth, copper, steel, silver, palladium, gold and platinum or a metal plated with said metals. It is thought, however, that for the purposes of the intended use of the present invention only those metals not subject to corrosive attack from the sulfuric acid electrolyte would be acceptable. Materials such as silver, palladium, platinum and gold or metals plated with said materials such as for example silver plated stainless steel, silver clad stainless steel and the like would be acceptable.

It is thought that any solder which is resistant to corrosion attack from the sulfuric acid electrolyte would be satisfactory. Since the sulfuric acid electrolyte boils at a temperature of about 105° C., the lower the melting point temperature of the solder, the more advantageous the solder. Of the several available solders, silver base solders are preferred because of their resistance to corrosive attack from the sulfuric acid electrolyte and because of their low melting point temperature. Of the silver base solders, silver-lead solder is most preferred. The preferred composition of the silver-lead solder is approximately 95% by weight or more lead, the remainder silver. Of the possible lead-silver alloys, however, a solder having approximately 97.5% by weight lead, the remainder silver is most preferred.

By way of example, a solderable glass-to-stainless steel and seal for an electrolytic capacitor using an electrolyte having about a 40% concentration sulfuric acid may be prepared by anodically cleaning a stainless steel ring of suitable dimensions in a suitable cleaning device. The stainless steel ring is immersed in a bath containing nickel and a "Woods Nickel Strike" is electro-plated thereon having a thickness of about 0.00005 inch. The temperature of the bath is room temperature and the current density during electro-plating is about 10 amperes per square foot. The electro-plated stainless steel ring is rinsed in water. The electro-plated stainless steel ring is immersed in a bath containing nickel sulfamate and having a temperature of about 125–135° F. Nickel is electro-plated onto the stainless steel using a current density of about 4 amperes per square foot. A nickel plate thickness of about 0.0003 inch is realized. The stainless steel ring or part is rinsed in water. A silver strike is electro-plated over the nickel plate by immersing the part in a bath at room temperature and electro-plating using a current density of about 5 amperes per square foot. The thickness of the silver strike is about 0.00005 inch. The part is rinsed in water. The part is immersed in a bath of Mallosil silver at about room temperature. The part is electro-plated with silver by using a current density of about 2.5 amperes per square foot. A silver thickness of about 0.001 inch is electro-plated on the part. The part is rinsed in water. The part is placed in a bath containing gold at room temperature. Electro-plating of gold is carried out using a current density of about 1 ampere per square foot. About 0.0001 inch of 24 k. gold is plated on the part. The part is removed from the bath, dried and the outer portion of the ring is filled with a suitable glass frit. The part and the glass frit are heated to about 940°C.±10° C. for 15 to 20 minutes to cause the silver and gold layers to diffuse into one another thereby forming a silver-gold alloy which is resistant to corrosive attack from a sulfuric acid electrolyte, which prevents the stainless steel from oxidizing during diffusion step, and which is solderable under normal conditions. The heating of the part and the glass frit for the specified time at the specified temperature cause the glass frit to melt and upon cooling, to form a glass-to-stainless steel seal.

Any one of several suitable glass frits may be used to provide the glass-to-metal seal. One of the limitations placed on the glass is that its melting point be less than the lowest melting point temperature of the metals electro-plated over the stainless steel. It is thought that the maximum upper limit on the working point temperature of the glass is about 960° C. since the melting point temperature of silver is about 961° C. A minimum working point temperature of the glass is about 800° C. since it is thought that at last this temperature is required to cause the second and third layers to diffuse into one another within practical lengths of time. It is recognized that the silver layer and the noble metal layer will diffuse into one another without heating if allowed to stand a sufficient length of time. If the diffusion step and the glass melting step are not to be performed simultaneously then the lower limit on the melting point temperature of the glass ceases to be of importance. A suitable glass for use when the diffusion and glass melting steps are performed simultaneously contains the following, by weight constituents: about 28–32% silicon, about 20–23% sodium, about 4–5% by weight potassium, about 10–12% barium the remainder oxygen with traces of lead, chromium, lithium, copper and tin.

It is thought that the diffusion step may be eliminated by electro-plating silver and gold simultaneously from a potassium cyanide solution to thereby form a silver-gold alloy over the barrier layer metal. It is recognized that the constituents of solution will vary in accordance with the percent by weight of the constituents of the silver-gold alloy.

FIG. 2 shows a stainless steel part **10** which may be a component part of a glass-to-metal seal electro-plated with a layer of nickel **11**, and a layer of an alloy of silver-gold **12** wherein the silver and gold are diffused into one another. The silver-gold alloy renders the stainless steel solderable to any suitable material solderable under normal conditions such as a silver-clad stainless steel piece **13** by the use of any suitable solder **14** such as lead-silver solder. The joint between the stainless steel body and the solderable metal part is mechanically strong and resistant to chemical attack from a sulfuric acid electrolyte. The stainless steel piece may be crimped and soldered in the manner shown in FIG. 2 thereby providing a mechanically strong seal construction.

FIG. 3 shows another embodiment of the present invention wherein flanged stainless steel part **30** and stainless steel part **31**, which are resistant to chemical attack by a sulfuric acid electrolyte, are each electro-plated with a layer of nickel **32** and **32'**, a layer of silver and a layer

of gold in accordance with the method taught hereinbefore. The plated stainless steel parts are placed in abutting relationship and then heated to a temperature of about 800–950° C. for approximately 15–30 minutes and allowed to cool slowly. During the heating of the pieces or parts to the temperature of about 800–950° C. there is effected a rapid diffusion of the gold and the silver layers carried by each of the stainless steel parts into one another to form gold-silver alloy **33** and **33'**. Upon cooling of the respective parts, there is effected by such diffusion of the gold and silver layers or films into one another, a resultant gold-silver alloy which provides strong mechanical joint which is resistant to chemical attack by the sulfuric acid electrolyte. It should be noted that the use of solder to effect the joining of the stainless steel parts is eliminated.

FIG. 4 represents an electrical component such as a capacitor **39** including a glass-to-stainless steel assembly **40** which closes the open end of cathode can **49** thereby providing an hermetically sealed wet electrolytic capacitor. The seal assembly **40** includes film forming metal wire **41** having an oxide layer **42** formed thereon which is sealed to a glass ring **43** by heating a glass frit to a temperature of about 930–950° C. to cause the glass to become molten and form a compressive seal with the metal wire projecting therethrough upon cooling. The glass ring **43** is in turn compressively sealed to the outer metal ring **44** of stainless steel. The stainless steel ring is resistant to corrosive attack from a sulfuric acid electrolyte **45**. The electrolyte **45** is a solution which contains about 39 to 40% sulfuric acid. Prior to the assembling of the capacitor an anode riser **46** of any suitable film forming metal having an oxide layer formed thereon projects from an anode **48** of film forming metal and is welded to the metal wire **41** at a point below the seal as shown at **47**. The metal wire, the anode riser and the anode are fabricated from the same film-forming metal, that is, tantalum or the like. The anode **48** may be fabricated from a film-forming metal foil rather than the pressed and sintered powder as shown in FIG. 4. Any resulting defects in the oxide film resulting from the weld operation are heated by the sulfuric acid electrolyte **45**. The seal assembly **10** is then positioned in the non-corrosive cathode can **39** and soldered or brazed in place with a silver base solder **37** as described hereinbefore. A conductive lead wire (not shown) is welded to the lead wire **41** to complete the capacitor. The capacitor may include an anode spacer **38** for seating the anode. The spacer is fabricated from any suitable resilient material which is not chemically attacked by the electrolyte.

FIG. 5 shows yet another embodiment of the present invention using the solderable glass-to-stainless steel seal assembly **50**. The capacitor **51** comprises a non-corrosive metal cathode can **52** having an anode spacer **53** fabricated from any suitable resilient material which is not chemically attacked by the electrolyte. The spacer is positioned in the closed end of the cathode can for heating the capacitor anode **54** fabricated from a suitable film-forming metal. The seal assembly **50** includes a hollow film-forming metal tube **55** having an oxide film **56** formed on the inner periphery thereof. A glass ring **61** circumscribes the tube and is compressively sealed to the outer periphery of the tube and to a non-corrosive metal ring **60** of stainless steel. The metals used in the can and the ring must be compatible with the entire system and capable of being welded together.

An anode riser **57** of a suitable film-forming metal extends from a film-forming metal anode **54** and projects through a portion of the aperture of the tube **55**. The tube, the anode riser and the anode are fabricated from the same film-forming metal, that is, tantalum or the like. The anode **54** may be fabricated from a film-forming metal foil rather than the pressed and sintered powder as illustrated in FIG. 5. It should be noted that the riser has an oxide film **56'** formed on the periphery thereof. The tube is pinched inwardly by any suitable means such as by crimping means and is welded at the crimp as shown at **58** in FIG. 5. The riser is not integral with and positioned by seal assembly **50**. The seal assembly and the anode are then positioned within the cathode can containing a liquid electrolyte such as sulfuric acid. The anode spacer **53** is optional, as anode **12** is securely positioned by the crimp and the weld joint at the crimp. The seal assembly is soldered to the cam using any suitable corrosive resistant solder such as lead-silver solder **62**. The can may be fabricated from any noncorrosive material such as stainless steel, silver, silver plated stainless steel, silver clad stainless steel and the like.

While the invention is illustrated and described in an embodiment it will be understood that modifications and variations may be effected without departing from the scope of the novel concepts of this invention and as set forth in the appended claims.

Having thus described our invention, we claim:

1. A solderable part comprising a strip of metal, a mass of glass circumscribing and fused to said strip of metal, a mass of stainless steel circumscribing said mass of glass thereby forming a glass-to-metal seal, a first layer of metal which is a barrier to diffusion of materials therethrough to said stainless steel, and a second layer of a solderable material over said first layer thereby rendering said stainless steel part solderable.

2. A solderable part as claimed in claim **1**, wherein said first layer is a metal selected from the group consisting of nickel and cobalt and said second layer is an alloy wherein one of the constituents thereof is selected from the group consisting of silver and copper and the other constituent of said alloy is selected from the noble metal group consisting of gold, palladium, platinum, rhenium and osmium.

3. A solderable metal part as claimed in claim **2**, wherein said first layer has a thickness of about 0.0002 to 0.0004 inch thick and said alloy layer consisting essentially of a first constituent having an original thickness of about 0.0008 to 0.002 inch and a noble metal constituent having an original thickness of about 0.00005 to 0.0002 inch.

4. A solderable metal part as claimed in claim **1**, wherein said alloy layer thickness is about 1–16% greater than the thickness of the layered constituents.

5. A solderable metal part as claimed in claim **2**, wherein said alloy constituents are silver and gold.

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L. DEWAYNE RUTLEDGE, Primary Examiner

E. L. WEISE, Assistant Examiner

U.S. Cl. X.R.

204—37

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,698,880 Dated October 17, 1972

Inventor(s) Edwin Russell Koons, et. al.

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 62, "electrolytes" should read -- electrolyte -- . Column 4, line 6, "electroplated" should read -- electro-plated -- . Column 7, line 41, "heated" should read -- healed -- ; line 43, "39" should read -- 49 -- ; line 57, "heating" should read -- seating -- . Column 4, line 2, "or" should read -- of --.

Signed and sealed this 8th day of May 1973.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents

FORM PO-1050 (10-69)

USCOMM-DC 60376-P69
* U.S. GOVERNMENT PRINTING OFFICE : 1969 O—366-334.