

July 9, 1968

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3,391,447

SOLDERING NONMETALS TO METALS

Filed June 10, 1964

2 Sheets-Sheet 1

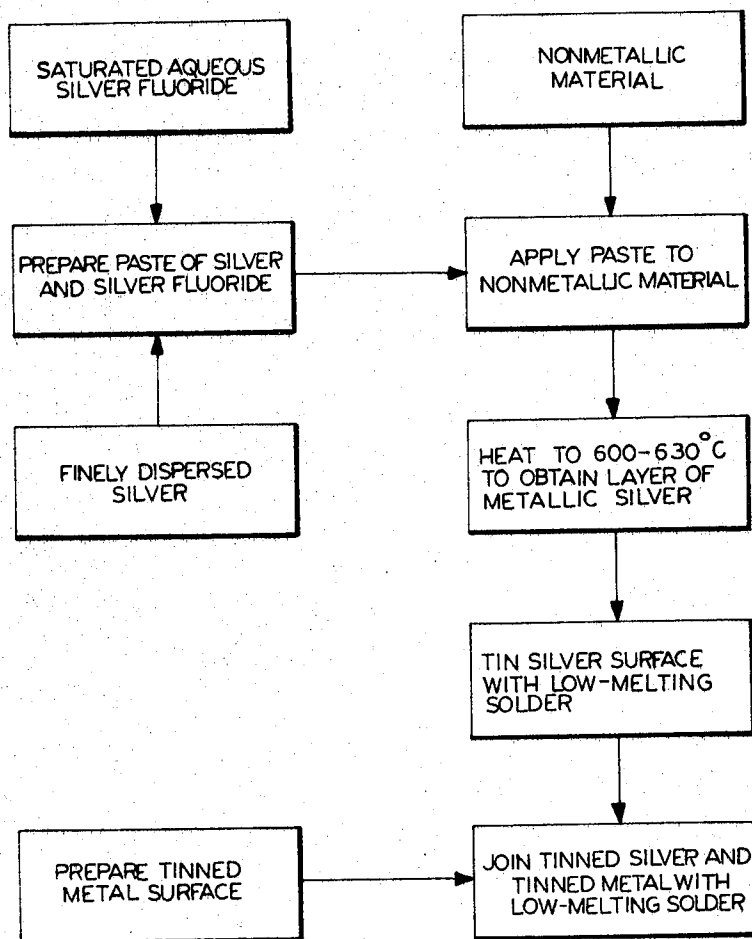


FIG.1

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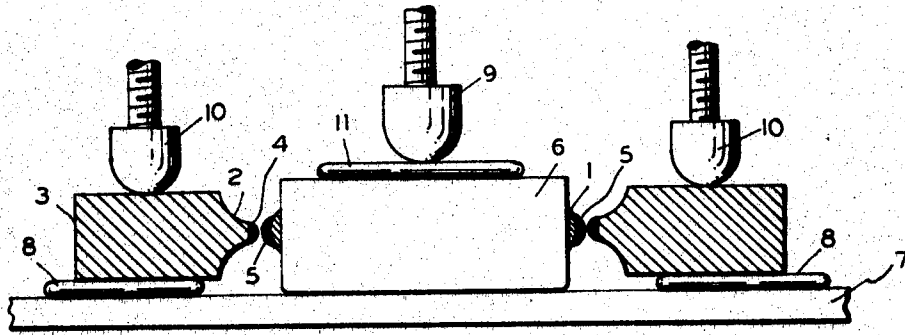


FIG. 2

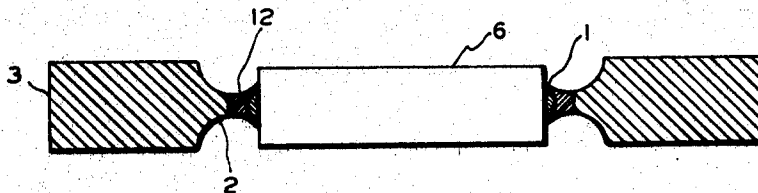


FIG. 3

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SOLDERING NONMETALS TO METALS

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Filed June 10, 1964, Ser. No. 374,215

7 Claims. (Cl. 29—473.1)

ABSTRACT OF THE DISCLOSURE

Silver that is finely dispersed and substantially water-wettable is combined with approximately saturated aqueous silver fluoride solution in such proportions to make a paste. The paste is applied to a nonmetallic material such as a water soluble salt, warmed and allowed to dry. While drying the silver and silver fluoride react to form silver subfluoride which, upon heating, decomposes to leave a layer of metallic silver bonded to the nonmetallic material thus providing a means by which the nonmetallic material can be soldered to a metal.

A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This invention relates to joining nonmetals to metals, and particularly relates to bonding a solderable metal layer to a nonmetallic material.

The term "nonmetal" is here used to describe physical characteristics of a substance and not its chemical composition. The majority of nonmetals hereinafter disclosed are, or contain, compounds such as metal salts and minerals.

While methods of bonding nonmetals to metals are available, none of the materials or processes currently available are satisfactory for soldering to a surface like that of sodium chloride. A solder bond is particularly desirable in the construction of certain devices, for example, radiation sample cells where the exposed seals of the cells should not dissolve in, soften with, or imbibe infrared samples, media or fluids used in cleaning the cell.

In currently available cells for use in instruments employing radiation to characterize the sample material the cell windows are typically mounted between two pieces of metal; a cylinder and retaining ring, or two rectangular frames etc.; and gaskets, spacers, or both are used to seal the cell and to cushion the window from the hard material applying pressure to hold the window in place.

Among the disadvantages of such cells are the possibility of contamination from the gasket material, the numerous rinsings necessary to insure removal of previous samples, and size and weight of some of the more sophisticated models.

An object of the present invention is to provide a novel means of bonding a metal to a nonmetallic material. Another object is to provide a layer of solderable metal on a nonmetallic material. A further object is to provide a layer of solderable metal such as silver on crystalline material used for windows in radiation sample cells as a means of mounting the windows in a metal frame. Other objects and a fuller understanding of the invention may be obtained from inspection of the specification and attached drawings in which:

FIG. 1 illustrates the steps of a process for affixing a solderable metal to a nonmetallic material and joining this solderable metal to a second solderable metal;

FIG. 2 is a generally cross-sectional view illustrating a means of securing a nonmetal such as a cell window in

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a metal support or frame prior to connecting the parts with solder, and;

FIG. 3 is a cross-sectional view illustrating one embodiment of a soldered cell window unit for use in a radiation sample cell.

I have discovered that an adhering layer of metallic silver can be applied to a crystalline material such as a metal halide or quartz, or to other nonmetallic material such as stone or fired clay, by a process comprising combining silver that is finely dispersed and substantially water-wettable with saturated aqueous silver fluoride solution to the consistency of a paste, applying a layer of paste to the nonmetallic material, and heating to at least about 600° C.

In an embodiment of the present invention in which the process is applied to the construction of radiation cells, the edge of the window is smoothed, water-wettable elemental silver, preferably colloiddally fine silver, is combined with approximately saturated aqueous silver fluoride solution, the amount of silver being sufficient to form a paste, the freshly prepared paste is applied to the edge of the window, the paste is warmed and allowed to dry, during which time the silver and silver fluoride react to form silver subfluoride, and the thus-treated window heated to a temperature sufficient to decompose the silver subfluoride and leave a layer of metallic silver bonded to the edge of the window. The silver layer and the opposing edge of the metal frame or supporting member designed to hold the window are individually "tinned." The window is positioned in the supporting element so that the tinned silver layer on the edge of the window and the tinned edge are adjacent to each other, then the tinned silver and tinned metal joined and sealed with a low melting solder.

Heat shock to sensitive materials was minimized by preheating them uniformly on a warming table to near the temperature of soldering before tinning, and to just above the melting temperature of solder for actions like readjustments of the spacing and flowing the solder uniformly in a filled spaced.

As described in the copending application entitled "Radiation Sample Cells," Ser. No. 374,216, filed June 10, 1964, the window may be of various compositions and may be of different sizes and shapes. The sides which will be the faces of the window are not finished, but the edge of the window and the lip of the frame to which the window is to be bonded are adapted to provide a slight clearance. The edge of the window is smoothed to provide a surface generally perpendicular to the plane of the faces.

Dispersion of silver in the aqueous silver fluoride solution and subsequent adherence of the silver plate to the window is assured by using water-wettable silver particles, preferably a colloiddally fine silver, substantially free of oil. Means of preparing the silver may vary; in practice formaldehyde was combined with ammoniacal silver nitrate solution to precipitate silver, the heavy sludge washed many times by centrifuging, and the precipitate stored wet. The explosive hazard of improperly precipitated silver should be recognized.

It is considered desirable to combine at least enough silver with silver fluoride to form silver subfluoride. An excess of silver is immaterial as the silver subfluoride is subsequently converted to silver. Since such an excess of silver is obtained when a paste is prepared by mixing the precipitated silver with saturated aqueous silver fluoride, no weighed amounts or specific volumes of components are required.

The paste must be applied to the window within a few minutes as the paste soon begins to stiffen and become difficult to apply.

As the paste stands, with application of mild heat, silver

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and the silver fluoride react in a matter of minutes to form silver subfluoride, which looks like gold.

When the paste is applied to a water soluble salt such as sodium chloride or potassium bromide the adhesion of the silver composition may be enhanced by a slight dissolving of the salt, but since the silver fluoride solution is a saturated solution originally, very little salt can be dissolved. Solution of a surface layer is not considered a requirement for obtaining an adhered coating of silver since the procedure has been successfully applied to materials such as quartz, arc-light carbon, clay brick, Alberine stone, and a silica-based common glass. Negative results were obtained with boron-containing glass and spectrographic carbon.

The metallic silver band is obtained by heating the silver subfluoride to temperatures of at least about 600° C., preferably to about 630° C. In practice the treated window was placed on an asbestos-board shelf in a muffle furnace, the temperature raised gradually over about an hour to prevent pore formation and heat shock, maintained at about 630° C. for a few minutes, and then cooled to room temperature at a similar rate. The window with freshly applied paste may be placed directly in a furnace at room temperature and the preliminary drying and reaction to form silver subfluoride will occur in the early stages of heating.

High spots of the silver layer (FIG. 2, 1) were smoothed with a small hand grinder operating at moderate speed and held so that the silver was always between the grinder and the salt window 6. The silver band was checked for adhesion and continuity, because any faults must be repaired now, not after adding solder. A silver band of the desired width and thickness is obtained by repeating the application of paste and heating step as often as necessary. One application may suffice, but for purposes as critical as preparation of these cells a second coat was usually applied.

Alternatively, the silver for the second coat was obtained by applying a layer of silver-filled organic paste, such as silver-filled epoxy paste (not the mixture including the catalyst for hardening), and heating to burn off the organic phase and leave a deposit of metallic silver.

Referring to FIG. 2, the lip face 2, preferably about 1 mm. wide, of the frame 3 was tinned (coated with solder) 4. Flux is removed and the solder cleaned to assure subsequent adherence with a low-melting solder. Typically a precoat 5 of low-melting solder, such as a commercial indium-tin solder softening at 116° C., was applied to the silver band. This is accomplished conveniently by clamping the window on a warming table, heating to a temperature a few degrees less than the softening point of the solder and a layer of solder flowed on to the silver with a soldering iron just hot enough to flow the solder quickly. Flux was used sparingly, and was cleaned off with acetone as soon as the window was cool.

The window 6 with the solder-precoated silver band was then assembled, as depicted in FIG. 2, with the frame 3 on a hard flat surface 7, such a flat glass, on a warming table (not shown), the frame adjusted with spacers 8 to align the surfaces to be joined, and the window and frame secured with clamps 9 and 10, respectively. The protective layer 11 is optional, but is desirable to secure the window 6 with minimum possibility of cracking. Soft, low melting solder was then flowed on between the precoated silver band and the soldered lip 4 to bond the aligned surfaces and the solder bridge was made neat by careful rubbing with a pointed instrument or soft rotary tool to provide the result shown 12 in FIG. 3. The soldered unit was removed from the clamps and the frame served as a matrix while one of the projecting window faces was ground and polished. The unit was then rewarmed until the solder was pliable, the window reset, the other face finished in the same manner, and, if necessary, the unit rewarmed for final adjustment of the window in the desired position to provide a unit as depicted in FIG. 3.

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Alternatively, only enough solder may be applied between the precoated silver 1-5 and the soldered lip face 4 to hold the window in the frame, the window faces finished, and the soldering completed.

Attempts to omit one or both tinning steps (solder pre-coats 4 and 5) was found impractical, because this sequence so often gave poor seals due to lack of adherence of solder or to disruption of the silver layer on the window.

The frame 3 is for illustrative purposes only. It may represent an actual body member of generally circular or rectangular shape or may be a window supporting element adapted for engaging or attaching to a body member.

The invention has been described in relation to working models in which the window supporting element and frame are typically made of a metal such as brass. It will be obvious, however, from the foregoing disclosure that the silver plating procedure is applicable to a variety of materials; that the frame could be nonmetallic, a ceramic casting, for example; in which instance the lip face 2 would be silver plated instead of tinned and the other steps of the process of the present invention would be substantially as described.

The silver plating procedure makes it possible to deposit an adhering layer of silver on a variety of materials that do not melt or become pliable at temperatures below about 650° C., so that the plated article can be soldered to either metals or nonmetals.

Heat applied at several steps of the process is a critical factor in obtaining a successfully bonded product. The silver subfluoride coating should be heated sufficiently to remove substantially all of the volatile materials and leave a layer of silver, but the silver should not reach a temperature high enough to allow it to coalesce into globules as can obtain at above about 680° C. The process fails if much silver is dissolved away from the nonmetallic window surface. The soldering step should be conducted carefully. Although a lead-tin solder may be applied by a deft, rapid soldering technique, the use of a low-melting solder is recommended.

Radiation sample cells in which the windows are bonded to a support according to the process of the present invention have many advantages. The cells have a relatively simple construction and can be designed for easy disassembly for cleaning or for resurfacing of window faces. Unlike organic plastic adhesives, the metallic bonds are completely insoluble in the cleaning fluids typically applied to salt optics. In tests with radiations transmitted adjacent to the bonding layer, no contamination of spectra could be detected over a range of 4000 to 650 cm^{-1} in cells constructed according to the process of the present invention.

It is to be understood that the invention is not limited to any specific embodiment herein illustrated and described, and accordingly all suitable modifications and equivalents may be restored to, falling within the scope of the invention as claimed.

I claim:

1. In a process of soldering a metal to a nonmetallic material which softens at a temperature higher than about 650° C., the improvement which comprises:

- (a) mixing a sufficient amount of finely dispersed, water-wettable silver with an approximately saturated aqueous silver fluoride to form a paste;
 - (b) applying a layer of said paste to the nonmetallic material;
 - (c) warming said layer for a time sufficient to cause the silver and silver fluoride to react and form a layer of silver subfluoride on the nonmetallic material; and
 - (d) then heating the layer of silver subfluoride on the nonmetallic material to a temperature of from 600 to 630° C., thereby causing the silver subfluoride to decompose and produce an adherent layer of metallic silver on the surface of the nonmetallic material.
2. The process of claim 1 in which the nonmetallic

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material is selected from the group consisting of sodium chloride, potassium bromide, quartz, arc-light carbon, A'berine stone, baked clay and silica-based common glass.

3. The process of claim 1 in which the nonmetallic material is sodium chloride.

4. The process of claim 1 in which the nonmetallic material is potassium bromide.

5. In the preparation of radiation cells having a cell window bonded by the edges thereof to a supporting surface of a metallic support member, said cell window being a nonmetallic material having a softening temperature higher than about 650° C., the improvement which comprises;

(a) mixing a sufficient amount of finely dispersed, water-wettable silver with an approximately saturated aqueous silver fluoride to form a paste;

(b) applying a layer of said paste to the nonmetallic material;

(c) warming said layer for a time sufficient to cause the silver and silver fluoride to react and form a layer of silver subfluoride on the nonmetallic material;

(d) then heating the layer of silver subfluoride on the nonmetallic material to a temperature of from 600 to 630° C., thereby causing the silver subfluoride to

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decompose and produce an adherent layer of metallic silver on the surface of the nonmetallic material;

(e) tinning both said silver layer on the nonmetallic window and the supporting surface of the metallic support member;

(f) aligning both tinned surfaces to abut one another; and

(g) joining said tinned surfaces with a low-melting solder.

6. The process of claim 5 in which the cell window consists substantially of sodium chloride and the metal of the support member is brass.

7. The process of claim 5 in which the cell window consists substantially of potassium bromide and the metal of the support member is brass.

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