United States Patent

LaChance

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[54] **METHOD OF HERMETICALLY SEALING SILICON TO A LOW EXPANSION ALLOY**

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[52] **U.S. Cl.** .....**287/189.365,** 29/473.1, 29/504, 29/590, 317/234 M

[51] **Int. Cl.** .....**C03c** , F16b 11/00

[58] **Field of Search**.....29/473.1, 589, 590, 504; 317/234 M; 287/189.365

[56] **References Cited**

UNITED STATES PATENTS

2,163,408	6/1939	Pulfrich	287/189.365
2,776,472	1/1957	Mesick, Jr.	287/189.365 X
2,974,761	3/1961	Patrichi	287/189.365
3,243,072	3/1966	Day	287/189.365 X
3,006,069	10/1961	Rhoades et al.	29/473.1
3,025,439	3/1962	Anderson	29/590 X
3,418,422	12/1968	Brodham	317/234 UX
3,462,349	8/1969	Gorgengi	317/234 UX
3,609,471	9/1971	Scace	317/234 UX

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[57] **ABSTRACT**

A surface of silicon is hermetically sealed to a surface of Kovar by coating the surface of silicon with several thin layers of silver, coating the surface of Kovar with a layer of copper, applying a tin-base sealing alloy between the two coated surfaces and then heating the tin-base sealing alloy so as to cause it to fuse to the two coated surfaces.

5 Claims, 2 Drawing Figures

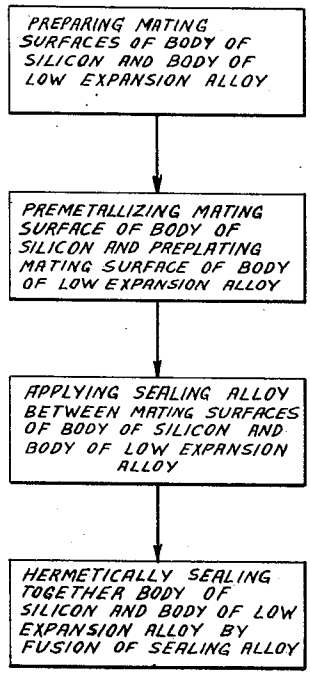


Fig. 1.

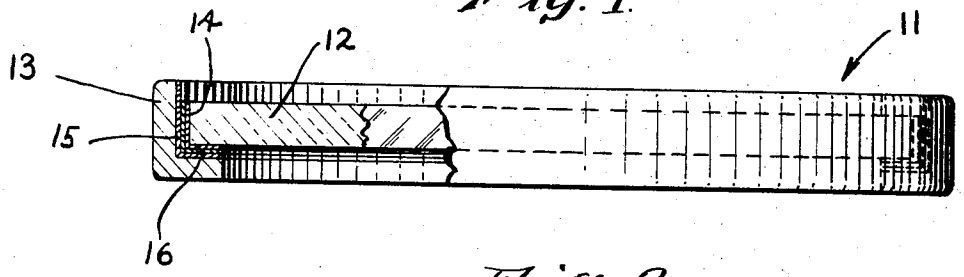
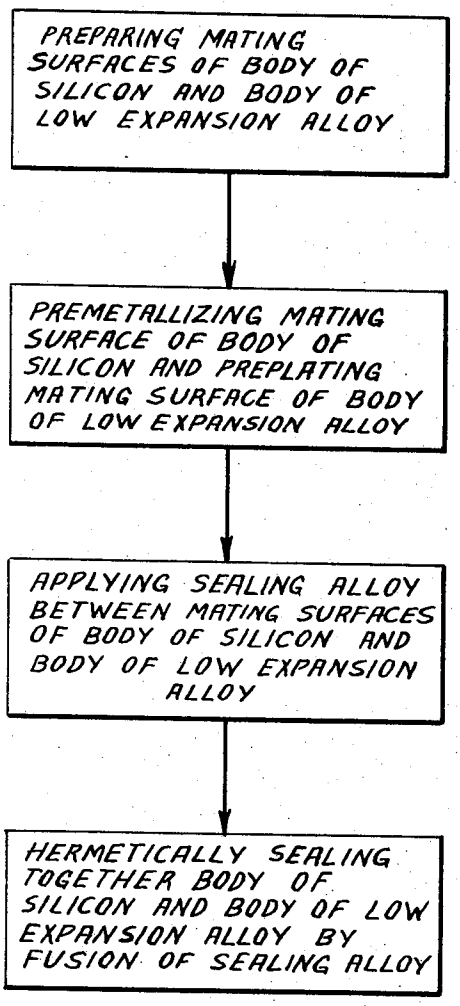


Fig. 2.



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METHOD OF HERMETICALLY SEALING SILICON TO A LOW EXPANSION ALLOY

This invention relates to joining silicon to a low expansion alloy. More particularly, this invention relates to hermetically sealing a surface of silicon to a surface of a low expansion alloy.

Silicon has a wide variety of uses and applications. Two of the more important uses for silicon are as a semiconductor material and as an optical material. Frequently, it is either desirable or necessary to hermetically seal an article or device that is made of or coated with silicon to an article or device that is made of a low expansion alloy. As is well known in the art, it is not simple to join metalloid material to metallic material. Some of the known techniques for joining, but not necessarily hermetically sealing, silicon to a low expansion alloy are disclosed in U.S. Pat. No. 2,856,681, U.S. Pat. No. 3,031,747 and U.S. Pat. No. 3,298,093.

It is an object of this invention to provide a new and improved method for joining silicon to a low expansion alloy.

It is another object of this invention to provide a new and improved method for hermetically sealing silicon to a low expansion alloy.

It is still another object of this invention to provide a new and improved method for hermetically sealing a surface of silicon on one body to a surface of a low expansion alloy on another body.

The above and other objects are achieved by joining a surface of silicon to a surface of low expansion alloy according to the method of this invention. Briefly, the method involves preparing the surface of silicon and the surface of the low expansion alloy, then coating the surface of the body of silicon with silver and the surface of the low expansion alloy with copper or other metal which will improve the brazing characteristic of the low expansion alloy, then applying a tin-base sealing alloy between the two coated surfaces and then heating the tin-base sealing alloy so as to cause it to fuse to the two coated surfaces.

Many features and advantages of the invention will become apparent from the following detailed description when taken in conjunction with the drawings wherein:

FIG. 1 is a view partly in elevation and partly in section of a silicon optical window hermetically sealed to a Kovar support member using the method of this invention; and

FIG. 2 is a flow chart illustrating the basic steps of the method of this invention.

Referring now to FIG. 1, there is shown an optical assembly **11**. The assembly **11** includes a silicon window **12** mounted in a Kovar support member **13**. Kovar, as is well known in the art, is the trade name of a low expansion alloy made of a mixture of iron, nickel and cobalt. The percentages of iron, nickel and cobalt in the mixture are nominally 55 percent iron, 28 percent nickel and 17 percent cobalt. The surface of the silicon window **12** in contact with the Kovar support member **13** is provided with a coating of silver **14** and the surface of the Kovar support member **13** in contact with the silicon window **12** is provided with a coating of copper **15**. A quantity of a tin-silver sealing alloy **16** is disposed between the two coated surfaces and is fused

to the two coated surfaces so as to hermetically seal the silicon window **12** to the Kovar support member **13**.

The method of assembling the silicon window **12** and the Kovar support member **13** so that the silicon window **12** is hermetically sealed to the Kovar support member **13** is as follows.

First, the surfaces of the silicon window **12** and the Kovar support member **13** which are to be connected to each other are properly prepared. The surface of the silicon window **12** is prepared by first grinding it with a number **9** or finer diamond grit or similar material so as to make it smooth, then swab etching it with a solution of 46 percent HNO_{30} — 27 percent HF — 27 percent Acetic Acid or other similar solution to remove silicon oxide and any other contaminants, then rinsing it first in deionized water or a similar liquid and then in a reagent grade ethanol or other similar solution to remove residue material. The surface of the Kovar support member **13** is prepared by first cleaning it ultrasonically in hot Oakite solution or other similar solution to remove surface contaminants, then rinsing it first in deionized water or a similar liquid and then in ethanol or other similar liquid to remove the Oakite.

Once the two surfaces are properly prepared, the surface of the silicon window **12** is premetallized and the surface of the Kovar support member **13** is preplated. The surface of the silicon window **12** is premetallized in the following manner. First a coating of silver **14** made up of about three to five separate layers of a silver-base slurry or equivalent mixture is applied by a brush or other similar means. Several layers rather than a single layer of silver are necessary to build a coating that is both non-porous and sufficiently thick to prevent it from being dissolved during the subsequent fusing process. After each layer is applied the surface is heated to about 120° C for about 3 to 4 hours and fired at around 600° C for about one hour in air ambient. Then one or more coatings of a 50 percent Pt — 50 percent Au or similar metallizing suspension may be applied by brush or other similar means over the multiple layer silver coating. This is done to improve the "wettability" of the surface. The surface of the Kovar support member **13** is preplated by coating it by electroplating or other similar means with a layer of copper **15** approximately 0.001 inches thick.

The next step involves applying a tin-base sealing alloy **16** such as a mixture of 95 percent tin and 5 percent silver by weight between the two coated surfaces. Before the tin-base sealing alloy **16** is applied between the two coated surfaces it is degreased in acetone or other similar liquid, rinsed in ethanol or other similar liquid, air dried, dipped into a flux such as Kester Electronic Flux No. 196 and then air dried.

Then the assembly **11** is heated so as to cause the sealing alloy **16** to fuse to the two coated surfaces. This involves heating the assembly **16** in an argon or other protective atmosphere to about 242° C or above the melting point of the sealing alloy and then, with minimum dwell time, causing the assembly to cool rapidly.

It is to be understood that the method described above with respect to hermetically sealing a silicon window and a Kovar support member is only by way of example and that the invention is applicable to items made of silicon other than windows, items made of

Kovar other than support members, items made of low expansion alloys other than Kovar and bodies of materials not made of silicon but coated with silicon and not merely bodies made out of silicon.

The basic steps of the method of this invention are shown in the flow chart in FIG. 2.

What is claimed is:

1. A method for forming a hermetic seal between the mating surfaces of a silicon member and a metal alloy comprising the steps of:

a. preparing the mating surfaces of the silicon member and said metal alloy.

b. forming a plurality of metal layers on the surface of said silicon member, each of said layers consisting essentially of silver,

c. coating the metal alloy mating surface with a layer of copper,

d. interposing a sealing alloy between the layered mating surfaces,

e. heating the sealing alloy to a temperature at least equal to the melting point of the sealing alloy, and

f. cooling the contact area of the mating surfaces and the sealing alloy to effect fusing of the sealing alloy to the silver and copper layers.

2. The method as defined in claim 1 wherein said metal alloy comprises Kovar.

3. The method as defined in claim 2 wherein said sealing alloy is heated in an argon atmosphere to a temperature of about 242° C.

4. A hermetically sealed assembly comprising a silicon member, a plurality of metal layers overlying one surface of said silicon member, said metal layers each consisting essentially of silver, a metal alloy member having a copper coating, and a sealing alloy sandwiched between the layered surface of said silicon member and said coated metal alloy member, said sealing alloy being fused to said copper and silver layers.

5. The assembly as defined in claim 4 wherein said metal alloy comprises Kovar.

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