

UNITED STATES PATENT OFFICE.

HENRY MILLER, OF NEWARK, NEW JERSEY, ASSIGNOR TO HANOVIA CHEMICAL AND MANUFACTURING CO., OF NEWARK, NEW JERSEY, A CORPORATION OF NEW JERSEY.

PROCESS OF SEALING MOLYBDENUM OR SIMILAR REFRACTORY METALS AND FUSED QUARTZ.

No Drawing.

Application filed January 8, 1926. Serial No. 80,066.

This invention relates to the process of fusing metallic molybdenum and its allied metals of the chemical periodic table, such as for instance, chromium, tungsten, uranium, vanadium and tantalum and fused quartz whereby an impermeable joint is obtained, completely proof against the passage of air, gases or liquids.

It also relates to a method for sealing metal through the walls of quartz or silica vessels for the primary purpose of conducting electric currents from the inside to the outside or vice versa. As many vessels of this type are evacuated, it is essential that the method should produce a gas tight union between the quartz and the metal.

For this purpose it has been found that metal molybdenum and its allied metals of the chemical periodic table are well suited under certain conditions. The most important of these conditions is that the metal shall be so treated with certain salts of the alkali metals to insure a satisfactory bonding between the metal and the quartz, and to produce a gas tight or vacuum tight seal.

It is the object of this invention to provide a process applicable to apparatus used for medical and electrical purposes, in which the joints may be subjected to severe test by a vacuum, air compression, various gases and the like and resist transmission of the same.

Molybdenum as well as its allied metals, are difficult to fuse, but they possess properties that render them extremely valuable in certain apparatus, notwithstanding the difficulty hitherto encountered in forming substantially integral connections with articles made of quartz and forming part of the apparatus.

The metal, which throughout is understood to mean molybdenum, and its allied metals of the chemical periodic table, such as for instance chromium, tungsten, uranium, vanadium and tantalum, may be in any preferred physical shape, as blocks, wires, ribbons, etc., is fitted to engage the article made of fused quartz or silica, irrespective of its shape or contour.

After such a fitting has been accomplished the metal is heated in an oxidizing flame to a bright red, in the presence of a nitrate,

nitrite, peroxide, or a chlorate of an alkali metal or mixture thereof; or

The metal is heated in an oxidizing flame to a bright red, and after removal from the flame, plunged immediately into an aqueous solution of a nitrate, nitrite, peroxide, or chlorate of an alkali metal, or a mixed solution of these salts; or

The metal is heated in an oxidizing flame to a bright red, and, after removal from the flame, plunged immediately into a bath of fused nitrate, nitrite, peroxide, or chlorate of an alkali metal or mixture of these, the fused salt or salts being maintained at a temperature at which no decomposition of the chemical compounds occurs; or

The metal is heated to a bright red in an oxidizing flame and then treated with a finely powdered nitrate, nitrite, peroxide, or chlorate of an alkali metal, or a mixture of these, suspended in an air current or a mixture of neutral and reducing gases and oxygen.

These steps may be repeated in accordance with the size or section of the metal treated which must be determined by the operator's experience. Thereafter the metal may be again placed for a short time in an oxidizing flame and eventually immersed in distilled water or a solution of hydrogen peroxide. Thereafter the metal is brought into contact with a reducing flame.

The metal is then brought into engagement with the fused quartz article, both being suitably supported, and rotated in the presence of a slightly reducing flame or in the vortex between several of such flames.

When the fused quartz article indicates a plastic flow under such treatment pressure is applied to the article in the direction of its axis, causing it to flow and fill any interstices between it and the metal.

The article, if in the form of a sleeve, may be evacuated by any means to create a vacuum and pressed into the metal.

By my improved process herein described the molybdenum metal and its allied metals of the chemical periodic table have undergone a chemical, molecular and crystalline change thus chemically making them ductile as well as giving them chemical reactivity with silica at higher temperatures.

It will be apparent that the foregoing disclosure is susceptible of minor changes without departing from the spirit and scope of the appended claims.

5 Having thus described my invention, what I claim as new and desire to secure by Letters Patent, is:—

1. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz consisting in
10 treating the metal previously fitted to the quartz, with certain salts of the alkali metals, applying heat to the metal in connection with the quartz until the latter becomes plastic and thereafter stressing or
15 pressing the quartz sufficiently to cause the latter to flow and blend with the metal.

2. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz articles to form
20 a gas and vacuum tight joint, consisting of treating the metal by heat in the presence of alkali metal salts, said metal having been shaped to conform with the quartz article,
25 placing the metal in juxtaposition with the article to which it is to be united, applying heat uniformly to the metal and article until the article becomes plastic, and thereafter pressing the article and metal into
30 forcible contact while the article is in a plastic state.

3. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz articles to form
35 a gas and vacuum tight joint, consisting in subjecting the metal repeatedly to an oxidizing flame to a bright red heat in the presence of alkali metal salts, applying the metal to a fused quartz article, heating the
40 metal and article together to a point caus-

ing the article to become plastic and causing a union between the heated metal and plastic fused quartz.

4. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz articles to form
45 a gas and vacuum tight joint, consisting in treating the metal with certain salts of the alkali metals in an oxidizing flame, placing the metal in contact with a reducing flame
50 and thereafter fusing and manipulating the quartz article into intimate bonding contact with the metal.

5. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz articles to form
55 a gas and vacuum tight joint, consisting in coating the metal with a compound resulting from treating said metal with certain salts of the alkali metals in an oxidizing
60 flame, and subsequently treating the metal in a reducing flame, and thereafter fusing and manipulating the quartz article into intimate bonding contact with the metal.

6. The process of uniting molybdenum metal or allied metals of the chemical periodic table and fused quartz articles to form
65 a gas and vacuum tight joint, consisting in treating the metal with certain salts of the alkali metals in an oxidizing flame, plunging the metal into an aqueous solution of
70 certain salts of the alkali metals, placing the metal in contact with a reducing flame and thereafter fusing and manipulating the quartz article into intimate bonding contact
75 with the metal.

This specification signed and witnessed this 31st day of December, 1925.

HENRY MILLER.