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METAL GLASS SEAL

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6 Claims. (Cl. 49—81)

My invention relates to methods of making a joint between a ferrous metal and an electrical insulating substance.

One object of my invention is to provide a joint between the metal and an electrical insulating substance, which is free from bubbles that result when heat is applied to the joint between the two substances.

Other objects of my invention will become apparent on reading the following specification.

The presence of bubbles in a ceramic material at the joint between it and a ferrous metal and electrical insulating substance cause trouble and materially weaken the joint between the two. By ferrous I means to include iron or cobalt or nickel or any combination of these or their alloys. The presence of these bubbles is not always apparent to one who is not highly skilled in the art. The electrical insulating substances commonly used in such joints are, of course, the glass used as a casing of electric discharge devices, and also ceramic materials applied to metals as well as enamel coatings. The presence of bubbles in the glass portion about the seal materially weakens the structure of the container, especially where the container has a high vacuum therein. The presence of bubbles also spoils the appearance as well as the insulating properties of enamel and ceramic materials.

These electrical insulating substances are generally applied to the metal with the application of heat sufficient to fuse the insulating material but not the metal in order to make a firm joint or seal between the two substances. In the case of glass, where a glass is sealed to the metal conductor or electrode, such as described in my copending application for glass metal seals Serial No. 376,291, filed July 5, 1929, a hole is melted in the glass wall and the metal is inserted therein and then the molten glass is squeezed around the metal and fused thoroughly in contact therewith. Other methods are used by glass blowers to join a tubular metal shape to a tubular glass shape. I have discovered that the bubbles resulting from these processes are apparently caused by carbon in the metal uniting with the oxide formed on the surface of the metal during the heating step of uniting the two substances, and the release of this gas into the insulating material causes bubbles to form therein.

It is an object of my invention to prevent the formation of these bubbles by removing the carbon from the surface of the metal.

In my copending application previously mentioned, namely Serial No. 376,291, I have de-

scribed the sealing of certain nickel-iron-cobalt alloys into specified boro-silicate and lead glasses. In particular, I have found that an alloy comprising about 30% nickel, 16% cobalt, 0.2% manganese and the remainder iron seal satisfactory through certain boro-silicate glass. In general, the range of materials is from 15% to 32% nickel and 12% to 45% cobalt, with less than 1% manganese and the remainder iron. This is only one specific instance but the invention is also applicable to any iron base alloy of proper expansion coefficient for sealing into glass.

Small amounts of carbon, however, cannot be avoided in these iron base alloys without elaborate precautions. Consequently, it is more convenient to remove the carbon from the alloy than to prevent its introduction therein. Carbon can be removed from the molten alloy by passing hydrogen through the molten metal. This treatment is preferable when the finished piece is bulky. When, however, the metal is finished to a small cross-section, the carbon can be removed conveniently from the solid metal. The treatment required is heating at an appropriate temperature in hydrogen atmosphere until the carbon is substantially removed or reduced in the surface. The temperature is between 700 and 1200° C. and is preferably 950° C. for a sufficient time to reduce the carbon in the surface of the metal. The carbon content in the surface should be less than 0.02% and it is preferable that this low content of carbon extend to a depth of 0.02 inch of the surface. The time of exposure of the metal to this hydrogen atmosphere of 950° C. should be well in excess of the time occupied in making the seal, that is at least one hour. Five hours are sufficient for small sections, but for joints requiring very careful and firm construction, I prefer to hold the temperature at substantially 950° C. for 16 hours and even up to 24 hours. I also prefer the use of hydrogen which is moist rather than the dry hydrogen, as the moist hydrogen atmosphere appears to expedite the carbon removal. After this annealing process, the articles are cooled either in hydrogen or air and may be further machined or fabricated provided that a sufficiently long annealing time has been given. The surface of the metal can be polished before contact with the glass or other electrical insulating substance, and, in fact, it may be desirable to so polish the metal.

The glass or other ceramic material and the metal are then heated so that the glass will become molten where it is to be fused to the metal. This heating of the metal produces an oxide sur-

face thereon and, if the carbon had not been removed by the application of my invention thereto, the oxide would unite with the carbon to produce gas that would cause bubbles in the glass adjacent to the joint with the metal. This oxide surface on the metal is desirable in order to have the glass firmly adhere to the metal. The presence of the carbon prevents the use of the adhering qualities of this oxide on the surface of the metal. Accordingly my invention not only prevents the undesirable bubbles but also ensures the possibility of making use of the desirable oxide coating on the metal.

While I have in the foregoing described certain particular embodiments of my invention, it will be understood that these are for the purposes of illustration only and that the principles may be otherwise utilized as will be readily apparent to those skilled in the art. I accordingly desire that the following claims shall be accorded the broadest interpretation of which their terms are susceptible in view of the terms which are imposed by the prior art.

I claim as my invention:

1. The method of making a bubble-free joint between carbon-bearing metals and alloys and an electrical insulating substance which comprises first heating the metal in the presence of hydrogen at a temperature and for a time sufficient to remove substantially all of the carbon from an appreciable depth of the surface, and then joining the said insulating substance to said decarbonized surface by so heating in an oxygen-bearing atmosphere as to produce a surface oxide to which the insulating substance intimately fuses.

2. The method of making a bubble-free joint between an oxidized surface of carbon-bearing metals and alloys and an electrical insulating substance which comprises first heating the unoxidized metal in the presence of hydrogen at a temperature and for a time sufficient to remove substantially all of the carbon from the surface, and then forming an oxide on said surface and intimately

fusing the insulating substance thereto by heating in the presence of oxygen.

3. The method of making a bubble-free joint between carbon-bearing ferrous metals and alloys and an electrical insulating substance which comprises decarbonizing the surface of said metal before joining by heating in a gaseous atmosphere containing hydrogen at a temperature between 800° and 1100° C. for at least 1 hour and thereafter applying said electrical insulating substance to said metal in an oxygen-bearing atmosphere with the application of heat.

4. The method of making a bubble-free joint between carbon-bearing metals and alloys and an electrical insulating substance which comprises first decarbonizing the surface of said metal to less than 0.02% carbon and then applying the electrical insulating substance to said surface in an oxygen-bearing atmosphere with the application of heat.

5. The method of making a bubble-free joint between carbon-bearing metals and alloys and an electrical insulating substance which comprises first decarbonizing the surface of said metal to less than 0.02% carbon for a depth of at least 0.02 inch and then applying the electrical insulating substance to said surface in an oxygen-bearing atmosphere with the application of heat.

6. The method of making a vacuum-tight bubble-free joint between carbon-bearing metals and alloys and an electrical insulating substance which comprises alloying from 15% to 32% nickel, 12% to 45% cobalt, less than 1% manganese and the remainder iron with small amounts of carbon, then heating the alloy in the presence of hydrogen at a temperature and for a time sufficient to remove substantially all of the carbon from an appreciable depth of the surface, and then joining the said insulating substance to said decarbonized surface by so heating in an oxygen-bearing atmosphere as to produce a surface oxide to which the insulating substance intimately fuses.

HOWARD SCOTT.