REMOVAL OF CONDUCTIVE COATING FROM DIMENSIONALLY STABLE ELECTRODES
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ABSTRACT OF THE DISCLOSURE
A molten salt bath consisting of an alkali metal hydroxide and an alkali metal salt of an oxidizing agent is used to completely remove the conductive coating from a dimensionally stable electrode. Another object of the present invention is to provide a method for removing the electrically conductive coating from a dimensionally stable electrode in such a manner that the valuable constituents of said coating may be recovered.

A further object of the present invention is to provide a method which rapidly removes the conductive coating from a dimensionally stable electrode with a minimum of damage to the electrode substrate.

These and further objects and advantages of the present invention will become apparent to those skilled in the art from the specification and claims which follow.

It has now been found that the conductive coating may be rapidly and efficiently removed from a dimensionally stable electrode, without substantial damage to the electrode structure itself, by contacting the dimensionally stable electrode with a molten salt bath consisting of a mixture of from 1 to 15 parts by weight of an alkali metal hydroxide and one part by weight of an alkali metal salt of an oxidizing agent. Upon removal from contact with the molten salt bath, coating and rinsing any adherent fused salt from the electrode structure, said structure is suitable for etching and application of a new electrically conductive coating.

DESCRIPTION OF THE PREFERRED EMBODIMENTS
By the use of the term "alkali metal hydroxides" in the specification and claims it is intended to refer to the hydroxides of sodium, potassium and lithium or mixtures thereof, notably sodium and potassium hydroxide and preferably potassium hydroxide. As is mentioned hereinabove this alkali metal hydroxide will be present within the range of 1 to 15 parts of alkali metal hydroxide per 1 part of oxidizing agent, all parts by weight. At this time the preferred range is from 3:1 to 10:1. A typical and especially preferred bath consists of 5 parts of potassium hydroxide and 1 part of an alkali metal salt of an oxidizing agent.

It has been found by experience that at least equal quantities of hydroxide and oxidizing agent are required to obtain satisfactory results. Preferably a large excess of hydroxide to oxidizing agent is used. When approaching the 1:1 ratio, from an excess of hydroxide, it is found that the amount of time required to effect complete stripping of the coating increases greatly, all other factors being equal. Conversely, when approaching the upper limits of the 15:1 ratio, no sacrifice in stripping rate is noticed, although replenishment of the oxidizing agent is more frequently required to maintain the minimum concentration.

By the use of the phrase "alkali metal salt of an oxidizing agent" it is intended to refer to sodium, potassium and lithium salts of such agents. Again, sodium and potassium salts are preferred, with potassium being especially preferred at this time. The oxidizing portion, or anion, of the salt is selected from the group consisting of nitrates, chlorates, peroxides, permanganates and perchlorates. Examples of suitable oxidizing agents are potassium nitrate, sodium nitrate, sodium chlorate, potassium perchlorate, potassium permanganate, sodium peroxide, and the like. Obviously, mixtures of oxidizing agents may be used in many instances. Especially preferred at this time are sodium and potassium nitrates.

An advantage of the present invention, which requires that at least a 1:1 ratio of ingredients be used, and preferably a large excess of hydroxide, is that the cost of the hydroxide is far less than that of the oxidizing agent.
That is, a bath requiring a reverse ratio of ingredients would be significantly more expensive to establish and maintain.

The term "dimensionally stable electrodes" is used throughout the specification and claims and is intended to refer not only to titanium or tantalum but also to coatings carrying an electrically conductive coating. In addition to "pure" titanium and tantalum, alloys of these metals are also useful, as are other conductive metals, such as copper and aluminum, clad or coated with titanium, tantalum or alloys thereof. Other valve metals not adversely affected by the bath may also be used, e.g., cast iron, and may be used in some applications. These electrode structures are generally in the form of a solid sheet, a perforated or slotted sheet, or an expanded mesh configuration, although the shape of the electrode has no bearing upon the practice of the present invention.

It has been found, somewhat surprisingly, that the identity of the electrically conductive coating is likewise not critical to the practice of the present invention, that is, a variety of electrically conductive coatings may be removed by this technique. Thus, noble metal coatings such as platinum, palladium, iridium and ruthenium metals and alloys thereof are rapidly and readily removed according to the invention. Likewise, the oxide, or "ceramic," coatings are also effectively and efficiently removed. These latter coatings include, in addition to the oxides of the noble metals themselves, mixtures of oxides of noble oxides of valve metals (e.g., titanium, tantalum, aluminum); mixtures of oxides of noble metals, valve metals and other metals; as well as mixtures of certain of the foregoing oxides with noble metals in the metallic form. Examples of such coatings include: ruthenium oxide; ruthenium oxide-titanium oxide; ruthenium oxide-titanium oxide-aluminum oxide: ruthenium oxide-titanium oxide-iridium; ruthenium oxide-titanium oxide-tantalum oxide, and others.

In order to effectivly and completely remove the electrically conductive coating from the titanium or tantalum base, it is only necessary to contact the coated structure with the molten salt bath for a period of time dependent upon the thickness of the coating to be removed and the temperature of the salt bath. A wide range of temperatures, e.g., 500° C. to 600° C. are effective in this invention so long as the temperature chosen is sufficient to insure maintenance of a molten state but not so high as to result in ignition of the substrate metal. Generally it has been found that the most effective stripping operation can be accomplished at a temperature within the range of 450° C. to 600° C. Using a salt bath within this temperature range to remove the coating from an average dimensionally stable electrode will require less than 15 minutes, generally less than 5 minutes and often as low as 1 minute.

A significant advantage of the present invention lies in the fact that, despite the speed of the stripping operation and the fact that a variety of coatings may be removed with equal ease and effectiveness, substantially no damage is incurred by the titanium or tantalum structure itself. Thus, experience to date has shown that less than 5 percent, and generally less than 2 percent, by weight of the metal is lost from the structure itself in each case.

After the stripping operation is complete, the electrode is removed from the molten salt bath and, after cooling, the fused salt adhering to the electrode structure may be readily removed by rinsing in water to dissolve the salt. Obviously, the electrically conductive coating remaining in the molten salt bath and at periodic intervals the components of the coating may be recovered in a variety of ways, provided of course that they are of such a nature as to be worth the cost of a recovery operation. An especially preferred manner of providing the salt bath in condition for economical recovery of the coating constituents involves the concentration of these constituents to a maximum level. This may be readily accomplished by adding fresh amounts of the hydroxide-oxidizing salt mixture, in the proper proportions, to the molten bath as it is depleted by us and by drag-out. This serves to effect the concentration of the recoverable components while still maintaining an effective and efficient stripping operation. When the "saturation point" of the bath is reached, with respect to its ability to accept more stripped coating without a sacrifice in stripping efficiency, recovery may be attempted. For example, the components of the coating, now in a highly concentrated state, may be recovered by dissolving the fused salt melt and precipitating and filtering the materials therefrom. Alternately, recovery can be by electrolytic means.

A problem of major concern when attempting to re-coat electrode structures after stripping of the old coating by prior methods, has been the difficulty of obtaining any degree of adherence of the subsequently applied coating to the "cleaned" electrode structure. According to the present invention however, not only is the degree of adherence obtained at least equal to that of a new electrode, but the degree of etching required prior to the application of the coating is significantly reduced.

In order that those skilled in the art may more readily understand the present invention and certain preferred embodiments by which it may be carried into effect, the following specific examples are afforded.

EXAMPLE 1

An anode comprising an expanded titanium mesh substrate having deposited thereon an electrically conductive coating of ruthenium dioxide-titanium dioxide, is first cleaned of extraneous material such as cell putty, salt and the like and thereafter heated to dryness. The structure is then immersed in a clear salt bath containing 5 parts by weight of KOH and 1 part by weight of KNO₃ and maintained at a temperature of 450° C. (±20° C.). A nickel tank is used to prevent corrosion. After 5 minutes in the molten salt bath, the anode structure is carefully removed, the still molten salt is permitted to drain back into the melt, and the anode is allowed to cool, at which time any adhering fused salt is removed by rinsing in water. Upon examination, the surface of the titanium is seen to be bright, smooth and uniform.

The thus-cleaned structure is then etched prior to the application of a fresh coating of electrically conductive material and it is found that only a 10 minute etch in boiling, azetroic, hydrochloric acid is required, compared with the normal 45-60 minute etch used when initially preparing the titanium for coating.

After recoating the titanium structure with a RuO₂-TiO₂ coating mixture, it is found that an electrode having voltage characteristics and a wear rate at least comparable to the original electrode is obtained.

EXAMPLE 2

An expanded titanium mesh anode having a platinum metal coating thereon is cleaned and dried as in Example 1. In this instance the molten salt bath is a mixture of 5 parts of sodium hydroxide and 1 part of sodium nitrate maintained at a temperature of about 350° C. After 5 minutes, the structure is removed from the melt, cooled and rinsed with water as before, at which time it is noticed that the surface of the mesh is perfectly clean with the exception of a few scattered white spots. The weight loss on the substrate during stripping is determined to be 2.1 percent. During the subsequent hydrochloric acid etching operation the white spots disappear and the cleaned, etched structure is totally acceptable for recoating with an electrically conductive material.

Although the invention has been described in reference to certain specific embodiments thereof, it is not to be so limited since changes and alterations may be made therein which are within the full and intended scope of the appended claims.
I claim:

1. A method for removing the electrically conductive coating from a dimensionally stable titanium electrode structure which method comprises contacting said structure with a molten salt bath consisting of a mixture of from 1 to 15 parts by weight of an alkali metal hydroxide and 1 part by weight of an alkali metal salt of an oxidizing agent, for a period of time sufficient to effect removal of said coating.

2. A method as in claim 1 wherein the alkali metal hydroxide is selected from the group consisting of potassium hydroxide and sodium hydroxide and the alkali metal salt of the oxidizing agent is selected from the group consisting of sodium and potassium salts of nitrates, chlorates, perchlorates, permanganates and peroxides.

3. A method as in claim 1 wherein the alkali metal hydroxide is potassium hydroxide and the alkali metal salt of the oxidizing agent is potassium nitrate.

4. A method as in claim 1 wherein the ratio of alkali metal hydroxide to alkali metal salt of an oxidizing agent is within the range from 3:1 to 10:1.

5. A method as in claim 1 wherein the ratio of alkali metal hydroxide to alkali metal salt of an oxidizing agent is 5:1.

6. A method as in claim 1 wherein the molten salt bath is maintained at a temperature within the range of 400° to 450° C.

7. A method for removing the electrically conductive coating from a dimensionally stable titanium electrode structure by stripping said coating from said electrode structure in a molten salt bath containing from 1 to 15 parts by weight of an alkali metal hydroxide and 1 part by weight of an alkali metal salt of an oxidizing agent and recovering the thus-stripped coating, which method includes the step of increasing the concentration of the stripped coating in the molten salt bath by periodic additions of the hydroxide and oxidizing agent as the bath becomes depleted in these components.

8. A method for removing the electrically conductive coating from a dimensionally stable titanium electrode structure by stripping said coating from said electrode structure in a molten salt bath containing from 1 to 15 parts by weight of an alkali metal hydroxide and 1 part by weight of an alkali metal salt of an oxidizing agent, which method includes the step of recovering the stripped coating from the salt bath.

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