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METHOD OF PREPARING SILVER BASE ALLOYS

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ABSTRACT OF THE DISCLOSURE

An improved method for preparing alloys for use in forming electrical contact members consisting essentially of cadmium oxide, silver and a reactive metal such as sodium involves the steps of first heating a mixture of cadmium metal and silver to a temperature within the range 1760° F. to 2100° F. to completely melt the mixture while maintaining a flux cover consisting of boric acid and rock salt on top of the mixture, adding the reactive metal carried, for example, within the end of a hollow carbon stirring rod through the flux cover and stirring the reactive metal into the melt for approximately 10 seconds to six minutes, and retaining the resulting melt under the flux cover for at least approximately fifteen minutes. This latter step permits the reactive metal to diffuse throughout the melt to form a homogeneous mixture. The cadmium is subsequently internally oxidized to cadmium oxide in the usual manner. By preparing the alloy in this manner, a substantially homogeneous alloy is produced without excessive loss of cadmium and/or the reactive metal through vaporization and the percentage of reactive metal retained in the melt and the final alloy may thereby be more precisely controlled.

This invention lies in the field of alloys and more particularly relates to improved methods of preparing silver-cadmium oxide alloys for use in forming electrical contact members.

U.S. Pats. Nos. 2,932,595, 3,472,654, and 3,477,845, and copending and coassigned application Ser. No. 607,034, filed Jan. 3, 1967, describe the preparation of silver-cadmium oxide alloys containing additives which regulate the grain size of silver-cadmium oxide particles and impart improved properties to the alloys for their intended use in making electrical contact members therefrom. As described in the above patent and applications, the additives may be reactive metals such as cobalt, potassium, sodium, ruthenium, magnesium and beryllium. It has been the practice to prepare such alloys by melting cadmium metal, silver and the reactive metal additive, with or without a flux cover, at a temperature within the range 1800° F.–2100° F. and then pouring the melt to form an ingot. The ingot was then further processed as described to internally oxidize the cadmium to cadmium oxide and to form electrical contact members. While this method of preparation has been generally satisfactory for the preparation of alloys containing reactive metal additives such as cobalt and magnesium, for example, it has proven generally unsatisfactory in the case of highly reactive metal additives such as sodium, potassium and lithium. Being highly reactive with the atmosphere and having lower melting points and boiling points than the other reactive metals mentioned above, vaporization causes excessive loss of these metals, and it has not been possible with the methods employed heretofore to control with any degree of precision the amount of reactive metal retained in the melt and in the final alloy. Also, alloys of cadmium oxide, silver and sodium, potassium or lithium prepared as described have not always exhibited the desired degree of homogeneity.

Among the objects of the invention, therefore, may be mentioned the provision of improved methods for preparing silver base alloys containing sodium, potassium or lithium as a reactive metal additive; the provision of such methods which permit control of the amount of reactive metal additive incorporated in the alloys and avoids excessive vaporization of the reactive metal and of cadmium during perparation of the alloys; and the provision of methods of this type which may be readily carried out to produce alloys of substantially homogeneous composition. Other objects will be in part apparent and in part pointed out hereinafter.

The present invention is thus directed to the method of preparing an alloy consisting essentially of cadmium oxide, silver and a reactive metal such as sodium, potassium or lithium by first heating a mixture of cadmium metal and silver to a temperature between approximately 1760° F. and 2100° F. to effect substantially complete melting thereof while maintaining a flux cover on top of said mixture, adding the reactive metal in at least a partially enclosed form through said flux cover and stirring the reactive metal into the melted mixture, retaining the resulting melt under the flux cover at said temperature for a period of a least approximately fifteen minutes to diffuse the reactive metal throughout the melt to form a substantially homogeneous mixture, and subsequently internally oxidizing the cadmium to cadmium oxide.

In accordance with the present invention, it has now been found that by carrying out the preparation of silver base alloys in the above manner, excessive vaporization and loss of the reactive metal is avoided and an alloy of substantially homogeneous composition is obtained. Thus, I have found that the shortcomings of prior art methods of preparing silver base alloys with highly reactive metal additives therein may be obviated by first effecting substantially complete melting of a cadmium metal-silver mixture while maintaining a flux cover on top of the mixture, adding the highly reactive metal in at least a partially enclosed form to the melted mixture through the flux cover and then retaining the resulting melt under the flux cover for at least fifteen minutes, all steps being conducted at temperatures within the range 1760° F. to 2100° F. The resulting alloy may then be cast into ingots, the cadmium internally oxidized to cadmium oxide and electrical contact members formed in the usual manner.

In carrying out the novel method of the invention, the desired amount of cadmium metal is first loaded into the bottom of a crucible, such as a silicon carbide crucible, and covered with the required quantity of silver. A flux cover is then added over the cadmium-silver mixture and the resulting combination is heated to a temperature between approximately 1760° F. and 2100° F. for a sufficient period to effect complete melting of the mixture. During heating, additional amounts of flux are added as needed to maintain a complete cover over the mixture. Various flux materials known to the art may be employed, and the use of boric acid or a mixture of boric acid and rock salt has been found to be especially advantageous. More particularly, mixtures of crystalline boric acid and rock salt in the range of ratios from approximately 2:1 to 5:1 are preferred for use as the flux cover. The rock salt functions to lower the viscosity of the boric acid and thereby causes the flux to more readily flow over and cover the melting metal combination.

When the cadmium metal-silver mixture has become completely molten under the flux cover, the highly reactive metal additive, such as sodium, potassium or lithium, is prepared for addition to the melt. In accordance with the invention, the reactive metal is added to the melt in a partially or wholly enclosed form so that excessive reaction between the metal and the atmos-

phere does not occur. Preferably, the reactive metal is first chopped into small pieces which are then stuffed into the end of a hollow stirring rod of carbon or other inert material. In this partially enclosed form, the reactive metal carried by the rod is then inserted through the flux cover and into the bottom of the crucible, and the melted mixture stirred for a period of from about 10 seconds to about six minutes to allow the reactive metal to work its way out of the rod and into the melt. Less preferably, small pieces of the reactive metal may be enclosed or wrapped within thin films of silver and introduced through the flux cover and into the melt with stirring, as for example through the use of forceps composed of an inert material. In either case, the reactive metal is partially or wholly protected from the atmosphere and is introduced into the melt through the flux cover and thereafter stirred into the melt.

After the addition of the reactive metal to the melt as described, the melt containing the reactive metal additive therein is retained under the flux cover at a temperature within the range previously stated (i.e., 1760° F. to 2100° F.) for at least fifteen minutes and preferably for a period of from fifteen minutes to two hours. It has been found that retention of the melt under the flux cover for this period permits the reactive metal to diffuse throughout the melt to form a substantially homogeneous mixture. In general, a one hour period of retention of the melt under the flux cover is sufficient to effect homogenization thereof at the preferred operating temperature of 1800° F–1900° F.

As stated, the method of the invention may be carried out at a temperature between approximately, 1760° F. and 2100° F. For best results, it is preferred, however, to employ a temperature between approximately 1800° F. and 1900° F. Temperatures within this range are sufficient to effect complete melting of the cadmium metal-silver mixture while at the same time avoiding excessive loss of cadmium through vaporiaztion. Similrly, after addition of the reactive metal to the molten mixture as described, it is preferred to retain the resulting melt at a temperature between approximately 1800° F. and 1900° F. to diffuse the reactive metal throughout the melt. If higher temperatures within the range 1760° F.–2100° F. are employed, then the period of retention is shortened to avoid excessive loss of cadmium and/or of the reactive metal through vaporization. Thus, for example, at temperatures within the preferred range of 1800° F–1900° F., one hour is the preferred period of retention whereas at higher temperatures such as 2050° F,. a fifteen minute period of retention would be adequate.

Following the homogenizing period, the melt is cast into molds and allowed to cool and set. After cooling, the molds are opened and the ingots removed. For convenience, the ingots are quenched in water to permit easier handling. The impurities, if any, are present in the gate and butt ends of each ingot and these are therefore cut off and discarded. The ingots are then further processed in the conventional manner as described, for example, in U.S. Pat. No. 2,932,595, including internal oxidation of the cadmium to cadmium oxide and forming of the alloys into electrical contact members.

In general, the novel methods of the invention may be used in the preparation of alloys consisting essentially of approximately 1.0%–20.0% by weight of cadmium oxide, approximately 0.001%–0.25% by weight of sodium, potassium or lithium as the reactive metal additive, and the balance silver. Preferably, the alloys may contain between approximately 0.0025% and 0.0035% by weight (25 to 35 parts per million) of the reactive metal to impart the desired properties thereto.

By effecting substantially complete melting of the cadium metal-silver mixture prior to the addition of the reactive metal through the flux cover as described and by retaining the melt, with the reactive metal therein, under

the flux cover for a sufficient period to permit diffusion of the reactive metal throughout the melt, the percentage of reactive metal retained may be more accurately controlled and an alloy of substantially homogeneous composition obtained.

The following examples further illustrate the invention.

EXAMPLE 1

An alloy consisting essentially of 10% by weight of cadmium oxide, 0.0025%–0.0035% by weight of sodium and the balance silver (approximately 90% by weight) was prepared as follows.

The bottom of a silicon carbide crucible was loaded with cadmium metal and the cadmium covered with the necessary quantity of fine silver grain and bars. A flux covered consisting of crystalline boric acid and rock salt (ratio of 4:1) was then added on top of the cadmium-silver mixture. The charge was then heated to a temperature of 1900° F. to effect complete melting of the cadmium-silver mixture, additional amounts of flux being added as necessary to maintain a complete flux cover during heating of the mixture. Small pieces of sodium were stuffed into the end of a hollow carbon stirring rod and the end of the rod was then inserted into the melted cadmium-silver mixture through the flux cover. The amount of sodium added was 0.05% by weight of the cadmium-silver melt. The rod was inserted into the bottom of the crucible and the sodium leaked out of the end of the rod and was stirred into the melt for a period of approximately three minutes. The rod was then withdrawn from the melt, and the melt maintained under the flux cover without stirring at a temperature of 1900° F. (+20° F., −50° F.) for a period of one hour. After the one hour period, the melt was cast into molds at a temperature of 1900° F. under natural gas flame, After cooling, ingots of the resulting alloy were removed from the molds and washed. The gate and butt ends of each ingot were cut off and discarded, and the ingots were thereafter processed in the conventional manner to internally oxidize the cadmium to cadmium oxide and to form electrical contact members from the alloy.

EXAMPLE 2

Example 1 was repeated to prepare an alloy consisting essentially of 15% by weight of cadmium oxide, 0.0025%–0.0035% by weight of sodium and the balance silver (approximately 85%). The same procedure was employed except that the cadmium-silver mixture was melted at a temperature of 1800° F. and, after addition of the sodium, the resulting melt was maintained at a temperature of 1800° F. (+20° F., −40° F.) for a period of one hour.

EXAMPLE 3

Example 1 was repeated except that potassium was employed as the reactive metal in lieu of sodium.

In view of the above, it will be seen that the several objects of the invention are achieved and other advantages results attained.

As various changes could be made in the above methods without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. The method of preparing a substantially homogeneous alloy consisting essentially of cadmium oxide, silver and a reactive metal selected from the group consisting of sodium, potassium and lithium which comprises the steps of heating a mixture of cadmium metal and silver to a temperature of between approximately 1760° F. and 2100° F. to effect substantially complete melting thereof while maintaining a flux cover on top of said mixture, adding said reactive metal in a least a partially enclosed

form through said flux cover and stirring the reactive metal into the melted mixture, retaining the resulting melt under said flux cover at said temperature for a period of at least fifteen minutes to diffuse said reactive metal throughout the melt to form a substantially homogeneous mixture, and subsequently internally oxidizing the cadmium to cadmium oxide.

2. The method according to claim 1 wherein said flux cover consists of a mixture of boric acid and rock salt.

3. The method according to claim 1 wherein said temperature is between approximately 1800° F. and 1900° F. and the resulting melt is retained under said flux cover for a period of approximately one hour.

4. The method according to claim 1 wherein said reactive metal is added through said flux cover by being carried in the end of a hollow stirring rod of inert material.

5. The method according to claim 4 wherein the resulting alloy contains between approximately 0.001% and 0.25 by weight of said reactive metal.

6. The method of preparing a substantially homogeneous alloy consisting essentially of approximately 1.0%–20.0% by weight of cadmium oxide, approximately 0.001%–0.25% by weight of a reactive metal selected from the group consisting of sodium, potassium and lithium, the balance silver, which comprises the steps of heating a mixture of cadmium metal and silver to a temperature of between approximately 1800° F. and 1900° F. to effect substantially complete melting thereof, maintaining a flux cover on top of said mixture during melting thereof, adding said reactive metal through said flux cover and

stirring the reactive metal into the melted mixture, the reactive metal being carried in the hollow end of a stirring rod of inert material, retaining the resulting melt under said flux cover at said temperature for a period of from approximately fifteen minutes to approximately two hours to diffuse said reactive metal throughout the melt to form a substantially homogeneous mixture, casting the melt into ingots and thereafter internally oxidizing the cadmium to cadmium oxide.

7. The method according to claim 6 wherein said flux cover consists of a mixture of boric acid and rock salt.

8. The method according to claim 7 wherein the ratio of boric to rock salt in said flux cover is between 2:1 and 5:1.

9. The method according to claim 6 wherein said reactive metal is sodium.

10. The method according to claim 9 wherein the resulting alloy contains between approximately 0.0025% and 0.0035% by weight of sodium.

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