

UNITED STATES PATENT OFFICE

2,431,479

RECTIFIER FOR CHLORIDE SALT BATHS

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1

This invention relates to metallurgy and more especially to a rectifier for a chloride bath, such as, for example, a bath comprising substantially sodium chloride 40% to 60%; potassium chloride 30% to 50%; and sodium fluoride 2% to 7%, preferably substantially sodium chloride 55%, potassium chloride 40% and sodium fluoride 5%. Another type of chloride bath may comprise sodium chloride 20% to 40%; potassium chloride 10% to 30%; calcium chloride 5% to 20%; and barium chloride 20% to 40%; preferably, sodium chloride 35%, potassium chloride 20%, calcium chloride 15%, and barium chloride 30%. A further example of a chloride bath to which the present invention applies is the No. 1 bath disclosed in Holden and Solakian Patent 2,237,434, patented April 8, 1941. This bath is sometimes referred to as a "pre-heat bath."

The chloride baths above referred to are intended to be used as neutral baths which are for the single purpose of heating the work to predetermined temperatures without the bath otherwise affecting the ferrous work parts being heated. These baths may be operated at temperatures from 1300° F. to 2000° F., except that the bath No. 1 of Patent No. 2,237,434 will fume and vaporize at 2000° F.

When the chloride baths specified are in use, particularly at the higher temperatures, the chlorides break down into alkali metal or alkaline earth metal oxides that are alkaline in character and the baths therefore become alkaline. An alkaline bath will decarburize ferrous metal parts and if the bath becomes very alkaline the work being heated therein may be decarburized to such an extent as to be ruined. It, therefore, becomes necessary to replenish the bath, which is expensive and wasteful, or to introduce therein chemicals that will reduce the alkalinity of the bath without affecting the fluidity of the bath or otherwise destroying the desirable characteristics of the neutral bath.

I have found a satisfactory rectifier for such chloride baths may comprise boric acid (H_3BO_3) 30% to 70%; titanium dioxide (TiO_2), 10% to 40%; silicon dioxide (silica sand (SiO_2)) 2% to 15%; and potassium chloride (KCl) 2% to 15%; preferably substantially boric acid 50%; titanium dioxide 30%; silica sand 10%; and potassium chloride 10%. In this rectifier the boric acid and the silicon dioxide, being on the acid side of reactions, tend to reduce the alkalinity of the bath, and the boric acid tends to neutralize the broken-down salts and to clean up metallic oxides on the work or in the bath, while the titanium

2

dioxide combines with the broken-down salts to form a sludge of inert stable titanates which tend to fall to the bottom of the pot in which the salts are melted.

Another satisfactory rectifier for these chloride baths comprises the introduction of silicon carbide (SiC) in conjunction with the boric acid (H_3BO_3). In this variation the boric acid acts to clean up any metallic oxides, as specified above and the silicon carbide acts to reduce the oxides of the salts to neutral stable compounds such as sodium silicate, calcium silicate, barium silicate, etc. The grain size of the silicon carbide is of some importance in that where the bath is operating at from 1300° F. to 1550° F. the grain size should be small, preferably such as will pass through a screen from 150 to 200 meshes per lineal inch, while with higher temperatures above 1550° F. to 2000° F. the grain size preferably is such as will pass through a 70 to 100 mesh screen. The silicon carbide-boric acid rectifier may vary in proportions as for example, silicon carbide 50% to 90%; and boric acid 10% to 50%. For lower temperature chloride salt baths operating at from 1300° F. to 1550° F. the preferred proportions are 50% to 60% silicon carbide, and 40% to 50% boric acid; for higher temperatures from 1550° to 2000° F. the proportions preferably are increased as to the silicon carbide and diminished as to the boric acid, as for example, 60% to 90% silicon carbide and 40% to 10% boric acid.

These salt baths, herein referred to as capable of rectification by the rectifiers specified, are to be used in ceramic pots, preferably of the silica alumina type, known in the art as the "Mulite" type.

Heat treating neutral salt baths of the character herein specified should be tested about every thirty-six hours of continuous operation, and if found to be alkaline, the rectifier should be added to the bath. This test may be made by dissolving one gram of the bath in one hundred cubic centimeters of water, filtering the solution, adding four or five drops of methyl red indicator solution, and titrating the solution with 0.1% normal hydrochloric acid until the solution turns red. The number of cubic centimeters of acid solution added to obtain the change to red is a direct indication of the alkalinity of the bath and indicates the amount of rectifier required to bring the bath back to neutral. Each cubic centimeter of acid used indicating four ounces of rectifier required per hundred pounds of the salt bath.

3

I claim as my invention:

1. A rectifier for normally neutral salt bath formed of chlorides adapted to decompose into alkaline compounds during the operation of the bath; said rectifier consisting essentially of silicon carbide 50% to 90% and boric acid 50% to 10%, said silicon carbide being adapted to react with said alkaline compounds to form neutral silicates, said rectifier reacting with said bath to reduce the alkalinity of said bath.

2. A rectifier for normally neutral salt baths formed of chlorides adapted to decompose into alkaline compounds at temperatures between 1300° F. and 1550° F.; said rectifier consisting essentially of boric acid 40% to 50% and silicon carbide 60% to 50%, said rectifier reacting with said bath to reduce the alkalinity of said bath.

3. A rectifier for normally neutral salt baths formed of chlorides and adapted to decompose into alkaline compounds at temperatures between 1550° F. and 2000° F.; said rectifier consisting essentially of boric acid 10% to 40% and silicon carbide 90% to 60%, said rectifier reacting with said bath to reduce the alkalintiy of said bath.

4. A rectifier for a normally neutral chloride salt bath for heat treatment of ferrous articles, the bath tending to become alkaline during operation; said rectifier consisting essentially of silicon carbide in amount sufficient to form corresponding alkali metal and alkaline earth metal silicates with the breakdown products of the

4

chlorides and boric acid in amount sufficient to clean the metal surfaces of metallic oxides by reacting with metallic oxides on the work and in the bath.

5. The method of neutralizing an alkaline salt bath for heat treatment of ferrous articles, said method comprising quantitatively determining the degree of alkalinity of said bath and introducing a rectifier in amounts regulated according to the determination, said rectifier consisting essentially of silicon carbide in amount sufficient to form corresponding alkali metal and alkaline earth metal silicates with the breakdown products of the chlorides and boric acid in amount sufficient to clean the metal surfaces of metallic oxides by reacting with metallic oxides on the work and in the bath.

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