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SALT BATH FOR HEAT TREATMENT AND RECTIFIER THEREFOR

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The present invention relates broadly to metallurgy and more especially to a salt bath for heat treatment of ferrous metals parts, and rectifier therefor.

The present invention is more particularly an improvement upon the salt bath designated as #2 bath in Holden and Solakian Patent #2,237,434, patented April 8, 1941. The #2 bath of the said patent comprised silica sand, calcium fluoride, calcium oxide, sodium carbonate and sodium fluoride. This bath absorbed iron oxide from the work which tended to make the bath alkaline and which is undesirable. The bath of the present invention does not absorb iron oxide and is inert to the ferrous metal parts being treated, as well as to the refractory or ceramic pots in which the bath is contained. There is a slight tendency of the #2 bath of Patent #2,237,434 to react with ceramic pots.

The ceramic pot preferably used with the present salt bath is a pot composed of aluminum oxide and aluminum silicate combinations well-known in the art.

The present invention is a neutral high temperature bath adapted to operate in a working range of 1750° F. to 2350° F. and is adaptable more especially for treating high speed steels, such as high carbon-high chrome types of steels as well as the various high speed steels specially referred to in the said Patent #2,237,434.

The preferred chemicals for the present bath are barium chloride, titanium dioxide, and a small percentage of silicon dioxide. The percentage ranges may be from 90% to 99% barium chloride; ½% to 5% titanium dioxide; and ¼% to 5% silicon dioxide. The preferred percentages are 98% barium chloride, 1½% titanium dioxide, and ½% silicon dioxide.

A satisfactory bath may comprise 95% to 99% barium chloride and 1% to 5% titanium dioxide, the preferred bath being 98% barium chloride and 2% titanium dioxide. The omission of the silicon dioxide does not seriously impair the operation of the bath, although it does remove a slight safety factor to the life of the bath.

Another satisfactory formula for this salt bath may comprise barium chloride, barium fluoride, calcium fluoride (alkaline earth metal halides), chromium fluoride, and titanium dioxide, plus a small amount of silicon dioxide or sand in proportions comprising 70% to 90% barium chloride; 3% to 7% barium fluoride; 3% to 8% calcium fluoride; 2% to 7% chromium fluoride; and ½% to 3% titanium dioxide, plus ½% to 5% silicon dioxide (which may be omitted), and the preferred bath of these ingredients comprises 84½% barium chloride; 5% barium fluoride; 5% calcium fluoride; 4% chromium fluoride; and 1% titanium dioxide, plus ½% silicon dioxide. The silicon dioxide may be omitted, in which case the barium chloride is increased to 85%.

In the operation of the baths above specifically set forth, the barium chloride when heated to the temperature specified, namely, 1750° F. to 2350° F., tends to break down into barium oxide plus chlorine which goes off as a gas. The presence of barium oxide in the bath tends to make the bath alkaline which is undesirable. The titanium dioxide reacts and combines with the barium oxide to form barium titanate. The formula being $BaCl_2 \rightarrow BaO + Cl$; and $BaO + TiO_2 \rightarrow BaTiO_3$. The barium titanate is neutral and at the lower temperatures the barium titanate will form a sludge and settle at the bottom of the pot. Thus it will be seen that in the operation of the bath the barium chloride is slowly decomposed and the titanium dioxide is slowly combined into a different inert chemical, namely, barium titanate.

During the working of the bath the percentages of the ingredients get out of balance, and the bath, instead of remaining neutral, becomes alkaline and gradually increases in alkalinity. An alkaline bath, at high temperatures, will decarburize steel parts, and this is very undesirable. In order to avoid this and to bring the bath back to neutral balance, the method of introducing into the bath the chemicals of the bath in abnormal percentage relationship, has been successfully employed. We have designated such a mixture as a "rectifier." The rectifier in the present case preferably comprises barium chloride, titanium dioxide and silicon dioxide, the range of percentages being 4% to 25% barium chloride, 60% to 95% titanium dioxide, 1% to 15% silicon dioxide, the preferred percentage for the rectifier being 20% barium chloride, 70% titanium dioxide, and 10% silicon dioxide.

A satisfactory rectifier may be obtained by omitting the silicon dioxide, under which conditions the percentages may be 5% to 25% barium chloride, 75% to 95% titanium dioxide, the preferred percentages being 20% barium chloride, and 80% titanium dioxide.

With reference to the bath including as ingredients the fluorides specified, namely, barium, calcium, and chromium fluoride, the above rectifiers are satisfactory to maintain neutrality of the bath for the reason that the fluorides decompose extremely slowly and may be replenished by adding to the bath the ingredients of the orig-

inal bath which is necessary to replace the material which is taken from the pot by the work as it is lifted out of the pot after heat treatment. However, in order to maintain the fluidity of the bath as well as neutrality, it may be desirable to add small percentages of the calcium fluoride and chromium fluoride to the rectifier. Both of these fluorides tend to lower the melting point of the bath and to overcome alkalinity. Where calcium fluoride and chromium fluoride are added, the rectifier becomes titanium dioxide, calcium fluoride and chromium fluoride in percentages of 60% to 90% titanium dioxide, 5% to 25% calcium fluoride, 2% to 15% chromium fluoride, the preferred percentages being 85% titanium dioxide, 10% calcium fluoride, 5% chromium fluoride.

The condition of the bath for neutrality can be checked by taking a 1 gram sample, dissolving the same in 100 cubic centimeters of water, filtering and then titrating the filtrate with .1% normal hydrochloric acid, using methyl red as an indicator; the pink color being the end point. If the number of cubic centimeters of hydrochloric acid required to bring the end point exceeds two cubic centimeters, then ¼% of 1% of rectifier needs to be added. In other words, if the pot contains 100 lbs. of salt bath, then 4 ounces of the rectifier should be added to bring the bath back to normal neutrality.

Rectification of the bath is not frequently required and may not be necessary more often than at intervals of thirty-six hours with a continuously operated bath.

The salt baths herein specified as comprising the present invention are normally neutral in character, operate at temperatures between 1750° F. and 2350° F., and comprise materials which are soluble in water to a much greater degree than the #2 bath specified in Patent 2,237,434. Such material of the present baths as adhere to the work is quickly washed away in #3A or #3B baths specified in said Patent #2,237,434.

What we claim is:

1. A substantially neutral salt bath for heat treatment of ferrous metals at temperatures from 1750° F. to 2350° F., consisting essentially of an alkaline earth metal chloride salt comprising the major portion of said bath and tending to become an alkaline earth metal oxide at the temperatures specified; and titanium dioxide to convert the alkaline earth metal oxide formed into a neutral alkaline earth metal titanate.

2. A salt bath for heat treatment of ferrous metals at temperatures from 1750° F. to 2350° F., consisting essentially of barium chloride as a major portion of said bath which slowly decomposes into chlorine gas and barium oxide at the temperatures specified; titanium dioxide to form the said oxide into barium titanate and ¼% to 5% of silicon dioxide.

3. A salt bath for heat treatment of ferrous metal parts at temperatures between 1750° F. to 2350° F., and comprising 95% to 99% barium chloride and 1% to 5% titanium dioxide.

4. A salt bath for heat treatment of ferrous metal parts at temperatures between 1750° F. to 2350° F., and comprising substantially 98% barium chloride and 2% titanium dioxide.

5. A salt bath for heat treatment of ferrous metal parts at temperatures between 1750° F. to 2350° F., comprising 90% to 99% barium chloride; ½% to 5% titanium dioxide; and ¼% to 5% silicon dioxide.

6. A salt bath for heat treatment of ferrous metals at temperatures of from 1750° F. to 2350° F., comprising substantially 98% barium chloride; 1½% titanium dioxide, and ½% silicon dioxide.

7. A rectifier for a heat treatment salt bath, in which a chloride salt tends to decompose into an alkaline oxide when heated, said chloride salt comprising more than 70% of the bath, and a quantity of titanium dioxide to convert said alkaline oxide into an inert neutral composition; said rectifier comprising 60% to 95% said titanium dioxide and 4% to 25% said chloride salt.

8. A rectifier for a heat treatment chloride salt bath, in which a chloride bath decomposes into an alkaline oxide when heated to the decomposing temperature of said salt, said salt comprising more than 70% of said bath, and a quantity of titanium dioxide to convert said oxide into an inert neutral composition; said rectifier comprising said chloride salt and more than 60% of said titanium dioxide.

9. A rectifier for a substantially neutral salt bath for heat treatment of ferrous metals, said salt bath comprising a chloride salt tending when heated to decomposing temperatures to break down into chlorine gas and an alkaline oxide, said chloride salt comprising more than 70% of said bath; said rectifier comprising titanium dioxide adapted to convert said alkaline oxide into an inert neutral composition, said titanium dioxide comprising more than 60% of said rectifier.

10. A rectifier for a salt bath for heat treatment of ferrous metals at temperatures from 1750° F. to 2350° F., said salt bath comprising 95% to 99% barium chloride and 1% to 5% titanium dioxide; said rectifier comprising 5% to 25% barium chloride and 75% to 95% titanium dioxide.

11. A rectifier for a salt bath for heat treatment of ferrous metals at temperatures from 1750° F. to 2350° F., said salt bath comprising substantially 98% barium chloride and 2% titanium dioxide; said rectifier comprising substantially 20% barium chloride and 80% titanium dioxide.

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