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HEAT TREATMENT BATH

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The present invention relates to metallurgy and more especially to a fused bath, for heat treatment of material known in the art as "high speed steel."

The invention is characterized by submitting the high speed steel to a series of fused baths of graduated predetermined temperatures.

A further characteristic of the present invention is that the highest temperature bath of the series, which is of a ceramic nature, shall be contained with a non-contaminating container also of a ceramic character.

There are several types of high speed steels and the following are used as illustrative examples. One type of tungsten high speed steel is a steel containing iron alloyed with materials in substantially the following proportions:

	Per cent
Tungsten	18
Chromium	4
Vanadium	1
Carbon	0.70

Another type of high speed steel which may be referred herein to as molybdenum type A comprises an alloy of iron with

	Per cent
Chromium	4
Tungsten	2
Vanadium	1
Molybdenum	8
Carbon	0.70

There is another molybdenum type high speed steel which may be herein designated as molybdenum type B in which iron is alloyed with

	Per cent
Molybdenum	10
Chromium	4
Vanadium	1
Carbon	0.75

These high speed steels have critical temperatures at which the carbides go into solid solution in such a manner as to improve the cutting qualities of tools which are heat treated above this critical point. The critical high point of the tungsten type of steel varies from 2250° F. to 2325° F. and the molybdenum types vary as to their critical high temperature point from 2150° F. to 2200° F. These steels also have two low criticals through which rapid quenching is important to obtain maximum austenitic microstructure. These lower critical points of the high speed steel are around 1450° and 700° F. The micro-structural change takes place below the latter critical point and the steel becomes hard.

From the foregoing, it will appear that it is necessary to retain the high speed steel articles at the higher critical temperature, around 2300°, just long enough for all parts of such articles to reach this temperature, then drop and hold the temperature below the low critical point, around 1400°, for a sufficient length of time to permit the work to fully attain this temperature. From this temperature, the steel may be cooled directly to the lower critical, around 700°, or cooled without interruption slowly in the air to normal air temperature. Since the high critical point is within a relatively narrow temperature range, it becomes important to be able to maintain the entire tool of high speed steel within this critical range without either over heating or under heating any part of the work.

The present invention carries out the desired conditions by using a plurality of independent fused salt baths to impart the desired heat to steel masses immersed in the baths. The process may be performed by the use of three baths but preferably four baths are used. The first bath is for the purpose of preheating or elevating the temperature of the high speed steels to a preliminary temperature from which temperature the steel may be advanced in the second bath to a higher temperature. The subsequent baths are quenching baths in which the temperature is reduced and whereby the water-insoluble material of the preceding bath is removed from the work and replaced by water soluble material so that in the finality the work may be washed in water or other suitable solvent to completely remove all traces of the heat treating fused salt baths from the work.

The bath No. 1 may comprise various materials capable of being fused and maintained at the desired temperature without fuming and wherein traces of this preliminary bath No. 1 will be removed when the work goes into the succeeding baths. Preferably, the preheating No. 1 bath shall be materials which are in a fused fluid state at from 1500° F. to 1700° F., and salts adaptable for this bath are alkali metal chlorides, preferably potassium chloride. In the event it is desired to preheat at a slightly lower range, 70% potassium chloride may be fused with 30% sodium chloride whereby a bath is produced which is fused and liquid at a temperature in the neighborhood of 1400° F. and still may be heated without undue fuming up to 1500° F. or even to 1700° F. The work is retained in this No. 1 bath until it has been heated uniformly to the temperature of the bath. A desirable characteristic of the No. 1 bath is that the salts thereof will vaporize at the temperature of a succeeding No. 2 bath. Since the succeeding No. 2 bath runs above 2000° F., it is desirable that the salts used in the preheating No. 1 bath shall volatilize at a temperature of 2000° F. or above, and this is the case with the chlorides used in the No. 1 bath.

The No. 2 bath which is the high temperature

bath preferably is comprised largely of silicates and is reasonably fluid at temperatures from between 2000° to 2400° F. The preferred No. 2 bath comprises the following ingredients: silica sand, calcium fluoride, calcium oxide, sodium carbonate, sodium fluoride. Other earth metal fluorides and oxides may be used, and other alkali metal carbonates and fluorides may also be used, instead of the specific materials designated. The specific percentages by weight which are preferred are as follows:

	Per cent
Silica sand	20 to 40
Calcium fluoride	20 to 40
Calcium oxide	10 to 25
Sodium carbonate	10 to 25
Sodium fluoride	5 to 15

As the above ingredients are brought up to operating temperatures, the following reactions, inter alia, occur:

$$SiO_2+CaO \rightarrow CaSiO_3$$
$$SiO_2+Na_2CO_3 \rightarrow Na_2SiO_3+CO_2$$

The proportion of silica to calcium oxide and sodium carbonate should not be substantially less than that required to carry out the above equations to avoid extensive erosion on the ceramic pots used to prevent contamination of the bath with iron. Preferably, the proportion of silica is maintained substantially in amounts required to combine with the lime and carbonate, as indicated above. As the carbonate acts like an oxide, reference to the silica to metallic oxide ratio is intended to include not only the calcium oxide but also the sodium oxide of the carbonate ingredient of the melt.

Where higher temperatures are desired, the sodium carbonate may be replaced by barium carbonate. The sodium and calcium fluorides in the bath reduce the melting point of the bath and increase its fluidity, so that at high temperatures the bath is quite fluid to facilitate immersions of the work in the bath and subsequent draining off of the salts when the work is removed.

In general, the proportion of silicate should exceed that of fluoride, and that of fluoride should exceed that of any other ingredient.

Since the liquid temperature for this No. 2 bath is above 2000° F., it will be apparent that any of the material of the No. 1 bath which sticks to the work will be volatilized in the temperatures at which the No. 2 bath operates so that all traces of the No. 1 bath completely disappear in the No. 2 bath. The composition of the No. 2 is essentially a refractory or ceramic material which when cold is not soluble either in water or in acid solutions. It, therefore, is very desirable that the materials of the No. 2 bath, which stick to the work when the work is removed therefrom, shall be removable by a succeeding No. 3 bath so that the work will not be coated with this insoluble material when the heat treatment is over. Accordingly, No. 3 bath, and also No. 4 if used, contain a considerable proportion of an alkaline compound of an alkali metal, such as the carbonate or hydroxide.

The No. 3 bath may be fused salts that can be arranged according to the following formulae, one of which shall be herein designated as No. 3A bath. These materials for the No. 3A bath in their preferred form are: sodium cyanide, sodium carbonate, potassium chloride, and potassium carbonate, and the preferred percentages by weight are:

	Per cent
Sodium cyanide	5 to 25
Sodium carbonate	15 to 25
Potassium chloride	10 to 15
Potassium carbonate	40 to 60

A bath which operates satisfactorily, but is not so desirable as the No. 3A bath is a bath which is herein designated as No. 3B, and which omits the potassium chloride. The No. 3B bath preferably comprises the following percentages by weight:

	Per cent
Sodium cyanide	5 to 30
Sodium carbonate	20 to 40
Potassium carbonate	40 to 55

It will be observed that the mineral salts in both the baths No. 3A and No. 3B are of the alkali metal family and it has been found that other salts of this same alkali metal family may be used in the place of those specifically designated, but for commercial reasons the above specified formulae are the preferred formulae. The No. 3A and No. 3B baths are both on the alkali side. The cyanide salt specified in these baths does not tend to case-harden the work except very slightly but does facilitate the dissolution of the No. 2 bath material which may stick to the work. These baths No. 3A and No. 3B are operated from 1200° F. to 1450° F. and act as quenching baths. It is possible under certain conditions to terminate the process with the removal of the material from the No. 3A or No. 3B baths and then continue the cooling either in air or in a suitable liquid. Where this process is utilized, it is preferable that the No. 3A and No. 3B baths be operated in the lower ranges of temperatures approximating 1250° F. Where these No. 3A or No. 3B baths are intermediate and are followed by another fused salt No. 4 bath, then it is preferable that the No. 3A and No. 3B baths be operated at the higher range of temperatures from 1350° F. to 1450° F.

In the preferred process, a fourth No. 4 bath is used as an additional quench bath to further dissolve the materials of the No. 2 bath and the No. 3A or No. 3B baths which may stick to the work, and to more gradually lower the temperature of the work. The final or No. 4 bath may also comprise two formulae which are herein designated as type No. 4A bath and type No. 4B bath. This No. 4 bath preferably is operated between 700° and 900° F. and the formulae are preferably as follows: For the formula of the bath No. 4A, the following are the preferred ingredients: sodium carbonate, potassium carbonate, potassium chloride, sodium chloride, sodium cyanide, sodium fluoride, and sodium hydroxide. The materials may be varied in percentages but the preferred percentages by weight are as follows:

	Per cent
Sodium carbonate	15
Potassium carbonate	8
Potassium chloride	7
Sodium chloride	5
Sodium cyanide	11
Sodium fluoride	7
Sodium hydroxide	47

As a second formula No. 4B, the potassium carbonate and potassium chloride may be omitted and the formula becomes then sodium carbonate, sodium chloride, sodium cyanide, sodium fluoride,

and sodium hydroxide and here again the percentages of the material may be varied, preferably keeping the sodium hydroxide at a substantially relatively high percentage. This formula No. 4B has a slightly higher melting point than the No. 4A formula. The No. 4B may be run at temperatures as high as 1200° F. The percentages may be varied but the preferred percentages by weight for the No. 4B bath are:

	Per cent
Sodium carbonate	28
Sodium chloride	10
Sodium cyanide	11
Sodium fluoride	13
Sodium hydroxide	48

It will be noted that both of the baths No. 4A and No. 4B are strongly alkaline and are easily soluble in water. These No. 4 baths remove any traces of the No. 2 bath which may not be removed in the No. 3 bath and leave the work clean so that it may be quenched in air and washed in water without any oxidation of the work.

There is a further advantage of utilizing the No. 4 baths, in that the No. 3 bath reduces the temperature of the high speed steel to between 1350° and 1450° F. which is at a temperature substantially at the intermediate low critical point for high speed steels and holding the work in the No. 3 bath until it has cooled to the temperature of this No. 3 bath produces a very desirable result. The No. 4 bath then quenches the intermediate work below the low critical of the steel and fixes the steel in the desired state so the steel may then be cooled to normal air temperature. The work as it leaves the fourth bath is free from scale and is sufficiently cold so that it does not tend to decarburize or oxidize.

In general, the greater part of baths Nos. 3, 3A, 3B, 4, 4A and 4B consists of alkaline compounds of the alkali metals. To lower the melting point and to increase the fluidity of the baths a neutral low melting point salt is added to the alkaline compounds, preferably the cyanide and/or chloride of an alkali metal. The cyanide is more useful than the chloride in cleansing the steel of adhering silicates. Any large proportion of alkaline earth metal chloride is to be avoided as it tends to reduce the solvent action of the quench on the high temperature salt. The addition of the alkaline earth metal chloride, in conjunction with the carbonates lowers the melting point of the quenching bath.

In connection with the use of the high temperature No. 2 bath or as herein designated, which is of a refractory nature, it has been found desirable to use this bath in a ceramic or a ceramic lined container which may be heated in any suitable way. There are two marked advantages in the use of such a ceramic pot. First, its ability to withstand high temperatures such as 2000 to 2400° better than a metallic container. Second it eliminates the danger of contaminating the high temperature bath with iron and its oxides as would be the case with an iron or iron alloy container. The contamination of the high temperature bath with iron and its oxides causes a soft skin to form on high speed steel tools and introduces a highly objectionable condition. The ceramic container shall be such that its characteristics are acid and neutral, but not alkaline, so that there is no reaction between the container and the high temperature No. 2 bath. Suitable containers with a high temperature bath may be constructed of the material disclosed in United States Farish Patent No. 1,561,641, patented November 17, 1925, which material is a non-recrystallized refractory composition consisting of silicon carbide, a carbonizing binder, and borax. The materials of the Farish Patent No. 1,561,641 may be used alone, but the pot is stronger when supported by some other material. It has been found that a container made in accordance with the United States Farish Patent 1,458,913, patented June 12, 1923 and then lined with the material of the Farish Patent 1,561,641 forms a desirable and practical container for the No. 2 bath. The material of Farish Patent 1,458,913 is a non-crystallized refractory material consisting of silicon carbide, crystalline graphite, a carbonizing binder and sand. This material provides the strength desired to support the lining. When the No. 2 bath is used at high temperatures, it is desirable that the inner surface of the container be glazed in accordance with the process disclosed in the Diamond Patent No. 1,828,767, patented October 27, 1931. This glazed lining produces a container which stands temperatures satisfactorily to the higher range of 2600° F. to 2900° F. These containers have a neutral or acid lining which contains no graphite, or other materials which at high temperatures might enter the bath to injure the work.

The foregoing baths and their relative temperatures comprise a complete method for heat treating of high speed steels of the character herein specified and wherein the baths No. 1 and No. 2 are heating baths in which the material is raised to and held at the upper limit of temperature desired. The primary purpose of the No. 1 bath is to preheat the work so that it will go into the No. 2 bath without a great differential in temperature and without chilling the No. 2 bath by the cold work as would be the case if work were introduced into the No. 2 bath from air temperature. Furthermore, the two stage heating results in substantial economy in heat penetration through the work. The No. 3 and No. 4 baths are quenching baths for reducing the temperature and are also washing and cleaning baths which remove the water insoluble material used for the high temperature No. 2 bath.

There is the further advantage of using the double quench stage by means of the No. 3 and No. 4 baths in that the work may be held at a temperature of substantially the second critical point of the steel which induces toughness and other desired characteristics to the steel which has come from the high temperature bath. Then the materials are subjected to the lower state of quenching and finally are air cooled and washed.

In using these several baths, it is desirable that the work be retained in each bath a sufficient interval of time so that all parts of each piece of work reaches the temperature of the particular bath in which the work is immersed. When the bath temperature is reached, then the work should be removed from the bath it is in and passed to the next bath until finally the work is taken from the last bath of the series and is allowed to cool to normal air temperature.

The final result of the various stages through which the work has passed is a hard tough high speed steel product which has not changed in dimensions and which is entirely free from scale,

oxidation, or decarburization so that the work at the end of the process is in its most desirable condition for further operations.

What we claim is:

1. A series of fused salt baths for steel treatment comprising a preheating salt bath, a high temperature salt bath and a quenching salt bath, the preheating bath consisting chiefly of alkali metal chlorides volatile at the temperature of the high temperature bath so that preheating salts adhering to the steel when it is immersed in the high temperature bath are removed by volatilization, the high temperature bath consisting chiefly of a silicate and a fluoride, the quenching bath comprising a predominate amount of an alkali metal carbonate to aid in the removal from the steel of any high temperature salts adhering thereto.

2. A series of fused salt baths for steel treatment comprising a high temperature bath consisting chiefly of a silicate and a fluoride and a quenching bath comprising a predominate amount of an alkali metal carbonate to aid in the removal from the steel of any high temperature salts adhering thereto.

3. A series of fused salt baths for steel treatment comprising a high temperature salt bath consisting chiefly of a silicate and a material to increase the fluidity of the bath, and a quenching salt bath consisting chiefly of alkali metal chloride, alkali metal cyanide and an alkali metal carbonate or hydroxide.

4. A high temperature fused bath for heat treatment of high speed steels comprising a fused mass of silica sand, an earth metal fluoride, an earth metal oxide, an alkali metal carbonate, and an alkali metal fluoride, the proportions of the materials being substantially as specified.

5. A high temperature fused bath for heat treatment of high speed steels comprising a fused mass of silica sand, calcium fluoride, calcium oxide, sodium carbonate, and sodium chloride, the proportions of the materials being substantially as specified.

6. A high temperature fused bath for heat treatment of high speed steels comprising a fused mass within the percentages by weight by silica sand 20 to 40%; calcium fluoride 20% to 40%; calcium oxide 10% to 25%; sodium carbonate 10% to 25%; and sodium fluoride 5% to 15%.

7. A quenching and washing bath for high speed steels which have been heated in a high temperature silicate bath, said quenching bath comprising a fused mass of an alkali metal cyanide, and an alkali metal carbonate.

8. A quenching and washing bath for high speed steels which have been heated in a high temperature silicate bath, said quenching bath comprising a fused mass of sodium cyanide 5% to 30%; sodium carbonate 20% to 40%; and potassum carbonate 40% to 55% by weight.

9. A quenching and washing bath for high speed steels which have been heated in a high temperature silicate bath, said quenching bath comprising sodium cyanide 5% to 25%; sodium carbonate 15% to 25%; potassium chloride 10% to 15%; and potassium carbonate 40% to 60%, by weight.

10. An alkali water soluble quenching and washing bath for high speed steels which have been heated in a silicate bath and preliminarily washed in a cyanide bath, said alkali water soluble bath comprising a fused mass of sodium carbonate, sodium chloride, sodium cyanide, sodium fluoride, and sodium hydroxide.

11. An alkali water soluble quenching and washing bath for high speed steels which have been heated in a silicate acid bath and preliminarily washed in a cyanide bath, said alkali water soluble bath comprising a fused mass of an alkali metal cyanide, carbonate, chloride, fluoride, and hydroxide; the said alkali metal hydroxide being by weight substantially one-half of the fused mass.

12. A high temperature salt bath with an operating range of from 2000° F. to 2400° F. for the treatment of high speed steels comprising a silicate and a material to increase the fluidity of the bath and to lower the melting point thereof, the proportions of said silicate and material being substantially as specified.

13. A salt bath for treatment of high speed steels comprising the fusing of silicon dioxide, a metallic fluoride, a metallic oxide, and a carbonate of an alkali metal, the proportion of the silicon dioxide to the metallic oxide plus the carbonate to be not less than their combining ratio whereby the fused bath comprises chiefly silicates and fluorides, as specified.

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