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METHOD OF BORONIZING STEEL

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2 Sheets-Sheet 1

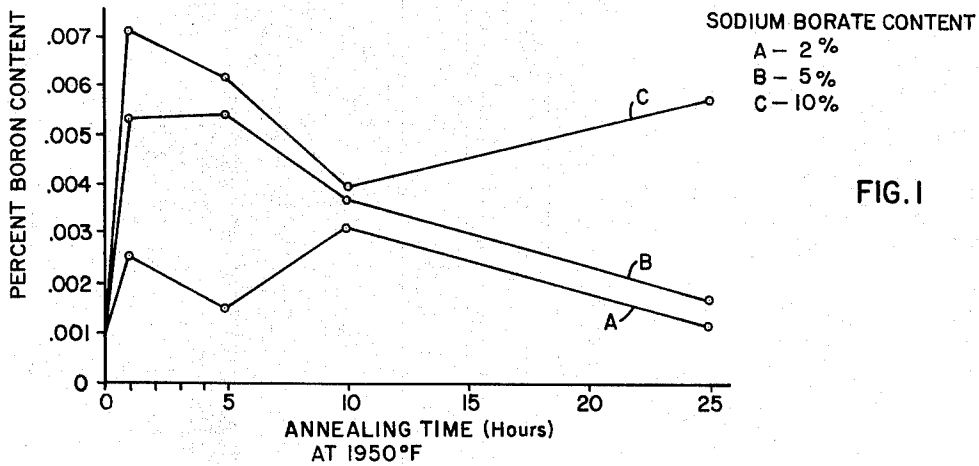


FIG. 1

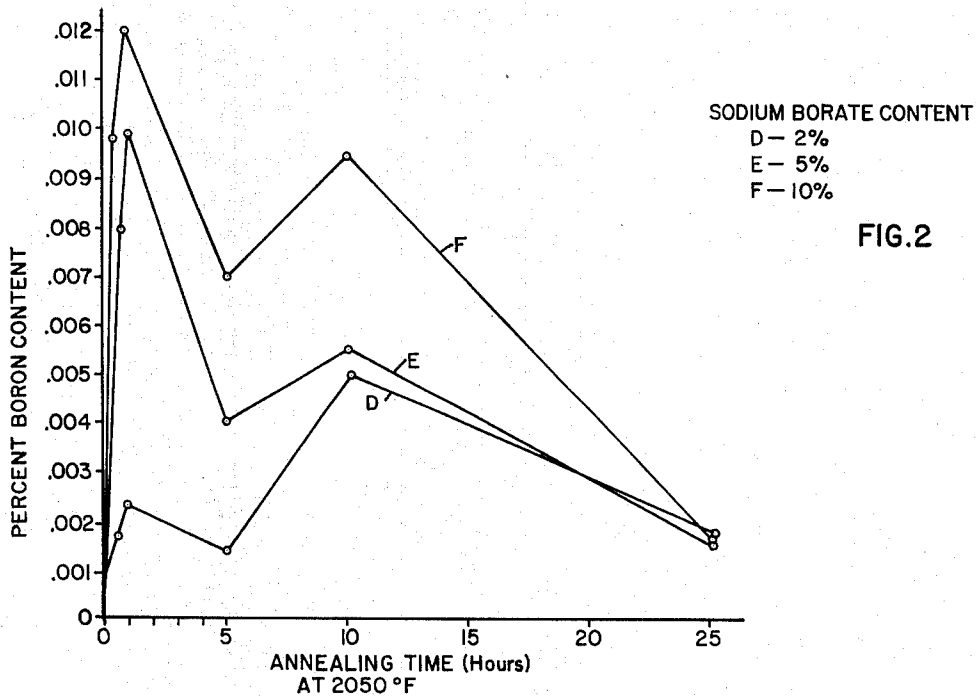


FIG. 2

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2 Sheets-Sheet 2

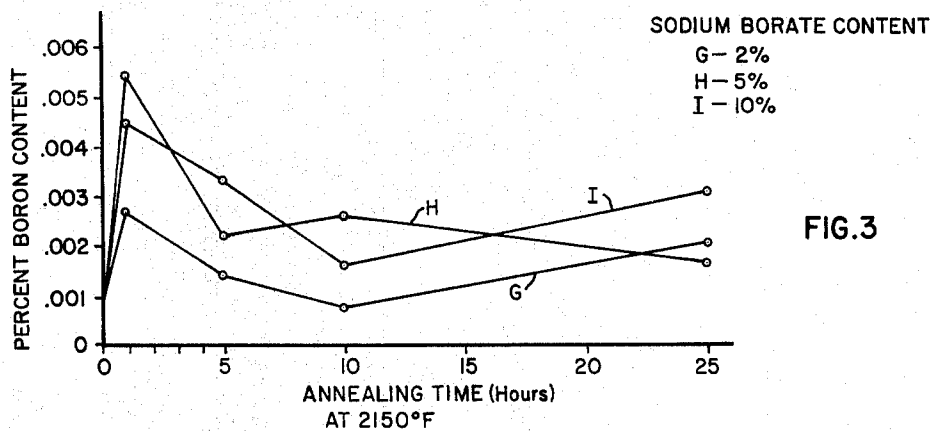


FIG. 3

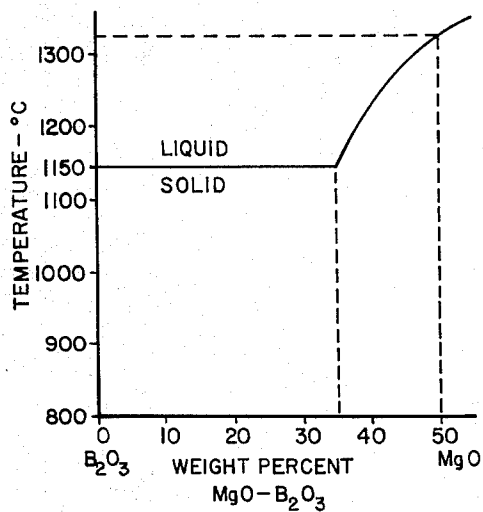


FIG. 4

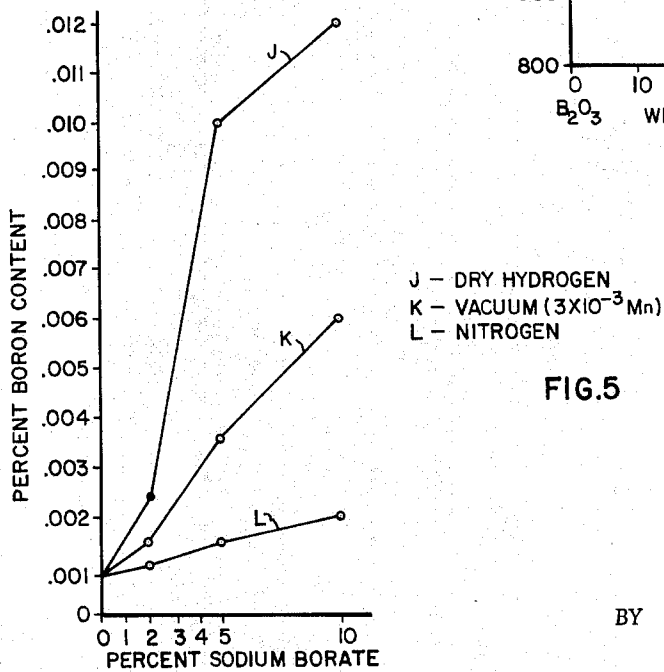


FIG. 5

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1

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METHOD OF BORONIZING STEEL

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5 Claims. (Cl. 148—16)

This invention relates to a method of treating metallic materials, such as carbon and alloy steels, to enhance certain properties thereof, such as stress-rupture properties, and, more particularly, to a method of accurately controlled boronizing of such materials.

The inclusion of boron, for example, in amounts on the order of 3×10^{-4} percent in carbon and low alloy steels is well known and commonly practiced in improving the hardenability of such steels. Boron is also on occasion introduced into high strength steels for use in the intermediate temperature range and, most recently, it has also been observed that small concentrations, i.e., a few thousandths of a percent of boron, are very helpful in improving stress-rupture properties of the high temperature alloys, such as the so-called superalloys, e.g., nickel base, cobalt base and iron base superalloys. As an example of the function of boron in the last mentioned alloys, it is known that a grain boundary constituent known as the lamellar phase, seriously lowers the stress-rupture life of the high temperature, iron base superalloy A-286. This alloy is an austenitic steel, which has good stress-rupture properties in the absence of the lamellar phase. However, in the presence of the lamellar phase the potential stress-rupture properties are not developed. It has been found, however, that if this steel contains over about 0.001% boron, the steel consistently exhibits high stress-rupture life together with improved notch strength. Such improvement in properties of this alloy is due to the absence of the lamellar phase and is attributable to the presence of boron in the steel.

The addition of boron during melting of the aforementioned steels is accompanied by certain disadvantages. Thus, the greater hardness and strength of the boron-containing steels contributes materially to the difficulty of fabricating these steels. Moreover, it is difficult to control the required small percentages of boron during melting.

Therefore, it is an object of the present invention to provide a method of producing metals and alloys having precisely controlled amounts of boron therein.

It is another object of the invention to provide a method of adding boron to difficultly workable metals and alloys subsequent to the initial melting and preliminary working thereof.

It is a further object of the invention to provide a method of accurately controlling boron additions to metals and alloys of the type having properties responsive to small additions of boron.

Still another object of the invention is to provide a method of strengthening metal and alloy articles subsequent to complete or partial fabrication thereof by controlled additions thereto of the element boron.

Exemplary of the method of the invention, a slurry comprising a volatilizable liquid vehicle, such as water, and a disperse solid phase comprising an inert, refractory ceramic carrier, such as magnesium oxide, together with a minor but effective amount of a source of boron, as sodium tetraborate, is applied to the surfaces of an article to be treated. The vehicle is volatilized and the coated

2

article then heated in the presence of a suitable reducing medium, such as dry hydrogen gas, whereby boron diffuses from the coating into the basis metal to effect the desired alteration of properties thereof. The foregoing and other objects of the invention will be apparent from a review of the following description taken in conjunction with the accompanying drawings, wherein

FIGURE 1 is a graphical representation relating heat treating time, at 1950° F., in a dry hydrogen atmosphere, with boron content introduced by diffusion into basis metal samples to which have been applied coating compositions comprising a magnesia carrier together with varying amounts of a boron compound, i.e., sodium borate;

FIGURE 2 is a graphical representation relating heat treating time, at 2050° F., in a dry hydrogen atmosphere, with boron content introduced by diffusion into basis metal samples to which have been applied coating compositions comprising a magnesia carrier together with varying amounts of a boron compound, i.e., sodium borate;

FIGURE 3 is a graphical representation relating heat treating time, at 2150° F., in a dry hydrogen atmosphere, with boron content introduced by diffusion into basis metal samples to which have been applied coating compositions comprising a magnesia carrier together with varying amounts of a boron compound, i.e., sodium borate;

FIGURE 4 is a simplified phase diagram of the system $MgO-B_2O_3$, and

FIGURE 5 is a graphical representation relating percent of sodium borate in a magnesia-containing coating composition versus boron content diffused into basis metal samples, for various atmosphere environments.

It has now been found possible to introduce accurately controlled amounts of boron into such metals as carbon steels, low alloy steels, high chromium alloy steels, and other materials, as nickel base alloys, cobalt base alloys and iron base superalloys, by a gas-metal boronizing reaction conducted under certain, limited conditions of temperature, time and atmosphere environments, by application to the material to be treated of certain specific coating compositions containing a source of boron. Thus, it has been found that a suitable coating composition for this purpose comprises an inert, refractory ceramic material, such as magnesia, together with a solid, dissociable boron compound, such as a boron oxide, e.g., boric anhydride (B_2O_3) or the hydrated form thereof ($B_2O_3 \cdot 3H_2O$); boric acid and salts thereof, as sodium borate, lithium borate, potassium borate, or magnesium borate, in anhydrous form or the various hydrated forms thereof. Refractory carriers other than magnesia are also contemplated, such as lime, dolomite, chromite, etc., and mixtures thereof. A mixture of carrier and boron compound is made, ground to a finely divided particulate form, and a slurry is prepared therefrom, using any suitable, non-reactive, volatilizable liquid, e.g., water, hydrocarbons, alcohols, ketones, etc., as a suspending vehicle. Water is desirable as a vehicle in view of its low cost, but other, non-aqueous vehicles are especially useful in maintaining a desired low percentage of water vapor in the atmosphere surrounding the coated article during subsequent heat treatment as described hereinbelow.

A coating slurry as above described may be applied to an article to be treated in any suitable manner, determined, in part, by the size and geometry of the article. Thus, in the case of irregularly shaped articles the coating composition may conveniently be applied by brushing, dipping, or spraying, whereas in the case of flat products such as sheet or strip, the coating composition is most conveniently applied by applicator rolls. Upon appli-

cation of the coating to the article to be treated, the coating is then dried, as by heating to a moderate temperature, to evaporate the liquid vehicle. In the case of coating compositions utilizing a chemical hydrate form of one of the constituents thereof, the heating temperature will, necessarily, be higher than in the case of coating compositions utilizing anhydrous materials.

The dried, coated article is then heated in the presence of a reducing agent, for example, dry hydrogen gas, whereupon, it is believed the boron compound dissociates and elemental boron thereupon diffuses into the underlying basis metal with the resultant advantageous effect on certain properties thereof as described hereinabove. It is also believed, but the invention is not limited by this theory, that the reducing, dry hydrogen atmosphere exerts its observed advantageous effect, i.e., enhanced rate of boron diffusion, due to its action in preventing formation and/or accumulation, as well as active removal, of relatively boron-impermeable surface films, as oxides, on the basis metal. When hydrogen gas is utilized as the reducing agent in the inventive process, it must be quite dry, e.g., the dew point should be at least -20°F . and preferably -50°F . It is in this aspect that suitable non-aqueous vehicles are especially useful in the preparation of the coating compositions.

Although dry hydrogen is the preferred atmosphere for use in the boronizing treatment of the invention, other non-carburizing and non-decarburizing reducing agents can also be employed as, for example, dissociated ammonia, etc.

In the course of the experimental work leading to the invention, slurry compositions were prepared, using a distilled water vehicle, with a dispersal aid comprising a magnesia (MgO) carrier, together with varying quantities of a source of boron, i.e., sodium tetraborate. Thus, slurries were prepared containing, by weight percent of dry coating composition, 2%, 5%, 25% and 50% of sodium borate, the balance of the dry composition being magnesia.

Basis metal samples comprised Type 302 (austenitic) stainless steel having dimensions of 6 inches x 1 inch x 0.04 inch. This steel, containing approximately 0.15% maximum carbon, 2% maximum manganese, 1% maximum silicon, 17 to 19% chromium, and 8 to 10% nickel, is representative of the high chromium steels usefully treated in accordance with the invention. These samples were brush coated, to an average thickness of approximately 0.02 ounce per square foot, with each of the aforementioned coating compositions and allowed to air dry. The samples were then inserted into a heat treating furnace containing a hydrogen atmosphere, and the furnace was held at a temperature of 1500°F . until the dew point was reduced below -20°F . Heat treating tests were then conducted at temperatures of 1950°F ., 2050°F ., and 2150°F . For each of these heat treating temperatures, test determinations were made of boron content introduced into the basis metal after varying times at the corresponding temperatures, as shown in Table I.

Table I

Coating Composition	Heat Treating Temp., $^{\circ}\text{F}$.	Atmosphere	Boron Content (Weight Percent) $\times 10^3$, in Sample After Heat Treatment, Time (Hours)				
			0.5	1	5	10	25
2% $\text{Na}_2\text{B}_4\text{O}_7$, balance MgO -----	1,950	Dry hydrogen-----		2.5	1.5	3.0	1.2
	2,050			2.3	1.4	5.0	1.8
	2,150		1.8	2.75	1.4	0.7	2.0
5% $\text{Na}_2\text{B}_4\text{O}_7$, balance MgO -----	1,950	do-----		5.3	5.4	3.7	1.6
	2,050			9.9	4.0	5.4	1.6
	2,150		8.0	5.5	2.2	2.7	1.5
10% $\text{Na}_2\text{B}_4\text{O}_7$, balance MgO -----	1,950	do-----		7.2	6.2	4.0	5.5
	2,050			12.0	7.0	9.4	1.8
	2,150		9.8	4.5	3.4	1.7	3.1
25% $\text{Na}_2\text{B}_4\text{O}_7$, balance MgO -----	2,050	do-----		1.0		30.0	
50% $\text{Na}_2\text{B}_4\text{O}_7$, balance MgO -----	2,050	do-----		1.4		(i)	

¹ B concentration too great for spectrographic resolution.

After treating at the temperatures indicated in Table I for the specified times, the samples were allowed to cool in their annealing atmospheres to $300-400^{\circ}\text{F}$. prior to removal from the furnace. Thereafter, the samples were removed from the furnace and the excess unreacted coating was scrubbed off with warm water and the samples were analyzed, in accordance with standard spectrographic analytical procedures, for the presence of boron. A standard curve for direct comparison of the spectrographic results of these tests was obtained from a series of wet chemical analyses of varying boron content and a sample of untreated Type 302 stainless steel was utilized as a standard.

The data given in Table I is graphically illustrated in FIGS. 1, 2, and 3, FIG. 1 consisting of graphs A, B, and C relating annealing time at 1950°F . against final boron content of the sample for coating compositions comprising magnesia and, respectively, 2%, 5% and 10% of sodium borate. FIG. 2 comprising graphs D, E, and F, similarly relates annealing time and boron content for various sodium borate concentrations of an annealing temperature of 2050°F ., and FIG. 3, comprising graphs G, H, and I, relates the same variables at an annealing temperature of 2150°F .

From the data of Table I and FIGS. 1-3 erected thereupon, it will be seen that the inventive method affords a means for introducing boron into a coated basis metal. It will be further noted that, at each of the annealing temperatures studied, generally the boron content of the basis metal increased as the amount of sodium borate in the coating was increased. Each of the test results of Table I is an average of a number of separate tests, each of which was obtained from a different area of a single specimen. These separate test values differed from one another only slightly, thereby indicating uniformity of diffusion of boron from the coating into the underlying basis metal. The samples also showed a remarkable uniformity of boron content between samples treated under the same process conditions of temperature, time and atmosphere. The uniformity of boron distribution within the relatively thin samples is probably due to the high diffusion rate of the interstitial element boron into the basis metal.

Importantly, it will be further noted that, in the case of FIGS. 1-3, the boron content introduced into the basis metal during the treatment thereof rises rapidly to a maximum value and, unexpectedly, thereafter rapidly decreases with increasing time exposure. Equally importantly, and again unexpectedly, it will also be observed, by a comparison of FIGS. 1, 2 and 3, that the annealing temperature is highly critical in respect of the rate and quantity of boron introduced into the basis metal. Thus, it will be seen that, for the tested borate concentrations over about 2%, the maximum obtainable boron concentration in the basis metal is much greater at temperatures of 1950°F . and 2050°F . than that obtainable at a temperature of 2150°F . Thus, whereas the maximum boron content was found to be about 7.2×10^{-3} weight percent at a tempera-

ture of 1950° F., and about $12 \times 10^{-3}\%$ at a temperature of 2050° F. The maximum boron content in the basis metal was only about $5.5 \times 10^{-3}\%$ under similar conditions at a temperature of 2150° F. Similar increases in boron concentration are also observed at the 5% borate level at temperatures of 1950° F. and 2050° F. as compared to that obtainable at 2150° F. Accordingly, therefore, the annealing temperature utilized in the inventive method is limited to between about 1925° F. and 2100° F., and preferably to between about 1950° F. and 2050° F. or 2075° F.

Moreover, the maximum boron concentrations obtainable in the basis metal are reached after about one hour's annealing time. Thereafter, and surprisingly, boron concentration in the basis metal generally decreases more or less rapidly, depending on the heat treating temperature and the borate concentration in the coating. Consequently, the time for which the treated metal is held at the heat treating temperature is restricted to a maximum of about two hours for obtaining maximum boron in the basis metal. Maximum boron concentrations in the basis metal are most effectively obtained by utilizing a heat treating time of about 0.5 to 1.0 hour. In the case of extremely massive basis metal bodies, the maximum time required for boronizing will generally be not greater than about 1 hour after temperature equilibrium (at the boronizing temperature) is reached. Periods of exposure less than these times are, of course, useful in those instances wherein it is not desired to obtain maximum boron concentrations in the basis metal.

Moreover, and importantly, however, the discovery herein that boron concentration in the basis metal actually decreases, and at a lower rate, upon continued heating for times greater than the maximum time, as aforesaid, for maximum boron incorporation in the basis metal, comprises a highly useful method of accurately controlling the boron content of alloys wherein small quantities of boron have effects, as upon mechanical properties, quite disproportionate to the boron percentage. Thus, should the boron level rise, during the initial heating period, to a value too high for best results, the quantity of boron may be reduced, and the desired properties of the alloy accurately established, by continued heating, in accordance with the reactions illustrated in FIGS. 1-3, for periods of time totalling up to about five hours.

Reference to FIGS. 1-3, and particularly to FIG. 2, representing the relationship between annealing time at 2050° F. and boron content in the basis material under optimum process conditions, shows that coating compositions containing from about 2 to about 10%, and preferably from about 5 to about 10%, by weight of dry coating, of sodium borate, are sufficient to introduce boron into the basis metal in amounts usually required for the necessary increases of strength and hardenability of commercial metals and alloys. Thus, in order to obtain a basis metal boron concentration of about 2×10^{-3} weight percent, borate concentrations over about 2% are required in the coating composition. Moreover, the maximum obtainable boron concentration is obtained within the aforesaid maximum heating time, i.e., 1-2 hours, only in those compositions containing over about 2% sodium borate. FIG. 2 also shows that borate concentrations up to about 0.012% are obtainable by utilizing coating compositions containing up to about 10% by weight of borate. In general such basis metal boron contents are sufficient for the intended purposes. Reference to Table I shows, however, that even higher basis metal borate concentrations are obtainable in accordance with the inventive method by utilizing higher borate concentrations in the coating. Thus, a coating composition containing 25% of sodium borate, balance magnesia, when applied to a Type 302 stainless steel sample and heated at 2050° F. in dry hydrogen for ten hours, resulted in a boron concentration in the basis metal of 0.030%. A similar coating composi-

tion containing 50% by weight of sodium borate, and tested under the same conditions, resulted in a borate concentration in the basis metal so high as to give black, unreadable lines on the spectrographic film, indicative of very large amounts of boron pickup in the basis metal under these conditions.

The mechanism responsible for the decrease in boron concentration in the basis metal with increasing exposure times is not fully understood. Nor is the mechanism responsible for the critical character of the heat treating temperature fully understood. With respect to the latter mechanism, however, it is believed, but the invention is not restricted to this theory, that the observed critical minimum temperature, i.e., about 1925° F., and preferably 1950° F., is required in order to effectuate dissociation of the boron compound and the release of the elemental metal for diffusion into the underlying basis metal. As stated hereinabove, it is believed, that the presence of a dry hydrogen atmosphere during treatment of the basis metal also serves to preclude or to remove boron-imperious films from the surface of the basis metal, thereby facilitating the diffusion therein of the boron. To this end, it is advantageous that the coating on the basis metal be permeable to hydrogen. In this connection, the contemplated coating films applied to the basis material in accordance with this invention are believed to remain in a hydrogen-permeable state throughout the treating process or at least for a sufficient period of time during the annealing-boronizing process to enable the hydrogen to accomplish its beneficial effects. By reference to FIG. 4, constituting a simplified phase diagram of the system

MgO-B₂O₃

it will be seen that the minimum liquidus-solidus temperature for the system MgO-B₂O₃ is about 1150° C. (2102° F.) for compositions containing up to about 35% by weight of B₂O₃ (about 61 weight percent Na₂B₄O₇), and rises with increasing percentages of B₂O₃. Thus, at the effective heat-treating temperatures contemplated herein, the contemplated magnesia-borate compositions are believed to remain substantially in solid form and pervious to hydrogen. However, at higher temperatures, e.g., 2150° F., a substantial portion of the coating is believed to liquefy, in accordance with the phase diagram of FIG. 4, thereby accounting, at least in part, for the lower boron content obtainable in the basis metal at such higher temperature, as shown in FIG. 3.

Further comparative tests were conducted on similar samples, coated, as aforesaid, with coatings comprising magnesia and varying weight percentages of sodium borate, in various environments, i.e., dry hydrogen, vacuum (3×10^{-3} mm. Hg) and nitrogen. The results of the latter tests, wherein the samples were heated for one hour at 2050° F., are given in FIG. 5 from which it will be seen that the rate of increase of boron content, as well as the maximum obtainable boron content, in the basis metal is much greater in the samples treated in a hydrogen atmosphere than in the case of the samples treated either in a nitrogen atmosphere or in a vacuum environment. It will be further noted from FIG. 5 that treatment in nitrogen atmosphere gives the lowest rate of boron pickup and the lowest total boron content of the environments studied, whereas treatment in vacuo gave results lying between those obtained for hydrogen and nitrogen, but still far below the boron levels obtainable with the use of hydrogen. Consequently, the use of a dry hydrogen atmosphere is preferred, especially in those instances wherein it is desired to incorporate relatively larger amounts of boron in the basis metal. However, in instances wherein it may be desired to incorporate only relatively smaller quantities of boron, environments other than hydrogen, e.g., nitrogen or a vacuum may be utilized.

The boronizing process of the invention is applicable to materials other than austenitic stainless steels such as

the Type 304 steel exemplified hereinabove. Thus, further tests were conducted on samples of a commercial, ferritic hot work steel, having a nominal analysis as follows: 0.40% carbon, 0.33% vanadium, 0.55% manganese, 1.00% silicon, 2.50% molybdenum and 3.25% chromium. The samples of this steel were in the form of strips, as in the case of the Type 304 samples, except that the ferritic steel samples had a thickness of 0.125 inch. As in the case of the stainless steel samples, the hot work steel samples were brush-coated with an aqueous slurry comprising a fused mixture of magnesia and 2% sodium borate. The thus-coated samples were annealed in dry (-30° F. dew point) hydrogen at various temperatures, cooled, scrubbed with water to remove the adherent coating, and spectroscopically analysed for boron. The results are given in Table II.

Table II

Sample No.	Heat Treatment	Boron Content, Weight Percent	Carbon Content, Weight Percent
1.....	Untreated.....	0.0008	0.33
2.....	1 hour at 1,950° F.....	0.001	0.33
3.....	1 hour at 2,050° F.....	0.002	0.33

From Table II it will be seen that, even with this relatively low borate concentration in the coating, i.e., 2%, substantial quantities of boron can be introduced into this basis metal in accordance with the invention.

It will be further noted from Table II that the process of the invention does not result in decarburization. This is, of course, an important consideration in the heat treatment of alloy carbon steels wherein any appreciable decarburization would result in drastically altered properties.

The invention is applicable to the boronizing of a wide variety of basis metals and of products of many different physical forms. A particularly useful application of the invention comprises the introduction of accurately controlled quantities of boron into products in sheet or strip form. Thus, cut sheets, requiring a final heat-treatment, are readily coated with a contemplated boronizing composition, as aforesaid, and then subjected to the required heat treatment, whereupon boron is introduced into the sheet, with the aforementioned attendant advantages. Moreover, elongated strip or sheet material may, with great advantage, be coated in a continuous or semi-continuous manner, coiled, simultaneously annealed and boronized at proper time and temperature, then, after cooling, cleaned, cold rolled to final gage or hardness, leveled and flattened. Introduction of boron, as herein contemplated, after substantial mechanical working has been accomplished, avoids the added working difficulties often attendant upon the presence of substantial quantities of boron during extensive mechanical reduction. Thus, the boronizing process of the invention is especially useful in respect of steels and superalloys which require boron and which are inherently difficultly workable. Exemplary of such alloys are the precipitation hardenable stainless steels, and nickel- and iron-base superalloys. Introduction of the required boron contents into thin section articles of these materials, after all or a substantial portion of the mechanical reduction thereof has been accomplished, materially reduces the difficulty of such reductions and the recovery yields of the alloys.

As heretofore set forth, it has been found that about 2% by weight of sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$) is required in the dry coating compositions in order to obtain the advantages of the invention and, further, that sodium tetraborate contents up to about 50% by weight may be utilized. The corresponding weight percentages of elemental boron are, respectively, about 0.4 and 10% and since, as has been also hereinabove noted, a variety of boron compounds can be utilized in the practice of the invention, the latter percentages are generally definitive of

the contemplated coating compositions vis-a-vis the active, boronizing constituent thereof.

In order to obtain enhanced bonding and adherence between the boronizing coating material and the basis metal during treatment thereof, suitable bonding agents may be incorporated in the slurry of boronizing agent. Exemplary of suitable bonding agents are organic materials such as carboxymethyl cellulose or analogs thereof.

The foregoing descriptive and exemplary matter is illustrative of the principles of the invention and it is to be understood that various modifications and additions thereto may be made by those skilled in the art without departing from the spirit and scope of the invention.

What is claimed is:

1. A method of combined controlled boronizing and annealing of ferrous metal sheet stock comprising apply-

ing to the surfaces of the stock a slurry comprising a volatilizable vehicle and a fused mixture of magnesium oxide and a material selected from the group consisting of boron oxides, borates of sodium, lithium, potassium, and magnesium, and mixtures thereof, heating the coated stock to volatilize the vehicle and continuing said heating in dry hydrogen at a temperature between about 1900° F. and 2100° F. for a period from about 0.5 to about 2 hours to anneal the stock and to effect impregnation of the stock with boron to a desired extent.

2. A method of annealing and alloying with boron an article comprising a metal selected from the group consisting of base alloys of iron, nickel and cobalt, comprising coating the article with a slurry comprising a volatilizable vehicle and a dispersoid comprising a particulate refractory carrier and a boron compound selected from the group consisting of boron oxides, borates of the metals sodium, lithium, potassium, magnesium and mixtures thereof, wherein the boron compound comprises, in weight percent of dispersoid, between about 0.4 and under 10% of elemental boron, volatilizing the vehicle to leave on the article surface a dry coating of dispersoid, heating the coated article in a nonoxidizing environment to a temperature of at least about 1900° F. and less than that at which the coating components form a liquid phase, and continuing the heating of the coated article for at least about 0.5 hour and less than about 5 hours whereby the article is annealed and alloyed substantially uniformly throughout with boron to a desired extent.

3. A method in accordance with claim 2 wherein the carrier is magnesium oxide, the environment is hydrogen having a maximum dew point of about -20° F., and the maximum heating time is about two hours.

4. A method in accordance with claim 3 wherein the boron compound is sodium tetraborate and the heating temperature is between about 1900° and 2100° F.

5. A method in accordance with claim 4 wherein the sodium tetraborate is present in an amount between about 2-25%, by weight of dispersoid, and the heating temperature is between about 1950° and 2075° F.

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