March 7, 1950 P. G. MABUS 2,499,764

PROCESSING STAINLESS STEEL

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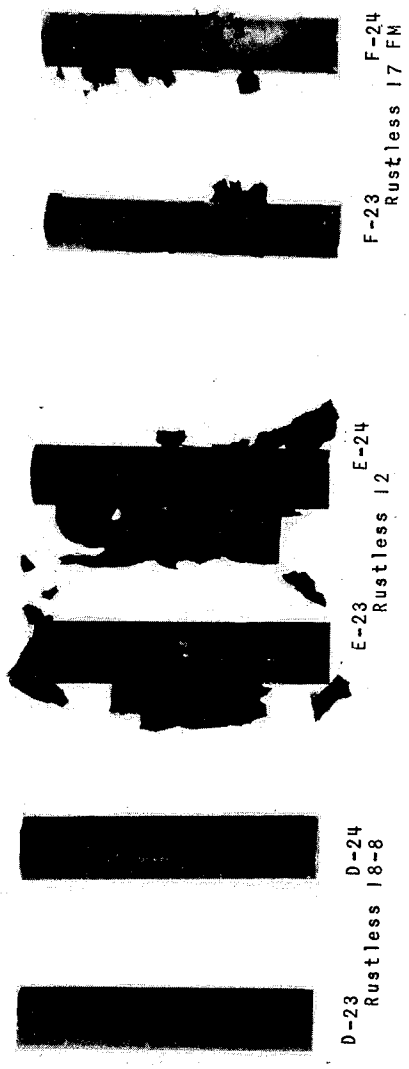


Fig. 2.

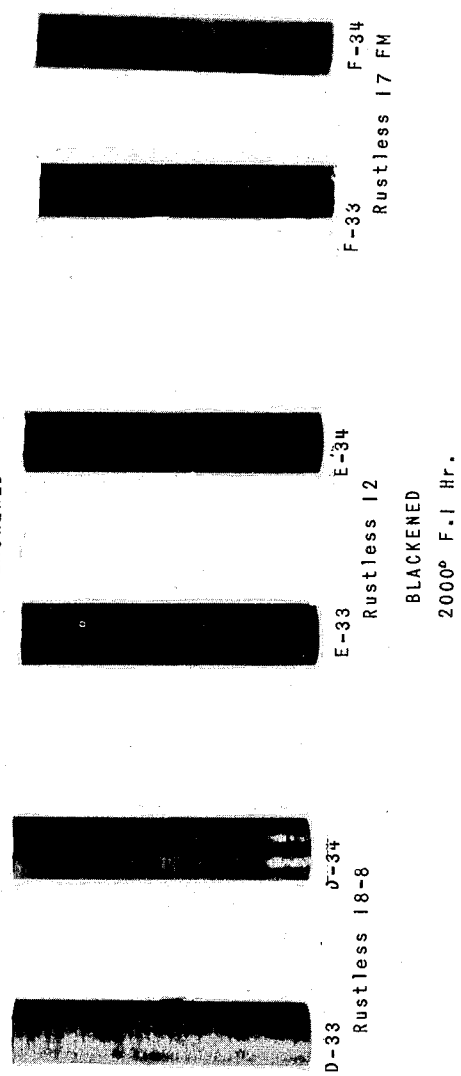


Fig. 1.

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PROCESSING STAINLESS STEEL

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6 Claims. (Cl. 148—6.11)

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My invention relates to processing stainless steels and particularly concerns the fabrication and heat-treatment of articles formed thereof.

Among the objects of my invention is to provide a method of treating stainless steel articles formed to nearly precise dimensions to permit subsequent heat-treatment without detrimental scaling, the surface of the metal being at all times thoroughly protected throughout the entire exposure to elevated temperatures; which method is simple, reliable, subject to faithful duplication of results and requiring a minimum of investment of plant, equipment, materials and labor; which is sure, certain, and is comparatively rapid and involves a minimum number of procedural steps; and which permits the construction of fabricated parts, particularly of the standard grades of stainless steels such as 12% chromium, 18–8 chromium-nickel, 17% chromium and the like, which parts have high finish and faithful correspondence to required dimensions, and which require no finishing reduction to size following the requisite heat-treatment.

All of these and many other advantages will in part be obvious and in part pointed out hereinafter during the course of the following description, taken in the light of the accompanying drawings.

My invention accordingly resides in each of the several manipulative and procedural steps, as well as in the relation of each of the same to one or more of the others, the scope of the application of which is more fully set forth in the claims at the end of this specification.

In the drawings, wherein I have disclosed several comparative tests illustrating the advantages of my new technique over practices as heretofore existant, Figure 1 discloses test bars of the 12% chromium, 18–8 chromium-nickel and 17% chromium free-machining grades of stainless steel which have been treated in accordance with my invention and subsequently heat-treated, while Figure 2 shows similar tests run with like bars which have not been so treated. In the tests the specimens were subjected to a heat-treatment at 2000° F. enduring for one hour. Strikingly superior results attended upon my new practice, as is evident from the comparison of the figures.

As conducive to a more thorough and ready understanding of my invention it is advantageous at this point to consider that stainless steels have come into greater and greater use through the past several decades for many and varied purposes. While certain superior grades of stainless steel, costly and time-consuming in production, display advantageous non-oxidizing qualities even when subjected to high temperature, oxidizing atmospheres over protracted intervals of time, this is by no means true of the standard grades of this metal. When these materials have heretofore been subjected to high temperature oxidizing atmospheres, wide-spread scaling has been observed, demonstrating a substantial and unpredictable variation of the surface dimensions of the ultimate product from that which was first subjected to heat-treatment.

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Now such heat-treatment is substantially a prerequisite in the production of numerous parts which are intended for specialized industrial applications. Heretofore, to accomplish the desired objective the parts had to be first shaped only roughly to allow for substantial yet unpredictable scaling, then subjected to heat-treatment, and thereafter ground or otherwise machined adequately to the required dimensions. This required two manipulative steps, which were time-consuming and costly.

Moreover, extensive machining subsequent to the heat-treatment is performed with difficulty and at substantial expense. The art, therefore, for a comparatively long period of time experienced a definite need for a method of heat-treating stainless steel in an oxidizing atmosphere in desired good manner without the necessity of appreciable subsequent machining and while retaining closely the machining and dimensioning imparted thereto prior to the heat-treatment.

An important object of my invention is to provide a method of processing stainless steel, especially the standard grades, in the substantial avoidance of the defects, drawbacks and deficiencies of the prior art, including those heretofore more specifically described; which method is comparatively rapid, simple and inexpensive, involving a minimum number of procedural steps and each of these simple and easy in themselves to effectuate; and which substantially suppresses detrimental scaling and permits preliminary forming of the end product to substantially its ultimate nicely determined dimensions, so that minimum machining or finishing is required subsequent to heat-treatment according to my new method of processing.

Briefly stated, my invention comprises passivating the metal, as by converting a microscopically thin surface layer of the metal to black oxide, and thereupon subjecting the passivated metal to prolonged heat-treatment at required high temperatures so as to impart the desired

structure to the metal, all in the substantial absence of detrimental scaling.

The particular application of my process is to the standard grades of stainless steels, as compared to what may be termed the high chromium and special grades thereof, all as more fully pointed out hereinbefore and hereinafter. It is necessary in producing commercial articles of these steels, after first roughing up the articles to an approximation of the final form thereof, to impart a proper structure thereto through suitable heat-treatment. The superior grades of stainless steel display a substantial degree of resistance to the highly oxidizing atmosphere which ordinarily prevails in such heat-treatment. Unfortunately, however, it has been practically impossible to subject the standard grades of stainless steel, say the 12% chromium, 18–8% chromium-nickel or 17% chromium free-machining steels to these oxidizing atmospheres without detrimental scaling.

It is to prevent this scaling that the passivating treatment heretofore referred to is employed. Preliminary to such passivation, however, my invention makes it entirely practicable and it is my practice according thereto, to form the articles constructed of these standard grade stainless steels to practically determine their final dimensions, with but little if any tolerances provided to allow for undesired scaling. Thus, after the heat-treatment heretofore referred to, little if any further machining is required to produce the final articles.

Passivation, which is the conversion of the surface layer of the metal to a black oxide coating, is accomplished by immersion in a bath consisting of molten sodium dichromate or molten potassium dichromate or mixtures of the same proposed in the U. S. application for Letters Patent of Irvine C. Clingan, Serial No. 470,853, filed December 21, 1942, now Patent 2,394,899 of February 12, 1946, entitled Stainless steel and method of coating same. The temperatures employed range from 320° C. to 500° C. The time of immersion is on the order of some two to fifteen minutes. Preferably the bath entirely consists of molten sodium dichromate at a temperature of about 400° C. The black oxide film, as more particularly noted in the patent referred to, is dense, hard and tough. It is flexible and does not crack, chip or spall off. It appears rich in oxides of chromium.

In the heat-treatment following surface oxidization or passivation the temperatures employed are those required to bring about the desired structure. They may amount to as much as 2000° F. They will endure for the required time intervals, ranging upwards of one or two hours.

Following heat-treatment in the manner described the black coating is removed according to the practice of my invention, by immersion in any standard descaling bath, such as a fused sodium hydride. The coating also may be removed by immersion in a standard pickling bath such as a nitric-hydrofluoric acid bath (10% nitric acid-1% hydrofluoric acid, remainder water). Five minutes treatment is found sufficient for this purpose. Because the oxide coating is so easily removed and because of its extreme microscopic thinness, detrimental pickling finish, etching or scale marks are avoided.

Having reference to the four figures of the drawings, it will be noted that according to Figures 1 and 2 like specimens have been subjected to heat-treatment. The three sets of bars shown

in Figure 1, however, have been first exposed to a passivating treatment in accordance with the practice of my invention. They were thereafter subjected to heat-treatment at 2000° F. for one hour. Substantially no scaling is observed to have occurred. The dimensions remain substantially unchanged.

Where no preliminary passivating treatment has been resorted to, however, similarly heat-treated bars, as shown in Figure 2, are subject to extreme and detrimental scaling. A substantial loss of metal occurs, and dimensions are appreciably changed. In such instances, it is necessary to allow additional metal in the roughed article, to compensate for such scaling, and subsequently to machine the metal down to final dimension. This of course may entail further structural change, both undesirable and unpredictable in nature.

It is apparent from the foregoing that according to the practice of my invention it is now possible to subject stainless steel, especially the standard grades such as 17% chromium, 12% chromium, or 18–8% chromium-nickel steel and the like which ordinarily are subject to oxidation at high temperatures, to prolonged heat-treating operations in the substantial absence of appreciable or detrimental scaling. The process of conditioning and heat-treating such steels is comparatively rapid, simple and easily carried into operation. Results are certain and predictable. Machining to close tolerances prior to the heat-treatment is made possible. All these and many other highly advantageous results attend upon the practice of my invention, both as to the standard grades of stainless steel and the superior grades as well.

Once the broad aspects of the invention are disclosed, many embodiments thereof of course will occur to those skilled in the art, all falling within the scope thereof. Similarly, many modifications of the present embodiment will readily suggest themselves to those skilled in the art. Accordingly, I intend the foregoing disclosure to be considered as merely illustrative and not by way of limitation.

I claim:

1. The method of processing stainless steel, comprising in combination, the steps of chemically producing on the surface of the stainless steel a flexible non-spalling black oxide coating rich in oxides of chromium, annealing the coated steel in an oxidizing atmosphere ordinarily causing scale formation, and removing the black oxide coating down to the annealed metal by immersing the coated metal in a descaling bath.

2. The method of processing stainless steel, comprising in combination, the steps of chemically producing on the surface of the steel a flexible non-spalling black oxide coating rich in oxides of chromium, heat-treating the coated steel at elevated temperatures under oxidizing conditions ordinarily causing scale formation, and removing the black oxide coating from the treated steel by pickling in a bath comprising 10% nitric acid and 1% hydrofluoric acid.

3. The method of processing stainless steel, comprising in combination, the steps of chemically producing on the surface of the steel a flexible non-spalling black oxide coating rich in oxides of chromium, thereupon heat-treating the steel at elevated temperatures under oxidizing conditions ordinarily causing scale formation for protracted periods of time, and thereupon removing the black oxide coating by immersing

the coated steel in a bath of fused sodium hydride.

4. The method of heat-treating stainless steel in the substantial absence of detrimental scale formation, comprising in combination, the steps of converting a surface layer of the metal to the black oxide thereof by immersion in a bath consisting of one or both of the group molten sodium dichromate and molten potassium dichromate, thereupon heat-treating the metal at elevated temperatures and in oxidizing atmosphere ordinarily causing scale formation, and then chemically removing the black oxide down to the underlying metal by immersing the coated steel in a descaling bath.

5. The method of treating stainless steel, comprising in combination, the steps of a heat-resistant black oxide coating on the surface of the steel by immersion in a bath consisting of one or more of the group molten sodium dichromate and molten potassium dichromate, heat-treating the coated steel in highly oxidizing atmosphere for achieving desired heat-treatment properties in the steel underlying said coating, and subjecting the heat-treated steel to a pickling bath comprising nitric acid and hydrofluoric acid for removing the black oxide coating.

6. The method of producing heat-treated accurately dimensioned stainless steel articles, comprising forming the article to substantially final dimension, producing a microscopically thin black oxide coating on the surface thereof by immersion in a bath consisting of one or more of the group molten sodium dichromate and molten potassium dichromate, thereupon heat-treating the coated article at temperatures sufficiently elevated and in oxidizing atmosphere ordinarily causing scale formation and enduring for a time sufficient to impart desired heat-treatment qualities to the metal, and then removing the coating from the dimensioned article by immersion in a descaling bath.

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