

1
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METHOD OF IMPROVING THE BONDING PROPERTIES OF STEEL SURFACES

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This invention pertains to a method of improving the bonding properties of mild steel surfaces.

Usefulness, serviceability and attractiveness of steel surfaces, particularly those present in consumer goods, are almost invariably greatly improved by application of a surface coating to these surfaces. In many cases such a surface coating is a necessary prerequisite, spelling the difference between a useful and a useless item.

The surface coatings considered here may be organic coatings, such as lacquer, paint, resinous, rubber and similar coatings, or they may be inorganic coatings, for example, electroplated metallic coatings or metallic coatings applied by non-electrolytic means. Not considered here and not within the scope of this invention are coatings applied at high temperatures, such as hot-dipped metallic coatings.

Any such coating is applied for the purpose of presenting to the environment, not a steel surface but the particular surface characteristics of the coating. Therefore, such coatings should completely cover the underlying steel surface and do so reliably during the service life of the item. Of utmost importance in this connection is the ability of the coating to adhere firmly to the steel surface under service condition. Ideally, the coating should be so firmly adherent, that a break, if it occurs at all, will be either in the steel or in the coating, but not at the interface.

It has been recognized for a long time that the condition of the steel surface, prior to the application of a coating, is of very great importance. Obviously, gross contamination of that surface by manufacturing residues, such as oxide scale, rust, grease and the like, will interfere with proper coating adhesion. Therefore, surface cleaning by a variety of procedures, to remove gross contamination, is standard practice. In these procedures, solvents are employed for grease and oil removal, acids, or alkalis for removal of other contaminants, and in many cases multi-step treatments using various such cleaning agents are employed. Generally, proper use of these and related procedures, some mechanical in nature, will result in rather complete removal of gross contaminants and give a steel surface termed "clean." Also, experience has shown that coatings applied to such a "clean" steel surface will adhere quite well to that surface. Therefore, industry quite generally concluded that "cleaning" procedures as illustrated above, if properly used, will give a steel surface with the best bonding characteristics practically obtainable.

More recent investigations, however, have shown that ideally clean surfaces should have bonding characteristics vastly superior to those obtained with the usual, though carefully applied, cleaning methods.

It has now been found, in accordance with the present invention, that a relatively very simple treatment, applied to steel surfaces, after the usual grease and/or scale removal, will produce a surface with bonding characteristics vastly superior to those obtained by heretofore

known cleaning procedures and approaching the bonding characteristics of ideally clean surfaces.

The treatment of this invention employs a treating bath comprising an aqueous solution of acids and oxidizing agents. Based on the discovery that in such systems, water must be regarded as an active reagent and not simply as a diluent, the treating bath must contain water, acid or acids and the oxidizing agent in certain critical concentrations and ratios to each other.

A typical bath, in accordance with my invention, might contain as essential ingredients, phosphoric acid, water and hydrogen peroxide. This bath will vastly improve bonding characteristics of mild steel by simply contacting the steel with it at ambient temperature for from 20 to 120 seconds, provided the bath constituents are present in certain concentrations and ratios to each other.

An operable system of this type can be defined clearly by means of a triangular graph in which each apex of the triangle represents a component concentration of 100% and each side of the triangle a component concentration of zero percent.

In such a graph, suitable bath compositions will be found to be between approximately 20 to 65 weight percent of water, 5 to 30 weight percent of H_2O_2 and 25 to 70 weight percent of mineral acid. The actual bath composition is then defined by the further requirement that the sum of percentages of water, H_2O_2 and acid must equal 100%.

This relationship is therefore as follows:

$$\begin{aligned} \text{Percent water} + \text{percent } H_2O_2 \\ + \text{percent mineral acid} = 100\% \end{aligned} \quad (1)$$

wherein

$$\text{Percent water is between 20\% and 65\%} \quad (2)$$

$$\text{Percent } H_2O_2 \text{ is between 5\% and 30\%} \quad (3)$$

$$\text{Percent mineral acid is between 25\% and 70\%} \quad (4)$$

Because results over the whole range are more or less the same, one will choose, for practical reasons, systems relatively low in H_2O_2 and/or mineral acid. For any system the proper percentage of water is then found from Equation 1 by inserting the chosen percentage figures for the other two components.

Outside the specified range, results will become more and more unsatisfactory, either in fact or in cost or both.

In making up the baths of this invention, one may employ a mineral acid. Practical considerations dictate the use of phosphoric acid or sulfuric acid, or a mixture thereof, as the acid component. However, sulfuric acid alone may be used, an example being a bath containing 40% water, 42% H_2SO_4 and 18% H_2O_2 . Other mineral acids are less desirable because of their incompatibility with H_2O_2 , HCl for example reacting with liberation of some chlorine. Mixtures of phosphoric acid and sulfuric acid are, however, advantageous and are preferred. In this case, bath composition is calculated as explained above for phosphoric acid, but in actually making up the bath, 5 to 20 percent of H_3PO_4 as calculated, is replaced by H_2SO_4 on a weight basis.

As oxidizing ingredient, it is preferred to use H_2O_2 , but compounds liberating H_2O_2 in contact with water in presence of acid may be used.

If desired, surface active agents may be added to the solution.

The steel to be treated must be free of grease and oil, and preferably free of mill scale. Particularly good results are obtained with cold rolled mild steel, but hot rolled steel will also respond to the treatment after proper oxide scale removal.

After degreasing and, if needed, de-scaling, the steel, preferably after a water rinse, is immersed in the treating bath at ambient temperature. Immersion time may range

3

from about 20 to 30 seconds to as much as two or three minutes and longer. Very good results can be obtained in about 30 to 60 seconds and are not substantially improved with longer treating times, which however may be more practical in certain cases.

Upon removal from the treating bath, the steel is rinsed promptly and thoroughly with cold water and dried. The improved brightened steel surface obtained will not change its characteristics over prolonged periods of time and may be coated immediately after drying, or days and weeks thereafter, without change in bonding behavior.

The following examples will serve to illustrate this invention. Percentage figures given are by weight.

Example 1

A mild steel panel was degreased in a conventional manner by treating with an organic solvent, rinsed with water and then treated at room temperature for 40 seconds with a solution containing:

35.0% water
7.5% H_2O_2
57.5% H_3PO_4

Thereafter the panel was rinsed thoroughly with water and dried. The originally dull steel surface was brightened very uniformly and was very well suited for subsequent application of a coating.

Example 2

Another mild steel panel was treated as in Example 1 with a solution containing:

40% water
42% H_2SO_4
18% H_2O_2

A very evenly brightened steel surface, highly suitable for subsequent paint coating, was obtained.

Example 3

Another mild steel panel was degreased as in Example 1 and then treated for 60 seconds with a solution containing:

45.5% water
7.3% H_2O_2
34.4% H_3PO_4
12.8% H_2SO_4

After rinsing and drying, the now brightened steel surface was coated with a 1.5 mil thick coating of a pigmented nitrocellulose lacquer.

For purposes of comparison, the same lacquer coating was applied to a steel panel which had been only degreased and to another panel which had been bonderized (phosphatized) by a commercial bonderizing process. Bonderizing of steel is done largely to improve the bonding properties of the steel surface.

The three panels, one treated in accordance with this invention, one degreased and one bonderized, after properly drying the lacquer coating, were then subjected

4

to a lacquer adhesion test, using a Gardner impact tester. In this testing method, a weight is dropped on to the surface to be tested and coating adhesion and chip resistance are judged by the impact in inch-pounds which the coating will suffer without damage. This test gave the following results:

Damaged by impact of

Panel treated according to this invention... 80 inch-pound
Panel degreased only 10 inch-pound
Panel bonderized 30 inch-pound

Example 4

Another mild steel panel was treated as in Example 2 and coated with a 3 mil thick coating of white alkyd type baking enamel. Upon impact testing as in Example 3, it was found that the treated panel withstood 80 inch-pound impact, compared to 10 inch-pound for a bonderized and 10 inch-pound for a degreased panel.

Example 5

Another steel panel was treated as described in Example 3. The treated panel was then electroplated in a Watt's bath and an approximately 3.5 mil thick nickel coating deposited.

A commercial standard test showed that nickel plate adhesion was excellent.

For comparison, a similar untreated panel was prepared for nickel plating by a widely used commercial cleaning procedure comprising degreasing, alkaline anodic cleaning, rinsing, acid dipping and rinsing, and then plated in the same way.

This cumbersome multi-step procedure resulted in a nickel plate adhering not as well to the panel as a nickel plate upon a panel treated in accordance with this invention.

What is claimed is:

1. The method of improving the bonding properties of mild steel surfaces which comprises treating the steel for a period of at least 20 seconds with a solution containing about 20 to 65 weight percent of water, 5 to 30 weight percent of H_2O_2 and 25 to 70 weight percent of mineral acid in such proportions that the sum of percentages of water, H_2O_2 and acid equals 100% and then rinsing the steel.

2. The method of claim 1 in which the mineral acid is phosphoric acid.

3. The method of claim 1 in which the mineral acid is a mixture of between 5 and 20% sulfuric acid and between 95 and 80% phosphoric acid.

4. The method of claim 1 in which the mineral acid is sulfuric acid.

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