TEST FOR DETERMINING CORROSION RESISTANCE OF STAINLESS STEEL

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TEST FOR DETERMINING CORROSION RESISTANCE OF STAINLESS STEEL

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This invention relates to a test which makes possible the rapid determination of the corrosion resistance of chromium-bearing stainless steels. Stainless steel, particularly chromium ferritic steel, is widely used in the automotive industry, for example, in the form of exterior fittings, such as wheel covers, window frames and other decorative trim. These exterior fittings are subject to corrosion attack particularly in northern climates during the winter months due to the water soluble salts which are often applied to road surfaces to mitigate the effects of ice and snow. As a result of these salts, road sludge contains such corrosive elements as sulfates, sulfides, thiosulfates, chlorides, alkaline earths and alkalies. These salts when splashed upon stainless steel parts cause pitting of the stainless steel. Accelerated tests for determining the corrosion resistance of a given stainless steel have been devised and they are a great improvement over time consuming actual road trials. However, such tests still require considerable time, sometimes up to 16 hours, as well as complicated equipment including accurate timing and cycling devices. Accordingly, it is the object of the present invention to provide a rapid, dependable test procedure upon which the corrosion resistance of the stainless steel, when subjected to actual road conditions, may be predicted. Broadly stated, the test of the present invention comprises an electrophoretic procedure which will rapidly generate a corrosive attack on a stainless steel test specimen and visually display the severity of same, so that the relative corrosion resistance of the stainless steel may be readily determined. More specifically, the electrophoretic test procedure of the invention comprises passing an electric current through a polished and degreased section of the particular stainless steel to be tested while it is in contact with a gelatin or other water dispersible colloidial gel coated, electrolyte saturated member. The electric current will generate a corrosive attack on the test specimen. The iron salts and chromium salts which are formed as a result of the electrolytic attack will remain embedded in the gelatin when the gelatin coated member and the test specimen are separated, and the extent and quantity of embedded salts is a visually observable measure of the relative corrosion resistance of the test specimen. For best results, the imbedded iron and chromium salts are then extracted and converted to salts which possess a distinctly different color so that the visual observation of the same is simplified. In the practice of the invention, the polished section of the surface to be tested may be thoroughly degreased by any suitable means such as washing with solvents, for example, acetone perchloroethylene, or carbon tetrachloride. Suitable gelatin coated members for use in the practice of the invention include dye transfer paper and the type printing paper used in the photographic industry from which the silver halides have been removed by treatment in a hypophosphite "fixing" solution. The electrolyte is suitably a 0.05 to 0.2 N aqueous solution of a chloride salt of an alkali or alkaline earth metal to which has been added from 0.5 to 3 grams, per liter of solution, of a nitrate composed such that the ratio of nitrate ion to chloride ion is about 6 to 1. An aqueous sodium chloride-sodium nitrate solution which is within the above limits is preferred.

The actual test procedure of the invention comprises intimately contacting a paper member which has been uniformly saturated with electrolyte with a clean and degreased section of a stainless steel specimen to be tested and subjecting the specimen to electrolytic attack by passing a current therethrough, utilizing the specimen as the anode. During this electrolysis period, if the steel is corrosively attacked iron salts and chromium salts will form. When the gelatin coated paper is separated from the test specimen, any iron and chromium salts which were formed will be carried away in the gelatin. The location and size of these salt deposits will correspond exactly to the areas of the stainless steel sample which were attacked and are proportional in number and area to the severity of the attack. The severity of the attack may be graphically illustrated by treating the salts with a potassium ferri cyanide solution to give the salts a distinct bluish color. The test procedure of the invention is most readily accomplished by forming a multi-layer assembly comprising a uniformly electrolyte saturated, gelatin coated member, a test specimen of stainless steel having a polished and degreased surface section, and a thin insulating member interposed therebetween, said insulating member being provided with an opening of know area across which the gelatin coated member and the test specimen are in intimate contact. The purpose of the insulating layer is to insure passage of the electrolyzing current through the specimen at a known current density. The required intimate contact between the gelatin coated paper and the specimen may be suitably obtained by loading the multi-layer assembly with a heavy weight or preferably by compressing it in a small hydraulic press. The invention will be more readily understood by reference to the accompanying drawing wherein the sole figure is a schematic view of a suitable assembly for carrying out the test procedure of the invention. Referring now to the drawing, an insulating layer 10, such as rubber sheet or the equivalent, is placed upon a platinum 12 of a press 14. An electrically conductive member 16 is placed adjacent the insulating layer 10 and the negative pole 18 of the power supply is attached thereto. Adjacent the electrically conductive member 16, a layer of a suitable electrolyte saturated member 20, such as blotting paper, is disposed and this member serves as an electrolyte reservoir. A suitable electrolyte saturated paper 22 which is coated on at least one surface with a layer of gelatin 24 is superimposed on the blotting paper member 20, with the gelatin layer 24 facing upward. The gelatin layer 24 is partially covered with a very thin insulating film 26, such as polyester film, in which an opening 28 of known dimensions has been provided. The opening 28 should be centered with respect to the electrolyte saturated paper 22. When the degreased and
polished surface of the stainless steel specimen 30 to be tested is placed in contact with the gelatin layer 24, on the electrolyte saturated paper 22, any entrapment of air bubbles should be carefully avoided. The specimen 30, of course, present a somewhat larger surface than the opening 23 in the insulating film 26. Since the test specimen 30 is generally a thin sheet it is preferably backed up by an electrically conductive member 32 so that the load of press 14 may be uniformly applied over the full area of the specimen 30. The positive pole 34 of the power supply is connected to the conductive member 32. Alternatively, if desired, the positive pole could also be applied directly to the test specimen.

After the assembly has been built up as set forth above, sufficient pressure is applied to the assembly to bring the gelatin layer 24 on the electrolyte saturated paper 22 and the test specimen 30 into intimate contact over the entire area of the opening 23, in the insulating film 26. While the specimen 30 and the gelatin layer 24 are in intimate contact, a current of known density is passed through the assembly to form a corrosive attack.

At the end of the electrolysis period, the pressure is released, and the electrolyte saturated paper 22 with the gelatin layer 24 are separated from the test specimen 30 and the intervening insulating film 26. The gelatin layer 24 is then treated, suitably by being dipped into or being sprayed with a potassium ferricyanide solution in order to impart a bluish color to any deposited salts. The location and size of these deposited salts will correspond exactly to the areas of the test specimen which were attacked, and are proportional in number and area to the extent of attack.

A specific example of the test of the invention utilizing the illustrated apparatus is the following:

**Example 1**

A multi-layer assembly comprising a AISI No. 430; 0.02 inch thick chromium stainless steel test specimen, a surface portion of which had been degreased with acetone and a layer of dye transfer paper which had been saturated with an aqueous 0.1 N solution of sodium chloride to which 1.0 g. of sodium nitrate per liter of solution was added, the facing surface of the paper having an approximate 0.002 inch thick layer of photographic film type gelatin, was assembled with a .0005 to .001 inch thick polyester insulating film layer between the test specimen and the gelatin surface. The polyester film was provided with an opening having an area of 7 cm², and this opening was centered with respect to other members.

This assembly was placed in the apparatus of the drawing and a load of 100 to 400 p.s.i. was applied to the assembly. While under this pressure, a current density of approximately 0.3 milliampere per square centimeter of exposed test specimen area was passed through the assembly for approximately one minute. The gelatin surface was subsequently dipped into a 1% aqueous potassium ferricyanide solution and held there until no further increase in blue color of the deposited and embedded salt was noted (approximately 15 seconds to 1 minute). The excess potassium ferricyanide solution was washed out in running water and the paper was dried. The paper may be kept as a permanent record, if desired.

The success of the test of the invention was determined by comparing the indicated degree of resistance or attack as measured by the test of the invention with the same as measured by the commonly practiced dip and dry test which consists of alternately dipping the specimen in a special salt solution and drying by blowing at a temperature of 100° F. to 120° F. This dipping and drying is continued for about 8 hours which is approximately 288 complete cycles.

These two tests were applied to a series of chromium steels with the following results:

<table>
<thead>
<tr>
<th>Steel, AISI No.</th>
<th>Invasive Test</th>
<th>Dip and Dry Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>301</td>
<td>Very resistant</td>
<td>Very resistant</td>
</tr>
<tr>
<td>302</td>
<td>Same attack</td>
<td>Heavily attacked</td>
</tr>
<tr>
<td>303</td>
<td>Medium attack</td>
<td>Medium attack</td>
</tr>
<tr>
<td>304</td>
<td>Heavily attacked</td>
<td>Heavily attacked</td>
</tr>
<tr>
<td>305</td>
<td>Medium attack</td>
<td>Medium attack</td>
</tr>
</tbody>
</table>

From the above, it will be appreciated that the test of the invention which may be completed in considerably less than 5 minutes gives the exact same results as the far more complicated and time-consuming dip and dry test.

As further appreciated, while any of the numerical conditions of the test as set forth above may be varied somewhat such as, for example, the current density or the duration of the electrolyzing period, depending upon the sensitivity of the particular stainless steel specimen being tested, all the variables should be held constant when comparing the relative corrosion resistance of a series of test specimens.

We claim:

1. A test for determining the relative corrosion resistance of a stainless steel specimen, said test comprising the steps of preparing a polished and degreased surface section of said stainless steel specimen, uniformly saturating an absorptive member, which has a gel coating on at least one surface, with a 0.05 to 0.2 N aqueous electrolyte solution of a chloride salt, chosen from the group consisting of alkaline salts and alkaline earth metal salts, to which has been added from about 0.5 to about 3 grams, per liter of solution of a nitrate compound such that the ratio of nitrate ion to chloride ion is about 6 to 1, intimately contacting the gel coated surface of said electrolyte saturated member and said polished and degreased surface section of said stainless steel specimen, making the stainless steel specimen anodic by passing a direct current of known density through said stainless steel specimen while maintaining said intimate contact, and then chemically treating any iron salts and chromium salts which were formed as a result of said direct current and which will be embedded in said gel to convert them to salts having a highly distinct and visible color.

2. The test of claim 1 wherein said electrolyte solution is an aqueous solution of sodium chloride and sodium nitrate and said deposited iron salts and chromium salts are treated with a potassium ferricyanide solution to impart a bluish color thereto.

3. A test procedure for determining the relative corrosion resistance of a stainless steel test specimen, said procedure comprising forming a multi-layer assembly which comprises an absorptive member which has a gelatin coating on at least one surface and which is uniformly saturated with a 0.05 to 0.2 N aqueous electrolyte solution of a chloride salt, chosen from the group consisting of alkaline salts and alkaline earth metal salts, to which has been added from about 0.5 to about 3 grams, per liter of solution of a nitrate compound such that the ratio of nitrate ion to chloride ion is about 6 to 1, a test specimen of stainless steel having a polished and degreased surface section, and a third immersing member interposed between the absorptive member and the specimen, said insulating member being provided with an opening of known area across which the gelatin coated member and the test specimen are in intimate contact making the stainless steel specimen anodic by a direct current of known density through said stainless steel specimen while maintaining said intimate contact, separating said member with said gelatin layer from said test specimen, and said insulating immersing member, and then chemically treating any iron salts and chromium salts which were formed as a result of said electric current and which will be embedded in said gelatin
to convert them to salts having a highly distinct and visible color.

4. The test procedure of claim 3 wherein said electrolyte solution is an aqueous solution of sodium chloride and sodium nitrate and said deposited iron salts and chromium salts are treated with a potassium ferrocyanide solution to impart a bluish color thereto.

5. The test procedure of claim 4 wherein intimate contact is provided by compressing said multi-layer assembly in a press.

6. The test procedure of claim 5 wherein an electrolyte reservoir member is disposed adjacent said electrolyte saturated absorptive member.

7. The test procedure of claim 4 wherein said electrolyte saturated absorptive member is paper, said insulating member is polyester film, and said direct current is at a density of approximately 0.3 milliamperes per square centimeter of exposed test area.

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