

In Situ Electrochemical Testing Of Stainless Steel Surfaces

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ABSTRACT

A novel approach for performing electrochemical testing on in-situ stainless steel surfaces is presented. This approach involves the use of an absorbent pad placed on the test surface to facilitate the application of standard electrochemical test methods. A detailed description of the experimental procedure is provided. The test method has the advantages of being nondestructive and can be used as a field test. Examples of the successful use of this approach for the in-situ evaluating the pitting resistance of 316L pharmaceutical tanks and for nondestructive detection of sigma phase in cast duplex stainless steel are provided. Other possible applications and limitations of the test method are discussed.

Key words: In-situ testing, electrochemical testing, stainless steel, pitting resistance, sigma phase

INTRODUCTION

Electrochemical testing has been a valuable tool for investigating the corrosion performance and corrosion mechanisms of stainless steels and other corrosion resistant alloys. Over the past 40 years many electrochemical test methods have been developed and are now adopted as ASTM Standards. For example, potentiodynamic anodic polarization measurements⁽¹⁾, cyclic polarization measurements for localized corrosion susceptibility⁽²⁾, electrochemical critical pitting temperature measurements⁽³⁾, and electrochemical reactivation measurements for detecting sensitization⁽⁴⁾. Although these test methods are valuable tools for the corrosion specialist, they do have the limitation of requiring a cut coupon to be placed in a laboratory test cell. Because of this, these techniques are either destructive to the component being examined or require significant fabrication procedures to cut coupons and repair the sampled area.

There have been efforts to develop cells for in-situ corrosion testing of metal surfaces and these approaches typically involve the use of clamp-on cells or small portable hand held sensors^(5 - 8). This investigation makes use of an absorbent pad, saturated with the electrolyte of interest, placed on the in-situ test surface to facilitate electrochemical testing. The use of absorbent pads to preform electrochemical processing is not new and has been used for many years in applications such as brush

plating⁽⁹⁾, on-site electropolishing⁽¹⁰⁾, and as a grid marking method on metal sheets⁽¹¹⁾. However, the use of a test cell consisting of an absorbent pad to perform electrochemical testing of metal surfaces is novel. It has the advantage of being simple, cost effective, and can be readily used with existing potentiostatic equipment.

Although various electrochemical tests can be performed using this technology, this paper will only discuss the use of this approach for two situations that are often encountered when using stainless steel equipment and components. The first example, Case Study 1, is the in-situ measurement of the relative pitting resistance of the inside walls of large Type 316L stainless steel processing tanks. The second example, Case Study 2, involves the use of this method to develop an in-situ, non-destructive, test to detect the presences of undesirable intermetallic phase in cast duplex stainless steel couplers.

EXPERIMENTAL PROCEDURE

The procedure for creating a test cell with an absorbent pad is relatively simple and a schematic representation is shown in Figure 1. The first step is to mask off a defined surface area for testing, which can be done using commercially available masks. Once a test area is determined, an absorbent pad is placed over the test area. The absorbent pad should be inert to the test solution and free of contaminants that might impact the testing. Clean, thick cotton pads are very effective for this testing. Other materials such as thick filter paper or laboratory paper wipes can also serve as pad materials. The size of the absorbent pad must be large enough to cover the test area and provide sufficient surface area to allow the placement of the reference and counter electrodes. In order to prevent signals from areas outside of the masked-off region, the absorbent pad must be small enough to stay within the masked-off area. Once the pad is in place, it is saturated with the desired test solution, and the reference and counter electrodes are placed on top of the pad so that the reference electrode is positioned over the test area. Details of the experimental setup are presented in Figure 2.

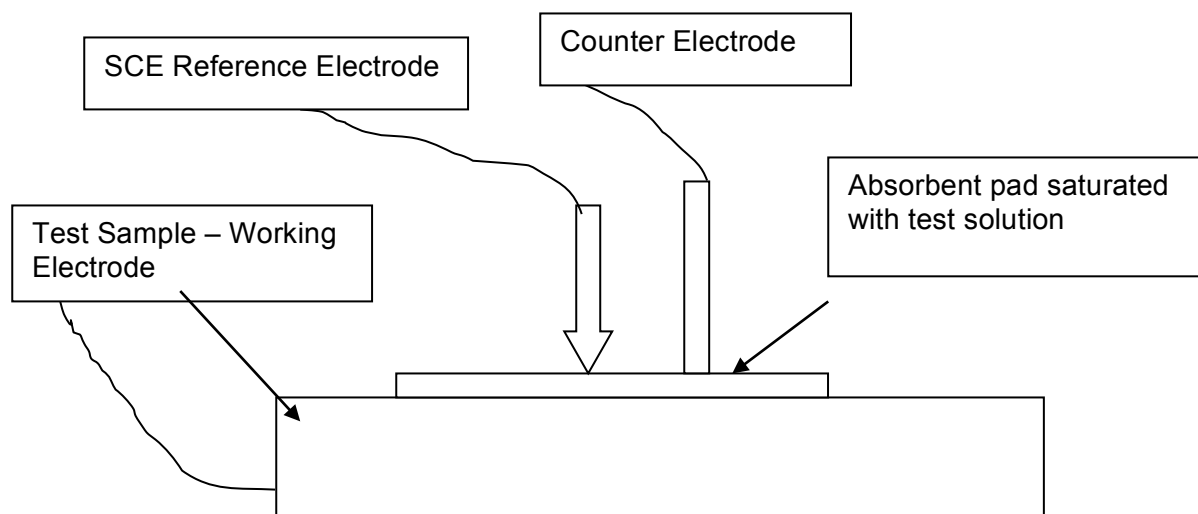


Figure 1: Schematic representation of the experimental setup.

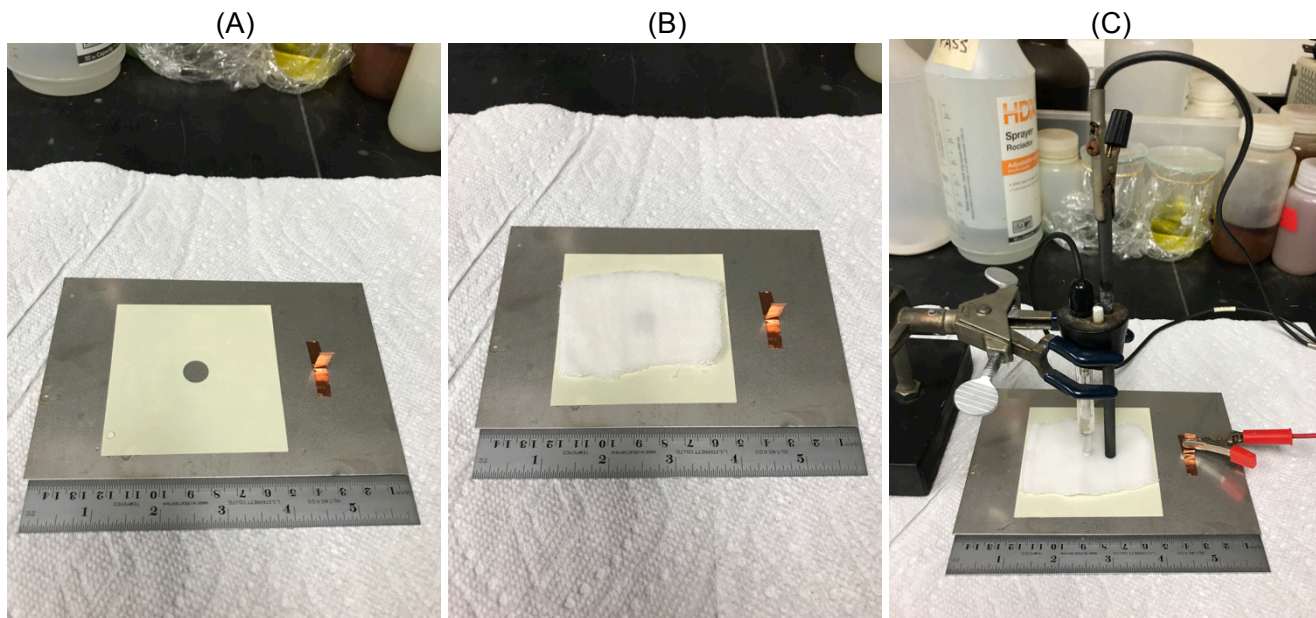


Figure 2: (A) – Masked off test area and copper tape for connection of working electrode lead. (B) – Absorbent cotton pad placed over the test area. (C) – Reference electrode and graphite counter electrode positioned on top of the absorbent pad.

The test cell can now be connected to a potentiostat for electrochemical testing. The component being tested is connected to the working electrode lead. When the test component has a geometric shape that complicates connection to the potentiostat, conductive copper tape can be applied close to the test area to facilitate the connection of the working electrode.

Case Study 1

A pharmaceutical facility that employs many large 316L processing tanks manufactured by various fabricators approached the author to see if the relative corrosion resistance of the inside walls of their tanks could be evaluated without cutting samples from the tank. Based on the performance of their tanks, they suspect that the electropolished surfaces on the inside walls of tanks supplied by one particular fabricator might have poor resistance to chloride pitting. In order to test this possibility, an in-situ test was developed and qualified in the laboratory using an electropolished and passivated 316L plate sample. The passivation treatment used on this 316L sample consisted of a 3-hour exposure to 15 volume % Astro Pak[†] passivation solution at 82.2 °C. This electropolished sample is in the optimal condition for resisting chloride pitting and the sample serves as a benchmark for comparison.

The test procedure used on the 316L sample employs an absorbent pad saturated with an electrolyte of 1000 ppm chloride at a pH of 5.0. A saturated calomel reference electrode (SCE) and graphite counter electrode were used with a Gamry model 600 potentiostat[†]. The test method consists of applying a constant anodic potential for 60 seconds and monitoring the resulting current to determine if the surface remains passive at the selected potential or shows initiation of chloride pitting as demonstrated by an increasing current well above the 10 microamp/cm² range. If the surface remains passive the potential is stepped to a more anodic value and the process is repeated.

The electropolished 316L sample was tested in the laboratory using the absorbent pad at potentials of 500, 700, and 900 mV (vs. SCE) at a temperature of 22 °C. A test area of 1.0 cm² was masked off for all measurements. The masks used in this investigation are the PTC1 PortHoles Electrochemical Sample Masks[†] supplied by Gamry Instruments. These masks were chosen because they are very

[†] Trade name

effective in avoiding crevices during electrochemical testing of stainless steel surfaces. The resulting currents measured during these potentiostatic treatments are shown in Figure 3. This test sample remained passive at 500 and 700 mV as demonstrated by the very low currents and pitted at 900 mV as indicated by the higher currents. These results show that the 316L sample has a pitting potential between 700 and 900 mV measured with this in-situ technique.

The potentiostatic exposure time at each potential was kept to 60 seconds. This short exposure time was chosen so that if any pitting initiates it would only result in a small amount of total charge, which ensures that any pits that may have initiated will be in the very early stages of pit growth. Because of this, areas that do experience pitting during this testing only suffer superficial attack and the attack would not be deep enough to damage the component being tested.

The results from the 316L sample are used as a benchmark for comparison with measurements taken from inside the tanks in question. During a site visit to the pharmaceutical facility the same test procedure was repeated inside two 316L tanks produced by the fabricator in question.

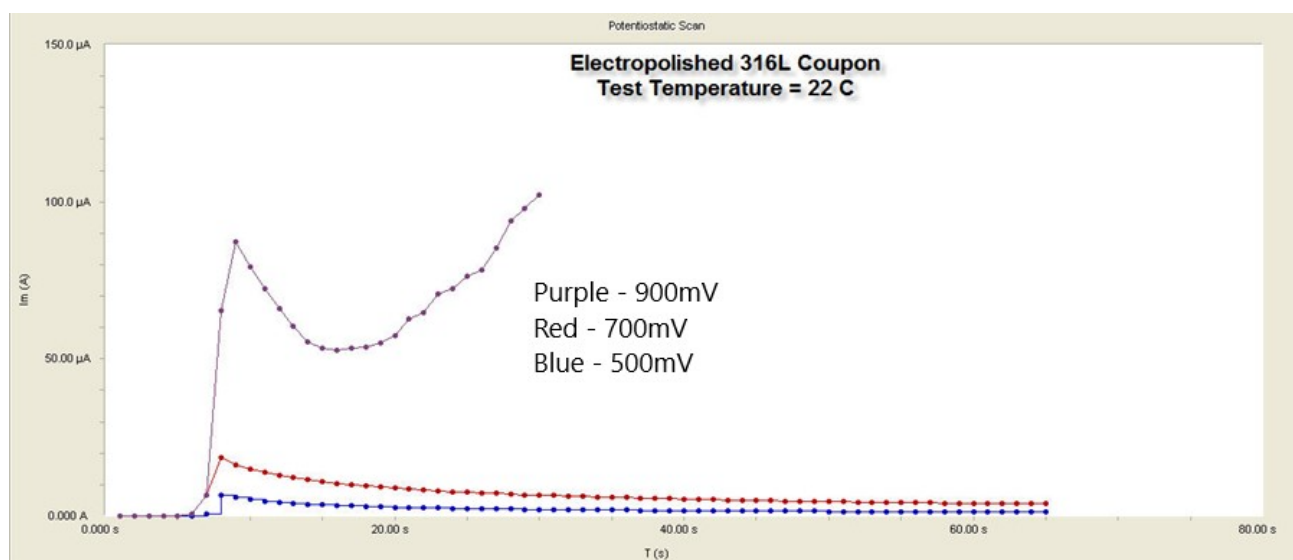


Figure 3: Currents measured during potentiostatic polarization at 500, 700, and 900 mV measured on an electropolished and passivated 316L sample.

Case Study 2

The author was approached by a manufacturer of cast duplex stainless steel couplers to see if a test could be developed that would detect the presence of undesirable sigma phase without destroying the casting. The couplers have sizes from 1.5 to 10 inches and are cast from a duplex stainless steel, ASTM A890⁽¹²⁾ Grade 2A. The coupler manufacturer imports the castings from various sources and occasionally receives product with improper heat treatment resulting in the precipitation of sigma phase in the cast microstructure. The presence of the sigma phase has produced occasional unexpected failures (see Figure 4). Although it was determined that the ASTM A923⁽¹³⁾ Method C ferric chloride test performed at a test temperature of 30 °C readily detects the presence of sigma phase in the A2 castings, this test typically requires the destruction of the casting to obtain a test coupon. Because of this the coupler manufacturer requested the development of an in-situ, nondestructive test for detecting the presence of the sigma phase.

In order to develop a nondestructive test procedure, a solution annealed coupler that was free of sigma phase was sectioned into three portions. One portion was left in the solution annealed condition and the other two portions were heat treated at 899 °C (1650 °F) for 5 minutes and 20 minutes, to produce varying amounts of sigma phase precipitation. These samples were designated HT-0, HT-5, and HT-