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A Comparative Study Of DLEPR Techniques For Cast Austenitic Stainless Steel

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ABSTRACT

Double-Loop Electrochemical Potentiodynamic Reactivation (DLEPR) is a useful technique to measure the degree of sensitization (DoS) in austenitic stainless steels, as it is fast, nondestructive, and accurate in identifying low to medium DoS. While its use has been well-established for wrought austenitic stainless steels, the same is not true for their cast equivalents. A modified version, with more aggressive conditions in order to more selectively cause reactivation in regions that had residual ferrite from solidification, was tested with cast austenitic stainless steel spanning both UNS J92500 and J92600 compositions. Testing included microstructural evaluation of polished surfaces after the electrochemical tests and DoS measurements. The modified DLEPR consistently measured higher DoS values, but the trends in the data were the same for both versions of the test. Microscopy was more difficult with the modified DLEPR, as the surfaces were more severely corroded, somewhat masking smaller features such as inclusions and twin boundaries.

Key words: Corrosion monitoring; Corrosion tests; Stainless steels; Test methods; Electrochemistry; Sulfuric acid

INTRODUCTION

Stainless steels are susceptible to sensitization in the temperature range of 500-900°C.¹⁻³ This is caused by the formation of Cr-rich $M_{23}C_6$ (carbide) particles on the grain boundaries (GBs), resulting in a local depletion of Cr in the surrounding area, leaving it vulnerable to corrosive attack in the form of intergranular corrosion (IGC).⁴⁻⁹

Electrochemical potentiodynamic reactivation (EPR) is a common method to evaluate the susceptibility of stainless steels to IGC caused by sensitization. ASTM⁽¹⁾ G108 is a standard method of EPR for use with wrought austenitic stainless steels, but it requires a 1 μ m surface finish to give reliable results and is strongly affected by the grain size of the sample.^{10, 11} A more robust EPR method is the Double-Loop

EPR (DLEPR) test and is insensitive to surface finish and only slightly sensitive to grain size.^{10, 12} These methods are stated to only apply to wrought austenitic stainless steels, such as UNS S30400 (304) and S30403 (304L).¹⁰⁻¹² In the literature, a modified DLEPR method exists for the cast equivalents of 304 and 304L, UNS J92600 (CF-8) and J92500 (CF-3) respectively. It is more aggressive in both its electrolyte and test parameters in order to compensate for the variations in microstructure that occur from casting.¹³ The cast materials often have residual ferrite due to the sequence of solidification, and no cold- or hot-working is done to homogenize the structure to pure austenite.¹⁴⁻¹⁷ As a result of the presence of ferrite, which contains higher Cr concentrations than austenite, the cast materials can passivate more strongly, necessitating the use of more aggressive corrosion tests.¹³

EXPERIMENTAL PROCEDURE

Samples of CF-8 and CF-3 were created by melting ingots of CF-3 and adding carbon in the form of pig iron, then pouring the metal into nobake sand molds. The compositions of the samples used in this work are shown in Table 1.

Table 1

Measured compositions of the chemistries poured, in wt%, balance Fe. Measured values represent average $\pm$ 95% Confidence Interval (CI).

Heat	C	Mn	Si	S	P	Cr	Ni	Mo	Cu
CF-3	0.016 ^a	0.77 $\pm$ 0.01	1.07 $\pm$ 0.00	0.000 ^b	0.034 ^b	18.1 $\pm$ 0.1	9.8 $\pm$ 0.1	0.23 ^c	0.19 ^c
CF-8	0.054 ^a	0.54 $\pm$ 0.02	0.77 $\pm$ 0.03	0.000 ^b	0.034 $\pm$ 0.001	17.7 $\pm$ 0.0	9.7 $\pm$ 0.0	0.23 ^c	0.19 ^c

^a Measurements did not report multiple runs, so the standard deviation and thus 95% CI could not be calculated

^b Measurements had a standard deviation of 0, so a 95% CI could not be calculated.

^c $\pm$ 0.00

The castings were plates measuring 3.5 in (8.9 cm) wide, 4.5 in (11.4 cm) long, and 0.375 in (0.953 cm) thick. These plates were then waterjet cut into square samples measuring 0.75 in (1.9 cm) on a side. In accordance with ASTM⁽¹⁾ A743, the samples were solution-annealed at 1065°C followed by a water quench.¹⁸ The samples were given a heat treatment intended to cause varying amounts of sensitization: 610°C for 23, 45, 90, and 180 minutes. Before corrosion testing, the samples were ground to a 600 grit finish, with samples used for microscopy polished to a 0.05 μ m finish, all per ASTM⁽¹⁾ E3-11.¹⁹

DLEPR tests were conducted in a Princeton Applied Research 4-channel Versastat MC Potentiostat/Galvanostat[†] using a flat cell test setup shown in Figure 1. The setup used a three-electrode configuration and a saturated calomel electrode (SCE) with KCl.

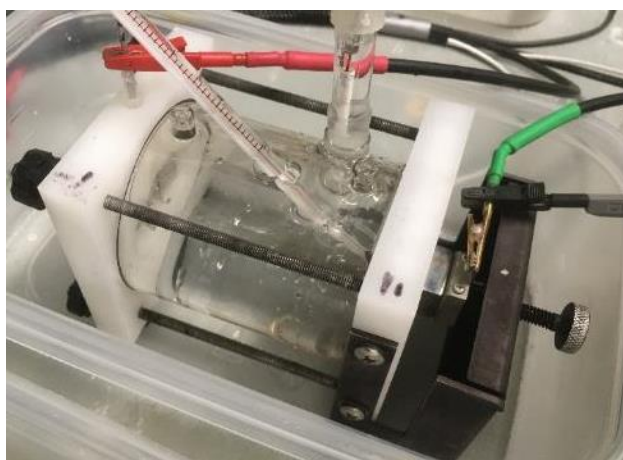


Figure 1: Flat cell setup, showing sample, reference and counter electrodes, thermometer, and leads.

The electrolyte for the wrought-only test (Akashi) was the same as ASTM⁽¹⁾ G108: 0.5 M H₂SO₄ + 0.01 M KSCN at 30±1°C, deaerated with nitrogen.¹¹ For each test, the sample was held at open-circuit (OC) for two minutes, then the potential is scanned anodically at 6 V/h until reaching +300 mV_{SCE}, where it is then scanned in the reverse direction until it reaches -450 mV_{SCE}.^{10, 12}

For the modified method for castings (Reichert), the electrolyte was 0.5M H₂SO₄ + 0.02 M KSCN, the scan rate was 3 V/h, and the maximum potential before reversing the scan was +450 mV_{SCE}.¹³

The data point obtained from a single test was the ratio between the maximum current during the forward scan (the activation current i_a), and the maximum current during the reverse scan (the reactivation current i_r). This i_r/i_a ratio is a good measurement of the degree of sensitization (DoS) of the sample.^{10, 12} For both types of test (wrought and cast), four samples of each chemistry-sensitization combination were tested in order to get valid statistical results.

Optical microscopy was performed on samples after corrosion testing in order to observe the corroded microstructures.

RESULTS AND DISCUSSION

As expected, the Reichert tests produced higher values of both i_a and i_r , owing to the more corrosive nature of the test. Figures 2 and 3 show typical runs for both types of test, with samples that gave low DoS results in Figure 2 and high DoS results in Figure 3. Time is not represented in Figures 2 and 3, so it is worth mentioning here that the Reichert test, due to the wider potential range to scan through and the scan rate being half that of the Akashi test, took more than twice as much time per run (38 minutes average vs. 17 minutes average).

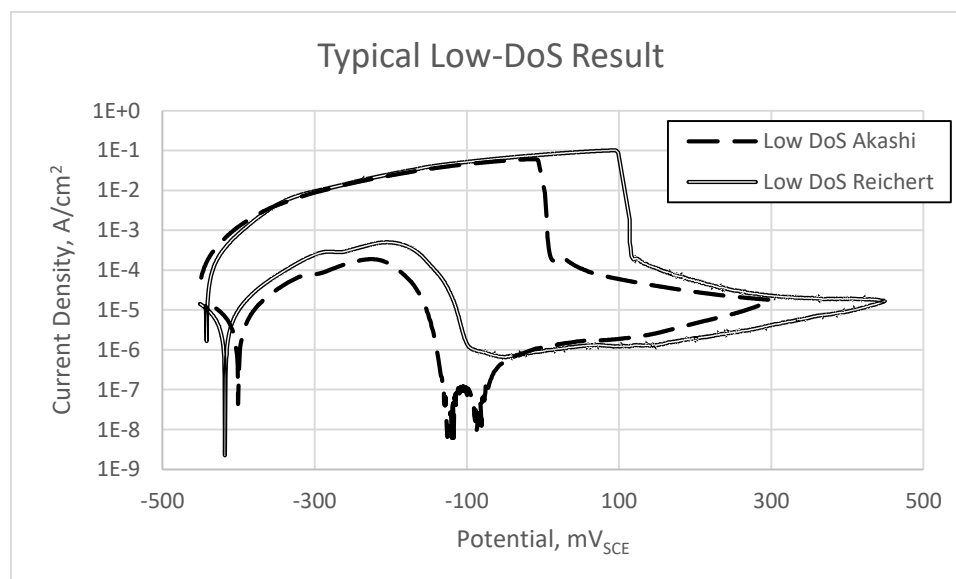


Figure 2: Graph of a typical low-DoS result for both tests, showing the increased i_r and i_a values of the Reichert test as compared to the Akashi test.