RAPID MACROCELL TESTS OF ENDURAMET® 2304 STAINLESS STEEL BARS

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A Report on Research Sponsored by
TALLEY METALS

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THE UNIVERSITY OF KANSAS CENTER FOR RESEARCH, INC.
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ABSTRACT

The corrosion resistance of EnduraMet® 2304 stainless steel bars was evaluated using the rapid macrocell test outlined in Annexes A1 and A2 of ASTM A955-10. Based on the test results, the 2304 stainless steel bars satisfy the requirements of ASTM A955-10.

Keywords: chlorides, concrete, corrosion, macrocell, reinforcing steel, stainless steel
ACKNOWLEDGEMENTS

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INTRODUCTION

This report describes the test procedures and results of rapid macrocell tests to evaluate the corrosion performance of EnduraMet® 2304 stainless steel reinforcing bars. Six specimens are tested in accordance with Annexes A1 and A2 of ASTM A955-10.

EXPERIMENTAL WORK

Materials

Tests were performed on No. 5 (No. 16) EnduraMet® 2304 stainless steel bars. The bars were inspected upon receipt and found to be in good condition.

The 2304 bars underwent pickling prior to shipment. According to the supplier, pickling consists of 30 minutes in hot Cleanox, a product of Henkel Corporation, followed by five minutes in nitric acid. Cleanox is a mixture of hydrofluoric and sulfuric acids with concentrations of 20g/L and 125g/L, respectively. The bars are removed from the acid and rinsed using high-pressure water every 10 minutes while in Cleanox. The temperature is maintained at 130°F for the duration of the process.

The chemical composition of the 2304 stainless steel is given in Table 1:

Table 1: Chemical Composition of 2304 Stainless Steel (Provided by Manufacturer)

<table>
<thead>
<tr>
<th>Material</th>
<th>Cr</th>
<th>Ni</th>
<th>C</th>
<th>Mn</th>
<th>N</th>
<th>P</th>
<th>S</th>
<th>Mo</th>
<th>Si</th>
<th>Cu</th>
<th>Co</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Enduramet 2304</td>
<td>22.81</td>
<td>3.62</td>
<td>0.02</td>
<td>1.68</td>
<td>0.18</td>
<td>0.03</td>
<td>0.01</td>
<td>0.26</td>
<td>0.44</td>
<td>0.29</td>
<td>-</td>
<td>0.002</td>
</tr>
</tbody>
</table>

Experimental Procedures

Rapid Macrocell Test

Six specimens were tested in accordance with the rapid macrocell test outlined in Annexes A1 and A2 of ASTM A955/A955M-10 and detailed in Figure 1. Each bar used
in the rapid macrocell is 5 in. long and is drilled and tapped at one end to accept a 0.5-in.,
10-24, stainless steel machine screw. Bars are cleaned prior to testing with acetone to
remove oil and surface contaminants introduced by machining. A length of 16-gauge
insulated copper wire is attached to each bar via the machine screw. The electrical
connection is coated with an epoxy to protect the wire from corrosion.

A single rapid macrocell specimen consists of an anode and a cathode. The
cathode consists of two bars submerged in simulated pore solution in a plastic container,
as shown in Figure 1. One liter of pore solution consists of 974.8 g of distilled water,
18.81 g of potassium hydroxide (KOH), and 17.87 g of sodium hydroxide (NaOH). The
solution has a pH of about 13.4. Air, scrubbed to remove carbon dioxide, is bubbled into
the cathode solution. The anode consists of a single bar submerged in a solution
consisting of simulated pore solution and 15 percent sodium chloride (NaCl). The “salt”
solution is prepared by adding 176.5 g of NaCl to one liter of pore solution. The

Figure 1: Rapid Macrocell Test Setup
solutions are changed every five weeks to limit the effects of carbonation. The anode and cathode are connected electrically across a 10-ohm resistor. A potassium chloride (KCl) salt bridge provides an ionic connection between the anode and the cathode (Figure 1).

The corrosion rate is calculated based on the voltage drop across the 10-ohm resistor using Faraday’s equation.

\[
\text{Rate} = K \frac{V \cdot m}{n \cdot F \cdot D \cdot R \cdot A}
\]

(1)

where the Rate is given in $\mu$m/yr, and

\(K\) = conversion factor = \(31.5 \cdot 10^4\) amp $\cdot$ $\mu$m $\cdot$ sec/$\mu$A$\cdot$cm$\cdot$yr

\(V\) = measured voltage drop across resistor, millivolts

\(m\) = atomic weight of the metal (for iron, \(m = 55.8\) g/g-atom)

\(n\) = number of ion equivalents exchanged (for iron, \(n = 2\) equivalents)

\(F\) = Faraday’s constant = \(96485\) coulombs/equivalent

\(D\) = density of the metal, g/cm$^3$ (for iron, \(D = 7.87\) g/cm$^3$)

\(R\) = resistance of resistor, ohms = 10 ohms for the test

\(A\) = surface area of anode exposed to solution, 39.9 cm$^2$

Using the values listed above, the corrosion rate simplifies to:

\[
\text{Rate} = 29.0V
\]

(2)

To satisfy ASTM A955, no individual reading may exceed 0.50 $\mu$m/yr and the average rate of all specimens may not exceed 0.25 $\mu$m/yr. In both cases, the corrosion current must be such as to indicate net corrosion at the anode. Current indicating a “negative” value of corrosion, independent of value, does not indicate corrosion of the anode and is caused by minor differences in oxidation rate between the single anode bar and the two cathode bars.
In addition to the corrosion rate, the corrosion potential is measured at the anode and cathode using a saturated calomel electrode (SCE). Readings are taken daily for the first week and weekly thereafter.

**RESULTS**

The individual corrosion rates of the six specimens tested are shown in Figure 2, and the overall average corrosion rate for all six specimens is shown in Figure 3. As shown in Figure 2, specimen 4 exhibited the highest corrosion rate by an individual specimen, 0.445 \( \mu \text{m/yr} \) at week 11. As shown in Figure 3, the maximum average corrosion rate was, in fact, negative, \(-0.014 \mu \text{m/yr}\) at week 4. “Negative” corrosion is caused by minor differences in oxidation rate between the single anode bar and the two cathode bars. Thus, no individual specimen exhibited a corrosion rate above the allowable maximum of 0.50 \( \mu \text{m/yr} \), and the average corrosion rate was well below the allowable maximum of 0.25 \( \mu \text{m/yr} \).

Individual corrosion potential data taken with respect to a saturated calomel electrode (SCE) for the bars in pore solution with salt (anode) and bars in pore solution (cathode) are shown in Figures 4 and 5, respectively. As shown in Figure 4, the bars in pore solution plus salt show potentials ranging from \(-0.050\) to \(-0.225\) V versus the SCE. The bars in pore solution have potentials, shown in Figure 5, within the range of \(-0.100\) to \(-0.200\) V. ASTM C876 states that a potential more negative than \(-0.275\) V with respect to an SCE (\(-0.350\) with respect to a copper/copper sulfate electrode) indicates a 90% probability that corrosion is occurring. Two important differences between this macrocell test and ASTM C876 prevent a direct comparison of this test to ASTM C876:
Figure 2: Individual Corrosion Rate of 2304 Stainless Steel, Specimens 1-6.

Figure 3: Average Corrosion Rate of 2304 Stainless Steel, Specimens 1-6.
the steel used is a stainless steel alloy instead of conventional steel, and the bars were placed in a pore solution, not concrete. Overall, the average potential, shown in Figure 6, was more negative for bars in pore solution than for bars in pore solution plus salt by 0.020 to 0.040 V throughout the test. This would further explain the apparent negative corrosion values.

**Figure 4:** Individual corrosion potential with respect to SCE. 2304 stainless steel bars in salt solution (anode), specimens 1-6.
**Figure 5:** Individual corrosion potential with respect to SCE. 2304 stainless steel bars in pore solution (cathode), specimens 1-6.

**Figure 6:** Average corrosion potential with respect to SCE. 2304 stainless steel bars, specimens 1-6.
Once the specimens reached week 15, the test was terminated, and the specimens were removed from solution, lightly scrubbed with a soft brush for removal of residual salt, and examined for corrosion. Photographs were taken at each stage. All six specimens showed a discoloration on the anode bars below the liquid level. An example of the discoloration is shown in Figure 7.

Of the six 2304 specimens tested, corrosion products were observed on specimens 3, 4, 5, and 6 before brushing. The most severe corrosion was observed on the top third of the specimen 4 anode bar (Figure 8). Much of the corrosion product and staining on this bar was removed during brushing, providing a clearer picture of the corrosion initiation site, which was located about one inch from the top of the bar (Figure 9). The corrosion products are most likely caused by crevice corrosion at the point where the bar was occasionally in contact with the supporting lid. Crevice corrosion is a function of the test configuration, not macrocell corrosion.

**Figure 7:** Specimen 3 one week after removal from solution, after brushing. Anode bar exhibits discoloration below the liquid level
Figure 8: Specimen 4 (opposite site from that shown in Figure 7) one week after removal from solution. Significant corrosion appears on the top third of the anode bar.

Figure 9: Specimen 4 one week after removal from solution, after brushing. A clear corrosion initiation site is evident, caused by crevice corrosion.

Specimens 3 and 5 exhibited moderate and slight corrosion, respectively, at the top of the bar near the epoxy-coated connection (Figures 10 and 11). Brushing removed much of the corrosion product from the bars, which proved to be staining, deposited from corrosion under the epoxy (Figures 12 and 13). In all likelihood, a galvanic couple formed between the bar and the copper wires under the epoxy. Specimen 6 showed slight staining of the anode along the longitudinal rib 1.5 to 2 in. from the top of the bar (Figure 14) – this was removed with brushing and was possibly caused by staining at a crevice.
formed with the lid. Despite the local galvanic and crevice corrosion, the EnduraMet® 2304 bars satisfied the requirements of Annex A2 of ASTM A955-10.

Photos of all specimens appear in Appendix A.

**Figure 10:** Specimen 3 one week after removal from solution, prior to brushing.

**Figure 11:** Specimen 5 anode one week after removal from solution, prior to brushing.
Figure 12: Specimen 3 one week after removal from solution, after brushing. Moderate corrosion occurred at the top of the bar.

Figure 13: Specimen 5 anode one week after removal from solution, after brushing.
SUMMARY AND CONCLUSIONS

The corrosion resistance of 2304 stainless steel bars was tested in accordance with Annexes A1 and A2 of ASTM 955-10. The following conclusion is based on the test results presented in this report:

The EnduraMet® 2304 stainless steel bars tested in this study satisfy the requirements specified in Annexes A1 and A2 of ASTM 955-10.

REFERENCES


Figure A.1a: Specimen 1, Side A, immediately after removal from solution.

Figure A.1b: Specimen 1, Side A, eight days after removal from solution.
Figure A.1c: Specimen 1, Side A, eight days after removal from solution, after brushing.

Figure A.2a: Specimen 1, Side B, immediately after removal from solution.
Figure A.2b: Specimen 1, Side B, eight days after removal from solution.

Figure A.2c: Specimen 1, Side B, eight days after removal from solution, after brushing.
Figure A.3a: Specimen 2, Side A, immediately after removal from solution.

Figure A.3b: Specimen 2, Side A, eight days after removal from solution.
Figure A.3c: Specimen 2, Side A, eight days after removal from solution, after brushing.

Figure A.4a: Specimen 2, Side B, immediately after removal from solution.
Figure A.4b: Specimen 2, Side B, eight days after removal from solution.

Figure A.4c: Specimen 2, Side B, eight days after removal from solution, after brushing.
**Figure A.5a:** Specimen 3, Side A, immediately after removal from solution.

**Figure A.5b:** Specimen 3, Side A, eight days after removal from solution. Corrosion product from under epoxy collects at the top of the bar is exhibited as well as residual salt.
Figure A.5c: Specimen 3, Side A, eight days after removal from solution, after brushing. Much of the corrosion residue is removed with brushing.

Figure A.6a: Specimen 3, Side B, immediately after removal from solution.
Figure A.6b: Specimen 3, Side B, eight days after removal from solution.

Figure A.6c: Specimen 3, Side B, eight days after removal from solution, after brushing.
Figure A.7a: Specimen 4, Side A, immediately after removal from solution.

Figure A.7b: Specimen 4, Side A, eight days after removal from solution.
**Figure A.7c:** Specimen 4, Side A, eight days after removal from solution, after brushing.

**Figure A.8a:** Specimen 4, Side B, immediately after removal from solution. Crevice corrosion caused by test configuration; it does not reflect on the corrosion resistance of the steel.
Figure A.8b: Specimen 4, Side B, eight days after removal from solution.

Figure A.8c: Specimen 4 anode bar, eight days after removal from solution.

Figure A.8d: Specimen 4 anode bar, eight days after removal from solution, after brushing. Crevice corrosion caused by test configuration; it does not reflect on the corrosion resistance of the steel.
Figure A.8e: Specimen 4, Side B, eight days after removal from solution, after brushing.

Figure A.9a: Specimen 5, Side A, immediately after removal from solution.
Figure A.9b: Specimen 5, Side A, eight days after removal from solution.

Figure A.9c: Specimen 5, Side A, eight days after removal from solution, after brushing.
Figure A.10a: Specimen 5, Side B, immediately after removal from solution.

Figure A.10b: Specimen 5 anode, eight days after removal from solution, prior to brushing.
Figure A.10c: Specimen 5, Side B, eight days after removal from solution.

Figure A.10d: Specimen 5 anode, eight days after removal from solution, after brushing.
Figure A.10e: Specimen 5, Side B, eight days after removal from solution, after brushing.

Figure A.11a: Specimen 6, Side A, immediately after removal from solution.
Figure A.11b: Specimen 6, Side A, eight days after removal from solution.

Figure A.11c: Specimen 6, Side A, eight days after removal from solution, after brushing.
Figure A.12a: Specimen 6, Side B, immediately after removal from solution.

Figure A.12b: Specimen 6 anode, eight days after removal from solution, prior to brushing.
Figure A.12c: Specimen 6, Side B, eight days after removal from solution.

Figure A.12D: Specimen 6, Side B, eight days after removal from solution, after brushing.