CORROSION AND ELECTROCHEMICAL BEHAVIOR OF A LOW ALLOY STEEL IN BORIC ACID SOLUTIONS

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ABSTRACT

The effects of temperature, solution composition and dissolved oxygen on corrosion and electrochemical behavior of A508III low alloy steel in boric acid solutions at 25-95 °C were investigated by weight loss tests, electrochemical measurements and scanning electron microscope (SEM) observations. In aerated solutions, increasing the concentration of boric acid and lithium hydroxide in proportion to the composition of the standard PWR primary water increased the corrosion rates and current densities in the anodic polarization curves, and decreased the EIS values. The shapes of cathodic polarization curves were affected by the solution composition. Corrosion rates of A508III steel in deaerated solutions increased monotonically with increasing temperature in the range of 25-95 °C. Corrosion rates of A508III steel in aerated solutions showed a peak value at about 75°C. Corrosion rate was strongly affected by the dissolved oxygen in the solutions. The corrosion product films formed in aerated solution at 95 °C were compact and uniformly distributed. The films formed in deaerated solution were cracked and less compact.

Keywords: Boric acid, Corrosion, Low Alloy Steel, Electrochemical Behavior, SEM

1. INTRODUCTION

Primary water in pressurized water reactors (PWRs) contains boric acid (H₃BO₃) and lithium hydroxide (LiOH). Boric acid corrosion (BAC) at many pressurized water reactor (PWR) plants has been a concern of utilities for many years because of the large number of potential leakage sources—flanged joints, valve packing, mechanical seals, and fittings. In nuclear power plants, some components such as reactor pressure vessels (RPV), piping, valves, pumps, threaded fasteners and other structural parts are constructed with carbon steels and low alloy steels (LASs) [1-5]. Such materials are usually not in direct contact with primary water, but could be exposed to primary water when stainless steel or nickel-based alloy corrosion resistant layers fail or as a result of small leaks in valves, fittings, etc. that can cause primary water to come in contact with the external surfaces of carbon and low-alloy steel components. Leakage from piping and pressure vessels has resulted in varying degrees of external corrosion of PWR components. There have been many cases of corrosion of reactor pressure vessel (RPV) materials by the leaking primary water [6-10]. Electric Power Research Institute (EPRI) reported some boric acid corrosion issues found in nuclear power plants, summarized the results of previous industry boric acid testing programs, and included a bibliography of related literature [1,2,11]. One instance was the discovery of a corrosion cavity (about 127 mm width, about 178 mm long and through the low-alloy steel RPV head up to the stainless steel cladding) in the vessel head at the Davis-Besse Nuclear Power Station in March 2002 [12-14].

A533B and A508III LASs are often used as RPV materials and other key components in nuclear power plants. Several investigators [12-14] have conducted research studies and presented experimental data on
corrosion rates of carbon and low alloy steels in boric acid solutions of varying concentrations and temperatures, aerated or deaerated, and some other test conditions. Argonne National Laboratory measured the electrochemical potential (ECP) and corrosion rates of RPV A533 steel and A508 steel in boric acid solutions under different operating conditions at temperatures of 95-316 °C [14]. The ECPs of all alloys generally decreased with an increase in temperature. The ECP of A533 Gr-B steel increased slightly as the concentration of boric acid in the solution was increased from 3500 ppm to 36000 ppm at 95 °C. For A533 Gr-B steel, average corrosion rate was ≈40 mm/y in aerated saturated solution of boric acid at 97.5 °C and ambient pressure. The corrosion rate in aerated half-saturated solution was ≈2 times lower than that in saturated solution. The rates in deaerated solution were ≈40% lower than that in aerated solution. The effects of boron concentration in conjunction with temperature and aeration on corrosion rates were investigated by Xu H.Q. and Fyfitch S. [12,13]. They found that the corrosion rates of LAS in concentrated boric acid solutions increased sharply with increasing temperature in the range of temperatures tested (21°C to 100°C). The corrosion rate of A533B low alloy steel varied between a few millimeters to tens of millimeters per year at about 97.5 °C.

In the present work, the effects of solution composition, temperature and dissolved oxygen on corrosion and electrochemical behavior of A508III LAS in boric acid solutions at temperatures from 25 to 95°C were studied by weight loss measurements, electrochemical measurements and surface observations. Studying the corrosion and electrochemical behaviors of low alloy steel in boric acid solutions would be helpful for understanding the fundamental factors and mechanism affecting the corrosion.

2. EXPERIMENTAL

The test material was an A508III low alloy steel for reactor pressure vessel in nuclear power plants. The chemical composition of A508III steel used in this work is given in Table 1. Coupons of 15×13×2 mm size with a 2 mm diameter hole for hanging were used for the corrosion weight loss measurements. The samples were spot-welded to copper wires for electrical contact and then mounted by transparent epoxy with a 10×10 mm exposure area to the test solutions. Prior to measurements, the surfaces were ground mechanically with SiC papers up to 1000 grit, and then rinsed using ethanol and acetone, respectively.

A solution containing 1200 ppm B in H$_3$BO$_3$ and 2 ppm Li in LiOH was used to simulate the composition of a standard PWR primary water. Throughout the text, adding B means adding H$_3$BO$_3$, and adding Li means adding LiOH. The solution with 6000 ppm B in H$_3$BO$_3$ and 10 ppm Li in LiOH was called 5-times concentrated PWR primary water, and the solution with 12000 ppm B in H$_3$BO$_3$ and 20 ppm Li in LiOH was called 10-times concentrated PWR primary water. These concentrated solutions were used to represent the local concentrated solutions near the leaking sites. In some cases boric acid solutions only were used. Test solutions were prepared using analytical grade reagents and deionized water. Test temperatures varied from 25 to 95 °C. There is a limited solubility of boric acid at ambient pressure. The solubility of boric acid at room temperature is 5 g, which increases with increasing temperature. Therefore, 12000 ppm B in H$_3$BO$_3$ cannot be dissolved in water at 25°C. Weight loss tests were performed in two types of solutions: naturally aerated (without bubbling N$_2$) or deaerated solutions (with bubbling N$_2$). Bubbling N$_2$ was used to investigate the effect of dissolved oxygen on corrosion rates. Nitrogen was supplied to the vessel 30 min prior to the immersion test and continued until the sample immersed in the solution for 30 min in order to reduce the effect of O$_2$. The surface films were analyzed using scanning electron microscope (SEM).

The surface areas of weight loss test specimens were calculated and the initial weight $W_0$ (g) were measured. In most weight loss tests, the samples were immersed for 24 h in the test solutions with a volume of 0.2 L. In some tests, other immersion periods were used. The final weight $W_1$ (g) of the specimen after the immersion by removing the corrosion products was measured. Corrosion rates $V_{corr}$ (mm/y) were calculated using Equation (1), where $s$ (mm$^2$) is the exposed area of the specimen, $t$ (h) is the immersion time, and $\rho$ (g/cm$^3$) is the density of the specimen. There were three parallel samples for each test condition. Corrosion rates were calculated by weight loss results.
A three-electrode cell was used for studying the electrochemical behavior of A508III steel. The working electrode was A508III steel, the counter electrode was a platinum plate and the reference electrode was a saturated calomel electrode (SCE) utilizing a porous plug tip. A Luggin capillary with a salt-bridge connected to the reference electrode separated the test solution from the saturated calomel reference electrode. All the potentials were measured and quoted against SCE except for the potential-pH diagram analysis. The measured potentials $E_{corr}(\text{V}_{\text{SCE}})$ were converted to the standard hydrogen electrode (SHE) scale $\phi_e(\text{V}_{\text{SHE}})$, by the expression (2). Potentials vs.SHE were used in the E-pH diagrams.

$$\phi_e = E_{corr} + 0.242 - 7.6 \times 10^{-4} \Delta T$$

Where $\Delta T$ is the temperature difference (in °C) of the salt bridge in the reference electrode (i.e., the test temperature minus ambient temperature) [14]. Potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) plots were measured using Zahner-Zennium electrochemical workstation. Prior to polarization, the working electrode was immersed in the test solution for 30 min to reach a steady open circuit potential ($E_{corr}$) value. Anodic polarization curves were measured at a sweep rate of 100 mV/min from $E_{corr}$ to 1.0 V. Cathodic polarization curves were measured at a sweep rate of 20 mV/min from $E_{corr}$ to -1.1 V. Each test was repeated three times and showed good reproducibility. EIS measurements were conducted at open circuit potentials in a frequency range between 1 MHz and 10 mHz. The perturbation amplitude was 10 mV.

The pH values of test solutions for various temperatures were measured using an oxidized yttrium wire. The electrode potentials of the oxidized yttrium wire in the Titrisol buffer solutions pH at temperatures of 25-90 °C are shown in Figure 1. The electrode potential of the oxidized yttrium wire showed a good linear relationship with pH. The parameters of buffer solutions and the corresponding relation of pH and temperature were referred to the information published by Merck Company [15]. The measured potentials of the oxidized yttrium wire in test solutions at different temperatures were converted to pH values according to the calibration curves in Figure 1.

3. RESULTS

3.1 Corrosion in aerated Solutions at Various Concentrations and Temperatures

3.1.1 The pH values and corrosion rates

Figure 2 shows the measured pH values of test solutions at temperatures of 25-95 °C. The pH value of 1200 ppm B + 2 ppm Li solution decreased with increasing temperature. However, pH increased with increasing temperature in the more concentrated solutions. Corrosion rates based on weight loss results of 24 h immersion for A508III steel in aerated solutions of various concentrations and temperatures are shown in Figure 3. In 1200 ppm B + 2 ppm Li solution, corrosion rate was about 0.0084 mm/y at 25 °C and increased slightly with increasing temperature. Increasing boric acid concentration increased corrosion rates. This suggests that corrosion rates were low in normal primary water at room temperature. Concentrated solutions could lead to enhanced corrosion of LAS. The corrosion rate increased with increasing temperature, reached a maximum value, and then decreased with increasing temperature. The effect of adding LiOH in boric acid solutions on corrosion was insignificant, as shown in Figure 3.

3.1.2 Electrochemical behavior

Values of $E_{corr}$ for A508III steel at 25 °C are shown in Figure 4. $E_{corr}$ in 1200 ppm B + 2 ppm Li solution was stable at about -0.155 V. $E_{corr}$ in 6000 ppm B + 10 ppm Li solution was stable at about -0.628 V. Figures 5 and 6 show the anodic and cathodic polarization curves obtained for A508III electrodes in boric acid with lithium hydroxide solutions at 25 °C, respectively. Increasing solution concentration increased the anodic current density. The shapes of cathodic polarization curves changed with the solution.
composition. In 1200 ppm B + 2 ppm Li solution, there were two potentials $E_2$ and $E_3$ at which the measured current density was zero. The reason will be discussed in section 4. $E_1$ was the measured $E_{corr}$ for low alloy steel. In 6000 ppm B + 10 ppm Li solution, there was only one stable $E_{corr}$. EIS Nyquist and Bode diagrams measured at $E_{corr}$ for A508III steel in boric acid with lithium hydroxide solutions at 25 °C are shown in Figures 7 and 8. The Nyquist plots of A508III steel exhibited two capacitive loops, one at high and intermediate frequencies and the other at low frequencies. In 1200 ppm B + 2 ppm Li solution, the capacitive loop at low frequencies was larger than that at high and intermediate frequencies. The Bode diagrams revealed that increasing concentration of boric acid with lithium hydroxide solution decreased the EIS values.

Values of $E_{corr}$ for A508III steel in various solutions at 70 °C are shown in Figure 9. $E_{corr}$ was stable at about -0.2 V in 1200 ppm B + 2 ppm Li solution and it was about -0.65 V in more concentrated solutions. Figures 10 and 11 show the anodic and cathodic polarization curves for A508III electrodes in boric acid with lithium hydroxide solutions at 70 °C, respectively. Neither current peak nor typical active-passive transition could be observed on the anodic polarization curves. The Nyquist and Bode diagrams measured at $E_{corr}$ for A508III steel in boric acid with lithium hydroxide solutions at 70 °C are shown in Figures 12 and 13. The Nyquist plots exhibited two capacitive loops: one loop at high and intermediate frequencies and the other small one at low frequencies. Increasing concentration of boric acid and lithium hydroxide solution in proportion to the composition of 1200 ppm B + 2 ppm Li solution decreased the values of the electrochemical impedance.

Values of $E_{corr}$ for low alloy steel at 95 °C are shown in Figure 14. $E_{corr}$ of A508III steel in 1200 ppm B + 2 ppm Li solution could be stable at two levels: high potential of about -0.21 V and low potential of about -0.68 V. $E_{corr}$ was stable only at about -0.67 V in 6000 ppm B + 10 ppm Li solution, which was stable at -0.65 V in 12000 ppm B + 20 ppm Li solution. For open circuit states with $E_{corr} < -0.6$ V, increasing solution concentration made $E_{corr}$ move slightly in the positive direction. Figures 15 and 16 show the anodic and cathodic polarization curves for A508III electrodes in boric acid with lithium hydroxide solutions at 95 °C, respectively. Increasing solution concentration increased the anodic current density. For cathodic polarization curves in 1200 ppm B + 2 ppm Li solution at 95 °C, there were two potentials $E_2$ and $E_3$ at which the measured current was zero if $E_{corr} > -0.25$ V. There was only one corrosion potential in concentrated solutions. Figures 17 and 18 give the Nyquist and Bode diagrams measured at $E_{corr}$ at 95 °C. The Nyquist plots exhibited only one capacitive loop in 1200 ppm B + 2 ppm Li solution at a high $E_{corr}$ and in 5-times and 10-times concentrated solutions. Nyquist plots measured in 1200 ppm B + 2 ppm Li solution at two levels of $E_{corr}$ were close in the frequency range of 1M to 1 Hz, which were significantly different in the frequency range of 1 to 10m Hz.

### 3.2 Corrosion in aerated and deaerated Concentrated Solutions

#### 3.2.1 Corrosion rates

Tests were run in aerated and deaerated 12000 ppm B + 20 ppm Li solutions at temperature of 50-95 °C to investigate the causes for the appearance of maximum corrosion rates in Figure 3. Corrosion rates at various temperatures are shown in Figure 19. In deaerated solutions, corrosion rates increased monotonically with increasing temperature. Corrosion rates in aerated solution at first increased with increasing temperature, reached a maximum value at about 75 °C, and then decreased with increasing temperature. Increasing temperature decreased the concentration of dissolved oxygen in aerated solutions, increased the diffusion coefficient of oxygen, and affected the protectiveness of the surface film, leading to two types of corrosion rate vs. temperature correlations for aerated and deaerated solutions. The saturated dissolved oxygen concentration in water was about 8 ppm at 25 °C and about 0.8 ppm at 95 °C. Increasing the solution concentration decreased the saturated dissolved oxygen concentration [16]. Figure 20 shows the changes in the corrosion rates with increasing immersion time from 12 h to 96 h in aerated and deaerated solutions. In aerated solutions, corrosion rates decreased with increasing immersion time and then became stable. This suggests that the dissolution of base metal and oxide film, and the growth of
film achieved a dynamic balance. In deaerated solutions, corrosion rates at first increased with increasing immersion time, and then decreased. This could be attributed to the lack of replenishing of the solution during the test. Fyfitch S. and Xu H.Q. [13] suggested that the solution was becoming saturated with corrosion products (i.e., dissolved iron) during the test, which in turn decreased the corrosion rates.

3.2.2 Surface morphology

The SEM micrographs of the corrosion product films formed on A508III steel after the immersion in aerated and deaerated solutions for 24 h at 95 °C are shown in Figure 21. There were significant differences between the morphologies of the corrosion product films formed in aerated and deaerated solutions. The corrosion products formed in aerated solution were compacted and uniformly distributed. However, several cracks were observed in the micrograph of the films formed in deaerated solution, which caused loose corrosion products that were easy to break off.

After removing the surface corrosion products formed in aerated and deaerated solutions, the surface morphologies of the samples are shown in Figure 22. From the SEM micrographs, no local corrosion sites were found on the surface. The main corrosion type was general corrosion.

4. DISCUSSION

The potential-pH (E-pH) diagrams of Fe-H2O system can be used to analyze the corrosion and electrochemical behavior of A508III steel in the test solutions. The E-pH diagrams of Fe-H2O system at 25, 70 and 95 °C were drawn with HSC Chemistry 6.0 software [17]. The experimental data of solution pH and Ecorr in aerated solutions were marked at each temperature, as shown in Figures 23-25. The measured Ecorr values were converted to values against standard hydrogen electrode (SHE) in the E-pH diagrams. Line (a) was the H+/H2 equilibrium line and line (b) was the O2/H2O equilibrium line. Oxygen reduction reaction could occur when the potential was located under line (b), and hydrogen evolution reaction could occur when the potential was located under line (a). The position of the boundary line changed with the changing of substances contained in the actual system such as dissolved hydrogen, dissolved oxygen and partial pressure of oxygen or hydrogen. In this study, A508III steel in aerated solutions were mainly located in the zone A for active dissolution and zone C for passivation. For A508III steel in concentrated solutions at 25-95 °C, Ecorr was located in zone A, showing primary active anodic dissolution with hydrogen evolution reaction as the main cathodic reaction. For A508III steel in 1200 ppm B + 2 ppm Li solution at 25 and 70 °C, Ecorr was located in zone C, showing passivity state. For A508III steel in 1200 ppm B + 2 ppm Li solution at 95 °C, Ecorr was mainly located in Zone C, but occasionally in Zone A. This behavior is an indication of an unstable state of A508III specimen surfaces. This is most probably due to a lower dissolved oxygen concentration at 95°C than those at 25 and 70°C. A low dissolved oxygen concentration decreased the oxide stability, thus made Ecorr move to zone A in the E-pH diagram for active dissolution. Corrosion rates at each temperature can be explained by E-pH diagram analysis. Increasing the concentration from 1200 ppm B + 2 ppm Li to 6000 ppm B + 10 ppm Li shifted the open circuit state from passivity state to active dissolution state, which resulted in the increase in corrosion rates. Increasing the concentration from 6000 ppm B + 10 ppm Li to 12000 ppm B + 20 ppm Li caused the acceleration of hydrogen evolution reaction on the electrode surface due to a lower pH, which significant increased the corrosion rates. The emergence of zero-current potentials in the cathodic polarization curves was mainly controlled by multiple elemental electrode reactions. From the E-pH diagrams, E1, in the passive zone, presented a balance between the formation and dissolution of oxidized iron surface. The oxygen reduction reaction on the A508III steel surface as a cathodic process coupled with the anodic dissolution of bare metal gives the second zero-current potential (E2), which could be affected by the dissolved oxygen and temperature. The hydrogen evolution reaction (HER) from H+ to H2 on the base metal surface due to cathodic activation and the active anodic dissolution of iron give the third zero-current potential (E3).
In deaerated solutions, increasing temperature effectively accelerated the anodic and cathodic reaction rates, which resulted in an increase of corrosion rate. In aerated solutions, when temperature was lower than 75 °C, anodic and cathodic reaction rates as well as corrosion rates all increased with increasing temperature. When temperature exceeded 75 °C and continued to rise, dissolved oxygen concentration had a great influence on the protectiveness of the formed surface films. The protective film would mitigate the further corrosion of the base metal therefore reduced the corrosion rates. As shown in Figure 21, the corrosion product films formed in aerated solution were compact and uniformly distributed. In deaerated solution, the corrosion products were easily detached therefore led to more severe corrosion of the base metal.

CONCLUSIONS

Corrosion rates and electrochemical behavior of A508III LAS in simulated PWR primary water environments at 25-95 °C were investigated. In aerated solutions, increasing the concentration of boric acid and lithium hydroxide in proportion to the composition of the standard PWR primary water increased the corrosion rates and current densities in the anodic polarization curves, and decreased the electrochemical impedance values. The shapes of cathodic polarization curves were affected by the solution composition. Corrosion rates of A508III steel in deaerated solutions increased monotonically with increasing temperature in the range of 25-95 °C. Corrosion rates of A508III steel in aerated solutions showed a peak value at about 75°C. This was related to the combinations of temperature and dissolved oxygen on corrosion and film properties. The corrosion product films formed in aerated solution at 95 °C were compact and uniformly distributed. The films formed in deaerated solution were cracked and easy to break off.

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REFERENCES


| Table 1. Chemical Compositions of A508III Steel (wt. %) |
|-------------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Element | C    | Si    | Mn    | P     | S     | Cr    | Ni    | Mo    | Cu    | Fe    |
| Composition | 0.216 | 0.228 | 1.39  | 0.012 | 0.0031 | 0.122 | 0.68  | 0.54  | 0.117 | Bal.  |

Figure 1. Corrosion potentials of oxidized yttrium wire in the Titrisol Buffer Solutions pH at temperatures of 25-90 °C
Figure 2. The pH values of test solutions at temperature of 25-95 °C

Figure 3. Corrosion rates for A508III steel in aerated boric acid solutions or boric acid with lithium hydroxide solutions at various temperatures

Figure 4. $E_{corr}$ for low alloy steel in aerated boric acid with lithium hydroxide solutions at 25 °C
Figure 5. Anodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 25 °C

![Anodic polarization curves](image)

Figure 6. Cathodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 25 °C

![Cathodic polarization curves](image)

Figure 7. Nyquist diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 25 °C

![Nyquist diagrams](image)
Figure 8. Bode diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 25 °C

Figure 9. $E_{corr}$ for low alloy steel in aerated boric acid with lithium hydroxide solutions at 70 °C

Figure 10. Anodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 70 °C
Figure 11. Cathodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 70 °C

Figure 12. Nyquist diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 70 °C

Figure 13. Bode diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 70 °C
Figure 14. $E_{corr}$ for low alloy steel in aerated boric acid with lithium hydroxide solutions at 95 °C

Figure 15. Anodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 95 °C

Figure 16. Cathodic polarization curves obtained for A508III steel in aerated boric acid with lithium hydroxide solutions at 95 °C
Figure 17. Nyquist diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 95 °C

Figure 18. Bode diagrams for A508III steel in aerated boric acid with lithium hydroxide solutions at 95 °C

Figure 19. Corrosion rates for A508III steel in aerated and deaerated 12000 ppm B + 20 ppm Li solutions at temperature of 50-95 °C
**Figure 20.** Corrosion rates vs. immersion time in aerated and deaerated 12000 ppm B+ 20 ppm Li solutions

**Figure 21.** The SEM micrograph of the corrosion product films formed on A508III steel after immersion in 12000 ppm B+ 20 ppm Li solutions for 24 h at 95°C (a) aerated, (b) deaerated
Figure 22. The surface morphologies of the A508III specimens surfaces after removing the corrosion products formed in 12000 ppm B+ 20 ppm Li solutions. (a) 50°C aerated, (b) 50°C deaerated; (c) 75°C aerated, (d) 75°C deaerated; (e) 95°C aerated, and (f) 95°C deaerated.

Figure 23. The E-pH diagram of Fe-H₂O system and $E_{\text{corr}}$ values in aerated solutions at 25 °C
Figure 24. The E-pH diagram of Fe-H₂O system and Eₘₙ values in aerated solutions at 70 °C

Figure 25. The E-pH diagram of Fe-H₂O system and Eₘₙ values in aerated solutions at 95 °C