



Regular article

Effects of zinc injection on stress corrosion cracking of cold worked austenitic stainless steel in high-temperature water environments

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ABSTRACT

In the nuclear power industry, few data exist to demonstrate the effectiveness of Zn injection in reducing the crack growth rate of primary component materials. This work shows that Zn injection reduced the stress corrosion cracks growth rate of cold worked 316L stainless steel (SS) by 3 times. ZnCr₂O₄ oxide was detected both on the crack walls and at the crack tip, showing that Zn substitution in the spinel-type oxide occurred and mitigated stress corrosion cracking.

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Zn injection is known to have beneficial effects on the reduction of radiation fields in the primary loop of nuclear power plants due to the mitigation of general corrosion rate and substitution for radioactive Co isotopes in the oxide films, which are primarily made of austenitic stainless steels (SS) and nickel base alloys. Zn injection also retards the initiation of stress corrosion cracks [1–3]. A large amount of data from laboratory experiments, test reactors, and field experience demonstrate that Zn incorporates in spinel-type oxides [3–9], creating a ZnCr₂O₄ type inner layer oxide [5–11]. However, the effect of Zn on stress corrosion crack growth rates (CGR) is much less clear due to limited data, data scatter and contradictory results [8,12–16]. Moreover, the compelling evidence does not exist that Zn can diffuse fast enough into a long, tight, growing crack to be present in the oxide film all the way to the crack tip.

Stress corrosion cracking (SCC) of SS is one of the greatest concerns in light water reactor nuclear power plants. The aim of present work is to clarify the effect of Zn addition on the SCC CGR of SS in high-temperature water. The oxides on the crack walls were analyzed by XPS, and oxides at the crack tips were analyzed by STEM-EDS to evaluate the presence of Zn in the crack tip.

The test material was a bar of mill-annealed commercial nuclear grade 316L SS with chemical compositions of 0.014%C, 0.5%Si, 0.012%P, 1.8%Mn, 2.6%Mo, 17.6%Cr, 12.4%Ni and Fe balance (wt%). A one-step compression at 140 °C was applied by forging to produce uniform plastic deformation of about 20%. Standard 12.7 mm thick compact

tension (CT) specimens with side grooves of 5% thickness were prepared in S-L orientation according to standard ASTM E399[17].

SCC CGRs were measured in 325 °C high purity water at a pressure of 15.5 MPa using a reversing direct current potential drop method as described in [18]. Stable water chemistry in the autoclave was maintained by a high pressure metering pump at a refresh rate of two autoclave volumes per hour. The electrochemical corrosion potential was monitored with a Cu/Cu₂O/ZrO₂ reference electrode [19,20] and a Pt coupon as the counter electrode. Water was deaerated with high purity hydrogen gas, which yielded a dissolved hydrogen concentration of 1.6 mg/kg, which ensured that the corrosion potential was low. Some testing was performed in water containing 2 mg/kg dissolved oxygen. Zn, in the form of zinc acetate (Zn(CH₃COO)₂), was injected into the autoclave feed water with a peristaltic pump to a concentration of 60 µg/kg Zn. Water samples were taken at the outlet of the autoclave for offline ICP MS analysis during Zn addition and showed that the Zn concentration was initially low and gradually rose above 50 µg/kg in 16 days. After the test, the specimen was cut into two halves in the thickness direction, one half was further sliced into 5 thin pieces to observe the traverse crack path and prepare a STEM sample that contained crack tips. The other half was fatigue fractured in air to expose the fracture surfaces. The oxides on the crack surfaces were analyzed by an AXIS ULTRA DLD XPS analyzer using Al K α radiation (14.0 kV, 75 W) at a pass energy of 160.0 eV. The C 1s peak from carbon at 284.6 eV was used as a reference to correct the charging shift. STEM-EDS analysis of the chemical composition of the crack tip oxide was performed with a JEOL 2100 (operating at 200 kV) equipped with an Oxford energy-dispersive X-ray (EDX) detector system for elemental imaging.

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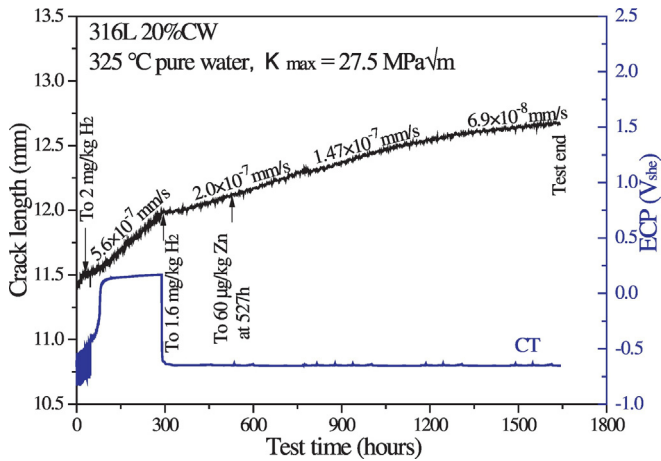


Fig. 1. Crack growth versus exposure time in high-temperature water.

As shown in Fig. 1, the SCC CGR in oxygenated water is very close to the results reported in the previous study [18,21], while the CGR in hydrogenated water is a little higher than that reported in the previous study under the corresponding conditions. Zn injection started after hydrogenated water chemistry in the autoclave was established and a steady state CGR was attained. However, no consequential change of CGR was observed during the initial 200 h after Zn addition at 527 h. However, over time, the crack growth gradually slowed down to 1.47×10^{-7} mm/s and eventually stabilized at 6.9×10^{-8} mm/s for 400 h, when the test was ended. The offline ICP MS measurement shows

that the concentration of Zn in the autoclave inlet water was maintained at about 60 $\mu\text{g}/\text{kg}$ continuously, while in the outlet water, Zn concentration rose slowly to 30 $\mu\text{g}/\text{kg}$ after 200 h and approached about 60 $\mu\text{g}/\text{kg}$ after 500 h of Zn injection. This was attributed to the adsorption of Zn by the large surface area of the system, including the 6 meters long co-axial regenerative heat exchanger tubing, autoclave, and autoclave internals.

The XPS results of the fracture surface films at three areas (Fig. 2(a)) are presented in Fig. 2 (b–e). In Fig. 2(b) the Fe2p3/2 spectra was deconvoluted at the three different analysis areas and shows a binding energy (BE) of 711.0 ± 0.2 eV, which is the characteristic of Fe^{3+} . Fig. 2(c) shows Cr2p3/2 spectra at three areas were deconvoluted into two main components at area 1: the BE signal of 577.1 ± 0.3 eV was attributed to $\text{Cr}(\text{OH})_3$ and the peak at 576.1 ± 0.3 eV to Cr^{3+} , by comparing with published data [22,23]. The Cr^{3+} signal comes from spinel oxides containing Cr, such as NiCr_2O_4 , ZnCr_2O_4 , and NiFeCrO_4 .

Fig. 2(d) shows Ni2p3/2 spectra at the three different regions, which was deconvoluted into two components: a BE peak at 855.5 ± 0.3 eV was assigned to spinel oxide NiFe_2O_4 , NiCr_2O_4 [9,23], and a BE peak at 861.9 ± 0.2 eV, 854 ± 0.4 eV assigned to $\text{Ni}(\text{OH})_2$ [23–25]. The deconvolution of the Ni2p3/2 peak at region 2 was similar to region 1, but the $\text{Ni}(\text{OH})_2$ peak disappeared at region 3 deeper into the crack.

Fig. 2 (e) shows the XPS spectra of Zn2p in the oxide films on 316 L SS. The spectra was deconvoluted into two peaks: one located at BE of 1022 ± 0.1 eV and the other one at a BE of 1045 ± 0.1 eV (ZnCr_2O_4) [9]. No ZnFe_2O_4 was detected, which is consistent with Beverskog's results [26]. Less Zn (lower peak height) was detected in the oxide films in region 3 than regions 2 and 1, suggesting that the Zn concentration in the oxide decreases deeper into the crack. The concentration of Zn incorporated in the oxide is also influenced by its release back into the solution as the autoclave cooled [26].

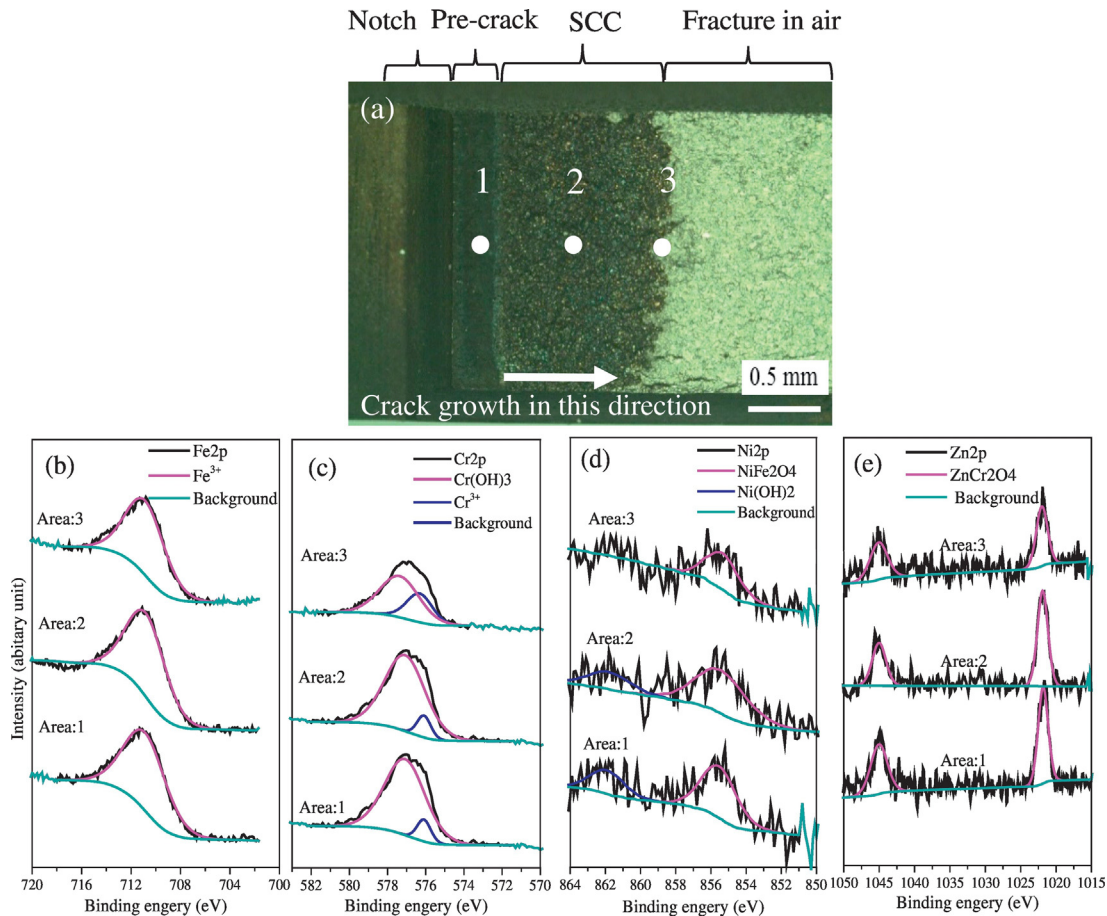


Fig. 2. Representative XPS spectra of Fe 2p, Ni 2p, Cr 2p and Zn 2p of the oxide films on 316L SS after SCC test in Zn-injection solution at 325 °C at three points. (a) the fracture surface of the specimen. (b–e) XPS spectra of Fe, Ni, Cr, and Zn.

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