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Correlation of microstructure and stress corrosion cracking initiation behaviour of the fusion boundary region in a SA508 Cl. 3-Alloy 52M dissimilar weld joint in primary pressurized water reactor environment

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ABSTRACT

Correlation of microstructure and stress corrosion cracking (SCC) initiation behaviour of the fusion boundary (FB) region in a SA508 Cl. 3-Alloy 52M weld joint in primary pressurized water reactor environment was investigated. Cr-depletion at the grain boundary and strength mismatch in the FB region are the two causes for SCC initiation in the FB region. The partially mixed zone between the dilution zone (DZ) of Alloy 52M and SA508 Cl. 3 has a retardation effect on SCC initiation. This is attributed to the decrease of strength mismatch and the alleviation of strain concentration adjacent to the grain boundary in DZ.

1. Introduction

Nickel base alloys such as Alloy 152/52 and Alloy 182/82 have been widely used as key weld metals to attach the stainless steel safe end of primary circuit piping to the solution annealed 508 (SA508) low alloy steel (LAS) reactor pressure vessel nozzle in nuclear power plants, due to their high resistance to corrosion and appropriate intermediate thermal expansion coefficients between those of low alloy and stainless steels. The fusion boundary (FB) region of the LAS-nickel base alloy dissimilar weld joint exhibits a complex microstructure, such as the dilution zone (DZ), the high residual strain adjacent to the FB, and the type-I and type-II boundaries in the FB region, etc. [1-12]. The evolution of type-II boundary that is parallel to the FB was likely due to the allotropic transformation in the base metal [[1–12],3,12,13]. The type-I boundary was the result of epitaxial growth of the grains in the FB region [[1-12],3,12,13], which links the FB to the type-II boundary. Further, the incomplete liquid state mixing in the FB region resulted in the formation of partially mixed zone (PMZ) between the DZ of austenitic alloy and LAS in an austenitic alloy-LAS dissimilar weld joint [11-14]. The PMZ adjacent to the FB mainly consists of martensite due to the formation of intermediate compositions [14]. The martensite in the PMZ that can be predicted by the Schaeffler Diagram showed a higher microhardness as well as more Ni and Cr than LAS [[1–12], [1-12], 9-14].

Due to the complexity of the microstructure of the FB region in a LAS-Alloy 182/82 dissimilar weld joint, a number of investigations have been conducted with a focus on the role of microstructure in the stress corrosion cracking (SCC) behaviour of the FB region [3–8,15–18]. The studies revealed that the type-I and type-II boundaries were a path for crack propagation, while the FB was a barrier to SCC growth in high temperature pure water [3,6–8]. In addition, while the Cr content of the DZ was lower than that of the bulk Alloy 182, the SCC growth behaviour of the DZ was similar to that of the bulk alloy in high temperature pure water with dissolved oxygen (DO) [7,8].

In recent years, Alloy 182/82 was increasingly replaced by Alloy 152/52 in nuclear power plants, which contains more Cr and exhibits better resistance to SCC [19–21]. To the authors' knowledge, there have been no reports on SCC failures of the LAS-Alloy 152/52 dissimilar weld joint during pressurized water reactor (PWR) service, primarily due to a high SCC resistance of Alloy 152/52 in primary water. Nevertheless, a couple of laboratory studies concluded that Alloy 152/52 was not immune to SCC [21–28]. Most of these studies focused on the resistance of

Abbreviations: ASTM E8/E8M-16a, standard test methods for tension testing of metallic materials; CSL, coincidence site lattice; DH, dissolved hydrogen; DO, dissolved oxygen; DZ, dilution zone; EBSD, electron backscatter diffraction; EDX, energy dispersive x-ray; FB, fusion boundary; FEM, finite element method; GTAW, gas tungsten arc welding; HAZ, heat affected zone; KAM, kernel average misorientation; LAB, low angle boundary; LAS, low alloy steel; OM, optical microscope; PMZ, partially mixed zone; PWHT, post weld heat treatment; PWR, pressurized water reactor; RGB, random high angle grain boundary; SA, solution annealed; SCC, stress corrosion cracking; SEM, scanning electron microscope; SSRT, slow strain rate tension; TEM, transmission electron microscope

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the weld metal to stress corrosion crack growth in simulated coolant environments of light water reactors. Investigations also found that the DZ of a LAS-Alloy 52 dissimilar weld joint has a moderate crack growth rate and a higher SCC susceptibility than the bulk weld metal in high temperature pure water with DO [4,5,10]. A more recent investigation of stress-strain behaviour of various regions in a SA508-52M-316LN dissimilar weld joint suggested that the FB region had a high SCC susceptibility in off-normal water chemistry of primary water with DO [12]. These research suggested that, due to the uniqueness of microstructure of the FB region, the SCC behaviour of the FB region for the LAS-Alloy 52M dissimilar weld joint may differ from that of bulk Alloy 52M weld metal in normal PWR primary water with dissolved hydrogen (DH) [12]. For example, the LAS-Allov 152/52 dissimilar weld joint was usually prepared by the automatic gas tungsten arc welding (GTAW). The change of welding procedure and increase of Cr content in Alloy 152/52 could result in a change of microstructure in the FB region such as the variation of chemical composition and the residual strain in DZ, both of which could affect the SCC initiation behaviour of the DZ in primary water. In addition, while the microstructure and mechanical property of the PMZ was different from the base metals, there is still lack of understanding of the role of PMZ in SCC initiation.

The present study aims to investigate the correlation of microstructure and SCC initiation behaviour of the FB region in a SA508 Cl. 3-Alloy 52M dissimilar weld joint in primary PWR environment. The microstructure of the FB region in the dissimilar weld joint was analysed by optical microscope (OM), scanning electron microscope (SEM), electron backscatter diffraction (EBSD), transmission electron microscope (TEM), and Vickers hardness tester. The SCC initiation behaviour of the FB region in simulated primary PWR environment was studied by the slow strain rate tension (SSRT) test, which is a convenient option for evaluating the SCC initiation behaviour of the nickel base alloys and stainless steels in high temperature water [20,29-31]. To correlate the SCC initiation behaviour with the applied strain, the SSRT test was interrupted at four strains of 5%, 7.5%, 10% and 15%. Since characterization of the microstructure in the FB region revealed that there was a local strength mismatch between LAS and Alloy 52M in the FB region, finite element method (FEM) was employed to simulate the tensile stress in the FB region to clarify the effect of the local strength mismatch on the stress distribution during SSRT.

2. Experimental

2.1. Materials and specimen

The SA508 Cl. 3-Alloy 52M dissimilar weld joint used for the study was cut from a mockup of the safe-end weld joint of the primary loop of PWR, as schematically shown in Fig. 1(a). The weld joint was prepared by GTAW (120–160 A, 10–12 V, interpass temperature < $203\,^{\circ}$ C). After welding, post weld heat treatment (PWHT) at 610 °C for 40 h was performed to relieve the residual stress, followed by furnace cooling at a rate of 25-40 °C/h. The composition of Alloy 52M and SA508 Cl. 3 is listed in Table 1. Four small-sized, round SSRT specimens extracted from the same location (i.e., bottom of the dissimilar weld joint) were used to investigate the local SCC initiation behaviour of the FB region, shown in Fig. 1(a). These specimens were in the circumferential orientation and thus the tensile direction was approximately parallel to the FB (Fig. 1(a) and (b)). The gauge section of the specimen is 20 mm in length and 3 mm in diameter. The ratio of length to diameter is 6.7:1, which is higher than the ratio of 5:1 required by ASTM E8 [32]. The specimens were ground using SiC papers up to 3000 grit and finally polished using 1 µm diamond paste. Plate type specimens with a dimension of $15\,\text{mm} \times 15\,\text{mm} \times 1\,\text{mm}$ containing the FB were also extracted from the weld joint for microstructure analysis, shown in Fig. 1(a).

2.2. Microstructure analysis

Metallographic microstructure of the FB region was characterized by OM. The Alloy 52M was electro-etched in 10 wt% chromic acid solution while the SA508 Cl. 3 was etched by 4% nital solution (4 mL of nitric acid and 96 mL of ethanol) for the OM observation. Chemical composition of the FB region was analysed by an FEI-XL30 field emission SEM equipped with an energy dispersive X-ray (EDX) spectroscopy detector.

An AMH43 microhardness tester was used for measuring the Vickers microhardness distribution in the FB region with a load of 50 g and a holding time of 15 s. Before the test, the specimens were slightly etched by 4% nital solution to show the FB and the PMZ.

Grain boundary character, residual strain and cracking path in the FB region were analysed by using an EBSD detector attachment in the SEM equipped with a camera in connection with the TSL software for analysing the misorientation. The specimens used for EBSD analysis were ground using emery papers up to 2000 grit, then polished by diamond paste to 1 μm , and finally polished with a 0.04 μm colloidal silica polishing slurry. Due to a huge difference in the grain size between SA508 Cl. 3 and Alloy 52M, the EBSD analysis was performed with a step size of 1 μm at a voltage of 25 keV to show the overall microstructure of Alloy 52M, while a step size of 0.15 μm was used for precise analyses of the FB region and SA508 Cl. 3. Local strain distribution was assessed using the kernel average misorientation (KAM), which has a linear relationship with the degree of strain [33]. The KAM is a local misorientation defined as an average misorientation of a point with all of its neighbours in a grain.

Grain boundary precipitation and grain boundary chemistry in the DZ adjacent to the FB were analysed using a JEOL 2100 TEM equipped with an EDX spectroscopy detector. The TEM specimens were prepared by a dual-beam focus ion beam -SEM technique using an FEI Quanta 200 3D system.

2.3. SSRT test

The SSRT tests were conducted in a 3-L, 316 stainless steel autoclave with a low flow rate of $100\,\text{mL/min}$ in simulated primary PWR water environment at $320\,^\circ\text{C}$. The primary water was prepared by highpurity water with $1.2\,\text{g/L}$ of B as H_3BO_3 and $2.3\,\text{mg/L}$ of Li as LiOH. The pH of the primary water at $320\,^\circ\text{C}$ is about $7.2\,$ [34]. DH in the influent water was controlled by bubbling H_2 into the water tank until equilibration occurred. The DH concentration used for the test was $2.6\,\text{mg/L}$, in order to make the electrochemical corrosion potential lower into the Ni metal regime [35]. The concentration of DO in the influent water was less than $5\,\mu\text{g/L}$. Conductivity, DH and DO of the influent water were continuously monitored during the test.

SSRT tests were performed on all specimens at a strain rate of $3\times 10^{-7}\,\text{s}^{-1}$ using a stepping motor in the constant displacement rate condition. The crosshead displacement was measured using a linear variable differential transformer to examine the strain rate. In order to correlate the SCC initiation behaviour with the applied strain, in total four interrupted strains at 5%, 7.5%, 10% and 15% were employed for the SSRT tests. Prior to strain the specimens, they were exposed to the simulated primary water for two days to stabilize the environmental condition with a load of about 50 N. This small load helped to ensure that there was no slack in the load train. At each interrupted strain, all specimens were removed from the autoclave for examinations of SCC initiation behaviour. Following the examination, the specimens were then pulled to the next target strain in the primary water after a twoday stabilization of the environment at 320 °C and DH = 2.6 mg/L. It should be mentioned that the SSRT is an accelerated test method that may oversee more corrosion dominated slow initiation mechanisms and underestimate the real SCC susceptibility [36].

Cracking behaviour of the FB region was analysed by counting the number of cracks and measuring the crack length on the surface of the

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