



Effects of phosphorus segregation on stress corrosion cracking in the heat-affected zone of a dissimilar weld joint between a Ni-base alloy and a low alloy steel



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ABSTRACT

Effects of phosphorus segregation on stress corrosion cracking (SCC) in the heat-affected zone of an Alloy 182-A533B dissimilar weld joint in high-temperature, oxygenated water doped with SO_4^{2-} and Cl^- were evaluated by creviced bent beam test. Results indicate that step cooling and the addition of Cl^- promoted SCC propagation and reactivation in the HAZ after cracks reach the fusion boundary. It is also suggested that phosphorus segregation at grain/packet boundaries and precipitate–matrix interfaces have promoted SCC, especially in coarse-grained HAZ and intercritically reheated coarse-grained HAZ.

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1. Introduction

The core shroud in a boiling water reactor (BWR) is a huge stainless steel cylindrical component within the reactor pressure vessel. It confines fuel assemblies, separates feed water, and helps to maintain control rod insertion geometry. It is supported by core shroud support plate, which is welded to low alloy steel (LAS) reactor pressure vessel by filling Ni-base alloy (usually Alloy 182) in between. In recent years, stress corrosion cracking (SCC) of Alloy 182 weld portion in such dissimilar weld joint has occurred in a number of BWR plants in U.S., Europe and Asia [1,2]. Although the cracks were usually confined within Alloy 182 weld metal because of high SCC resistance of LAS in operating BWRs with neutral high-purity (<1 ppb sulfate and chloride) water, concern has been raised on the possible growth of these cracks into LAS reactor pressure vessel when more aggressive combination of material, stress/strain, and environment is present. As a consequence, different water

Abbreviations: BWR, boiling water reactor; LAS, low alloy steel; SCC, stress corrosion cracking; FB, fusion boundary; HAZ, heat-affected zone; GB, grain boundary; IG, intergranular; AFM, atomic force microscope; APT, atom probe tomography; CBB, creviced bent beam; BM, base metal; PWHT, post-weld heat treatment; SC, step-cooled; NSC, non-step-cooled; CGHAZ, coarse-grained heat-affected zone; FGHAZ, fine-grained heat-affected zone; ICCGHAZ, intercritically reheated coarse-grained heat-affected zone; G/PB, grain/packet boundary; P-M, Precipitate–matrix; TG, transgranular.

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chemistries and loading conditions were applied to test the susceptibility of SCC in the Alloy 182-A533B dissimilar weld joint [3–7]. Fast propagation of interdendritic cracks was found parallel to the solidification direction in Alloy 182. Most of the cracks ceased on the fusion boundary (FB) by pitting, but minor crack growth and reactivation of cracks, i.e. cracks reinitiated at the bottom of pits were also observed in the heat-affected zone (HAZ) of A533B [3,4,7]. It is also revealed that high sulfate contents well above the action level 3 of the EPRI water chemistry guidelines [8] did not lead to significant SCC growth in LAS, but the addition of very small amounts of chloride in the range 5–15 ppb (slightly above the EPRI action level limit 1) were already sufficient to induce fast SCC growth into LAS [6]. Of particular interest is that the cracks in HAZ seem to favor grain boundary (GB) and are usually deflected when GBs intersect [3,4,7]. This is indicative of the importance of microstructure on the SCC susceptibility of HAZ. Due to the complex thermal history induced by multi-pass welding, the microstructure of HAZ is highly inhomogeneous. To the author's knowledge, little knowledge is available on the relationship between HAZ microstructure and SCC susceptibility.

On the other hand, it is well known that LAS is susceptible to thermal ageing embrittlement during prolonged exposure in the temperature range of 375–600 °C [9,10]. The cause of this embrittlement is attributed to GB segregation of nonmetallic impurities of groups IV–VI with average composition of 0.005–0.05 wt.%, essentially phosphorus (P) but also tin, antimony and arsenic

[9–11]. Segregation of P to GBs in reactor pressure vessel steels is a relatively common occurrence and has been observed in base material, weld metal, and HAZ [12,13]. In addition, P segregation was found to promote intergranular (IG) SCC of ferritic steels in caustic [14,15] and nitrate [16,17] solutions. Although so far no segregation-induced failure has been reported in BWRs, possible synergism may exist between thermal ageing (especially P segregation) and SCC after prolonged service time of BWRs. To date, little knowledge is available on this aspect and further investigation is necessary for the evaluation/extension of plant service time.

Therefore in this study, a systematic approach is carried out to clarify the potential synergism between thermal ageing, represented by P segregation, and SCC in the HAZ of an Alloy 182-A533B dissimilar weld joint, with a special focus on HAZ microstructure. Step cooling treatment was conducted on part of the dissimilar weld joint to accelerate P segregation to GBs, which was used to represent the material after long-term isothermal ageing at lower temperatures. The difference in the level of P segregation between the acceleratedly-aged material and non-aged one were characterized using atomic force microscope (AFM) and atom probe tomography (APT). SCC behavior of these materials was investigated with creviced bent beam (CBB) test in oxygenated high-temperature water doped with SO_4^{2-} and Cl^- . The results of CBB test were correlated to the P segregation data to evaluate the effects of P segregation on SCC susceptibility of the HAZ of reactor pressure vessel steel.

2. Experimental

2.1. Materials

The dissimilar weld joint investigated in this study was fabricated by filling Alloy 182 into a machined groove in an A533B plate using shielded metal arc welding. Alloy 182 filler metals provided by two different manufacturers were used: INCONEL Alloy 182 rod ($\varphi = 3.2$ mm) for the welding process from bottom of the groove up to a height of 7.2 mm (12 passes), and then YAWATA WELD Alloy 182 rod ($\varphi = 4.0$ mm) for the rest of welding process (39 passes). The nominal chemical compositions of A533B base metal (BM) and Alloy 182 weld metals are presented in Table 1. A typical post-weld heat treatment (PWHT) was applied to reduce residual stresses present after the welding and fast cooling process. This consisted of 25 h at 615 °C and cooling to 315 °C with a cooling rate of ~ 180 °C/h in still air, followed by cooling to room temperature after being pulled out from the furnace. An additional step cooling treatment was conducted on part of the weld joint to promote P segregation [14,18,19]. The sequence for this process was 593 °C/1 h \rightarrow 538 °C/15 h \rightarrow 524 °C/24 h \rightarrow 496 °C/48 h \rightarrow 468 °C/125 h. For the sake of simplicity, the materials that received the step cooling sequence are hereafter denoted as step-cooled (SC) materials, and the others are denoted as non-step-cooled (NSC) materials.

2.2. Characterizations of microstructure and microchemistry

2.2.1. Microstructural characterization

Etching was applied to identify the microstructural morphologies of the HAZ in A533B. One plate specimen containing the dissimilar weld joint from both NSC and SC materials were used for

this purpose. After grinding by emery paper down to 1000 grit, the specimens were polished in four steps with a diamond paste of 9 μm , 6 μm , 3 μm and 1 μm granulation consecutively. Then the specimens were immersed in picric acid-based aqueous solution at room temperature for 3 min to reveal the microstructures of the HAZ.

2.2.2. AFM measurement

As picric acid-based etchants have been reported to preferentially attack prior austenite GBs enriched with P [20–22], the depth of the grooves resulted by etching at GBs were measured by AFM (KEYENCE VN-8000) for both NSC and SC materials, with respect to the different HAZ microstructures identified by etching. The measurement was done at dynamic force mode after immersing the specimens in picric acid-based aqueous solution for 12, 14 and 16 min. The locations of measurement were randomly chosen and ~ 200 GBs were measured for each identified type of HAZ microstructure.

2.2.3. APT test

While AFM provides an averaged estimation of P segregation over a large number of GBs, the actual P segregation behavior was investigated using APT at sub-nanometer scale and later compared to the AFM results. The specimens were prepared separately for the different types of HAZ microstructure identified by etching. The preparation methods are detailed elsewhere [23]. APT tests were performed using a Cameca local-electrode atom probe 3000HR operating in high voltage mode, with a pulse repetition rate of 200 kHz, a 15% pulse fraction, and a sample temperature of 50 K. 3D reconstruction and data analysis of these samples were performed with Imago Visualization and Analysis Software, version 3.6. The segregation level was quantified in terms of Gibbsian interfacial excess [24,25] and enrichment factor, which equals the ratio of the maximum segregation concentration of a solute at GBs over its concentration in the bulk where the GB regions are excluded.

2.3. CBB test

In order to obtain representative data for analysis, CBB test was selected for its potential of producing a large number of cracks. In addition, the constant small strain applied on specimens can enable a more delicate investigation on crack retardation and reactivation.

As shown in Fig. 1(a), two types of specimens of size L35 mm \times W8 mm \times T2 mm were prepared from both NSC and SC materials. The first type contains the dissimilar weld joint with a very thin layer ($< \sim 500$ μm) of Alloy 182 at the top from which cracks are expected to initiate and grow in the direction perpendicular to the FB, while the second type contains only BM of LAS. It should be noted that in the dissimilar weld joint specimens, the distance from specimen surface to the FB is not constant but fluctuates continuously with weld passes. Cold work was introduced at the top surface of Alloy 182 by roughly grinding the specimens with emery paper up to 120 grit, in order to accelerate crack initiation in Alloy 182. The specimens were then sonicated in acetone, and fastened in pair onto each stage of the test fixture (Fig. 1(b)). The upper and lower surface of the fixture contacting the

Table 1
The chemical compositions of LAS base metal and Alloy 182 weld metals (wt.%).

	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	V	Co	Ti	Nb + Ta	Fe
A533B	0.20	0.24	1.41	0.009	0.004	0.65	0.13	0.54	0.12	0.001	/	/	/	Bal.
Inconel Alloy 182	0.053	0.44	6.50	0.004	0.002	68.90	14.70	/	/	/	/	0.52	1.55	7.23
Yawata weld Alloy 182	0.033	0.65	5.70	0.011	0.006	69.95	13.85	/	0.01	/	0.01	0.60	1.70	6.98

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