

Contents lists available at ScienceDirect

Materials Science & Engineering A



journal homepage: www.elsevier.com/locate/msea

Effect of solid solution carbon on stress corrosion cracking of Alloy 600 in a primary water at 360 $^\circ C$



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ARTICLE INFO

ABSTRACT

Article history: Received 9 September 2013 Received in revised form 18 February 2014 Accepted 18 February 2014 Available online 26 February 2014 Keywords:

Alloy 600 Primary water stress corrosion cracking Steam generator tube Solid solution carbon The effect of solid solution carbon in Alloy 600 steam generator tube materials on the stress corrosion cracking behavior was evaluated using microstructure controlled materials with a carbide-free and similar grain size structure in a hydrogenated primary water at 360 °C. Stress corrosion cracking tests were performed using a slow strain rate tensile test method for specimens with a hump in the middle of the gauge length. The time to failure and maximum load of Alloy 600 containing 0.04% carbon increased by about a factor of two relative to those of Alloy 600 containing 0.01% carbon. The role of solid solution carbon was discussed with respect to the interaction between carbon and dislocation during the deformation process.

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1. Introduction

The stress corrosion cracking (SCC) behavior of an Alloy 600 steam generator tube in nuclear power plants has been related with chromium carbides along the grain boundaries. That is, the tube materials with intergranular chromium carbides showed a superior resistance to SCC in both primary and secondary water chemistry. The extent and morphology of the chromium carbide precipitates at the grain boundaries depend on the amount of solid solution carbon and heat treatment conditions [1,2]. A sufficient amount of solid solution carbon is needed to precipitate chromium carbides well decorated at the grain boundaries. The solubility of carbon in the alloy matrix shows a linear relationship to a heat treatment temperature [3,4]. Therefore, to precipitate semicontinuous intergranular chromium carbides along the grain boundaries, the heat treatment temperature after cold work in the tube manufacturing process should be higher than a solvers line of carbon. In a reverse condition, intragranular chromium carbides are predominantly precipitated, resulting in a decrease in the SCC resistance [5,6].

The effect of intergranular chromium carbides on SCC cannot be differentiated from the effect of solid solution carbon or grain boundary carbon. This is because the precipitation of intergranular chromium carbides inevitably accompanies a reduction of the carbon content in the matrix or at the grain boundary. Therefore, it is not clear whether the exact factor affecting SCC resistance is the presence of intergranular chromium carbides or a subsequent reduction of carbon.

There is no consensus regarding the relationship between the amount of carbon in Alloy 600 and the SCC behavior. Some reports have shown that high carbon is beneficial to SCC resistance [7–9], while other reports could not find any relationship between them [10–12]. This seems to be due to a difference in the microstructures of the test materials. That is, low carbon alloys have a grain boundary with a chromium carbide-free and large grain sized structure, whereas high carbon alloys have a structure with chromium carbide precipitates and a relatively small grain size. Since the extent of grain boundary chromium carbides is a governing factor of SCC, the real effect of carbon on SCC cannot be evaluated through these results obtained from the test materials with such different microstructures.

In addition, among the various factors affecting the SCC behavior of Alloy 600, cold working degree and yield strength have been found to have a significant effect on the crack growth behavior SCC of Alloy 600 [13–16]. Especially, degree of cold working correlated with strain concentration at grain boundaries [17]. Cold working path could also accelerate the SCC rate by localization of strain gradients [18,19]. Therefore, test alloys with the same microstructure and stress status should be used for investigations of solid solution carbon effect, to exclude the effects of the grain carbides and residual stresses.

In this work, the effect of carbon on SCC was evaluated using microstructure controlled materials with a carbide-free and similar grain size structure in a hydrogenated primary water at 360 °C.

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2. Experimental methods

2.1. Materials

Alloy 600 steam generator tubes with a carbon content of 0.01% and 0.04% were used in all tests. The tubes had a 22.22 mm outer diameter and 1.23 mm wall thickness. The chemical compositions are listed in Table 1. Each alloy was annealed at 1070 °C to control the microstructure to a similar grain size and dissolve the carbon in the alloy matrix, and was then quenched in water to prevent the chromium carbide precipitation.

2.2. Stress corrosion cracking tests in primary water

Stress corrosion cracking tests were performed using a slow strain rate tensile test (SSRT) method for specimens with a cold deformed hump in the middle of the gauge length. After longitudinally splitting the tubes into four pieces, the tensile specimens were machined as shown in Fig. 1(a). Then, the hump in the middle part of the gauge length of the specimens was made by cold deformation as shown in Fig. 1(b), using a special die in Fig. 2. This type of specimens with a hump was proved to be more reliable and more sensitive to SCC than the reverse U-bend specimen [20,21].

The specimens for SSRTs were set in a 1 l Type 316 stainless steel autoclave connected to a solution circulation system and were electrically isolated from the pull rod by zirconia. Solutions simulating a primary water condition of nuclear power plants were prepared with high purity water, nuclear-grade lithium hydroxide and boric acid. The test solutions contained 2.0 ppm Li and 1200 ppm B. The amount of dissolved hydrogen and oxygen

Table 1

Chemical composition of Alloy 600 tube materials.

	С	Cr	Fe	Ni	Si	Mn	Cu	Ti	S
0.01% C Alloy 600	0.01	15.4	8.0	bal.	0.1	0.3	0.2	0.18	< 0.001
0.04% C Alloy 600	0.04	15.8	8.03	bal.	0.28	0.22	0.12	0.19	< 0.001



Fig. 1. Dimension of SSRT specimen.



Fig. 2. Dimension of hump press die.

was controlled to be 35 cm³ STP/kg of water and below 10 ppb, respectively. The solutions were flowed through the circulating loop in a once-through mode at a flow rate of 160 ml/min. All tests were conducted at a constant extension rate of 0.3 μ m/min at 360 °C.

3. Results

3.1. Microstructure

Fig. 3 shows the microstructures of the two test alloys after etching in a 2% bromine–98% methanol solution. Chromium carbides were observed neither inside the grains nor along the grain boundaries. Some massive particles in this figure were found to be Ti-carbonitrides [22]. An experimental equation related to the carbon solubility curve for Alloy 600, which was proposed by Sarver et al. [3], is as follows:

$^{\circ}C = 1449 + 130.3 \ln (\% \text{ carbon content})$

According to this equation, chromium carbides in the alloy containing 0.04% carbon content can be fully solutionized at 1030 °C. Since the carbon contents of the test alloys were 0.01% and 0.04% and the heat treatment temperature was 1070 °C, all the carbon was expected to be in a solid solution in the matrix.

Fig. 4 shows the grain size distribution measured by an image analyzer. The average grain size was $38 \,\mu\text{m}$ for the 0.01% carbon alloy and $46 \,\mu\text{m}$ for the 0.04% carbon alloy. Both alloys showed almost the same tensile stress–strain behaviors in air at room temperature, as shown in Fig. 5.

3.2. SCC behavior in primary water

Fig. 6 shows the SEM micrographs of the fracture surfaces after the SSRTs. The crack surfaces indicated a significant intergranular nature. The area fraction of intergranular SCC was not affected by the carbon content: 90.1% for 0.01% C alloy and 87.9% for 0.04% alloy. It should be noted that slip bands were observed on the surfaces of the grains, which were predominant in 0.04% carbon alloy.

However, the relationship between the load and displacement measured during the SSRTs showed a significant difference, as shown in Fig. 7. As the carbon content increased from 0.01% to 0.04%, the maximum load and fracture time increased from 50 kg to 95 kg and from 75 h to 139 h, respectively. This means that a low carbon alloy is much more susceptible to SCC in the primary water environment. On the other hand, the SSRTs using the hump

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