Verification of Intraspecimen Method Using Constant Stress Tension Test of Sensitized Alloy 600

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1. Introduction

Stress corrosion cracking (SCC) occurring at the Ni-base alloy 600 used in the nuclear power plant SG tubes and CRDM penetration nozzles had been reported after long-term operation in the harsh environment. Intraspecimen method was developed to predict the SCC initiation time statistically. [1] By dividing a test area into a number of smaller regions (intraspecimens) having homogeneous physical and chemical condition each SCC initiation in each intraspecimen could be counted as an independent outcome to provide enough number of statistical data.

Earlier work of intraspecimen method had many problems in test method and didn't agree with Weibull statistics which is the theoretical base of intraspecimen method. The test method is improved in this intraspecimen test. To find out the root causes of the problems in earlier work and improve the accuracy of intraspecimen method, two kinds of materials are introduced, which are different in grain size but same in chemical composition. Ni-base alloy 600, heat no. J313 and J323 are used as test materials.

Specimens of sensitized Alloy 600 are tested under the condition of constant tensile stress and well defined chemical environment therefore we can easily observe typical intergranular stress corrosion cracking (IGSCC). Material with finer grain (J323) showed the area-dependence in agreement with theoretical prediction. But material with coarser grain (J313) did not show any significant area-dependence. While SCC initiates earlier at grain boundaries that are oriented close to normal to the stress axis, crack initiation time showed no correlation with grain boundary misorientation estimated by Electron Back Scattered Diffraction (EBSD). From the SCC initiation tests with two test materials, it is concluded that the number of grains in an intraspecimen, degree of sensitization and uniform stress distribution are important parameters to meet Weibull statistics.

2. Test Methods

2.1. Test procedure

The material used in this test is alloy 600, with heat number J313 and J323 that had been produced after PWR reactor head penetration nozzle for ICG-EAC-T round robin program. These materials are sensitized at 704°C for 30 minutes and then surface were polished mechanically up to 0.3um alumina powder.

Sensitization behavior was examined by the modified Huey test. The weight loss, yield strength and grain size of sensitized J313 is 40.2 mg/day-cm^2, 365MPa, 310um, respectively, whereas those of sensitized J323 is 10.82 mg/day-cm^2, 373.8MPa, 145um, respectively. The corrosion rate of test materials is adequate to SCC test. [3]

After sensitization, specimen is covered with acryl by screen masking method except test area to avoid the interference between adjacent intraspecimens.

To maintain constant load of 90% of yield strength in one axial tension in the error of ±1N, interface IEEE-488 for Instron was provided to achieve precise load control by computer programming. For the observation of microstructure in real time, a long focal microscope that has focus length of 10~20cm is used.

To accelerate SCC, test environments are managed. The solution used in this test is 0.1M sodium tetrathionate ($Na_2S_4O_8 \cdot 2H_2O$). To control the corrosion potential, stable potential region is measured continuously to confirm the stability of the potential. Saturated calomel electrode is used as reference electrode. Test temperature is held up at 30°C.

After SCC test, finally, the misorientation at a grain boundary between two neighboring grains was examined with EBSD.

2.2. Modified Weibull Distribution

The SCC initiation time has a large scatter, it is often described by the Weibull cumulative distribution function. [3] If we consider the effect of small area of intraspecimen, the Weibull distribution for the crack initiation can be rewritten as following equation (1).

$$F(t) = 1 - \exp\left[-\left(\frac{A}{Ai}\right)^{a_i} \left(\frac{t - t_{oi}}{\theta_i}\right)^{b_i} \right] \qquad (1)$$

Where t_{0i} = incubation time for SCC initiation
θ_i = characteristic time
b_i = shape parameter
A = total test area
A_i = area of an intraspeicmen
a_i = the power to area ratio(A/Ai)

3. Result and Summary

Intraspecimen method was successfully examined by using a constant stress tension test of sensitized Alloy 600 in well defined chemical environment. We performed four tests with two kind of material. Figure 1 and table 1 shows typical one Weibull plot and test summary for crack initiation time.

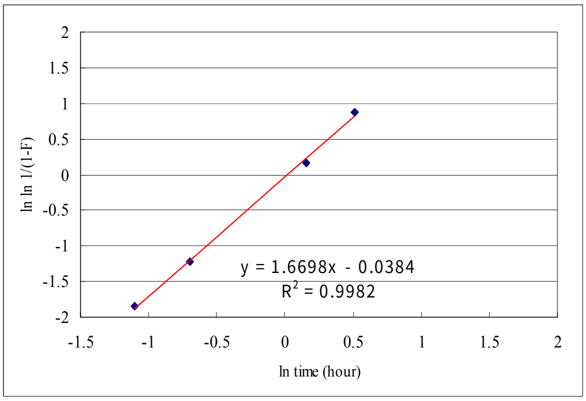

Figure 1. Linear form of Weibull distribution for J313 material test #2

Table 1. The summary of test result

Test #.	Material (heat number)	b (shape parameter)	Θ (characteristictime)
1	J313	1.7	1.0
2	J313	0.6	1.8
3	J323	2.9	3.9
4	J323	2.0	2.4

From EBSD, crack initiation time has lower dependency on grain boundary misorientation than that of our thought. Because we tested highly-sensitized-material, its grain boundary was already susceptible to SCC regardless of misorientation angle of grain boundary.

Due to the limited speed of observation in this work, it is recommended to design the experiment with slower crack initiation. Thus it is need for the future test that the elevated temperature test without a sensitization.

Before EAC test, EBSD analysis should be carried out because of surface deformation and therefore the needs of polishing.

4. Aknowledgement

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