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Full Length Article

The effect of cold rolling on grain boundary structure and stress corrosion cracking susceptibility of twins in alloy 690 in simulated PWR primary water environment

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ABSTRACT

The effects of cold rolling on the structure and stress corrosion cracking (SCC) susceptibility of twin boundaries in alloy 690 were studied. Most of the twin boundaries were transformed into random high angle boundaries (RHABs) following cold rolling. The transformed twin boundaries (TTBs) showed increased susceptibility to carbide precipitation during subsequent aging at 475 °C. The SCC initiation test in 360 °C hydrogenated water indicated that the prior RHABs exhibited increased SCC susceptibility after cold rolling. Moreover, TTBs become susceptible to SCC due to the promoted outward diffusion of Cr and can enhance the connectivity of susceptible grain boundaries.

1. Introduction

Annealing twin boundaries are ubiquitous in low stacking fault energy (SFE) face centered cubic (FCC) alloys. Austenitic stainless steels and Ni base alloys are highly twined alloys that have been widely used in nuclear power plants. The intergranular stress corrosion cracking (IGSCC) resistance of both alloys is an ongoing concern and has been intensively investigated. Twin boundaries have very low energy due to their high degree of order at the interface, providing some superior properties compared to random high angle grain boundaries (RHABs). They are highly resistant to carbide precipitation during aging treatments [1–4] and have much lower IGSCC susceptibility than RHABs [5,6]. Besides twin boundaries, coincidence site lattice boundaries (CSLBs) also have low energy and high resistance to intergranular cracking. The high SCC resistance of CLSBs motivated the development of “grain boundary engineering” by thermal mechanical processes [7] that has been extensively investigated ever since [8,9]. The optimization of the grain boundary structure by grain boundary engineering centers on the twinning process and on twin boundaries. The twin–twin interaction and the interaction between twin boundaries and RHABs could induce the formation of other CSLBs [10]. Recently, research attention has been paid to the connectivity of RHAB networks because the propagation of intergranular cracks can be interrupted at the nodes of the network. Kumar et al. [11,12] proposed that the fraction of CSLBs is not sufficient to account for the optimization of microstructure and the breakup of the connectivity of random boundary network is also critical. Lehockey et al. [13] improved the geometric model for IGSCC susceptibility proposed by Palumbo [14] by discounting neutral twins from the total CSLBs because such twins do not yield other low-Σ CSLBs at neighboring boundaries and have no effect on the connectivity of the network of random boundaries.

A high fraction of CSLBs was found to increase the intergranular corrosion resistance [15–17] and also improve the intergranular cracking behavior of materials [18,19]. However, some researches [5,20] suggest that only low angle and twin boundaries are strong enough to resist cracking. Gertsman and Bruemmer [6] studied the grain boundary character along IGSCC paths in austenitic alloys and found that only twin boundaries were truly special in term of IGSCC resistance while other CSLBs did crack. So it seems that only twin boundaries are reliable barriers for IGSCC.

Some results show that the twin boundary structure can be damaged by cold working induced during fabrication and installation of components. Asgari et al. [21] studied the strain hardening behavior and microstructure evolution of low stacking fault energy FCC alloys and found that during compression tests the twin/matrix interface deviated from the {111} plane in the matrix of the grain, indicating that

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annealing twins lost their coherency. Electron back scattering diffraction (EBSD) measurements on both alloy 600 and alloy 690 from Hou et al. [22,23] suggest that some Σ3 twin boundaries transformed into RHABs and the fraction of special boundaries mainly composed of Σ3 twin boundaries decreased with increasing tensile strain. Zhang et al. [24] analyzed the residual strain and Σ3 twin boundary structure on C-ring alloy 690 samples with EBSD and reported that higher residual strain caused larger deviation from ideal twin boundary misorientation. These results support the observations that cold working can damage the low-energy twin grain boundary structure. The IGSCC behavior of such damaged twin boundaries should be also affected since its high resistance is dependent on its low-energy structure.

This work focuses on alloy 690, which is a widely used structural material in nuclear power plants. In this work, the grain boundary character distribution of this alloy was analyzed before and after cold rolling. The precipitation behavior and IGSCC susceptibility of both samples were studied. Special attention was paid to the boundary structure and performance of twins.

2. Experiment

2.1. Alloy

The chemical composition of alloy 690 studied in this work is 57.6 wt.% Ni, 32.7% Cr, 8.64% Fe, 0.25% Mn, 0.315% Al, 0.08% Si and 0.02% C. The alloy was received as a forged bar with a diameter of 185 mm. It was hot rolled at around 1100 °C to an 8 mm thick sheet, then solution annealed (SA) at 1100 °C for 1 h and water quenched. Some of the stock was cold rolled (CR) to 20% thickness reduction. Part of both SA and CR samples were further aged at 475 °C for 10000 h. Samples are designated by the cold work condition and aging treatments. For example, CR-475-10000 denotes a sample that has been cold rolled and aged at 475 °C for 10000 h, and SA-00-00 denotes a solution annealed sample without aging treatment. The sample conditions used in this work along with the grain size and mechanical properties are listed in Table 1. The difference in grain size is within the statistical error so its possible effect on SCC behavior is negligible. The four samples used in this work were divided into two pairs and each pair contains a cold rolled sample and its non-cold rolled counterpart. As described in our previous studies [25,26], round tensile bars with 20 mm gauge length and 2 mm diameter were machined from the sheet with the sample axis in the cold rolling direction. The samples were mechanically ground to 4000 grit and electropolished for 30 s at 30 V in a solution of 10% (volume fraction) perchloric acid in methanol solution. Some coupons measuring around 8 × 8 × 4 mm were also prepared using the same procedure for grain boundary character and intergranular precipitate analysis. All the samples were cleaned three times alternately with methanol and acetone immediately after electropolishing. No intergranular surface cracks due to electropolishing were found on those coupons.

2.2. Apparatus and methodology

The grain boundary character distribution and precipitates were analyzed using a FEI Helios Nanolab 650 and an EDAX EBSD detector. The analyzed surfaces were perpendicular to the transverse direction of the cold rolled bulk samples. For EBSD measurements, the acceleration voltage was 20 kV and the electron current was 6.4 nA. The binning size was 4 × 4 pixels, the step size was 1 μm, and the scanned surface area was 400 × 600 μm. The EBSD data was analyzed using the TSL OIM software. The rotation angle of low angle boundaries (LABs) was set between 2 and 15 ° and that of RHABs was set above 15°. The rotation angle tolerance for CSLBs was given by $\Delta = 15/\Sigma^{1/2}$ following Brandon's criterion [27]. For twin boundaries, in addition to the rotation angle tolerance defined by Brandon's criterion, the angular tolerance between the {111} twin planes on either side of the boundary was set to 5°. Such restriction provides a partial check that the examined grain boundary is a coherent twin although it is not feasible to strictly differentiate between coherent and incoherent twin boundaries in this software. So the twin boundary defined in this software includes both. Some electron transparent cross sections were made using focused ion beam (FIB) milling on the Nanolab 650. Those samples were used for transmission electron microscopy (TEM) analysis on a JEOL 3011 microscope and for scanning transmission electron microscopy (STEM) analysis on a JEOL 3100 R05 microscope operated at 300 kV and a JEOL 2100F analytical electron microscope equipped with a EDAX energy dispersive detector (EDS).

Constant extension rate tensile (CERT) tests were performed in 360 °C high purity water containing 18 cm^3 (STP) H_2/kg H_2O which provides an electrochemical potential near the Ni/NiO boundary to evaluate the SCC susceptibility of those samples. The experimental details were described in ref. [25,26]. As pointed out in ref. [25], CERT is well suited for assessing the relative SCC susceptibilities between conditions, and by virtue of dynamic straining, is especially effective on SCC resistant alloys like alloy 690. Once the environment was stable, the samples were loaded to just below the yield point at a rate of $1.24 \times 10^{-5}\ s^{-1}$. Thereafter, all the samples were strained at $1 \times 10^{-8}\ s^{-1}$. The choice of such low strain rate was based on the high SCC resistance of this material and the limited uniform elongation of cold rolled samples [25]. It should be mentioned that the applied uniform plastic strain is the determining parameter reflecting the severity of active loading and was found to affect the SCC initiation process directly [28]. So the two samples of each pair were uniformly strained to similar levels, as shown in Table 2. In order to yield a significant amount of cracking, the applied uniform strains on the unaged pair are close to the maximum uniform strain achievable on CR-00-00 which is around 1.9%. The aged pair was strained more as CR-475-10000 showed larger uniform elongation than CR-00-00. After the straining tests, the gauge sections of tensile bars were examined with a JEOL JSM-6480 scanning electron microscope (SEM). More than 40 equally spaced areas on the uniformly strained gauge section were imaged at 1000×. The images were magnified to 4500 × to analyze intergranular (IG) cracks. Crack lengths were measured and the crack numbers were counted. From those data, crack length per unit area, crack density (number of cracks per unit area) and average crack length were calculated.

3. Results

3.1. Grain boundary structure

Fig. 1 shows the grayscale quality maps from EBSD measurements

Table 1
Conditions, grain size and mechanical properties of tested samples.

Sample designation	Condition	Grain size (μm)	Yield strength at 360 °C (MPa)	Uniform strain at 360 °C (%)
SA-00-00	Solution annealed	139 ± 68	186	> 20
CR-00-00	Solution annealed and 20% cold rolled	128 ± 45	840	1.9
SA-475–10000	Solution annealed and aged at 475 °C for 10000 h	144 ± 46	204	> 20
CR-475–10000	Solution annealed, 20% cold rolled and aged at 475 °C for 10000 h	129 ± 21	697	8.7