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Surface grain boundary engineering of Alloy 600 for improved resistance to stress corrosion cracking

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ABSTRACT

In this paper, we demonstrate a novel method for grain boundary engineering in Alloy 600 using iterative cycles of ultrasonic nanocrystal surface modification (UNSM) and strain annealing to modify the near surface microstructure (∼250 μm) for improved stress corrosion cracking (SCC) resistance. These iterative cycles resulted in increased fraction of special grain boundaries whilst decreasing the connectivity of random grain boundaries in the altered near surface region. A disrupted random grain boundary network and a large fraction of low CSL boundaries (Σ3–Σ27) reduced the propensity to sensitization. Slow strain rate tests in tetrathionate solutions at room temperature show that surface GBE lowered susceptibility to intergranular SCC. Detailed analysis of cracks using Electron Back-scattered Diffraction showed cracks arrested at J1(1-CSL) and J2 (2-CSL) type of triple junctions. The probability for crack arrest, calculated using percolative models, was increased after surface GBE and explains the increase in resistance to SCC.

1. Introduction

Grain boundary engineering (GBE) has been demonstrated as a viable method for improving the resistance to creep [1,2], hydrogen embrittlement [3], fatigue [4,5], corrosion [6–8] and stress corrosion cracking [2,9–14] (SCC) in austenitic stainless steels (SS), Ni based alloys and superalloys. GBE involves increasing the frequency of coincident site lattice (CSL) grain boundaries whilst disrupting the random grain boundary network through thermomechanical processing routes. Low grain boundary energy, resistance to grain boundary sliding and intergranular degradation, less susceptibility to impurity or solute segregation are some reasons that contribute to the “special” nature of CSL boundaries.

Thermo-mechanical processing routes involving cold rolling or uniaxial tension/compression and subsequent annealing have been used to increase the frequency of CSL boundaries [6,15]. One approach involves a single cycle of pre-straining the material followed by annealing at comparatively lower temperature for a long time. A multi-cycle approach including steps of moderate strains (6–30%) followed by relatively high temperature annealing for short times has also shown to increase the special grain boundary fraction [16,17]. In addition, the multi-cycle approach results in a disrupted random grain boundary network that correlates to improvements in fatigue, creep and corrosion resistance.

Detailed studies carried out by Bi et al. [18] have established that twin boundaries (especially coherent Σ3) are more resistant to carbide precipitation and corrosion because the atomic structure is highly coherent as compared to high angle grain boundaries. In particular, Σ3 and Σ9 boundaries in grain boundary engineered SS304 have been observed to more resistant to sensitization while Σ27 and other CSL boundaries were not really “special” in terms of their resistance to sensitization and thus intergranular stress corrosion cracking (IGSCC) [19]. Thus, it has been suggested that increased fraction of Σ3 and Σ9 boundaries would likely improve the corrosion and stress corrosion resistance.

Alloy 600 and austenitic stainless steels have been known to be susceptible to stress corrosion cracking (SCC) in polythionic acid environments [20–25]. Susceptibility to SCC at low temperature in tetrathionate and thiosulfate environments has been attributed to Cr depletion in the area surrounding the grain boundary. A reduction in Cr depletion by disrupting the random grain boundary network or increasing the fraction of special boundaries should decrease the susceptibility to sensitization and SCC [6,18,19].

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While GBE has been studied extensively to improve resistance to intergranular cracking, surface GBE has not been explored to the same extent.

In this paper, we propose a novel approach to engineer the near surface region by using ultrasonic nanocrystalline surface modification (UNSM)/ultrasonic peening followed by annealing to increase the fraction of special boundaries. Further, we present and discuss the effect of this surface grain boundary engineered material on the SCC behavior in tetrathionate solution. To the best of our knowledge, there are no studies investigating the effects of surface GBE on SCC behavior of Alloy 600.

2. Materials and methods

2.1. Materials

Alloy 600 plate (2 mm thickness) with chemical composition as shown in Table 1 was sectioned into 15 mm × 15 mm coupons using a wire EDM. The as received material was in annealed condition with a grain size of ~10 μm. UNSM is an advanced surface treatment that uses ultrasonic energy to strike a target (material surface) with a WC tip at a frequency of 20 kHz to induce strain in the near surface region of the material.

The amount of strain can be controlled by modifying the static and dynamic loads. A schematic of the UNSM process is shown in Fig. 1. Static load (P_{st}), amplitude of ultrasonic vibration, scan speed and overlap ratio can be controlled during processing. Details of UNSM have been reported elsewhere in literature [26,27]. For grain boundary engineering, coupons were peened using a LM20 UNSM system (DesignMecha) and subsequently annealed in a lab furnace for 10 min at 950 °C or 1000 °C, then water quenched (WQ). Processing details for surface GBE are listed in Table 2. AR and ARGBE conditions have been grouped together as Set 1 while SA and SAGBE are categorized as Set 2. The static load was 20 N and the amplitude of ultrasonic vibration was 8 μm. A scan speed of 3000 mm/minute and overlap interval of 30 μm was used for UNSM processing in this study.

After GBE treatments, samples were sectioned and cross sections were mounted in a conducting epoxy. For EBSD, each sample was ground to 1200 grit, electropolished in 87.5:12.5 vol% CH_3OH:H_2SO_4 solution at 24 V, 15 s and finally polished with 0.05 μm colloidal silica suspension. EBSD orientation mapping was performed in a FEI XL-30 SEM with step size of 2 μm at 30 kV. OIM scans were analyzed with the TSL OIM Analysis (version 7.1) package to calculate grain boundary character distribution (GBCD), grain size, boundary fractions and triple junction fractions. CSL grain boundaries were categorized according to Brandon criterion of $\Delta\theta \le 15°\Sigma^{-1/2}$ [28]. Boundaries with $3 < \Sigma < 29$ were considered to be CSL boundaries whereas boundaries with $\Sigma > 29$ were considered random high angle boundaries (HABs) and $\Sigma = 1$ as low angle boundaries (LABs). For triple junction analysis, only Σ3, Σ9 and Σ27 were considered as CSL boundaries.

2.2. Residual stress and FWHM

Residual stresses were measured using $\sin^2\psi$ technique with a Proto LXRD system, MnKα radiation and (311) peak of the austenite phase. To measure residual stress through depth, coupons were electropolished using 87.5:12.5 vol% CH_3OH:H_2SO_4 solution to remove 10–50 μm layers. Full width at half maximum (FWHM) data was also recorded for each depth.

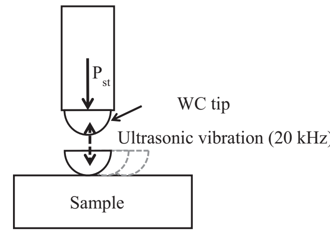

Fig. 1. Schematic for UNSM setup.

Table 2
Designation and corresponding details of processing used in this study.

Designation	Detail
AR	As received
ARGBE	AR+3 cycles of (UNSM+annealing at 950 °C, 10 min, WQ)
SA	AR+Solution annealing at 1050 °C,10 min, WQ
SAGBE	SA+3 cycles of (UNSM+annealing at 1000 °C, 10 min, WQ)

2.3. Double loop electrochemical potentiokinetic reactivation (DLEPR) tests

Baseline and grain boundary engineered samples were given a sensitization treatment at 650 °C, 2 h (water quenched) to induce precipitation of carbides. These samples were mechanically ground to 1200 grit, wet polished with 1 μm diamond suspension and finished with 0.05 μm colloidal silica suspension DLEPR tests were performed in accordance with ASTM G108-94 in a solution composed of 0.01 M H_2SO_4+20 ppm KSCN using a Gamry Potentiostat (Reference 600). Samples were kept immersed in the test solution for 1 h at open circuit potential before the start of each test. The scan rate was set at 0.5 mV/s for activation and reactivation loop and the sample size was 1 cm^2. Freshly prepared solution was de-aerated with high purity Ar gas before and during each test. All tests were performed at room temperature.

The following procedure was used to quantify sensitization in the annealed and GBE material after sensitization [29]. The degree of sensitization is reported as DL-EPR value (designated as R in %) which is the ratio of the current density in reactivation loop to that in the activation loop times 100.

$$R = \frac{\mathrm{Ir}}{\mathrm{Ia}} \times 100 \tag{1}$$

The DL-EPR value obtained is normalized with various parameters like grain boundary area (GBA), grain size, mean lineal intercept length (MIL). It should be noted that twins have been excluded from grain size analysis. The DL-EPR value of a given alloy condition (with ASTM grain size number of G′) is normalized with the grain size (with ASTM grain size number of G) of the as-received material (SA) and is given by:

$$R' = R \times \sqrt{2^{G'-G}} \tag{2}$$

The DL-EPR values were also normalized with grain boundary area (S_v, expressed in mm^2/mm^3):

Table 1
Chemical composition of the Inconel Alloy 600 used in this study.

C	Mn	Si	S	Cr	Fe	Co	Cd	Ti	Cu	P	Al	Ni
0.08	0.16	0.18	0.001 max.	15.05	8.05	0.16	0.01	0.18	0.1	0.001 max.	0.08	Bal.