Iterative Thermomechanical Processing of Alloy 600 for Improved Resistance to Corrosion and Stress Corrosion Cracking

Abhishek Telang¹, Amrinder S. Gill², Mukul Kumar³, Sebastien Teyssyre⁴, Dong Qian⁵, S.R. Mannava¹, Vijay K. Vasudevan¹

¹Department of Mechanical and Materials Engineering, University of Cincinnati, Cincinnati, OH, USA 45221-0072

²AK Steel, Research Center, 705 Curtis Street, Middletown, OH, USA

³Lawrence Livermore National Laboratory, Livermore, CA, USA

⁴Idaho National Laboratory, Idaho Falls, ID, USA

⁵The University of Texas at Dallas, Richardson, TX, USA

Abstract

The effects of thermomechanical processing (TMP) with iterative cycles of 10% cold work and strain annealing, on corrosion and stress corrosion cracking (SCC) behavior of alloy 600 was studied. The associated microstructural and cracking mechanisms were elucidated using transmission (TEM) and scanning electron microscopy (SEM), coupled with precession electron diffraction (PED) and electron back scatter diffraction (EBSD) mapping. TMP resulted in increased fraction of low coincident site lattice (CSL) grain boundaries whilst decreasing the connectivity of random high angle grain boundaries. This disrupted random grain boundary network and large fraction of low CSL boundaries reduced the propensity to sensitization, i.e. carbide precipitation and Cr depletion. After TMP, alloy 600 (GBE) also showed higher intergranular corrosion resistance. Slow strain rate tests in 0.01 M Na₂S₄O₆ solution at room temperature show TMP lowered susceptibility to intergranular SCC. To better understand the improvements in corrosion and SCC resistance, orientation maps of regions around cracks were used to analyze the interactions between cracks and various types of grain boundaries and triple junctions (TJs). Detailed analysis showed that cracks were arrested at J1 (1-CSL) and J2 (2-CSL) type of TJs. The probability for crack arrest at special boundaries and TJs, calculated using percolative models, was found to have increased after TMP, which also explains the increase in resistance to corrosion and SCC in GBE alloy 600. A clear correlation and mechanistic understanding relating grain boundary character, sensitization, carbide precipitation and susceptibility to corrosion and stress corrosion cracking was established.

1. Introduction

Grain boundary engineering (GBE) has been demonstrated as a viable method for improving the resistance to corrosion [1–3] and stress corrosion cracking (SCC) [4–8] in austenitic stainless steels (SS) and Ni based alloys (alloy 600, alloy 690). GBE involves increasing the frequency of coincident site lattice (CSL) grain boundaries whilst disrupting the random grain boundary network through thermo-mechanical processing (TMP) routes. These routes involve cold rolling or uniaxial tension/compression followed by annealing and have been used to increase the frequency of CSL boundaries [1,3,9–11]. One approach involves pre-straining the material followed by annealing at comparatively lower temperature for a long time (24-72 hours). Another multi-cycle approach uses two or more iterations of cold work (6-30%) followed by relatively high temperature annealing for short times (10-30 minutes) and has also been shown to increase the low CSL boundary fraction [11,12]. In addition, the multi-cycle approach results in numerous disruptions in the random high angle grain boundary (HAB) network. Further, a significant improvement in the corrosion and stress corrosion cracking has been attributed to these changes in the microstructure [13,14].

Alloy 600 and austenitic stainless steels have been known to be susceptible to SCC in polythionic acid environments [15–18]. 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/low CSL boundaries decreases the susceptibility to SCC [19].

Twin boundaries are more resistant to carbide precipitation and corrosion because the atomic structure is highly coherent as compared with high angle grain boundaries. In particular, $\Sigma 3$ and $\Sigma 9$ boundaries in GBE SS304 have been observed to be more resistant to sensitization, whereas $\Sigma 27$ and other CSL boundaries were not really “special” in terms of

their resistance to sensitization and thus intergranular stress corrosion cracking (IGSCC). Thus, it has been suggested that increased fraction of $\Sigma 3$ and $\Sigma 9$ and other low CSL boundaries would likely improve the corrosion and SCC resistance [20–23].

The purpose of this study was to evaluate the susceptibility of grain boundaries of known character to intergranular corrosion and their resistance to SCC. In this study, we use multi-step TMP to increase the fraction of low CSL boundaries. Electron back-scattered diffraction (EBSD) is used to characterize changes in the microstructure after TMP. Transmission Electron microscopy (TEM) and Precession Electron Diffraction (PED) were also used to characterize the microstructure and precipitation of carbides after sensitization. Double loop Electrochemical Potentiokinetic Reactivation (DLEPR) and intergranular corrosion tests were used to evaluate the corrosion resistance. We also performed slow strain rate tests to evaluate the susceptibility to intergranular SCC after TMP. EBSD mapping of regions around crack tips after DLEPR tests, corrosion (ASTM G28) tests and interrupted slow strain rate tests (SSRTs) were used to study the interaction of cracks with various types of boundaries and triple junctions to explain the improvements in grain boundary engineered Alloy 600.

2. Materials and Methods

2.1 Thermo-mechanical processing and characterization

Alloy 600 plate (2 mm thickness) with chemical composition as shown in Table 1 was given an annealing treatment at 1050°C for 10 minutes and then water quenched (WQ). For TMP, these plates were cold rolled 10% and subsequently annealed in a furnace for 10 minutes at 900 °C or 1000 °C, then water quenched. This cycle was repeated 3 times to obtain 2 heats of thermo-mechanically processed Alloy 600, designated GBE1 and GBE2. Processing details for all conditions are listed in Table 2.

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.	14.99	8.05	0.18	0.01	0.18	0.1	0.001 max.	0.08	Bal.

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

Designation	Detail
SA	As received + Annealing at 1050°C, 10min, WQ
GBE1	SA + 3 cycles of (10% cold work + 1000°C, 10min, WQ)
GBE2	SA + 3 cycles of (10% cold work + 900°C, 10min, WQ)

For EBSD, each coupon was electropolished in 12.5:87.5 sulfuric acid to methanol solution at 24V, 10s and finally polished with 0.05 μ m colloidal silica suspension. EBSD orientation mapping was performed in a FEI XL-30 scanning electron microscope (SEM) with step size of 0.5-1.5 μ m at 30kV. OIM scans were analysed with the TSL OIM Analysis 7 package to calculate grain boundary character distribution, grain size, types of triple junctions. CSL grain boundaries were categorized according to Brandon criterion of $\Delta\theta \leq 15^\circ \Sigma^{-1/2}$ [24]. Boundaries with $3 < \Sigma < 29$ were considered to be CSL boundaries whereas boundaries with $\Sigma > 29$ were considered HABs and $\Sigma = 1$ as low angle boundaries (LABs) [25]. Triple junctions (TJs) with no CSL boundaries, 1 CSL boundary, 2 CSL and 3 CSL boundaries have been classified as J0, J1, J2 and J3 respectively. For TJ analysis, only $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ were considered as CSL boundaries.

TEM was used to characterize the precipitation behavior of carbides on grain boundaries of known character. Thin foils were prepared by conventional method including grinding, dimpling and finally ion-milling. Foils were obtained from SA and GBE1 samples after sensitization (650°C, 2h) and analyzed with a Phillips CM-20 TEM operating at 200 kV.

Nanoscale lateral spatial resolution OIM analysis was performed by automated acquisition and indexing of precession electron diffraction (PED) patterns with a JEM 2100F TEM equipped with the DIGISTAR/ASTAR system from NanoMEGAS at the University of Pittsburgh. Precessed illumination, 0.6° precession angle, and electron beam focused to ~ 3 nm in diameter at the TEM specimen section surface was scanned across a pre-selected area of interest with step-size of 20 nm to obtain maps of PED patterns, which were indexed automatically by optimized matching to computer generated reciprocal lattice based templates of the Nickel and Cr_7C_3 (carbide) phases of interest here. The PED based TEM OIM data sets provide information akin to that available via EBSD based OIM in the SEM but with lateral resolution in areal maps being on the order of 2 nm, limited essentially by the electron beam diameter used in the TEM instrument. The raw data sets of the PED based orientation indexed areal maps were processed and analyzed further with the TSL OIM Data Analysis software.

2.3 Double loop electrochemical potentiokinetic reactivation (DLEPR) and corrosion tests

All samples (SA, GBE1 and GBE2) were given a sensitization treatment in a laboratory furnace at 650°C , 2h and water quenched (WQ) 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 a solution composed of 0.01 M H_2SO_4 + 20 ppm KSCN using a Gamry Potentiostat (Reference 600) [26,27]. 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 Argon gas before and during each test and all tests were performed at room temperature. Samples were kept immersed in the test solution for 1 hour at open circuit potential before the start of each test. The following procedure was used to quantify sensitization in the annealed and GBE material after sensitization [28].

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.

$$R = \frac{I_r}{I_a} \times 100 \quad (1)$$

The DL-EPR value obtained is normalized with various parameters like grain boundary area (GBA), grain size, mean lineal intercept length (MIL). 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:

$$R' = R \times \sqrt{2^{G'-G}} \quad (2)$$

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

$$R_{GBA} = \frac{R}{S_v} \quad (3)$$

The DL-EPR values were also normalized with mean intercept length (designated by l):

$$R_{MIL} = \frac{R}{l} \quad (4)$$

Here l is expressed in μm . The grain boundary area and mean lineal intercept length can be obtained from the number of intercepts per unit length (N_L) of the test line [29]. The N_L can be calculated from the following equation [29]:

$$G = -3.2877 + 6.6439 \log_{10} N_L \quad (5)$$

where N_L is a number of intercepts per unit length and G is the ASTM grain size number. The mean lineal intercept (l) and the GBA, S_v , are given by [29]:

$$l = \frac{1}{N_L}, S_v = 2N_L \quad (6)$$

To evaluate susceptibility to intergranular corrosion after TMP, all samples were given a sensitization treatment at 650°C , 2h and then tested as per ASTM G28 standard in

boiling ferric sulfate (25%)- sulfuric acid (50%) solution for 24 hours [30]. All samples were given a sensitization treatment at 650°C, 2h, WQ before testing. Samples were mechanically ground to 1200 grit finish and weighed before and after testing. On completion of tests, samples were sectioned and polished for SEM and EBSD analysis. For a few tests, samples were polished before testing for EBSD analysis. This allowed for orientation mapping of the sample after the test without introducing polishing artifacts.

2.4 Slow strain rate tests (SSRT)

Flat tensile samples of alloy 600 (all three conditions after the same sensitization treatment as above), with dimensions as shown in Figure 1, were fabricated using a wire EDM. Subsequently, these samples were polished to 1200 grit, degreased with acetone and ethanol, dried and immersed in the test solution for 1 hour prior to straining. A strain rate of 2×10^{-6} /s was chosen for all tests and test solutions were prepared using reagent grade sodium tetrathionate ($\text{Na}_2\text{S}_4\text{O}_6$). SSRTs were performed with a system driven by a servo motor and fitted with a custom built environmental chamber. Load and displacement values were recorded periodically and samples were tested to failure. For interrupted SSRTs, samples were prepared using the same procedure that was used for EBSD sample preparation mentioned previously. SSRTs were interrupted at 11% and 20% nominal strain and the samples were examined using SEM/EBSD and orientation maps were recorded from areas on the gage section.

Figure 1. Schematic of the sample used for all SSRTs. All dimensions in mm.

3. Results

3.1 Microstructure and sensitization

EBSD generated grain boundary maps from the SA condition (initial microstructure) are shown in Figure 2 (a, b). The microstructure obtained as a result of repetitive strain annealing

cycles, designated GBE1 and GBE2 are shown in Figures 2 (c, d) and 2 (e, f) respectively. All orientation maps were obtained from the surface and are representative of the microstructure after TMP. Grain boundary fractions (length), grain size and TJ fractions for all conditions has been summarized in Figures 3 (a-c). The HAB fraction (length) decreased from 50% to ~ 20% after TMP while the CSL fraction increased from 37% to ~ 70%. The fraction of $\Sigma 3$ boundaries increased from 30% in the SA condition to 55-60% for the GBE1, GBE2 conditions. More importantly, fractions of $\Sigma 9 + \Sigma 27$ increased from 2% (SA) to 10.2% (GBE1 and GBE2). In addition, TJ fractions extracted from the orientation maps show a sharp drop in fraction of J0 type from 43% to 6-10%. J3 fractions increased from 3% (SA) to 38% for GBE1 and 35% for GBE2. These changes in TJ fractions are consistent with the GBE model proposed by Kumar et al. [11,31]. Grain size after TMP increased modestly from ~11 μm in the SA condition to ~17 μm for GBE1 and 14 μm for GBE2 respectively.

Figure 2. Skeletonized grain boundary maps showing the microstructure for (a, b) SA (c, d) GBE1, (e, f) GBE2 conditions. Black lines denote random high angle grain boundaries and grey lines denote CSL boundaries ($\Sigma \leq 27$).

Figure 3. (a) Grain boundary character distribution and grain size, (b) CSL boundary fraction and (c) triple junction fraction for each processing condition.

TEM micrographs from SA and GBE1 samples (after sensitization) are shown in Figures 4 (a, b) and (c, d) respectively. In the SA condition, carbides were observed to have precipitated along grain boundaries. Further, PED was used to resolve the nature of grain boundaries and the type of carbides and these maps are presented in Figure 5 and 6 for SA and GBE1 conditions respectively. In case of SA sample, Figure 5(a) shows the inverse pole figure (IPF) map overlaid with grain boundaries. These boundaries were indexed as HABs (colored black). Figure 5(b) is an IPF map overlaid with the image quality (IQ) map showing carbides on grain boundaries that were indexed as HABs. These carbides were confirmed by

electron diffraction to be Cr_7C_3 type of carbides as shown in Figure 5(c, d) that are known to precipitate in alloy 600 when sensitized in the 600-800 °C temperature range. In case of GBE1 condition, no carbides were observed along grain boundaries but intra-granular carbides were present as shown in Figure 6a. The carbides in Figure 6b were indexed as Cr_7C_3 type and electron diffraction patterns from the matrix and carbide and their counterpart computer generated patterns are labelled 1 and 2 respectively.

Figure 4. TEM micrographs from SA (a, b) and GBE1 (c, d) conditions after sensitization.

Figure 5. (a) PED results from SA condition after sensitization including (a) IPF; (b) IQ overlaid with IPF; (c) IQ map showing carbides; (d) virtual bright field map and corresponding spot patterns from the grains and the Cr_7C_3 carbide.

Figure 6. PED results from GBE1 condition after sensitization including (a) IQ; (b) grain boundary map and corresponding spot patterns from the grain (1) and the Cr_7C_3 carbide (2) as well as simulated diffraction patterns. GBs colored black are random high angle boundaries while those colored red are CSL ($\Sigma 3$, $\Sigma 9$, $\Sigma 27$) boundaries.

Trillo and Murr observed that carbides (M_{23}C_6) precipitate first on HABs then at non-coherent twins in stainless steels, whereas no carbides were observed on coherent twin boundaries [32]. Zhou et al. also reported a higher resistance to carbide precipitation in special GB ($\Sigma \leq 29$) as compared with random GB [33]. In the SA condition, carbides GBE1 condition, shown in Figure 5, intra-granular carbides were observed. No carbides were observed along other random high angle GBs. The spot pattern #2 in Figure 6 confirms that the carbide is of Cr_7C_3 type. A number of $\Sigma 3$ twins show no carbide precipitation.

The area observed in the TEM was limited due to grain size as well as lower random HAB fraction, which made the quantitative, statistical analysis of the precipitation behavior on grain boundaries of known character difficult. Hence EBSD-based orientation maps were

recorded after DL-EPR tests to provide both a larger area for analysis and better statistics of the precipitation behavior at various types of boundaries in the SA and GBE conditions.

3.2 Double loop electrochemical potentiokinetic reactivation (DLEPR) and corrosion weight loss tests

After a sensitization treatment of 650°C, 2h (water quenched) and DLEPR tests were used to quantify the degree of sensitization (DOS) for each condition. The DOS value (R) for SA was 0.56%, but much lower for GBE1 and GBE2 conditions, being 0.06 and 0.12%, respectively. As the CSL fraction in the microstructure increased from 36% to ~ 70%, the susceptibility to sensitization was lowered as seen by the DOS or R value in Table 3. SEM micrographs recorded after DLEPR test from the samples are shown in Figure 7 (a-c). The corresponding normalized DOS values (Table 3) i.e. R' , R_{GBA} , and R_{MIL} all follow similar trends. It should be noted that twin boundaries were excluded from grain size analysis used for normalizing these values.

Table 3. CSL fraction and corresponding degree of sensitization values for each processing condition.

Sample	CSL fraction, %	DOS (R), %	R'	R_{GBA}	R_{MIL}
SA	37.7	0.56	0.4195	0.0070	0.0224
GBE1	72.9	0.06	0.0157	0.0021	0.0008
GBE2	65.3	0.12	0.0404	0.0033	0.0022

The SEM micrograph and its corresponding skeletonized grain boundary map in Figure 7(d, e) recorded after DLEPR test from the SA sample shows the presence of localized attack on random HABs (colored black). No attack was observed on low CSL boundaries ($\Sigma 3$ colored red and $\Sigma 9$, $\Sigma 27$ colored blue). The precipitation of carbides during sensitization results in Cr depletion in Alloy 600 and the DLEPR test gives good correlation with SCC susceptibility in tetrathionate and thiosulfate solutions [27,34]. The DLEPR test parameters used in this study have been optimized to attack the deeper Cr depletion zones as compared with shallower broad zones, which are more immune [27]. Cr_7C_3 type of carbides precipitated

along HABs during the sensitization treatment (shown in Figure 4 and 5) resulted in localized Cr depletion zones that were preferentially attacked during the DLEPR test. In both the GBE1 and GBE2 conditions, no localized attack was observed on HABs (colored black) or CSL boundaries ($\Sigma 3$ colored red and $\Sigma 9$, $\Sigma 27$ colored blue). A representative set of SEM and corresponding grain boundary maps from the GBE2 condition sample are shown in Figure 7 (f, g). These results indicate that the discontinuous random HAB network in the GBE conditions reduced the propensity for Cr carbide precipitation and Cr depletion as compared with the SA condition. The decrease in sensitization has been well correlated with CSL fractions in other instances in alloy 600 as well as austenitic stainless steels in literature [1,2,20,33,35,36].

Figure 7. SEM images showing microstructure after DLEPR test for (a) SA, (b) GBE1, (c) GBE2 conditions. SEM micrographs and corresponding GB map showing locations of localized attack (circles) for SA (d, e) and GBE2 (f, g) conditions. Random HABs are in black while $\Sigma 3$ and $\Sigma 9$, $\Sigma 27$ boundaries are colored blue respectively.

Corrosion weight loss tests were also performed to evaluate the effect of increased CSL fractions and disrupted grain boundary network on the corrosion behavior. Baseline and GBE1 and GBE2 samples (after sensitization) were immersed in boiling $\text{Fe}_2(\text{SO}_4)_3$ -50% H_2SO_4 solution for 24 hours as per ASTM G28 standard [30]. SEM micrographs from the surface of samples after the test are shown in Figure 8 (a-f). Weight loss after tests and corrosion rates calculated after testing are summarized in Table 4. Weight loss after 24 hours decreased from 5% (SA) to 0.67% while corrosion rates decreased from 16 mpy (SA) to ~ 2 mpy for GBE1 and GBE2 conditions. The attack was noticeably lower on the GBE1 and GBE2 samples as compared with the SA condition as seen in Figure 8. Grain dropping was clearly observed in the SA condition while GBE1 and GBE2 showed discontinuous attack along grain boundaries.

Table 4. Results of ASTM G28 weight loss tests for all conditions.

Sample	Weight Loss, %	Corrosion rate, mpy
SA	4.989	0.411
GBE1	0.656	0.035
GBE2	0.664	0.041

Like DLEPR tests, ASTM G28 corrosion weight loss tests are commonly used as quick tests to evaluate Cr depletion susceptibility to intergranular cracking. Figure 9 shows the SEM micrograph and corresponding grain boundary map for GBE1 condition after corrosion test (G28) for 24 hours. J0 type junctions consisting of all random HABs were attacked in all instances. J3 types of junctions were the most resistant with minimal or no intergranular attack. In J1 and J2 types, the CSL segments showed no attack in a majority of cases. This is consistent with other observations of termination of intergranular attack at low CSL boundaries during G28 tests [37].

Figure 8. Micrographs after ASTM G28 test for (a, b) SA, (c, d) GBE1 and (e, f) GBE2 conditions.

Figure 9. (a) SEM micrograph and (b) corresponding EBSD map recorded from GBE2 sample after ASTM G28 test for 24 hours. Circles show locations of J0, J1, J2 and J3 type of triple junctions.

3.3 Slow strain rate tests

The increase in the CSL fractions and lower DOS are indicative of less Cr depletion after TMP and may reduce the SCC susceptibility of alloy 600 in thiosulfate and tetrathionate solutions. Hence, SSRTs were performed to observe and quantify the susceptibility to IGSCC after TMP. Stress-strain curves obtained from SSRTs in tetrathionate (0.01 M Na₂S₄O₆) solution are as shown in Figure 10. Tests were performed at the same strain rate and in the same environment for direct comparison of SCC behavior.

In case of SA, the elongation to failure in 0.01 M Na₂S₄O₆ solution was only 15% for SA condition while those for GBE1 and GBE2 condition were about 66% and 35% respectively. Analysis of the fracture surface after tests showed ductile and mixed mode of failure for GBE1 and GBE2 conditions respectively while SA condition showed 100% intergranular cracking. The significant difference in elongation to failure as well as failure analysis indicates that TMP improved resistance to SCC in alloy 600. Further, secondary cracks from the gage sections of samples were analyzed in greater detail and the results are discussed in the next section.

Figure 10. Stress-strain curves for (SA) and thermo-mechanically processed (GBE1, GBE2) samples (after sensitization at 650°C, 2h) in 0.01 M Na₂S₄O₆ solution. Strain rate: 2 x10⁻⁶ /s.

3.4 Crack Analysis

Some SSRT tests were interrupted to analyze intergranular cracks along grain boundaries of known character. Cracks were observed on the gage section of the GBE2 sample and orientation, grain boundary maps from the gage section after 11% strain are shown in Figure 11(a-e). Conventional inverse pole figure (IPF) and grain boundary map with low CSL and HABs show locations of crack arrest in Figure 11(a) and 11(b) respectively. Cracks were observed to have been mitigated or arrested at a J1 or J2 type of TJs and a few locations have been circled (black). In addition, orientation and grain boundary maps, in Figure 11(c) and 11(d) respectively, have been colored according based on the technique proposed by Patala and Schuh [38]. With the continuous mapping of color to misorientation allowing for grain boundaries with same misorientation being colored similarly, this method provides information about both the misorientation angle and the misorientation axis. For example, $\Sigma 3$ boundaries (60° (111), colored blue) and $\Sigma 9$ (39° (110), colored green) were parts of J1, J2 type of junctions that were crack arrest locations circled in

black. This analysis indicates that disruptions in the cracks along along HABs, encounter greater resistance to propagation at J1 and J2 type of junctions. More statistical analysis that examines the susceptibility of certain axis/angle orientations to intergranular corrosion would be necessary to elucidate if other grain boundaries boundaries exhibit relatively higher resistance.

Figure 11. (a) Inverse pole figure (IPF) image and (b) corresponding grain boundary map showing intergranular cracks on the gage section of GBE2 sample interrupted after 11% strain during SSRT. (c) Misorientation map, (d) grain boundary map in quaternion color from the same area and (e) Fore scattered detector (FSD) image from the same area. (f) color legend for homophase misorientations, built using stereographic projections of surfaces of constant misorientation angle used to interpret the (c) and (d) plots. Locations showing crack deflection or mitigation are circled in black and the loading direction is horizontal.

Figure 12. (a) Kernal average misorientation map, (b) grain orientation spread map, (c) Taylor factor map and (d) Schmid factor map from the same region of GBE2 sample interrupted after 11% strain during SSRT. Locations showing crack deflection or mitigation are circled in black. Loading direction is horizontal.

Figure 12 (a, b) show the kernal average misorientation (KAM) map and grain orientation spread map from the same area area of a GBE2 sample interrupted after 11% strain during SSRT. KAM values were relativley higher around the crack tips (Figure 12(a)) as compared with the grain interior indicating higher local strain as the crack encounters a resistant TJ. The crack arrest locations in Figure 12b suggest that as the crack front encounters a resistant TJ, grains adjoining the crack paths are strained resulting in higher misorientations (lattice rotation). In addition, the low Taylor factor grains (Figure 12(c)) correspond well with high lattice rotation in the strained grains adjoining the crack arrest locations. Furthermore, the Schmid factor (Figure 12(d)) for grains adjoining the crack front are very high, which indicates high critically resolved shear stress on the slip planes. Therefore, we can infer that the grains adjoining and ahead of the crack front undergo severe plastic deformation. Similarly, Figure 13 (a, b) shows intergranular cracks on the gage section

after 20% strain. As in the case of the 11% strained sample, numerous instances of the crack arrest at TJs can be seen.

Figure 13. (a) FSD image and (b) corresponding grain boundary map, (c) IPF map and (d) KAM map showing intergranular cracks on the gage section of GBE2 sample interrupted after 20% strain during SSRT.

4. Discussion

The results of this comprehensive study have led to a number of new findings on the behavior of grain boundaries on their sensitization, carbide precipitation, corrosion and SCC behavior of alloy 600 and related mechanistic aspects, which are discussed below.

The present results confirm a decrease in DOS after TMP has been attributed to increased fraction of twin boundaries and its variants [1,2,20,28]. Additionally, CSL boundaries have been shown to be more resistant to precipitation of carbides and the resulting Cr depletion. TEM micrographs in Figures 4 and 5 show Cr carbides precipitated along random high angle boundaries. These Cr_7C_3 type carbides precipitated during the sensitization treatment. EBSD on SA sample surface after DLEPR tests showed local attack on random HABs. In contrast, CSL boundaries particularly $\Sigma 3$, 9 and 27 did not show any attack. For the GBE2 sample, no attack was observed along either HABs or CSL boundaries. This is consistent with the absence of Cr carbides along GBs in the GBE1 sample during TEM analysis. Bi et al. [19] reported precipitation of M_{23}C_6 type of carbides in austenitic SS304 along random HABs while CSL segments were precipitation free [19]. In addition to reduced Cr carbide precipitation, Cr depletion was suppressed. Zhou et al. [33] reported that more than 90% of special GBs ($\Sigma \leq 29$) were resistant to carbide precipitation. Kurban et al. [36] also observed higher resistance to carbide precipitation in grain boundary engineered SS304. The decrease in DOS by almost an order of magnitude suggests that the higher CSL fraction as well as

numerous disruptions in HAB network reduced Cr depletion. In the GBE1 condition, no instances of localized attack were observed along random HAB or CSL boundaries, indicating that the Cr depletion was suppressed as seen from micrographs recorded after DLEPR tests (Figure 7).

The probability for crack arrest at a TJ was calculated according to the three models proposed by Kumar et al. [41], Palumbo et al. [39] and Marrow et al. [40] and have been plotted in Figure 14.

Palumbo et al. [39] also proposed the following relation (Equation 7) to calculate the probability (P) of crack arrest,

$$P = (f_{sp}^2) + 2 [(f_0)(f_{sp})(1 - f_{sp})] \quad (7)$$

where f_{sp} is the fraction of special boundaries and f_0 is the fraction of interfaces in the distribution that are unfavorably oriented to the stress axis.

Assuming J3 type of junctions to be non-entities in crack propagation, Kumar et al. [11] proposed (Equation 8) that the percolative paths in the microstructure would be broken if,

$$P_R = \frac{J2}{(1-J3)} \geq 0.35 \quad (8)$$

Marrow et al. [40] observed cracks arrested at both J2 and J1 type of TJs and proposed the following (Equation 9),

$$P = \frac{[f_a J2 + f_b J1]}{(1-J3)} \quad (9)$$

where f_a and f_b (considered 1 and 0.5 respectively) are geometrical factors to account for unfavorably oriented sensitized boundaries at the triple junction. These probabilistic models provide a basis to evaluate microstructures that are indicative of the resistance to SCC.

Figure 14. Probability for crack arrest as proposed by Kumar et al. [11], Palumbo et al. [39] and Marrow et al. [40] for all conditions.

The probability for crack arrest was observed to increase from 0.3 to about 0.5 after TMP according to the model proposed by Marrow et al. and from 0.3 to 0.6 according to the model proposed by Palumbo et al. Similarly, P_R was observed to increase from 0.03 to 0.18 as per the model proposed by Kumar et al. Only $\Sigma 3$, 9 and 27 CSL boundaries have been considered for TJ analysis. This allows for a more conservative approach to evaluate the relative SCC resistance. These models provide a basis for comparing relative resistance of microstructures to percolation of intergranular cracks. Tsurekawa et al. also reported higher resistance to percolative intergranular corrosion in SS304L for microstructures with higher P_R [14]. They also found that triple junction distribution fraction was a more important parameter to rank than maximum random boundary cluster length or grain boundary character distribution (GBCD). In this study, TMP increased the ratio (P_R) by five times that in the baseline microstructure and this reduced the SCC susceptibility significantly. The increase in probability of crack arrest (P) after TMP explains the increase in resistance to SCC observed experimentally. Cracks were more likely to encounter a resistant junction, and the reduced sensitization of random grain boundaries due to altered Cr diffusion behavior contributed to reduced SCC susceptibility.

A number of crack tips were used for statistical analysis of crack arrest at TJs of specific kind. Figure 15 shows observed fractions of crack arrest at a TJ, probability of finding a crack tip/TJ of a specific type, and fraction of types of TJs in the microstructure. About 52 crack arrest locations were identified in the GBE2 sample and considered for this analysis. The probability of finding a TJ of a specific type was calculated as per the procedure used by Seita et al. [42]. The assumption for this calculation is that for a given TJ, each GB (including low CSL such as $\Sigma 3$, $\Sigma 9$, and $\Sigma 27$) is likely to be a cracked boundary. Subsequently, each of

the three possible permutations of cracked vs. non-cracked GBs for each TJ type are considered to determine the fraction of permutations that result in each type of crack tip. These fractions are then summed as shown in Equation 10(a-c) below to determine the total probability of finding each type of crack tip/TJ.

$$P(0\Sigma) = \frac{1}{3}P(J1) + P(J0) \quad 10(a)$$

$$P(1\Sigma) = \frac{2}{3}P(J2) + \frac{2}{3}P(J1) \quad 10(b)$$

$$P(2\Sigma) = P(J3) + \frac{1}{3}P(J2) \quad 10(c)$$

Here, $P(Ji)$ is the fraction of TJs with i low- Σ GBs in the microstructure [42].

Figure 15. (a) Probability of crack arrest at a triple junction of specific type, fraction of TJ junction type that were observed to be crack arrest locations and occurrence of TJ types in the bulk microstructure is summarized. (b) Inclination angle with respect to tensile axis for cracked boundaries.

Although no CSL ($\Sigma 3$, $\Sigma 9$, $\Sigma 27$) boundaries were observed to have cracked in this study, $\Sigma 9$ and 27 are likely to crack at higher strain and in a more aggressive environment [22]. It is clear that disruption of HAB connectivity deflects and/or arrests cracks at some J0, but almost all J1 and J2 types. About 51% of crack arrest locations were J1 type of TJs while 32% were J2 type and the rest (15%) were J0 type (Figure 15). The probability for crack arrest at J0, J1 and J2 type of TJ is 0.22, 0.37 and 0.42, respectively, and shows good correlation with the values of crack arrest locations determined from the EBSD observations and analysis of interactions of cracks with the different types of TJs. the observed values. In addition, a majority of cracked boundaries (all of them were random HABs) were observed to have inclination angle close to 70-90° with respect to the tensile axis. This is consistent with observations of intergranular cracks along boundaries inclined at angles close to 90° along the tensile axis [42–44]. It is evident that random HABs may be most susceptible to localized

intergranular attack, crack initiation and cracks tend to propagate along the random HAB network till they encounter a J1 or J2 type of junction.

Serial sectioning combined with 3D EBSD is currently being applied to further investigate the interaction between cracks and the 3D grain boundary network to further develop and extend the percolative models for quantification and prediction of crack arrest in 3D.

5. Conclusions

Iterative cold rolling and annealing was used to modify the GBCD in alloy 600 and its effect on carbide precipitation, Cr depletion behavior, intergranular corrosion and SCC was investigated in this study. The following conclusions can be drawn from this study,

1. The fraction of low CSL boundaries was increased after iterative steps of cold work and high temperature annealing and the random HAB network was disrupted.
2. TMP of alloy 600 decreased the degree of sensitization significantly (by an order of magnitude), carbide precipitation and chromium depletion at grain boundaries and, in turn, the corrosion rates were also significantly lower as compared with those in the SA condition.
3. SSRT results show an increase in elongation to failure and significant decrease in SCC susceptibility following TMP. Detailed analysis of cracks with the microstructural elements (different types of grain boundaries and TJs) on the gage section of the samples after interrupted SSRT show cracks being deflected or arrested at J1 and J2 type of TJs.
4. The probability for crack arrest at low CSL grain boundaries as well as at a particular type of TJ (J1, J2), calculated using percolative models, was found to have increased after TMP, which also explains the increase in resistance to corrosion and SCC.

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Figure 1. Schematic of the sample used for all SSRTs. All dimensions in mm.

Figure 2. Skeletonized grain boundary maps showing the microstructure for (a, b) SA (c, d) GBE1, (e, f) GBE2 conditions. Black lines denote random high angle grain boundaries and grey lines denote CSL boundaries ($\Sigma \leq 27$).

Figure 3. (a) Grain boundary character distribution and grain size, (b) CSL boundary fraction and (c) triple junction fraction for each processing condition.

Figure 4. TEM micrographs from SA (a, b) and GBE1 (c, d) conditions after sensitization.

Figure 5. (a) PED results from SA condition after sensitization including (a) IPF; (b) IQ overlaid with IPF; (c) IQ map showing carbides; (d) virtual bright field map and corresponding spot patterns from the grains and the Cr₇C₃ carbide.

Figure 6. PED results from GBE1 condition after sensitization including (a) IQ; (b) grain boundary map and corresponding spot patterns from the grain (1) and the Cr₇C₃ carbide (2) as well as simulated diffraction patterns. GBs colored black are random high angle boundaries while those colored red are CSL ($\Sigma 3$, $\Sigma 9$, $\Sigma 27$) boundaries.

Figure 7. SEM images showing microstructure after DLEPR test for (a) SA, (b) GBE1, (c) GBE2 conditions. SEM micrographs and corresponding GB map showing locations of localized attack (circles) for SA (d, e) and GBE2 (f, g) conditions. Random HABs are in black while $\Sigma 3$ and $\Sigma 9$, $\Sigma 27$ boundaries are colored blue respectively.

Figure 8. Micrographs after ASTM G28 test for (a, b) SA, (c, d) GBE1 and (e, f) GBE2 conditions.

Figure 9. (a) SEM micrograph and (b) corresponding EBSD map recorded from GBE2 sample after ASTM G28 test for 24 hours. Circles show locations of J0, J1, J2 and J3 type of triple junctions.

Figure 10. Stress-strain curves for (SA) and thermos-mechanically processed (GBE1, GBE2) samples (after sensitization at 650°C, 2h) in 0.01 M Na₂S₄O₆ solution. Strain rate: 2 x10⁻⁶ /s.

Figure 11. (a) Inverse pole figure (IPF) image and (b) corresponding grain boundary map showing intergranular cracks on the gage section of GBE2 sample interrupted after 11% strain during SSRT. (c) Misorientation map, (d) grain boundary map in quaternion color from the same area and (e) Fore scattered detector (FSD) image from the same area. (f) color legend for homophase misorientations, built using stereographic projections of surfaces of constant misorientation angle used to interpret the (c) and (d) plots. Locations showing crack deflection or mitigation are circled in black and the loading direction is horizontal.

Figure 12. (a) Kernel average misorientation map, (b) grain orientation spread map, (c) Taylor factor map and (d) Schmid factor map from the same region of GBE2 sample interrupted after 11% strain during SSRT. Locations showing crack deflection or mitigation are circled in black. Loading direction is horizontal.

Figure 13. (a) FSD image and (b) corresponding grain boundary map, (c) IPF map and (d) KAM map showing intergranular cracks on the gage section of GBE2 sample interrupted after 20% strain during SSRT.

Figure 14. Probability for crack arrest as proposed by Kumar et al. [11], Palumbo et al. [39] and Marrow et al. [40] for all conditions.

Figure 15. (a) Probability of crack arrest at a triple junction of specific type, fraction of TJ junction type that were observed to be crack arrest locations and occurrence of TJ types in the bulk microstructure is summarized. (b) Inclination angle with respect to tensile axis for cracked boundaries.

Graphical Abstract