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Through-thickness microstructural evolution during grain boundary engineering type thermomechanical processing and its implication on sensitization behavior in austenitic stainless steel

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ABSTRACT

Through-thickness microstructural variations and their synergistic influence on degree of sensitization in grain boundary engineering (GBE) treated 304L stainless steel was compared with solution-annealed and marginally cold-worked (~3% thickness reduction) specimens. The solution-annealed specimen has shown higher degree of sensitization due to the presence of lower fraction of Σ3 (as well as other low Σ CSL) boundaries and dominant random high-angle boundaries (RHAGBs) connectivity. However, it has shown thickness-independent sensitization behavior as grain boundary character distribution, residual strain and grain boundary network topology towards thickness direction are similar. Owing to higher residual strain, as revealed by local misorientation analysis, the cold-worked specimen showed maximum degree of sensitization (higher than solution-annealed condition) at surface. The GBE specimen has shown much lower degree of sensitization at surface due to higher-fraction of Σ3 boundaries and discontinuous RHAGBs connectivity. However, increasing fraction of RHAGBs and their connectivity, as confirmed by twinning-related domain and fractal analysis, cause more sensitization at interior of this specimen. In spite of this, the degree of sensitization at the interior of GBE specimen is lower than the solution-annealed or cold-worked specimen. Even though the residual strain in GBE specimen is higher than the solution-annealed condition, the lower degree of sensitization indicates that the grain boundary character distribution and grain boundary network topology have higher implication than residual strain in controlling sensitization.

1. Introduction

Sensitization has been recognized as an important problem in austenitic stainless steels which leads to inter-granular corrosion (IGC) and inter-granular stress corrosion cracking (IGSCC) [1–4]. Many conventional techniques like solution annealing followed by quenching, reducing carbon and/or adding non-chromium carbide forming elements (i.e. Ti, V or Nb) etc. have been employed to minimize sensitization [5]. In recent times, grain boundary engineering (GBE) approach has emerged as an effective method to alleviate sensitization in austenitic stainless steels without the need of altering the chemical compositions of the alloy [6–10]. The GBE approach is essentially based on the philosophy of optimizing the grain boundary character distribution (GBCD) and this is often realized through suitable thermomechanical processing (TMP) [6,11–15]. Shimada et al. [6] and Michiuchi et al. [15] have observed remarkable IGC resistance in austenitic stainless steels by introducing low Σ (Σ ≤ 29) coincident site lattice (CSL) boundaries in austenitic stainless steels.

Apart from GBCD, other microstructural features like grain size, residual strain etc. have also been found to influence the sensitization behavior in austenitic stainless steels [7,16–19]. An inverse relationship between the grain size and the degree of sensitization (DOS) was observed by Li et al. [20]. In contrast to this, it has been argued by some researchers that specimen with larger grains tends to exhibit more DOS [16]. Lower level of residual strain with a higher fraction of annealing twins has shown higher resistance to sensitization which was confirmed by double loop electrochemical potentiokinetic reactivation (DL-EPR) test followed by atomic force microscopic examination [21]. Moreover, the percolation related phenomena are not only dependent on the GBCD, but also on the grain boundary network topology of the materials. Therefore, various microstructural parameters viz. triple junction distribution, grain-cluster etc. were proposed to quantify the random high angle grain boundaries (RHAGBs) connectivity [9,10,22]. Based on the percolation theory, Schuh et al. [23] have theoretically quantified the grain boundary network topology in 2D as well as 3D microstructures. The specimens of large size grain-clusters associated with

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high proportion of the $\Sigma3^n$ boundaries and the $\Sigma3^n$-type triple junctions have shown high resistance to IGC [9]. Kobayashi et al. [22] have studied the correlation of the IGC susceptibility with the fractal dimensions of maximum random boundary connectivity (MRBC) in SUS316L austenitic stainless steel and reported that the lower fractal dimension of MRBC implies the higher resistance to IGC. More recently, researchers are focusing on the study of evolution and characterization of twin related domain (TRD) during material processing because it is expected that the inter-granular related properties can be manipulated/controlled by introducing large TRD [24–26].

Thermomechanical processing comprising of small deformation + annealing (single step or iterative) is often employed to realize GBE microstructure in low to medium stacking fault energy materials [27–29]. The evolution of microstructural features (viz. grain size, residual strain, GBCD and RHAGBs connectivity) in the thickness direction of the specimens during these GBE type TMP has not been investigated so far. It is obvious that such a variation of these microstructural parameters in the thickness direction would significantly influence the percolation related phenomena. Although researchers have reported the effect of grain size and GBCD on the sensitization behavior, the individual or combined effects of GBCD, RHAGBs connectivity and residual strain on the sensitization behavior is not well reported in literature. Therefore, the present study is emphasized on two objectives. The first objective is to study the evolution of through-thickness microstructural features (i.e. GBCD, residual strain and grain boundary network connectivity) during thermo-mechanical processing in extra-low carbon type 304 L austenitic stainless steel. The second objective is to evaluate the synergistic influence of these microstructural parameters on the sensitization behavior of the same alloy.

2. Experimental Procedure

2.1. Specimen Preparation

The material used in this study is an extra-low carbon type 304L austenitic stainless steel containing Fe-17.7Cr-13.7Ni-0.0147C-0.038Mn-0.003S-0.02Si-0.0045 N-0.001Co (all in wt%). To homogenize the steel, hot rolling (at 1373 K) and solution annealing treatment (at 1323 K for 60 min) was performed on the as-cast material. The solution annealed as-received (abbreviate hereafter as AR) specimen of 4 mm thick strip was subjected to iterative thermo-mechanical processing (ITMP) in order to realize GBE microstructure. The ITMP comprised of two iterations; a first iteration of 10% reduction followed by a second iteration of 5% reduction imparted through laboratory cold rolling. After each reduction, the specimen was annealed at 1173 K for 1 h prior to water quenching. This specimen is termed as GBE specimen. The AR specimen was also subjected to 3% thickness reduction through cold rolling in order to introduce residual strain and this specimen is termed as AR-3R. Sections were cut from each specimen to study the corrosion behavior at different level of thickness. All the three specimens (i.e. AR, AR-3R and GBE) were sensitized at 945 K for 77 h. Three surfaces at a depth of 50 μm (S-50), 200 μm (S-200) and 350 μm (S-350) of each specimen were prepared following standard metallographic procedure to study the microstructure and then conduct the DL-EPR tests. The schematic representation of the specimen's surface is shown in Fig. 1. Microhardness of each specimen at S-50 section was determined using UHL VMHT microhardness tester by employing a load of 200 gf for 15 s dwell time.

2.2. Characterization of Microstructure

Electron back scatter diffraction (EBSD) based orientation imaging microscopy (OIM) scans were performed on all the surfaces of the specimens using a TSL-OIM system attached to field emission scanning electron microscope (model: ZEISS MERLIN) operating at 20 kV. EBSD maps were collected using a step size of 0.5 to 1 μm with hexagonal grid. The EBSD data was analyzed using TSL OIM (version 7.2) software and standard clean-up procedure (grain dilation for single iteration) was applied before analyzing the EBSD data. The EBSD analysis was performed to calculate the GBCD, average grain size, kernel average misorientation (KAM)[1] and grain average misorientation (GAM).[2] To identify CSL boundaries, Brandon's criterion [30] is used. RHAGBs are defined as those with misorientation $\theta > 15^\circ$ and which are not low Σ ($\Sigma \le 29$) CSL boundaries. The grain size was measured using linear intercept method (average of horizontal and vertical intercept lengths) and a misorientation of 5° was used for determining the grain size. To ensure statistical significance, the GBCD, local misorientation and average grain size from all the investigated surfaces in each specimen was analyzed from at least an area of 1 mm × 1 mm.

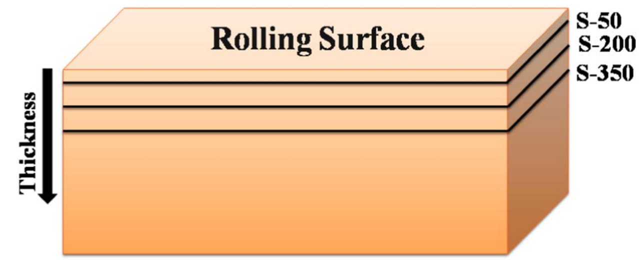

Fig. 1. Schematic representation of the specimen surfaces. Here, S-50, S-200 and S-350 represent the interior surfaces of the rolled specimen with a depth of 50, 200 and 350 μm, respectively.

In addition to OIM analysis, the newly developed ARPGE (Automatic Reconstruction of Parent Grains from EBSD data) software was used for the reconstruction of parent grains from original EBSD data [31,32]. ARPGE software routine groups the grains together to a single twin related domain (TRD) which are connected by the $\Sigma3^n$ ($n \le 3$) CSL boundaries. Hence, ARPGE reconstructs TRD that are linked by RHAGBs. The software automatically calculates the number of grains contained in each TRD and assigns an arbitrary color to it to differentiate between TRDs in the TRD reconstructed map.

To study the fractal dimension, the largest mass cluster (D_{max}) of RHAGBs was extracted from the OIM generated grain boundaries map of each surface for all the three specimens [33]. This has been done by using connected-component labeling algorithm (i.e. graph theory application) [34,35]. The steps performed in fractal dimension calculation are schematically shown in Fig. 2. Due to the self-similar nature of the D_{max}, the fractal dimension of the D_{max} has been calculated through the eq. 1 by using box-counting technique [36,37].

$$N(x) = x^{-D_f} \tag{1}$$

where $N(x)$ is the total number of unit square box with edge length x required for complete coverage of the D_{max} and D_f is the fractal dimension of D_{max}, which is the slope of the linear function of log $N(x)$ and $log\ x$ as represented in Eq. 2.

$$D_f = \frac{-log\, N(x)}{log\,(x)} \tag{2}$$

2.3. Corrosion Tests

In order to quantitatively evaluate the DOS, the DL-EPR test was carried out on each surface of all the three specimens using an electrolyte of 2.5 M H_2SO_4 + 0.01 M KSCN at ambient temperature employing a potentiostat workstation (Model: Biologic science-SP 200)

[1] KAM is the average misorientation of each measurement point with all of its neighbors, excluding misorientations exceeding some tolerance value (specified as 5° in the present study).

[2] GAM is the misorientation between neighboring measurement points in a grain.