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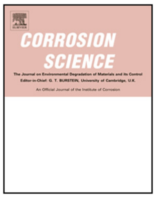

Short communication

Implication of grain boundary engineering on high temperature hot corrosion of alloy 617

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ABSTRACT

The role of grain boundary engineering (GBE) on high-temperature hot corrosion behavior of alloy 617 was evaluated by exposing both the as-received (AR) and GBE specimens in a salt-mixture of (75% Na_2SO_4 + 20% NaCl + 5% V_2O_5) at 1273 K for 24 h. The AR specimen having continuous network of random high angle grain boundaries (HAGBs) has undergone hot corrosion and substantial depletion/segregation of alloying elements through the entire cross section. The GBE specimen exhibited significantly reduced hot corrosion and depletion/segregation of alloying elements. This is attributed to the high fraction of 3-CSL triple junctions which break the percolation in the random HAGBs network.

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1. Introduction

Grain boundary engineering (GBE) has been shown to be an effective approach to improve the properties and performance of bulk polycrystalline materials [1–7]. The GBE involves manipulation of grain boundary character distribution to enhance the fraction of special boundaries in the microstructure and this is often realized by designing optimized thermo-mechanical processing schedule [2,3,6,8,9]. Special boundaries are those that have relatively better properties when compared to the random high-angle grain boundaries (HAGBs) and are often described in terms of the coincidence site lattice (CSL) model. It is generally assumed that low Σ ($\Sigma \leq 29$) CSL boundaries are 'special' even though there is no physical basis for this assumption [1–3,5,9,10]. Recent results have indicated that only a subset of low Σ CSLs are special [6,11–15]. In spite of this assumption, GBE approach has been shown to be effective in enhancing the properties in a variety of low-to-medium stacking fault energy materials [1–3,5,10]. This is essentially due to the fact that these materials exhibit prolific multiple twinning during GBE-type thermo-mechanical processing leading to higher fraction of 'special' $\Sigma3$ boundaries terminating on low-index plane [12]. In addition to this, multiple twinning also leads to the generation of $\Sigma9$ and $\Sigma27$ boundaries through the following interactions: $\Sigma3 + \Sigma3 = \Sigma9$ and $\Sigma3 + \Sigma9 = \Sigma27$ [16,17]. These higher order twin boundaries take part in the reconfiguration of the existing grain boundary network that eventually breaks down the random HAGBs connectivity [18,19].

Although GBE approach has been extensively employed in the past to mitigate the intergranular corrosion of a variety of low-to-medium stacking fault energy materials in aqueous corrosion medium [2,4,6,20], very limited effort has been made to explore the implication of GBE on high temperature hot corrosion of an alloy. In this study, we evaluate the hot corrosion behavior of GBE alloy 617 by exposing it to a salt mixture of (75% Na_2SO_4 + 20% NaCl + 5% V_2O_5) at high temperature and compare it with the as received (solution annealed) specimen of the same alloy. The above salt mixture is commonly used to simulate corrosion environment in gas turbines [21].

2. Experimental

The alloy 617 (with a nominal composition of 55Ni–21.8Cr–11.5Co–8.7Mo–1.07Al –0.38Ti–1.02Fe–0.06C, all in wt%) used in the present investigation was received from VDM Metals GmbH, Germany, solution annealed (at 1448 K) and quenched with water (abbreviated hereafter as AR specimen). The

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AR specimens were subjected to 5%, 10% and 15% reduction in thickness in a laboratory rolling mill. Deformed specimens were annealed at three different temperatures (1273, 1323 and 1373 K) for three different durations (0.5 h, 1 h and 2 h) and subsequently quenched in water [22]. A 15% reduction and subsequent annealing at 1373 K resulted in a GBE microstructure (termed hereafter as GBE specimen) having high fraction of Σ3 boundaries and notable disruption in random HAGBs connectivity [22]. Both the AR and the GBE specimens were polished employing standard metallography procedures described elsewhere [22]. The electron back scattered diffraction (EBSD) scans were done using Hitachi S-4300SE scanning electron microscope (SEM) at a voltage of 20 kV with a step size of 1 μm.The collected EBSD data were analyzed using TSL OIM analysis software (version 7.2). To identify CSL boundaries, Brandon's criterion [23] is used. Random HAGBs are defined as those with the misorientation angle $\theta > 15^\circ$ and which are not low Σ ($\Sigma \leq 29$) CSL boundaries.

The AR and GBE specimen ($10 \times 10\,\text{mm}^2$ dimensions) were subjected to high temperature hot corrosion by completely immersing them inside a corrosive salt mixture (75% Na_2SO_4 + 20% NaCl + 5% V_2O_5) in a preheated (at 1273 K for 1 h) crucible. The melting point of Na_2SO_4 is 1157 K which is lowered by the addition of NaCl due to the formation of an eutectic with melting point around 893 K [24]. It is to be noted that the addition of 5% V_2O_5 to the salt mixture increases the severity of the environment [25]. The crucible was covered by a lid to prevent contamination by any foreign elements or water vapor and placed inside a box furnace at 1273 K for 24 h. Upon removal of crucible from the furnace, the specimens were taken out from the molten salt melt before the salts could solidify. The specimens were subsequently kept separately in another crucible to cool down to room temperature. The cross sections of the AR and GBE specimens were analyzed using SEM and combined EBSD/energy dispersive spectroscopy (EDS) to assess the extent of hot corrosion attack.

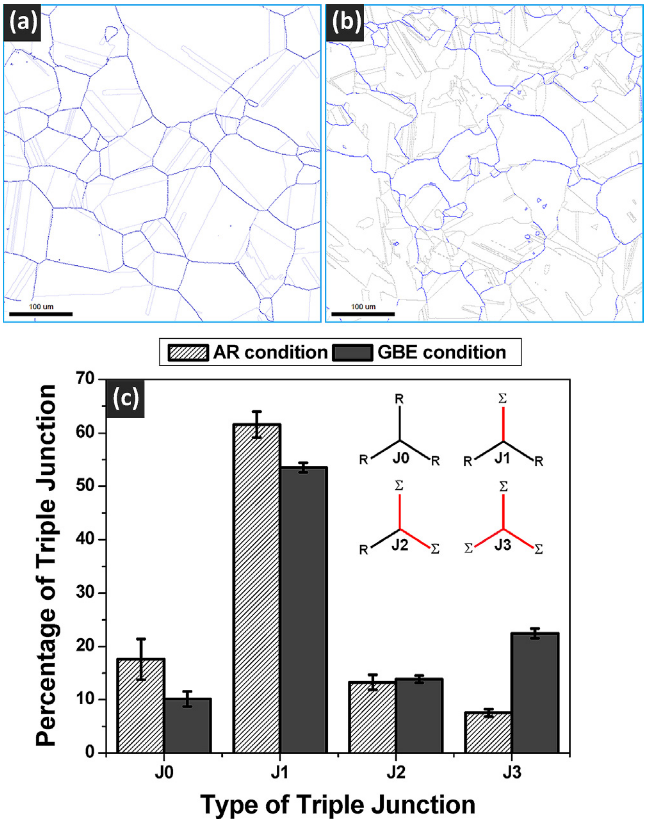

Fig. 1. Grain boundary maps showing random HAGBs connectivity in (a) AR and (b) GBE specimen (grain boundary color code: Σ3 & its variants – grey, random HAGBs – black); (c) Percentage of each type of triple junction in both the AR and GBE specimen. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

3. Results and discussion

The grain boundary character map reveals that random HAGBs connectivity is significant in the AR condition (Fig. 1a) whereas it is visibly disrupted in GBE specimen (Fig. 1b). The average grain size (measured by linear intercept method) in the AR condition was found to be 27 ± 1.4 μm (considering twin boundaries as grain boundaries). The grain size after GBE processing has been reduced to 18 ± 1.4 μm due to the introduction of large number of twin segments into the microstructure. The Σ3 fraction in the AR and GBE specimen was found to be 53 ± 2.5% and 67 ± 2.1%, respectively. The (Σ9 + Σ27) fraction, on the other hand, in AR and GBE specimen is 2.1 ± 0.6% and 6.5 ± 1.2%, respectively. It is obvious that fraction of Σ3 and its variants (i.e., Σ9 and Σ27) has been considerably enhanced following thermomechanical processing. However, enhanced fraction of Σ3 and its variants is a necessary but not sufficient condition to achieve the optimized GBE microstructure [26]. The distribution of these boundaries in the triple points of the microstructure (i.e., triple junction distribution) is also important [26,27]. This is due to the fact that intergranular corrosion propagates from the surface into the interior of the material preferentially through the random HAGBs and the resistance to intergranular corrosion depends on the degree of connectivity of these boundaries. Therefore, we have analyzed the triple junction distributions to quantify the microstructures in AR and GBE specimen in order to assess the effectiveness of the GBE approach. There are essentially 4 types of triple junctions, namely 0-CSL, 1-CSL, 2-CSL and 3-CSL (n-CSL refers to a triple junction having n number of CSL boundaries at the intersection). The triple junction distribution analysis involved exporting the triple junction data using TSL-OIM software to a customized Microsoft excel program and calculating the fraction of n-CSL ($\Sigma \leq 29$) boundaries present. The analysis shows that the fraction of 3-CSL boundary triple junctions has increased significantly following thermomechanical processing (Fig. 1c). A leveling off of 2-CSL junctions and a rapid increase in 3-CSL junctions would indicate a GBE microstructure. The saturation of 2-CSL junctions exists because if there are two near-exact CSLs at a junction (often 2 Σ3s) then by the product rule of CSL boundaries, the third boundary will also be a CSL [17].

Simultaneous EBSD + EDS scans were performed across the cross-section of the hot corrosion tested samples to analyze the elemental distribution and correlate with the grain boundary character. This would essentially give a clear idea about the type of grain boundaries that are susceptible to hot corrosion. It is important to note here that both the AR and GBE specimens did not show any kind of preferential segregation/depletion of alloying elements at grain boundaries before hot corrosion test. Fig. 2a shows the image quality plus grain boundary map of the cross-section of the AR specimen after hot corrosion whereas EDS elemental distribution maps of Mo, S, Co, Ni, Cr, Al, and O_2 are shown in Fig. 2b through h. The hot corrosion has led to the segregation of Mo (Fig. 2b), S (Fig. 2c) Co (Fig. 2d) and Ni (Fig. 2e) at the random HAGBs (see Fig. 2a) along with a depletion of Cr (Fig. 2f) in the AR specimen. In contrast to this, Σ3 boundaries do not show any preferential enrichment/depletion of any alloying element; thus indicating that these boundaries are resistant to hot corrosion. Five parameter stereological analysis has revealed that majority of these Σ3 boundaries terminate on (1 1 1) plane and hence they have very low interfacial energy [22]. Some of the higher order Σ3 boundaries were corroded (shown by yellow arrow in Fig. 2a through c) whereas few of them are not attacked

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