Materials Science & Engineering A 626 (2015) 51–60

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

The effect of thermo-mechanical processing on grain boundary character distribution in Incoloy 800H/HT

Hamed Akhiani *, Majid Nezakat, Mohsen Sanayei, Jerzy Szpunar

Department of Mechanical Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon, Canada S7N 5A9

ARTICLE INFO

Article history:
Received 16 October 2014
Received in revised form
3 December 2014
Accepted 13 December 2014
Available online 19 December 2014

Keywords:
Incoloy 800H/HT
Cross-rolling
Grain boundary engineering
Grain boundary character distribution

ABSTRACT

In this study, we applied a thermo-mechanical process to alter the grain boundary characteristic distribution (GBCD) with a view to the feasibility of grain boundary engineering in Incoloy 800H/HT. In order to optimize the GBCD through increasing the low Σ coincidence-site lattice (CSL) boundaries, we applied various thickness reductions with two different rolling modes followed by annealing. We used Electron Backscattered Diffraction (EBSD) to analyze the GBCD and CSL boundaries. We found that the coincidence-site lattice boundaries, particularly $\Sigma 3$ and its variants, increased with the pre-deformation level in cross-rolled (CR) samples. In contrast, the fraction of these CSL boundaries had an optimum in 50% reduction for unidirectionally rolled (UDR) samples. In fact, different $\Sigma 3^n$ interactions led to different GBCD in UDR and CR processed samples. Low to medium deformation with UDR and medium to high deformation with CR on the Incoloy 800H/HT samples showed potential for grain boundary engineering.

1. Introduction

Incoloy 800H/HT is an austenitic Fe–Ni super alloy, which is currently used in industrial chemical processing and power generation units due to its high resistance to high temperature oxidation and corrosion [1,2]. With these properties, application of Incoloy 800H/HT in other industries, such as nuclear power generation with its severe operating conditions, appears to be feasible. Recently, new approaches and designs have been implemented in Gen IV nuclear reactors to enhance their performance and safety. Two of the seven types of Gen IV reactors, the Very High Temperature Reactor (VHTR) and Super-Critical Water-cooled Reactor (SCWR), are being developed to work at high temperature (VHTR: 750–950 °C, SCWR: 550–650 °C) for a higher thermodynamic efficiency [3]. Moreover, improvement of the high temperature properties of construction materials is an enduring interest for materials scientists and engineers. This interest would become more focused by introducing these materials in new challenging industrial applications such as structural components in Gen IV nuclear reactors. According to the Gen IV roadmap [4] Incoloy 800H/HT is being considered as one of the candidate materials to be employed in construction of different components of Gen IV reactors such as the intermediate heat exchanger, hot duct, steam generator and fuel cladding [5]. However, to be suitable as a nuclear structural material in the Gen IV designed environment, some modifications of the material's structure should be considered [1,3]. Several studies state that grain boundary characteristics strongly influence the alloy's physical and mechanical properties [6–9].

Based on the misorientation of the adjoining grains, grain boundaries can be categorized as low-angle grain boundaries (LAGBs), high-angle (random) grain boundaries (HAGBs) and special/coincident-site lattice (CSL) boundaries [10]. Different CSLs can be categorized by Σ value, which is defined as the inverse of common lattice points in the boundaries between the two adjoining grains or crystals. The relative fraction of CSL and random boundaries as well as the deviation of the special boundaries from the exact CSL boundaries and connectivity between these boundaries can be derived from the grain boundary character distribution (GBCD) [9,11–15]. Altering the GBCD, as a relatively new microstructural feature, can effectively change the material's properties. This fact was initially proposed by Watanabe in the early 1980s as "grain boundary design and control" which has been referring to grain boundary engineering (GBE) afterwards [16,17]. Since then, many attempts have been made to improve the physical and mechanical properties of various materials by this method. In most studies, the GBE process is comprised of a combination of deformation and consequent annealing, which is known as thermo-mechanical processing (TMP) [9,11,12,18]. Fundamentally, the main concept of GBE is to generate and increase the low Σ CSL density through changing the geometry of grain boundaries, which consequently improves various material properties [14–16]. Several investigations show that low-Σ CSL boundaries (e.g., $\Sigma 3^n$) exhibit higher

* Corresponding author. Tel.: +1 306 370 0320.
E-mail address: hamed.akhiani@usask.ca (H. Akhiani).

http://dx.doi.org/10.1016/j.msea.2014.12.046
0921-5093/

resistance to various types of intergranular degradation compared with random boundaries [17]. Consequently, the presence of a high concentration of CSL boundaries enhances the properties of the materials in various aspects including lower rates of grain boundary sliding during creep [19], resistance to high temperature fracture [6], resistance to solute segregation, precipitation, and intergranular embrittlement [20], weldability [21], resistance to corrosion and stress corrosion [6,22], resistance to intergranular corrosion [23], and resistance to radiation damage [20,24]. The most successful experiments were done in low Stacking Fault Energy (SFE) materials [11,25] and some of them are now commercially employed [26,27].

In our previous papers, in addition to deformation and annealing textures evolution, we discussed in detail the recrystallization behavior of Incoloy 800H/HT during the proposed TMP [28]. In the present study, we aim to characterize the GBCD evolution with the same TMP procedure (different strain paths with various deformation strains) as we performed before. Studies investigating the effect of deformation path on the CSL boundaries are scarce. Lee et al [30] reported an improvement in the CSL density due to applying CR in comparison with UDR deformation. However, they did not address the reason for this improvement when they changed the strain path. Understanding the role of deformation mode and strain in manipulating the GBCD would strongly contribute to increase of the low Σ-CSL fractions and consequently improving the material's properties.

2. Experimental procedure

A commercial Incoloy 800H/HT from Special Metals® with the chemical composition listed in Table 1, was used in this study. The alloy was fully annealed at 1420 °C and water quenched in the as-received condition. We performed thermo-mechanical processing (TMP) on the as-received sample by a series of cold rolling followed by annealing at 1050 °C for a time period of 25 min/cm thickness, commensurate with the size of the samples. A true strain of 0.2 was applied per rolling pass for all the samples using a laboratory roll machine. We employed two different rolling modes, unidirectional rolling (UDR) and cross-rolling (CR) to decipher the role of strain path in altering the grain boundary structure. We cut samples with the dimensions of $50 \times 50 \times 13.25$ mm^3 and $50 \times 25 \times 13.25$ mm^3 from the same plate for UDR and CR, respectively. In order to study the effect of strain amount, we rolled the samples with 10%, 30%, 50%, 70% and 90% thickness reductions.

In pursuance of investigating the grain orientations and grain boundary character distribution (GBCD), we used Electron Backscattered Diffraction (EBSD). We prepared the samples with a general metallographic procedure to a mirror-like surface followed by the final polishing step with 50 nm colloidal silica on a Beuhler Vibromet® for 24 h. We employed an Oxford Electron Backscatter Diffraction (EBSD) setup interfaced to a Hitachi SU6600 Field Emission Gun Scanning Electron Microscope (SEM) to carry out Orientation Imaging Microscopy (OIM). We operated the SEM at 30 keV with the automatic EBSD scan on the transverse direction (TD) plane of the samples. We utilized HKL AzTech and Tango software to acquire and analyze the EBSD data, respectively. EBSD measurements were obtained from 5 different locations/scans with relatively good statistics of grains using the same scanning conditions for all the samples. The measurement uncertainty for grain boundary fraction was less than 5% and mean angular deviation (MAD) was used as statistical measure of accuracy of indexing. In our study, MAD values were less than 0.5°, which resulted to 98% indexing for all the scans. The Brandon criterion was adopted for the classification of Σ boundaries. Coherent and non-coherent twins were separated using a misorientation threshold of 2°, which was successfully used before [18]. In measuring the grain sizes, twins were not counted as grains. The Vickers hardness was measured using a Mitutoyu Vickers hardness testing machine with a load of 500 gf.

3. Results

Fig. 1 shows the microtexture, microstructure and grain boundary structure of the as-received Incoloy 800H/HT. The inverse pole figure (IPF) EBSD map illustrates a weak texture and a random dispersion of differently oriented grains. Coarse grains and twinning resulted from hot rolling deformation and solution annealing. High angle grain boundaries (HAGB: $\delta > 15$), low angle grain boundaries (LAGB: $5 < \delta < 15$) and CSL boundaries: $\Sigma 3$, $\Sigma 9$, and $\Sigma 27$ are respectively shown in black, gray, violet, blue and green lines.

Figs. 2 and 3 illustrate the EBSD maps of the processed samples after two different rolling modes with various rolling strains. In order to obtain statistically reliable data from EBSD images, the scan areas were selected in a way to contain a certain number of grains. Therefore, the scan areas varied based on the grain size range of the samples. To focus on the grain boundary character distribution and ease of comparison between the samples, an EBSD grain boundary map including the HAGB, LAGB, $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ CSL is shown for each sample.

Fig. 2a shows the EBSD grain boundary map of the annealed samples after 10% UDR deformation. The grain size decreased by almost half its size in the starting material. The microstructure of the 10%CR sample (Fig. 3a) resembles the starting sample; the grain size decreased by about 30% from the as-received sample. By increasing the rolling strains to 30%, the grain size was dramatically decreased, particularly for the UDR sample. The trend of decreasing the grain size continued as the prior deformation strain increased until the grain size finally reached about 10 and 17 μm for the 90% UDR and CR samples, respectively. The evolution of the grain size of the samples and their associated hardness with increasing the rolling strain is depicted in Fig. 4. The major difference in grain size evolution was that below 50% reduction, UDR samples had smaller grain size while after this deformation strain the CR samples exhibited lower grain sizes. This point can also be observed by comparing 50% UDR (Fig. 2c) and 50%CR (Fig. 3c) samples, where both show almost the same average grain size. In general, the CR samples possessed higher hardness when compared to UDR samples. This has been referred to as dislocation tangling, resulting from changing the strain path in CR [29,30]. Indeed, we previously discussed in detail the evolution of deformation and annealing texture, the texture differences between UDR and CR, as well as the recrystallization behavior of these samples [28,31]. Despite the difference in hardness number of UDR and CR samples, they both demonstrated an increasing trend upon decreasing the grain size. This is in agreement with the Hall–Petch relationship in the current grain size range [9].

Table 1
Chemical composition (wt%) of Incoloy 800H/HT.

Fe	Ni	Cr	Mn	Al	Ti	Mo	Cu	Si	Co	P	C	S
45.41	30.19	20.5	0.98	0.55	0.54	0.42	0.4	0.32	0.3	0.22	0.07	0.0001