



Texture and microstructure evolution of Incoloy 800H superalloy during hot rolling and solution treatment



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ABSTRACT

In this paper, we have implemented a specific hot rolling and solution treatments on Incoloy 800H to examine the evolution of texture and microstructure during this process. We also focused on the analysis of local misorientation (LM) and grain boundary character distribution through electron backscattered diffraction (EBSD) method. The results showed that the hot-rolled microstructure is mainly composed of elongated grains with typical texture components of B, Cu and S at the mid-layer of plates, while substantial inhomogeneous fine grains can be observed between the deformation grains at the surface-layer showing a combination of R-C and R-Cu components. Following solution treatment, the original deformed grains are fully substituted by the fine recrystallized grains and the grain size increases significantly at higher solution temperatures. An alternant lamellar structure of fine and coarse grains forms along the rolling direction at 1050 °C due to the pinning effect of TiC at fine grains boundaries. Besides, C and G are the dominant texture components after solution treatment. With increasing solution temperature, the overall level of LM and micro-hardness decrease stably and a higher ratio of $\Sigma 3:\Sigma(9+27)$ indicates that $\Sigma 3$ boundaries proliferation mechanism transforms from “ $\Sigma 3$ regeneration” to “new twinning”.

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1. Introduction

In metalworking, rolling is termed as the process of plastically deforming metal through passing it between rolls. It is well known that rolling is the most widely used forming process, which provides high production and close control of final product. Generally, the rolling process can be categorized into hot rolling and cold rolling according to the metal-rolling temperature. Hot rolling is a metalworking process that occurs above the recrystallization temperature of metallic material. The initial breakdown of ingots into billets is usually accomplished by hot rolling which is followed by further processing into plate, sheet, rod and bar and also provides raw stuff for consequently cold rolling. In order to obtain the desired microstructure and improved mechanical properties, solution treatment is often performed after hot rolling through holding a material long enough at a sufficiently high temperature to allow the constituents to be dissolved into the solid solution [1–3].

Incoloy 800H is a typical austenitic solid-solution superalloy and

composed of iron (~45 wt %), nickel (~31 wt %) and chromium (~20 wt %) as major constituent elements. It is extensively used as a candidate structural material for high-temperature service due to its excellent creep strength and good oxidation/corrosion resistance [4–6]. In the previous studies, much work has been focused on the texture and microstructure evolution of Incoloy 800H during cold rolling and annealing. H. Akhiani et al. [7] investigated the influence of different rolling paths on the recrystallization mechanism and subsequent annealing texture in a cold-rolled Incoloy 800H. L. Tan et al. [8] also studied and proposed a thermo-mechanical processing method (cold rolling with a thickness reduction of ~7% followed by annealing at 1050 °C for 90 min) to optimize the grain boundary character distribution in Incoloy 800H. However, in comparison, little work has been reported on the effects of hot rolling procedure and solution treatment on the microstructure and texture evolution of Incoloy 800H.

Our previous investigations [9–12] examined the microstructure evolution and hot working properties during hot deformation of Incoloy 800H through uniaxial compression tests. We discussed the influence of deformation parameters on the microstructure (dynamic recrystallization [9], precipitation behavior [10] and grain boundary character distribution [11]) and also established the hot

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Table 1
Chemical composition of as-received Incoloy 800H.

Element	C	Mn	Si	Ni	Cr	Al	Ti	Cu	N	Fe
wt%	0.068	0.08	0.31	30.5	20.2	0.36	0.34	0.02	0.013	Balance

processing maps to optimize the hot working procedure [12]. However, all of the above work is only related with simulation process and the processing method is totally different from the practical rolling production. In this paper, the Incoloy 800H plates were hot rolled by 80% thickness reduction in seven passes at different rolling temperature ranges according to the hot rolling parameters of industrial production. Afterwards, the hot-rolled Incoloy 800H plates were solution treated with different methods. The consequent development of texture and microstructure has been the subject of our research.

2. Experimental procedure

Incoloy 800H plates in the form of hot rolled and annealed condition with a thickness of 26 mm were used as initial materials in this study. The chemical composition of the investigated alloy is given in Table 1. These plates were soaked at 1200 °C for 2 h and then subjected to hot rolling with the accumulated reduction of 80% through seven passes reverse rolling on a laboratory rolling mill. The details of rolling procedure are presented in Table 2 and the hot-rolled samples were labeled according to the start rolling temperatures, namely HR1050 and HR950. The hot-rolled plates with a target thickness of 5 mm were quenched into water immediately to retain deformation microstructure. Samples with the dimensions of $15 \times 15 \times 5 \text{ mm}^3$ were cut from the central part of hot-rolled plates and further subjected to different heat-treatment schedules, during which the samples were solution treated for 30 min at 950 °C, 1050 °C, and 1150 °C before water cooled to the room temperature.

Microstructure observations were carried out on the plane consisting of rolling direction (RD) and normal direction (ND). The electron backscattered diffraction (EBSD) analysis was performed using an Oxford HKL Channel 5 system attached to a Zeiss UltraPlus analytical field emission gun scanning electron microscope (FEG-SEM). The samples for EBSD were electrochemical polished in a solution of 4% HClO₄ with alcohol at 25 V for 25–30 s and Channel 5 software by HKL technology was used to analyze EBSD data. For the standard noise reduction of EBSD data, wild spikes were extrapolated first and then a medium level of zero solution extrapolation was conducted and iterated until no zero solution remained. A minimum misorientation threshold of 2° was set for the EBSD analysis. The grain boundaries with misorientation angle of 2–15° and greater than 15° were defined as low-angle grain boundaries (LAGBs) and high-angle grain boundaries (HAGBs), respectively. The local variation in orientation was evaluated by the local misorientation (LM) component based on the filtered crystal orientation. The LM value was determined through calculating the average misorientation between a pixel and its surrounding pixels and then assigning the mean value to the pixel. The distribution of

LM was illustrated with a filter size of 5×5 and sub-grain angle of 5°. The boundaries with misorientation angles exceeding 5° were discarded in this calculation. Brandon's criterion was applied to identify the "coincidence site lattice" (CSL) grain boundary [13]. Orientation distribution functions (ODFs) were calculated after correction and symmetrization using the coefficient calculation (series expansion method) with expansion to $L_{\max} = 22$ and a cluster size of 5°. The distribution of different crystallographic orientations over the entire scanned region was displayed by ODFs inside the $\varphi_2 = 45^\circ$ and $\varphi_2 = 65^\circ$ sections of the Euler space. Thin foils for transmission electron microscope (TEM) investigation were prepared by jet polishing with perchloric acid and methanol solution (1:9) at -30°C and $\sim 40 \text{ mA}$. The TEM examination was performed on a FEI Tecnai G² F20 microscope operated at 200 kV. The distribution of different chemical elements was characterized using a JXA-8530F electron probe microanalyzer (EPMA). The samples for EPMA were prepared by standard mechanical polishing and chemically etched in a solution of 4 g CuSO₄, 20 mL HCl and 20 mL H₂O. Vickers hardness testing was conducted under a load of 50 gf with a dwell time of 10 s on a FM-700 micro-hardness tester. An average value of ten points was used to determine the hardness of each sample.

3. Results and discussion

3.1. Microstructure and texture of hot-rolled structures

The ND-IPF map (inverse pole figure map with colour legend) from EBSD scan for the pre-deformation sample is depicted in Fig. 1 (a), showing large austenitic grains and annealing twins that are ordinary in alloys with high nickel content. As shown in Fig. 1 (b) and (c), the ND-IPF maps at the mid-layer of hot-rolled plates are predominantly composed of severely elongated deformation grains. Within some of these grains, shear bands are pronounced indicating the occurrence of localized inhomogeneous deformation. It is worth noting that the length of these bands mostly remains within the level of deformed grain scale and the distribution of bands is almost parallel to each other with a certain angle (30–35°) to RD. As shown in Fig. 1 (d) and (e), most of the annealing twin boundaries will lose their special crystallographic orientation relationship with the sustained external loading. There is also some evidence to suggest that the distorted and discrete twin boundaries can be transformed into general HAGBs. Therefore, the fraction of $\Sigma 3^n$ ($1 \leq n \leq 3$) boundaries has a significant decrease after hot rolling. The LM measured by EBSD can be used to evaluate the local change in the crystal orientation. In fact, the plastic deformation can induce crystallographic slip and geometrically necessary dislocations (GNDs) on a microstructural scale, indicating the crystal orientation may fluctuate of several degrees within the same grain

Table 2
Parameters for hot rolling experiments.

No.	Rolling temperature range, °C	Rolling reduction every pass						
		1	2	3	4	5	6	7
HR1050	1050–850	15%	18%	17%	20%	25%	22%	28%
HR950	950–810							

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