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Effects of prior deformation and annealing process on microstructure and annealing twin density in a nickel based alloy

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ABSTRACT

The nickel based alloys with different Σ3 boundary density were achieved by cold-rolling and subsequent annealing treatment. Electron backscattered diffraction analysis showed that the grain size distribution changed with the processing parameters, and the discontinuous Σ3 boundary became continuous with the increase of prior deformation level. Furthermore, the Σ3 boundary density was found to be manipulated by both grain size distribution and Σ3 boundary density per grain which showed an increasing trend with prior deformation level and annealing temperature.

1. Introduction

It is believed that the properties of a material generally depend on composition and microstructure. Besides the development of new alloys, microstructure optimization is an important strategy to improve properties [1]. Grain boundary engineering (GBE) is considered to be an effective method to manipulate grain structures [2]. As a special boundary, the Σ3 boundary effectively enhances high temperature mechanical properties of nickel based alloys [3–5], and plays an indirect but irreplaceable role in obtaining desired properties through grain boundary engineering [6]. Therefore, optimizing of Σ3 boundary is an attractive way to achieve improved properties.

Recently, there have been a large number of studies on the relationship between Σ3 boundary and processing parameters. However, most of the relevant works primarily dealt with the distribution of Σ3 boundary and its content defined as the length ratio of Σ3 boundary to all boundaries, and prior deformation was limited to low or medium level [1,7–10]. More recent works employed a thermo-mechanical processing to study the effects of precipitates on Σ3 boundary in alloys with low stacking fault energy [11–13]. Some of the latest works focused on the mechanism and some affecting factors of annealing twinning at relatively low temperature, and therefore a wide range of annealing temperatures and heating rates were applied in these works [14–18]. However, limited works have reported the effects of processing history on grain structure and Σ3 boundary in a nickel based alloy, with a special emphasis on the relationships among processing parameters, grain size distribution and Σ3 boundary density defined as the length of Σ3 boundary per unit area [12].

It is known that the grain size and its distribution are closely related to the prior deformation level and subsequent annealing process. This provided an opportunity of insight into the effects of processing history on grain structures and Σ3 boundary density. In this work, a nickel based alloy was cold deformed to various thickness reductions and then annealed under various annealing conditions. The grain size and grain boundary character distribution (GBCD) were measured in annealed microstructures. The effects of prior deformation level and annealing process on grain structure were investigated. Furthermore, the relationships between Σ3 boundary density and processing parameters were studied, and a classical model for prediction of Σ3 boundary density was briefly discussed [19].

2. Materials and Experimental Methods

The studied material was a nickel based alloy from a forged pancake, whose composition was listed in Table 1. As-received specimens measuring 65 × 20 × 10 mm in dimension were cut from the pancake. Some as-received samples were annealed at various temperatures for

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Table 1
The main elemental analysis for the studied alloy (wt/%).

Ni	Cr	Mo	Nb	Ti	Al	C	Fe
46.8	17.8	3.2	5.8	1.0	0.52	0.03	Bal

30 min. Others were firstly annealed at 1000 °C for 60 min, and were cold rolled from 30 to 70% in the 10 mm direction respectively, and then annealed at 1020 °C for 30 min. The rolled 65% sample was annealed at 1020 °C, and the annealing time increased from 30 to 180 min. All samples were annealed in vacuum atmosphere and then cooled in air.

Samples for characterization were cut from the center of processed samples and metallographically polished, and then polished electrolytically with 20% solution of H_2SO_4 in methanol for electron backscattered diffraction (EBSD) analysis [9]. EBSD mapping was performed in a scanning electron microscope (JSM-7600F) operating at 20 kV accelerating voltage. The step size was chosen to be 0.8–2 μm which was about a tenth of grain size. For each specimen, more than 10 pieces of EBSD maps were acquired in order to ensure adequate statistical accuracy, and only one piece of these maps was shown here. The analysis of EBSD data was accomplished using the TSL-OIM software.

Both isolated pixels and isolate non-indexed pixels were preliminarily corrected. The interface with a misorientation larger than 15° was defined as a grain boundary. Grain size determination was achieved by HKL Channel system without considering twin boundaries. A tolerance of 2° or 8.6° disorientation was used in the detection of Σ3 boundary, regardless of coherent/incoherent character. As definition in a previous study [12], the Σ3 boundary density is defined as the length of Σ3 boundary per unit area.

3. Results and Discussion

In this work, we mainly focused on grain structure and Σ3 boundary, and thus other characters of microstructure were not addressed in detail. In EBSD maps, the high angle boundaries are illustrated in black lines, the Σ3 boundaries are drawn in red lines, and other low angle boundaries are drawn in lime green ones.

3.1. Grain Structure and Grain Boundary Character Distribution

An inhomogeneous equiaxed grain structure was achieved in the as-received sample after annealing at 1000 °C for 1 h, as shown in Fig. 1. The average grain size was 13.8 μm with 79 μm for large grains and 4 μm for small grains. The equiaxed grain structure and the sharp acquired diffraction pattern indicate that the specimen has been fully recrystallized after annealing at 1000 °C for 1 h. It was found in Fig. 1(b) that the density of precipitate (δ phase) in large grains was lower than that in small grains. Thus, the inhomogeneous grain structure is the result of selective growth (γ phase) due to the inhomogeneous spatial distribution of δ precipitates which is directly related to the chemical segregations in the studied alloy [12].

Fig. 2 shows the effect of annealing temperature on the recrystallized grain structure. Despite the fact that an equiaxed grain structure was achieved in these samples annealed at various temperatures for 30 min, all samples exhibited a significant diversity in the grain size and its distribution. The grain size increased slightly as the annealing temperature raised from 1000 to 1020 °C (Fig. 2(a) and (b)), but increased obviously when the annealing temperature increased to 1040 °C (Fig. 2(c)). In addition, the grain size distribution changed from a bimodal at 1000 °C (Fig. 2(a)) to a unimodal distribution at 1020 and 1040 °C (Fig. 2(b) and (c)), with an obvious increase in the width of the grain size distribution. According to previous studies [20,21], the solubility of Nb atoms in the γ-phase matrix increases with the raise of annealing temperature, and thus the density and size of δ precipitates decrease. This leads to a decreased drag force which fails to effectively impede boundary migration when the annealing temperature increases. Therefore the grain structures became homogeneous after annealing at 1020 and 1040 °C.

Fig. 3 shows the results of EBSD maps and the corresponding grain size distributions of the samples annealed at 1020 °C after various prior deformation levels. With increasing the prior deformation, the average grain size remained nearly unchanged, but the grain size distribution gradually changed from a bimodal to a unimodal distribution, without an obvious change in the width of the grain size distribution. According to a previous study [22], the grain size distribution mainly relies on the combination of initial microstructure and subsequent processing history. Since the grain size distribution was inhomogeneous in the as-received sample (Fig. 1(a)), the multiple peak distribution cannot be eliminated due to the heterogeneity of plastic deformation, especially as the deformation amount was limited to a low level. As shown in Fig. 3, with increasing the prior deformation level, a homogeneous grain structure was achieved due to the relative homogeneous distribution of strain energy during plastic deformation [22]. The present result indicates that prior deformation level has an important effect on grain size distribution.

The grain structures in rolled 65% samples as a function of annealing time are shown in Fig. 4. With increasing annealing time, the grain size increased gradually and the grain size distribution changed from a

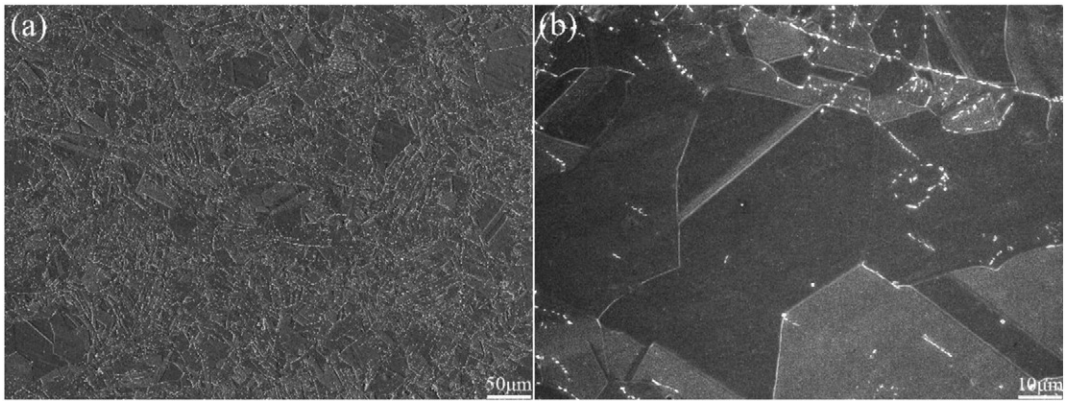

Fig. 1. Microstructure of the as-received sample annealed at 1000 °C for 60 min. (a) bimodal grain structure; (b) precipitated δ dispersed in the nickel matrix.