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Misorientation related microstructure at the grain boundary in a nickel-based single crystal superalloy

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ABSTRACT

The mechanical properties of nickel-based single crystal superalloys deteriorate with increasing misorientation, thus the finished product rate of the casting of single crystal turbine airfoils may be reduced due to the formation of grain boundaries especially when the misorientation angle exceeds to some extent. To this day, evolution of the microstructures at the grain boundaries with misorientation and the relationship between the microstructures and the mechanical properties are still unclear. In this work a detailed characterization of the misorientation related microstructure at the grain boundary in DD6 single crystal superalloy has been carried out using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques; the elemental distribution at the grain boundaries has been analyzed by energy dispersive (EDS) X-ray mapping; and the effect of precipitation of μ phases at the grain boundary on the mechanical property has been evaluated by finite element calculation. It is shown that the proportion of γ phase at the grain boundaries decreases, while the proportion of γ′ phase at the grain boundaries increases with increasing misorientation; the μ phase is precipitated at the grain boundaries when the misorientation angle exceeds about 10° and thus it could lead to a dramatic deterioration of the mechanical properties, as well as that the enrichment of Re and W gradually disappears as the misorientation angle increases. All these factors may result in the degradation of the mechanical properties at the grain boundaries as the misorientation increases. Furthermore, the finite element calculation confirms that precipitation of μ phases at the grain boundary is responsible for the significant deterioration of the mechanical properties when the misorientation exceeds about 10°. This work provides a physical imaging of the microstructure for understanding the relationship between the mechanical properties and the misorientation of the grain boundaries in the single crystal superalloy. Based on our study we hope that we can offer some scientific basis for further improvement of the nickel-based single crystal superalloys.

1. Introduction

Nickel-based single crystal superalloys are crucial materials for turbine blades manufacture and the depletion of grain boundaries in directionally solidified single crystal turbine blades has led to striking improvement of the turbine performance [1–3]. However, grain boundaries are inevitably formed during directional solidification of turbine blades due to the failure of grain selector or nucleation of spurious grains around re-entrant features such as platforms and shrouds [4]. Owing to the removal of grain boundary strengthening elements such as C, B and Hf, the mechanical properties of nickel-based single crystal superalloys could be dramatically deteriorated by the grain boundaries especially when the misorientation of the grain boundaries exceeds to some extent [5,6]. Therefore, many single crystal turbine airfoils are scrapped due to the formation of large angle grain boundaries so that the casting yield is lowered.

The misorientation tolerance of actual single crystal turbine blades may vary from each other due to the species of superalloys, their special shapes, applications, and so on. For the simplicity of scientific research, bars or slabs were usually used in the study of grain boundaries in single crystal superalloys. Many previous

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studies have been conducted on the effects of grain boundary misorientation on the mechanical properties of single crystal superalloys and showed that the mechanical properties of single crystal superalloys gradually decrease with increasing misorientation of grain boundaries and a dramatic drop of mechanical properties occurs when the misorientation exceeds some extent which is usually regarded as the tolerance of misorientation [7–10].

As far as reported, the misorientation tolerance of most unmodified single crystal superalloys is less than or equal to about 10°, for example, 6° for René N superalloy [7], 6° for CM 186 LC superalloy [9], and 10° for PW1483 superalloy [8]. In order to increase the casting yield of single crystal turbine blades, many researchers have devoted to increasing the misorientation tolerance of grain boundary in single crystal superalloys. One of the obvious ways is to add a limited amount of grain boundary strengthening minor elements (such as C, B, Hf and so on) to get a trade-off between increasing the tolerance of misorientation in grain boundary and corresponding loss in mechanical properties [11–14]. As the components and process condition differ from one superalloy to another, the tolerance and the modification mechanism of grain boundary in different single crystal superalloys vary from each other [12–15].

The microstructure of grain boundaries in modified single crystal superalloys has been studied by some researchers [15–17]; however, up to now, there is little report about the evolution of grain boundary microstructures with increasing misorientation and their relationship to mechanical properties in nickel-based single crystal superalloy. DD6 is a second generation single crystal superalloy containing about 2 wt% Re developed for areoengine blade applications [18], and numerous studies have been taken to study the mechanical properties of grain boundaries with different misorientation angles and showed that both the tensile and creep properties of DD6 single crystal superalloy were reduced by the formation of grain boundaries. The mechanical properties of DD6 single crystal superalloy gradually decrease with increasing misorientation angle when the misorientation angle is less than about 10°, while a significant deterioration of mechanical properties takes place when the misorientation exceeds about 10° [17,19–23]. In this study, we aimed to study the misorientation related microstructure at grain boundary in DD6 single crystal superalloy and their relationship to mechanical properties of the single crystal superalloy. Based on these studies, we hope that we can offer some scientific basis for further improvement of nickel-based single crystal superalloys.

2. Experimental procedure

The nominal composition of the second generation single crystal superalloy DD6 is shown in Table 1. Inverse pole figure (IPF) maps extracted from the electron backscatter diffraction (EBSD) maps across the grain boundaries were used to determine the misorientation angles. And the misorientation angles of the studied grain boundaries were determined as 3.2°, 5.6°, 7.1°, 10.5°, 12.2° and 31.7°. All bicrystalline slabs were cut from blades except for that with 31.7° grain boundary which was cast by seeding technique. And all slabs with grain boundary were fully heat treated before further observing and the heat treatment regime is as follows: 1290 °C/1 h+1300 °C/2 h+1315 °C/4 h/AC+1120°C/4 h/AC+870 °C/32 h/AC [18]. The normals of specimens for scanning electron microscopy (SEM) and transmission electron microscopy (TEM) observation are parallel to [001] direction for the 3.2°, 12.2° and 31.7° grain boundaries, while for the 5.6°, 7.1° and 10.5° grain boundary, the normal is parallel to [110], [100] and [100] directions respectively. SEM specimens were prepared via mechanical polishing, followed by chemical etching using a reagent containing 80 ml H_2O+100 ml HCl+20 g $CuSO_4$, and a JEOL 6301F field emission gun scanning electron microscope attached with an EBSD system was used to observe the microstructure of the grain boundaries. All TEM foils were prepared by spark erosion cutting, mechanical grinding and subsequent electropolishing with solution of 8% perchloric acid in ethanol at 25 V and −25 °C, and TEM observations were carried out on a FEI TECNAI G20 and a JEOL 2010F electron microscope.

Table 1
Nominal composition of DD6 superalloy (wt%) [10].

Cr	Co	Mo	W	Ta	Re	Nb	Al	Hf	C	Ni
4.3	9	2	8	7.5	2	0.5	5.6	0.1	0.006	Balance

3. Results

The microstructure of a series of grain boundaries with different misorientation angles has been carefully investigated in both SEM and TEM from three aspects: the γ/γ' configuration, the grain boundary precipitates and the elemental distribution at the grain boundaries.

3.1. The γ/γ' configuration

The SEM images of grain boundaries with different misorientation angles in DD6 are shown in Fig. 1. One thing to be mentioned here is that γ' phase was etched out during the chemical etching process of SEM specimen preparation, so γ phase shows bright contrast while γ' phase shows dark contrast in the SEM images. In Fig. 1a–c (misorientation angle smaller than 10°), the two grains seemed to be combined together and the grain boundary showed almost continuous bright contrast, which means surplus γ phase at these grain boundaries. While in Fig. 1d–f (misorientation angle larger than 10°), the two grains seemed to be separated from each other by the dark contrast along the grain boundaries, which indicates that these high angle grain boundaries are mainly formed by γ' phase and γ phase was depleted at these grain boundaries. Additionally, some abnormally coarse γ' phases could be found at the 31.7° large angle grain boundary.

More details could be observed from the TEM images in Fig. 2. When the misorientation angle is small as shown in Fig. 2a (3.2°) and b (5.6°), γ' particles adjacent to the grain boundary have almost the same size as those away from the grain boundary, while more γ phases (marked by yellow arrows) are detected at the grain boundary compared to the matrix regions away from the grain boundary, and small round secondary γ' particles (marked by red arrows) are precipitated in these surplus γ phases. In Fig. 2d, some coarse γ' particles (indicated by yellow arrows) are occasionally observed at the 12.2° grain boundary. In Fig. 2e, coarse γ' particles (indicated by yellow arrows) at the 31.7° grain boundary become much more prominent, γ' particles adjacent to the grain boundary show some extent of coalescence thus γ phase is eliminated from the grain boundary. These observations indicate that the proportion of γ phase at the grain boundary decreases as misorientation increases; on the contrary the proportion of γ' phase increases with increasing misorientation. In addition, coarse γ' particles are formed when the misorientation angle is high and both the size and quantity of coarse γ' particles increase with increasing misorientation of the grain boundaries.