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A quantitative approach to investigate discontinuous precipitation on grain boundary of Ni-based single crystal superalloys

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ABSTRACT

We designed a [001] tilt grain boundary by self-diffusion bonding technique in a single crystal superalloy. The effects of the grain boundary plane as well as its misorientation on discontinuous precipitation (DP) were investigated quantitatively. The anisotropic formation of DP colonies was associated with the anisotropic mobility of grain boundary plane. The critical misorientation for DP transformation is in between 20 and 25° for SXG3 alloy. The method proposed in this work can be used to investigate the misorientation tolerance of grain boundaries that occurs during casting of single crystal turbine blade.

1. Introduction

Nickel-based single crystal superalloys are widely used materials for turbine blades of aircraft engines due to their superior mechanical properties at high temperatures. However, the formation of low angle grain boundaries are difficult to avoid during the solidification process of hollow turbine blades, which are detrimental to the creep properties of blades in long-cycle services [1]. The supersaturated $\gamma + \gamma'$ two phase microstructure of Ni-based superalloys containing high levels of refractory alloying elements transforms into a coarsening lamellar $\gamma + \gamma' +$ TCP microstructure during the discontinuous precipitation (DP) along grain boundaries. This transformation would degrade the high temperature creep resistance of Ni-based single crystal superalloys by depleting the refractory alloying elements (i.e. Re and W) in γ phase [2–5]. The DP transformation is initiated with heterogeneous nucleation on grain boundaries and grain boundary migration [6]. High angle grain boundaries play a dominant role by serving as heterogeneous nucleation sites, high diffusion paths as well as the moving reaction front of DP colonies. Therefore, it is of great significance to elucidate the effect of grain boundaries on DP nucleation and growth.

The geometry of a grain boundary is normally described by five degrees of freedom. Two of these freedom degrees are assigned to the misorientation axis, one to the misorientation angle and two to the normal vector of the grain boundary plane [7]. In previous study, the effect of grain boundary misorientation on DP transformation was investigated by polycrystalline [8], directionally solidified [9] and bi-crystal [3] Ni-based superalloys. It was reported that the extent of DP transformation in bi-crystal superalloys was directly related to the grain boundary misorientation; DP occurs along high angle grain boundaries whereas only TCP phases precipitate along low angle grain boundaries [3,10]. Since, the grain boundary misorientation in polycrystalline superalloys is random, it is difficult to exclude the effect of misorientation axis and grain boundary plane on DP transformation. Although grain boundaries with specific misorientation can be obtained in directionally solidified bi-crystal samples, it is not a convenient approach to prepare a series of grain boundaries with different misorientations.

Furthermore, grain boundary cannot be defined by the misorientation alone. Since the interfacial mobility of grain boundary is anisotropic in the cubic crystal system [11], the effect of orientation of grain boundary planes on DP colony growth should be taken into account. However, relevant studies are quite limited due to the difficulty of obtaining grain boundaries of specific crystallographic planes with the same misorientation. Besides, grain boundary curvature induced by dendritic solidification cannot be avoided in bi-crystal samples. Since the direction of the initial curvature dominates the growth direction of DP colony [6, 12], the ideal grain boundary for studying the influence factor on the growth of DP colonies should be planar.

Herein, a simple, efficient and low cost approach is developed to obtain planar grain boundaries of specific crystallographic planes with a series of different misorientations. The effect of grain boundary misorientation and crystal-plane index on DP transformation and DP growth direction were investigated quantitatively in Ni-based single crystal superalloys, which offers valuable information on the misorientation tolerance of low angle grain boundaries for DP transformation in single crystal superalloys.

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2. Experimental procedure

The investigated SXG3 alloy is a 3rd generation Ni-based single crystal superalloy with a basic composition of Ni-12.0Co-4.0Cr-2.0Mo-6.0W-7.0Ta-5.0Re-5.0Al-0.15Hf-0.02C in wt.%. A single crystal bar, 16 mm in diameter and 180 mm in length, was directionally solidified by Bridgeman method at Beijing Institute of Aeronautical Materials. The growth orientation of single crystal bar was determined by Laue diffraction and is along [001] within 5°. This single crystal superalloy was used after standard heat treatment [13].

Fig. 1(a) and (b) are schematic diagrams of the sectioning and bonding of the single crystal superalloy. The (*hk*0) plane was sliced as the base plane (plane I), and plane II was selected and sectioned by rotating a specific degree α from plane I around [001] orientation. Then, planes I and II were bonded together by high temperature self-diffusion [10]. These [001] tilt grain boundaries based on the (*hk*0) plane with misorientation degree α were termed as (*hk*0)-α. In the current study, (010)-10°–45° and (110)-20°, (110)-25°, (110)-45° (equivalent to (010)-45°) grain boundaries were prepared and studied.

The surfaces of bonding planes were sanded by 400–2000# SiC sand papers and polished by 0.05 μm Al_2O_3 powder. After ultrasonically cleaned in acetone for 15 min, the bonding surface of single crystals were held together, and put in a superalloy fixture. A superalloy wedge was driven into the fixture to make these single crystals bonding tightly. As shown in Fig. 1(c), Al_2O_3 wafers were inserted between contact surfaces to avoid bonding at high temperature. This fixture with contacted single crystals were heated at 1290 °C for 12 h in 10^{-3} Pa vacuum and then cooled down in furnace.

To investigate the precipitation and the growth of DP colonies, the bonded samples were sealed within vacuum quartz tubes and then heat treated at 1100 °C for 0–400 h followed by air cooling. After heat treatment, samples were cut into vertical slices across the bonding plane for microstructural characterizations. The microstructures were observed under SEM. Grain boundary misorientation and the DP colony growth was verified by EBSD. The deviation from the target misorientation of these bonded grain boundaries determined by EBSD are <2°. In this study, the average width of a DP colony zone along grain boundaries is defined as the total area of the DP colony zone divided by its length. To obtain statistically significant results, the continuous DP colony were measured with a total length of >2 mm.

3. Results

Fig. 2 shows the typical microstructures of bonded (010) based grain boundaries of SXG3 alloy after a supersolvus heat-treatment as well as after a subsequent heat-treatment at 1100 °C for 200 h. The microstructure of (010)-20° and (010)-45° grain boundary before aging (Fig. 2(a)) shows the irregular and coarse γ′ phases (~1 μm in width and ~2 μm in length) precipitated at these planar grain boundaries (marked by black arrows) after bonding at 1290 °C for 12 h and subsequent furnace cooling. Fig. 2(b) shows that (010)-20° grain boundary are enveloped by a layer of γ′ film and several isolated large TCP phases distributed along the grain boundary after heat treatment at 1100 °C for 200 h. DP colony was not detected while the γ′ phase in the matrix coarsened and coalesced during the heat treatment. The microstructural features (γ′ film and isolated large TCP phases) of (010)-20° (Fig. 2(b)) and (110)-20° (image not shown) grain boundaries are quite similar with a boundary width of ~5 μm. The heterogeneous nucleation of a chain of TCP phase was observed along the initial planar grain boundaries of both (010)-25° and (110)-25° grain boundaries after heat treatment for 25 h. Extending heat treatment time to 200 h, the DP colony with a width of ~14 μm was observed along both (010)-25° and (110)-25° grain boundaries, and the TCP phase chain still remained at initial grain boundary. As shown in Fig. 2(c) and (d), the initial planar grain boundaries can be identified by a chain of TCP phases which precipitated in the earlier stage of DP, as marked by black arrows. The final boundary of the DP colony is curved and its moving front pinned by TCP phase was also detected, as pointed by white arrows. It is clearly demonstrated that the original fine γ + γ′ microstructure transformed into γ + γ′ + TCP lamellar microstructures during the DP reaction along grain boundaries. It is worth noting that the DP colony develops along both sides of the initial (110)-25° grain boundary (Fig. 2(d)) rather than primarily on one side of the (010)-25° grain boundary (Fig. 2c).

Fig. 3 shows the microstructures and IPF maps in x direction (the coordinate system is shown in Fig. 3(a) and (c)) of (010)-25° and (110)-25° grain boundaries by using EBSD in SXG3 alloy after heat treatment at 1100 °C for 400 h. The initial grain boundary is identified by a chain of TCP phases and marked by the dashed line and its migration direction is indicated by arrows. Fig. 3(a) and (b) shows that the reaction front of DP colonies on (010)-25° grain boundaries grew primarily along [010] orientation. However, Fig. 3(c) and (d) presents that the reaction front of DP colonies migrate in both side equally from the initial (110)-25° grain boundary. The migrated grain boundary becomes the reaction front of DP colonies and accompanied by the orientation change of grains. Therefore, the migration direction of DP colonies on (010)-25° and (110)-25° grain boundaries are different.

Fig. 4 illustrates the width variation of γ′ films or DP colonies along grain boundaries in SXG3 alloy as a function of the misorientation degree along (010) and (110) grain boundaries after heat treatment at 1100 °C for 200 and extended to 400 h. The average width of γ′ films or DP colonies increases with the increasing time and misorientation. The mild increment at 10–20° and 20–45° and abrupt increment at 20–25° is demonstrated that the misorientation tolerance for DP is 20–25° on both (010) and (110) based grain boundaries.

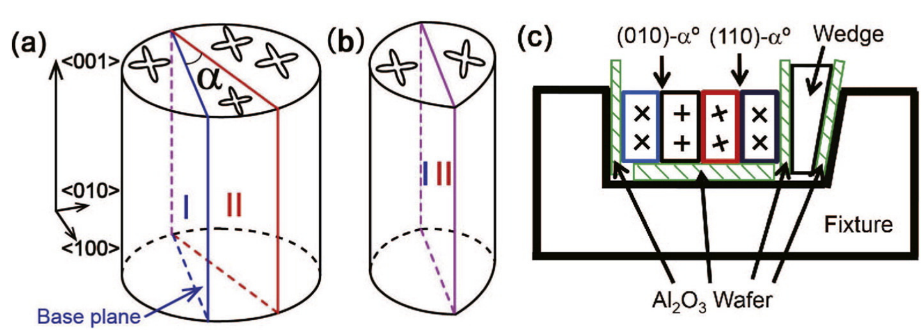

Fig. 1. (a) and (b) are schematic diagrams of sectioning and bonding single crystal superalloy, respectively. Dendrite core is shown at the top of single crystal bar, blue and red planes shown in (a) are the grain boundary plane I and plane II. The grain boundary misorientation is α; (c) schematic diagram of the (010)-α and (110)-α grain boundaries, which are assembled by single crystals with different orientations in a fixture. Dendrite core is also shown at the profile of single crystals. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)