RAPID PUBLICATION

Abnormal Grain Growth in Oxide Dispersion Strengthened Ni-Base Superalloy

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To examine the predominant factor controlling abnormal grain growth of Ni-base oxide dispersion strengthened superalloys, microstructures were investigated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Three kinds of particles were recognized; (1) fine, round shape of yttria particles of 10 to 80 nm in diameter, (2) large, round shape of Al and Ti oxide particles of 80 to 200 nm diameter, (3) flake shape of Cr rich phase with sizes of 0.2 to 0.4 μm in width and 0.4 to 2.5 μm in length. It was found that the major pinning obstacles of abnormal grain growth were the Cr-rich phase.

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I. Introduction

Oxide dispersion strengthened (ODS) superalloys for high temperature applications involve mechanical alloying of the starting powders together with the dispersoid particles, followed by consolidation processing by hot extrusion and directional recrystallization for abnormal grain growth⁽¹⁾⁻⁽⁴⁾. The practical merit of the ODS alloys is that the high strength is kept up to just below their melting points where precipitation hardening and solid solution strengthening rapidly lose their effectiveness. It is important to obtain highly elongated coarse grains in the ODS superalloys exhibiting excellent high temperature strength⁽⁵⁾⁻⁽⁸⁾. Some studies have been done on the beginning temperature of secondary recrystallization in relation to the gamma prime dissolution and yttria dispersoid coarsening⁽⁹⁾⁻⁽¹¹⁾. However, very little is known about the decisive factor which controls size and morphology of secondary grains⁽¹²⁾⁻⁽¹⁶⁾. Therefore, the mechanism of secondary recrystallization has not been well understood, especially on the main pinning site to abnormal grain growth.

In the present study, Inconel^{††} MA6000 as an ODS superalloy was undertaken to examine the abnormal grain growth behavior.

II. Experimental Procedure

The ODS superalloy MA6000 was manufactured in the process of mechanical alloying and subsequently con-

solidated by hot extrusion. An as-received extruded bar of 38 mm diameter was employed in this investigation. The chemical composition of MA6000 is shown in Table 1.

From the extruded bar, the rod specimen 5 mm in diameter was produced by an electro-discharge machine along the extrusion direction. The directional recrystallization was carried out in an argon-hydrogen mixture gas at the high temperature gradient of 70 K/mm and the maximum annealing temperature of 1553 K. The interspacing between the hot zone and cooling water jacket is 20 mm and the travel speed of an induction coil is 5 mm/h. The specimen heated by the induction coil was cooled directly by the water jacket. Thus, it was possible to get a high temperature gradient.

Microstructures were studied by means of scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The grain boundary characterization was made by observing selected area channeling patterns (SACP). Foils for TEM observations were prepared by mechanical polishing of electro-discharge machined 3 mm diameter discs and then electrolytical polishing using a twin jet technique at 268 K and 20 V in a solution of perchloric acid and ethanol.

Table 1 Nominal composition of Ni-base superalloy, MA6000, in mass%.

Cr	W	Mo	Ta	Fe	Mn	Al	Ti	Mg	C	Y ₂ O ₃	B (ppm)	Ni
14.68	3.91	1.96	2.07	1.19	4.09	4.32	2.38	0.15	0.05	1.1	105	bal

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III. Results and Discussion

The as-extruded MA6000 was found to consist of fine equiaxed grains having an average grain size of about $0.25\text{ }\mu\text{m}$. These grains included a low density of dislocations and a lot of annealing twins as well as uniformly dispersed fine yttria particles of 10 to 80 nm in diameter. However, it was difficult to find the directional alignment of large round shaped oxide particles in TEM observations, which will be discussed later. The similar observations have been reported in MA6000⁽⁹⁾⁽¹²⁾⁽¹⁴⁾.

In order to examine the distribution of oxide particles, the SEM observations were carried out in longitudinal and transverse sections of the as-extruded bar. It was revealed that large round shaped oxide particles were distributed inhomogeneously in the mechanical alloying and extrusion process. As shown in Fig. 1, round shaped oxide particles are well aligned along the extruded direction in the longitudinal section, while in the transverse section they delineate cell structure. However, uniformly dispersed fine yttria particles could not be observed at this magnification.

Figure 2(a) shows a typical example of SEM micrographs indicating a grain boundary between secondary grains grown by directional recrystallization at 1553 K and 5 mm/h, where the wavy grain boundary

Fig. 1 Scanning electron micrograph of as-extruded MA6000 showing the distribution of oxide particles, (a) longitudinal section and (b) transverse section.

exists along the arrows. One can see a white dotted row of oxide particles below the grain boundary in a similar way to Fig. 1(a). High magnification views of the squared regions A and B in Fig. 2(a) are shown in Fig. 2(b) and (c), respectively. Careful observations of the orientation of

Fig. 2 (a) Scanning electron micrograph of directionally recrystallized MA6000 in the condition of 1553 K and 5 mm/h, showing the grain boundary and the alignment of oxide particles to the extrusion direction. (b) and (c) are high magnification views of the squared regions A and B in (a), respectively.

Fig. 3 Selected area channeling patterns taken at the positions above (a), on (b) and below (c) the grain boundary shown in Fig. 2(a).

Fig. 4 (a) Transmission electron micrograph showing the secondary recrystallized grain boundary with oxide particles and flake-like phase. (b) is a high magnification view at the grain boundary of (a).

cuboidal gamma prime particles in Fig. 2(b) reveal the location of a grain boundary. However, there is no change in orientation of the cuboidal gamma prime particles above and below the oxide particle row in Fig. 2(c).

Figures 3(a), (b) and (c) are the SACP's taken from areas above, on and below the grain boundary in Fig. 2(a), respectively. It is evident that the pattern is changed at the grain boundary in Fig. 3(b). It was confirmed that

the pattern of Fig. 3(c) was not changed above and below the oxide particle row. Here, it should be noted that the grain boundary does not exist on the row of oxide particles but on the flake-like phase. This means that the main obstacle to abnormal grain growth is not the round shaped oxide particles but the flake-like phase.

Figure 4(a) shows the flake-like phase on the grain boundary, which was observed by TEM. Figure 4(b) is a high magnification view of Fig. 4(a). The matrix contains cuboidal gamma prime particles. However, because of the small mismatch between the two aligned fcc lattices of gamma and gamma prime, little strain contrast can be seen and it is difficult to discern the gamma prime particles clearly under the bright field condition. In these

figures round-shaped oxide particles 80 to 200 nm in diameter are present both on the grain boundary and inside the grains. This suggests that the oxide particles play a minor role in secondary grain growth. The size of the flake-like phase was distributed in the range of 0.2 to 0.4 μm in width and 0.4 to 2.5 μm in length.

Figure 5 shows the results on analytical TEM of the phases appeared in Fig. 4(b). Figures 5(a), (b), (c) and (d) correspond to Y_1 , P_1 , O_1 and M in Fig. 4(b), respectively. Analytical TEM results of Y_n , P_n and O_n ($n=2, 3$) were similar to those of Y_1 , P_1 and O_1 , respectively. Since the size of yttria particles is too small, Fig. 5(a) contains Ni peaks of the matrix. But Y_1 in Fig. 4(b) is determined to be yttria in comparison with Fig. 5(d). It is apparent that a flake-like phase is Cr rich, while a large round-shaped particle is Ti-rich phase. Large round-shaped Al oxides were frequently observed both on the grain boundaries and inside the grains in other areas. Therefore, it is concluded that an important pinning site to abnormal grain growth is the Cr-rich phase. Thus, the size and morphology of secondary grains in Inconel MA6000 are controlled by the distribution of the Cr-rich phase which is probably introduced during mechanical alloying and extrusion process. We have observed the Cr-rich phase in the as-extruded bar by analytical TEM, although it has not been found by SEM. The details of these results will be published elsewhere.

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Fig. 5 Analytical TEM results of the fine oxide particle (Y_1), large oxide particle (O_1), flake-like phase (P_1) and matrix (M) in Fig. 4(b). (a), (b), (c) and (d) correspond to Y_1 , P_1 , O_1 and M, respectively.