Materials & Design 130 (2017) 69–82

Contents lists available at ScienceDirect

Materials & Design

journal homepage: www.elsevier.com/locate/matdes

Effect of thermal stability of γ′ phase on the recrystallization behaviors of Ni-based single crystal superalloys

Lu Qin[a], Yanling Pei[a], Shusuo Li[a], Xinbao Zhao[b], Shengkai Gong[a,*], Huibin Xu[a]

[a] School of Materials Science and Engineering, Beihang University, No. 37 Xueyuan Road, Beijing 100191, PR China
[b] Xi'an Thermal Power Research Institute Co., Ltd., Xi'an 710032, PR China

ARTICLE INFO

Keywords:
Recrystallization
Superalloy
Thermal stability
Order

ABSTRACT

The recrystallization may deteriorate the mechanical properties of single crystal superalloys greatly, which concerns the engineers and scientists in the aviation industry. Recrystallization behaviors after grit-blasting and annealing at different temperatures of a γ′-richen experimental single crystal alloy IC32 (the as-cast and heat-treated) were investigated. Samples of the as-cast ones are less prone to recrystallization than the heat-treated ones. In the as-cast state, the interdendritic regions show a stronger resistance to recrystallization than the dendritic regions. According to the results, we can conclude that the size characteristics and thermal stabilities of the γ′-phases play important roles in deciding the recrystallization resistances of the Ni-based single crystal superalloys.

1. Introduction

Ni-based single crystal superalloys, which exhibit increased creep strength in comparison to conventionally cast and directionally solidified superalloys due to the complete elimination of grain boundaries, are used for turbine blades and vanes in order to increase the performance and efficiency of aeroengines and industrial gas turbines [1]. However, the recrystallization (RX), which is caused by plastic deformation and subsequent heat treatment or high temperature application, may deteriorate the mechanical property of superalloy vastly [2–6].

High solubility and diffusivity assume different importances in the recrystallization behaviors of the nickel-base superalloys with varying γ′ volume fractions [7]. The longest rupture life was attained by an alloy with γ′ contents in vicinity of 65–70% in 900 °C/392 MPa and 1100 °C/137 MPa [8]. The creep strength of the γ′-strengthened nickel based superalloys, especially at temperatures above 1100 °C, is dependent on the γ′ volume fraction [9]. Therefore γ + γ′ alloys with high volume fractions of γ′ phases are extremely meaningful in practical use. The research on recrystallization behaviors of these alloys is necessary.

In order to clarify the underlying key factors influencing the recrystallization resistances of γ + γ′ alloys with high volume fractions of γ′ phases, many researchers have devoted themselves to the study of the recrystallization behaviors of conventional nickel-based superalloys (CMSX-11B [5], PWA1483 [5], CMSX-2 [10–11], CMSX-6 [5], SRR99 [5], CMSX-4 [11], DD6 [12–14] et al.) and γ′-richen alloys with higher volume fractions of γ′ phases (IC6 [15–16], IC21 [17–19]). According to previous studies [20–22], the secondary phases such as carbides and eutectics in the interdendritic regions can help to suppress the recrystallization process by hindering the migration of the large angle boundary or promote the recrystallization process by stimulating the PSN (particle-stimulated nucleation) mechanism besides the interdendritic phases. Besides there have been extensive studies about the influence of macroalloying elements related to γ + γ′ microstructures on the recrystallization behaviors of γ + γ′ phases with high volume fraction of γ′ phases [19,23–25]. R. Yoda et al. [23] reported that the recrystallization temperature of Ni–20Cr alloy can be raised to the similar extent by including up to about 2%Al or about 3%Ti. Besides, the research on the influence of the matrix solute strengthening elements on the single crystal superalloys [19,24,25] indicated that the matrix solute strengthening elements tend to enrich at dislocations and grain boundaries and prevent dislocation movement and grain boundary migration. In addition, the difference of microstructures can also result from the alloy state. The as-cast alloys show stronger resistance to recrystallization than the heat-treated alloys [12,13,25]. L. Wang et al. [26] concluded that recrystallization nucleation may take place by strain induced boundary migration in the dendrite cores and particle stimulated nucleation occurs in the interdendritic regions.

However, the key factors which influence the recrystallization resistances of γ + γ′ alloys with high volume fractions of γ′ phases need to be further clarified on the basis of previous study. For this purpose, a carbon-free γ′-richen experimental single alloy IC32 with

* Corresponding author.
E-mail address: gongsk@buaa.edu.cn (S. Gong).

http://dx.doi.org/10.1016/j.matdes.2017.05.044
Received 11 March 2017; Received in revised form 28 April 2017; Accepted 14 May 2017
Available online 17 May 2017
0264-1275/

higher γ′ volume fraction, which is promised to show excellent thermal stability, is chosen. By comparing the recrystallization behaviors of the three microstructures in the two alloy states, a deeper understanding of the effect factors of recrystallization behaviors of these alloys can be acquired.

2. Experimental

A γ′-richen experimental single crystal alloy, IC32, was used in the present study with the nominal composition of Ni-(7–9) Al-(7–9) Mo-(2–4) Re-(2–4) Ta (in wt%). The master alloy was firstly prepared by a vacuum induction furnace, and then the single crystal alloy was produced by screw crystal selection method in a DZG-0.025 directionally solidified furnace. A near-[011] direction rod was used to cut the (010) plane, which can be the reference plane to eliminate the influence of orientation. One half of the samples were used as the as-cast ones and the other half of the samples were heat treated. The as-prepared alloy was cut into small pieces mechanically ground and polished, and then grit blasted by Al_2O_3 balls for 1 min at 0.4 MPa. The deformed samples were encapsulated in a quartz tube under argon atmosphere and annealed at different temperatures in the range of 950–1340 °C to determine the temperature dependence of microstructural evolution in the recrystallization behavior of the γ′-richen alloy.

Measurements of the γ′ phase area fraction were performed by image analysis on black and white images of samples quenched after heat treatment for 1 h at 1000 °C, 1050 °C, 1100 °C, 1150 °C, 1200 °C, 1250 °C, 1300 °C, respectively.

The microstructures of the specimens were characterized by a Leica DM4000 optical microscope (OM), a SU8010 field emission-scanning electron microscopy (FE-SEM) and Quanta 200F field emission-scanning electron microscopy (FE-SEM). The overall average recrystallization depths were measured from at least eight optical metallographs for each sample.

Differential scanning calorimetry (DSC) was used to determine the γ′ solvus temperatures. The temperature program comprised heating at 20 °C*min^{-1} to 1000 °C, and subsequently at 10 °C$*$$min^{-1}$ to 1480 °C with a dwell of 10 min. Experiments were carried out in a dynamic Ar environment.

The chemical compositions of the microstructures were quantified on the as-polished sample surface by JXA8230 electron probe microanalyser with wavelength dispersive spectroscopy (EPMA-WDS), at least 5 points with similar values were detected in the dendrite core in the as-cast state, the interdendritic regions in the as-cast state and the heat-treated state, respectively. As analyzer crystals of EPMA-WDS test, TAP crystal is for Al, LIFH crystal for Re, Ta, PETJ crystal for Mo, LIF crystal for Ni. The accelerating voltage, beam current and beam diameter for this analysis were 20 kV, 20 nA and 3 μm, respectively.

JMatPro 7.0 was used to calculate the chemical composition of γ′ phases of γ + γ′ alloys and the misfit of the γ + γ′ alloys.

The effective atom model based on Embedded Atom Method (EAM) potential [27] was used to study the relationshop between the structural stability and the long range order parameter of γ′ phases. The calculation range of Ni_3Al reaches the fourth nearest neighbor (4NN), which is determined by the cutoff radium.

3. Results

3.1. Original microstructure

In Fig. 1(a), the primary arms of the [011] direction dendrites arrange parallelly and the secondary arms on the two sides are symmetric, which is consistent with the characteristics of the (010) face. The Laue pattern indicates the cutting face is close to (010), which can be regarded as the reference plane. The microstructures are inhomogeneous and comprise of dendritic regions (DR) and interdendritic regions (IDR). Primary dendritic arms (PDA) and secondary dendritic arms (SDA), which compose the dendritic regions (DR), cover most of the (010) plane. Fig. 1(b–d) shows the microstructures of the IC32. In the as-cast state, γ′ phases in dendrites are butterfly like distorted cubes with poor alignment, as is shown in Fig. 1 (b). While in interdendritic regions, γ′ phases are irregular and blocky γ′ exists (Fig. 1(c)). Standard heat treatment dissolves the γ′ phases and improves γ′ cube morphology and alignment (Fig. 1(d)). Four different kinds of morphologies of γ′ are displayed in the microstructures of the alloys of as-cast and heat-treated state, which may determine the recrystallization characteristics of different alloy states.

3.2. Deformation state

Fig. 2 shows the morphology of slip bands on (001) section of the samples grit-blasted on the (010) plane. The distribution of the slip bands in the as-cast samples is inhomogeneous. Near the zone of the IDR, only vague single slip bands can be seen on the surface (Fig. 2(d)). Further away from the IDR, a little multiple slip bands appear besides the slip bands (Fig. 2(b)). While in the dendritic cores (Fig. 2(c)), obvious multiple slip bands can be seen on the surface. In comparison, dense multiple slip bands distribute on the surface in the heat-treated samples. While deeper into the interior of the samples, the multiple slip bands change into single slip band owing to the less deformation. The depths of the total deformation layer of IC32-HT and IC32-DR are 35.77 μm and 38.29 μm, which are larger than that in the IC32-IDR (23.65 μm). Besides, the depths of multiple slip layer in the three kinds of microstructures are 22.60 μm, 28.16 μm and 15.74 μm, respectively. Among them, the depth in the IC32-IDR is obviously shallower than the ones in the other two conditions.

3.3. Recrystallization behavior

Fig. 3 shows the surface morphology of the grit blasted samples after annealing at three different temperatures at which recrystallization behaviors have changed greatly. Fig. 3(a) shows recrystallization grains nucleating locally both in the dendritic regions and the interdendritic regions. In the interdendritic regions, recrystallization grains locate besides the blocky γ′ phases, while in the dendritic regions, recrystallization grains distribute dispersively. Specifically, strain energies are stored in the dendritic cores or intensively besides the blocky γ′ phases in the interdendritic regions after grit-blasting. In the former case, subboundary which form should can transform into large angle boundary. While in the latter case, PSN (particle-stimulated nucleation) mechanism should act [26]. In Fig. 3(b), surface recrystallization (SRX) appears in the dendritic regions and cellular recrystallization (CRX) locates in the interdendritic regions. The boundary of the surface recrystallization (SRX) clearly draws the boundary of the dendrites. As the temperature rises, the grains in the dendritic regions grow gradually and the cellular recrystallization in the interdendritic regions reduces continuously. At 1300 °C, a little cellular recrystallization is still located in the interdendritic regions. In comparison, the temperature dependence of the microstructural evolution in the heat treated samples shows a quicker characteristics. Generally, the recrystallization microstructure of the heat treated samples is more uniform. At 1200 °C, most of the surface is covered by the cellular recrystallization (CRX) (Fig. 3(d)). When the temperature is raised, the grains are replaced by the surface recrystallization (SRX) quickly and the sizes of the grains are larger apparently. At 1250 °C, the surface is covered by the surface recrystallization and the primary recrystallization behavior has completed (Fig. 3(e)). The grains continue to grow at 1300 °C (Fig. 3(f)). Twins are appearing in the recrystallization grains in both of the two states. Twins are helpful to promote the production of the large angle boundary and the nucleation of the recrystallization [25,28–29].

Fig. 4 shows the recrystallization behaviors of the deformed samples after annealing at different temperatures. The characteristics of the recrystallization behaviors of the as-cast samples can be seen in