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Thermal evolution behavior of carbides and γ′ precipitates in FGH96 superalloy powder

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ABSTRACT

The characteristics of rapidly solidified FGH96 superalloy powder and the thermal evolution behavior of carbides and γ′ precipitates within powder particles were investigated. It was observed that the reduction of powder size and the increase of cooling rate had transformed the solidification morphologies of atomized powder from dendrite in major to cellular structure. The secondary dendritic spacing was measured to be 1.02–2.55 μm and the corresponding cooling rates were estimated to be in the range of 1.4×10^4–4.7×10^5 K·s^{-1}. An increase in the annealing temperature had rendered the phase transformation of carbides evolving from non-equilibrium MC′ carbides to intermediate transition stage of $M_{23}C_6$ carbides, and finally to thermodynamically stable MC carbides. The superfine γ′ precipitates were formed at the dendritic boundaries of rapidly solidified superalloy powder. The coalescence, growth, and homogenization of γ' precipitates occurred with increasing annealing temperature. With decreasing cooling rate from 650 °C·K^{-1} to 5 °C·K^{-1}, the morphological development of γ' precipitates had been shown to proceed from spheroidal to cuboidal and finally to solid state dendrites. Meanwhile, a shift had been observed from dendritic morphology to recrystallized structure between 900 °C and 1050 °C. Moreover, accelerated evolution of carbides and γ' precipitates had been facilitated by the formation of new grain boundaries which provide fast diffusion path for atomic elements.

1. Introduction

FGH96, a nickel-base superalloy, is a strong candidate for high temperature gas turbine disc applications owing to its combination of excellent high temperature rupture life, creep strength, and fatigue crack growth resistance [1,2]. Superalloy components are prepared by powder metallurgy route. Firstly, the highly alloyed superalloy powder is produced by Ar gas atomization, which can virtually eliminate macrosegregation due to extremely fast cooling rate of small molten droplets. Subsequently, the fully densified specimens with homogeneous microstructure are obtained by hot isostatic pressing and reasonable heat treatment. An essential merit of powder metallurgy route is its capability to yield the higher degree of homogeneity, the higher levels of supersaturation, and refined grain size [3,4].

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The characteristics such as particle size, particle size distribution, and microstructure of the atomized powder as well as the thermal evolution behavior of the powder particle exert much influence on the resulting properties of the powders [5,6]. The investigation of loose powder is very important because it provides the valuable information for the powder production and the subsequent thermo-mechanical processing. On the one hand, the powder microstructure reveals the processing conditions used in gas atomization. The dendritic structure reflects the solidification condition. Moreover, the secondary dendrite arm spacing is closely associated with particle size and cooling rates. Therefore, the characterization studies of powder microstructure are of fundamental importance to optimize the atomization parameters. On the other hand, the microstructural features of the atomized powder are often correlated with the densification behavior as well as the formation of prior particle boundary [7,8]. Powder heat treatment is one of the techniques that reduces or even eliminates the prior particle boundary. The information on the microstructural evolution provides valuable information not only for the selection of consolidation parameters but also for the optimization of powder heat treatment. The fundamental mechanism of powder heat treatment is based on the control of carbides precipitation. Dahlen et al. [9] have deduced that powder heat treatment can stabilize the carbides in the form of MC and $M_{23}C_6$ at the interior of powder, resulting in the hindrance of reprecipitation at the prior particle boundary. However, the underlining mechanisms are not yet fully comprehended. There is still a considerable potential for the optimization of PM superalloys by improving the knowledge regarding the response of powder microstructure to heat treatment.

The precipitates in the nickel-base superalloy are primarily the γ' phase and carbides. The knowledge about the γ' is of fundamental importance for devising the proper thermo-mechanical processing parameters. The microstructural formation of Ni-base superalloy is based on the evolution of carbides and γ' precipitation. However, there exist a complex interaction between carbides and γ' precipitates during heat treatment. Up to now, a large amount of researches have investigated the evolution of γ' precipitates following the solution treatment [10,11]. However, very little attention has been paid to the phase transformation of carbides and the precipitation behavior of γ' phase in the rapidly solidified superalloy powder without solution treatment. The aim of the present investigation is to characterize the rapidly solidified superalloy powder and the thermal evolution behavior of γ' precipitates and carbides in loose FGH96 superalloy powder.

2. Experimental

Argon atomized superalloy FGH96 powders was supplied by Institute of Aeronautical Materials (China). The chemical composition of the starting powders is expressed in wt%, 0.03C, 16Cr, 13Co, 4Mo, 4W, 0.8Nb, 2.2Al, 3.7Ti, 0.011B, 0.036Zr, balance Ni. Powder particles were canned in vacuum (1×10^{-5} Pa) and annealed in the temperature range of 750–1150 °C for 1 h. In order to investigate the influence of cooling rate on the precipitation behavior of γ' phase, superalloy powder was canned in steel tubes and cooling tests were conducted on a Gleeble-1500 heat simulating equipment. The specimens were heated up to 1170 °C, held for 5 min, and then cooled at four cooling rates: 5, 50, 250 and 650 °C·min^{-1}.

The separation of the carbides from the particles was performed by electroless extraction method by using the solution composed of 10% $CuCl_2$ and1% tartaric acid. A filtering process was performed by using a special filtration unit fitted with a glass microfibre filter paper. The powder was accurately weighed before and after extraction. In order to study the powder microstructure, the powder particles were mounted, polished, and etched in a solution of 5 g $CuCl_2$, 100 ml of HCl and 100 ml of ethanol. The phase analysis of the extracted residue was studied on Siemens D 5000 X-ray diffraction meter using Cu radiation. As for γ' precipitate observation, the specimens were prepared by electroless etching using the solutions of 33% HNO_3, 33% acetic acid, 33% H_2O, and 1% HF. The examination of γ' precipitates was conducted on a ZEISS-SUPRA55 field emission scanning electronic microscope (FESEM). The powder samples for TEM observations were prepared by mounting the particles in nickel foils through the electrodeposition method. From the electrodeposited foils, 3 mm disks were punched and thinned by ion milling. The observation of the extracted residuals was performed on JEM-2100 transmission electron microscope (TEM) equipped with energy dispersive X-ray spectroscopy (EDS).

3. Results and Discussion

3.1. *Characteristics of Atomized Powder*

In order to establish the relationship between solidification microstructure, particle size, and cooling rates, the microstructural characterization of powders have been performed. Fig. 1 shows the microstructure of atomized powder with varying particle size. The higher cooling rate results in the smaller particles with the size range of 22–41 μm exhibiting a totally dendrite structure (Fig. 1a and b). Moreover, a large amount of white precipitates are distributed in interdendritic interstices or at cell boundaries. On the contrary, the smaller cooling rate results in the large particles (93 μm and 127 μm) rendering the formation of a mixed microstructure composed of dendrite structure and cellular morphology (Fig. 1c and d).

The main advantage of using the high gas velocity atomization to produce superalloy powder is the refining of the microstructure. The degree of refinement has been characterized by the secondary dendrite arm spacing. Fig. 2 shows the variation of secondary dendrite arm spacing (S) with powder size (d). The secondary dendrite arm spacing is measured to be 1.02–2.55 μm in the powder particle size range of 22–127 μm. The relationship between secondary arm space and particle size is expressed as:

$$\lg S = -0.714 + 0.536 \lg d \quad (1)$$

A key factor in gas atomization controlling the powder particle size and the powder microstructure is the cooling rate. The cooling rate has been estimated through the convection heat transfer principle. During the solidification process, the