

# Microstructure evolution and mechanical properties of disk superalloy under multiplex heat treatment

C.L. Jia<sup>a,b,\*</sup>, C.C. Ge<sup>a</sup>, Q.Z. Yan<sup>a</sup>

<sup>a</sup> School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China

<sup>b</sup> Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, Beijing 100095, China

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## ABSTRACT

Turbine disks of a powder metallurgy (PM) nickel-based superalloy FGH100 were subjected to multiplex heat treatment (MHT) and investigated in terms of the improvements in their mechanical properties, which are critical for the application of these alloys in aero-engines. The effects of MHT were assessed by microstructure evolution and tensile test measurements. The results show that the microstructural characteristics of the constituent  $\gamma'$  phase in the alloy change after repeated heat treatment, while the average grain sizes vary slightly. An optimized microstructure and excellent overall properties were achieved for FGH100 disk forgings by employing cyclic MHT. This study reports significant implications for the maintenance of turbines.

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## 1. Introduction

Powder metallurgy (PM)-processed nickel-based superalloys are an important class of high-temperature structural materials used for the manufacturing of turbine disks in aero-engines [1,2] because they exhibit characteristics such as high strength, ductility, fracture toughness, and good fatigue-resistant properties at both room and elevated temperatures [3,4]. These properties of PM disk superalloys are attributed to the precipitation of the ordered  $\gamma'$  phase within a disordered  $\gamma$  matrix [5]. The microstructure of disk superalloys, especially in terms of the morphology, as well as the spatial and size distribution of  $\gamma'$  precipitates, plays a very important role in determining the mechanical properties of disk superalloys [6,7].

The heat treatment of the turbine disk components is an important step for achieving an optimum microstructure and mechanical properties [8,9], which can be achieved by the typical processing of turbine disk blanks by single conventional heat treatment, termed as solution and aging treatment [10]. The microstructure evolution of disk superalloys is significantly affected by heat treatment. Previous studies have reported that the mean size of  $\gamma'$  precipitates increases with thermal exposure time during aging treatment(AT) [11,12]. Some researchers have pointed that

the cooling rate after solution treatment(ST) affects the size and shape of  $\gamma'$  precipitates [13–15]. The more rapid the ST cooling rate, the finer the intragranular  $\gamma'$  precipitates [9,16]. However, the rapid cooling rate after ST, e.g., quenching in oil from ST, can increase the propensity for the quench cracking of disk forgings [17]. Therefore, the ST cooling rate is a key parameter of heat treatment. In theory, the desired final mechanical properties can be obtained by controlling the process parameters for conventional heat treatment.

However, in practical operation, the poor control of the critical processing parameters in conventional heat treatment often leads to a non-preferable mechanical performance. On the other hand, it is being explored if the sequencing of a second step or multiple heat treatment (MHT) steps could improve and optimize the microstructure or performance of disk superalloys. Recently, in addition to several studies of conventional heat treatment [18–25], studies focusing on the dual microstructure heat treatment (DMHT) of PM disk superalloys have also been reported [26–29]. However, thus far, the MHT of PM disk superalloys have not been reported. For addressing the above-discussed issues, the effect of optimizing the processing parameters of MHT on the microstructure and mechanical properties of a spray-formed 3rd generation PM superalloy FGH100 was investigated. This study aims to provide proof and experimental basis for the development of various heat treatment methods for the purpose of optimizing the microstructure and restoring the performance in disk superalloys.

\* Corresponding author at: Science and Technology on Advanced High Temperature Structural Materials Laboratory, Beijing Institute of Aeronautical Materials, Beijing 100095, China.

E-mail address: [biamjcl@163.com](mailto:biamjcl@163.com) (C.L. Jia).

## 2. Materials and processes

FGH100 billets were prepared using spray-forming deposition. The spray-deposited billets were processed by hot-isostatic pressing (HIP) at 1140 °C and 150 MPa for 3 h for achieving a high degree of densification. The pressed preforms were isothermally forged (IF) to produce several pancake-shaped forgings having a diameter of approximately 210 mm and a height of 40 mm. The main chemical composition of disk forgings is C 0.04, Cr 12.80, W 4.32, Mo 2.78, Co 21.34, Al 3.52, Ti 3.34, Nb 1.36, Ta 1.61, B 0.015, Mg 0.02, and Ni Bal (mass fraction,%). The disk forgings were heat-treated by a process with specific parameters (Process-A). The parameters of process-A are as follows: a step heating rate (solution treatment) of 1130 °C (1 h), followed by two-step aging at 850 °C (4 h) and 770 °C (8 h), with slow cooling in air. Each of the treated disk forgings were cut into two symmetrical parts using a line-cutting machine. Out of the two sliced halves per disk, one was used as the sample for performance tests, while the other was further heat-treated using a different set of parameters (Process-B). The heat treatment procedure of process-B was as follows: a step heating rate (solution treatment) of 1130 °C (1 h), followed by an aging step identical to that in process-A but with a different cooling rate after ST. Effectively, the second halves of the disk blanks were heat-treated by process-A+process-B. Fig. 1 shows the schematics of Process-A and Process-B, in which the ST cooling rates of Process-A and Process-B are 5 °C/min and 110 °C/min, respectively.

Later, for tensile and creep tests, specimens were prepared out of the two groups: A and A+B-heat treated pieces. The machining of tensile test specimens, with a gage diameter of approximately 5 mm and gage length of approximately 25 mm, was performed by the Beijing Institute of Aeronautical Materials (BIAM). Tensile properties at room temperature (RT) were tested on an MTS810.22 m hydraulic universal material testing machine at the BIAM. Tensile tests at 705 °C were performed at the BIAM in using the Instron5982 tensile machine equipped with a resistance heating furnace and an axial extensometer. The tensile tests at room and elevated temperatures were performed according to HB5143 and HB5195 (the Aviation Standard of China), respectively. The machining and testing of creep specimens were performed at the BIAM. Special creep specimens with a gage diameter of 5 mm and gage length of 26.6 mm were machined and tested in uniaxial lever arm constant load creep frames using resistance heating furnaces and shoulder-mounted extensometers. Creep tests were performed according to HB5151 (the Aviation Standard of China). Two samples were tested for each test. For achieving the dispersion of the same type of measurements, the standard deviation of the test results was calculated, and the Origin 8.0 software was used to analyze all data.

Samples were cut from the HIPed alloy blocks and the tensile specimens for microstructural observation. Specimens for the

observations of  $\gamma'$  precipitates were prepared by electropolishing and electrolytic etching after mechanical grinding. The electrolytic polishing reagent used was 20 vol%  $\text{H}_2\text{SO}_4$ +80 vol%  $\text{CH}_3\text{OH}$ , and the polished samples were further electrolytically etched in 9 g  $\text{CrO}_3$ +90 mL  $\text{H}_3\text{PO}_4$ +30 mL  $\text{C}_2\text{H}_5\text{OH}$  at 5 V for 3–5 s. The  $\gamma/\gamma'$  microstructure was characterized using the secondary electron mode and the backscattered electron mode (BEM) of a Zeiss Ultra 55 field-emission gun scanning electron microscope (FEGSEM) and FEI Nova NanoSEM450 FEGSEM equipped with an energy-dispersive spectrometer (EDS).

TEM foils were prepared by the conventional route, which consist of mechanical grinding and polishing of a lamella with a diameter of 3 mm, followed by twin-jet electropolishing using a 10% perchloric acid and 90% ethanol solution at –30 °C. TEM characterization was conducted on an FEI Tecnai G2 F20 field-emission gun TEM operating at 200 kV. Representative regions were imaged at different magnifications for capturing the relevant secondary and tertiary  $\gamma'$  precipitates in the alloy.

The lattice constants of the  $\gamma$  and  $\gamma'$  phases in the block specimens were measured using a D8 DISCOVER X-ray diffractometer (XRD). The  $\gamma/\gamma'$  mismatch was calculated by the following equation:

$$\delta = \frac{2(a_{\gamma'} - a_{\gamma})}{a_{\gamma'} + a_{\gamma}}$$

Here,  $\delta$  represents the lattice mismatch, and  $a_{\gamma'}$  and  $a_{\gamma}$  represent the lattice constant of  $\gamma'$  phase and  $\gamma$ , respectively.

The  $\gamma'$  precipitates were subjected to image analysis. ImageJ software was used for determining the  $\gamma'$  area fraction and  $\gamma'$  particle size distributions (PSD). The diameter of individual precipitates was calculated on the basis of equivalent circles, which have the same area as the measured  $\gamma'$  phase [30]. Origin 8.0 software was employed for obtaining PSDs for  $\gamma'$  phases. Intragranular precipitates with a diameter above 100 nm were classified as secondary  $\gamma'$  precipitates and those below a diameter of 100 nm were considered as tertiary  $\gamma'$  precipitates.

Specimens for grain structure observations were chemically etched in a solution of 5 g  $\text{FeCl}_3$ +100 mL HCl for revealing the grain morphology and grain boundary characteristics. The grain structures were photographed using a Leica DMR optical microscope.

Finally, the experimental results of the spray-formed superalloy FGH100 disk forgings under two heat treatment processes were compared and analyzed.

Incidentally, this study aims to judge and verify whether the heat-treated disk components can be subjected to repeated heat treatment for improving the performances of the alloy, not to determine the optimum heat-treatment system of the FGH100 alloy. Thus, we emphasize and focus on the comparison between the results of latter MHT with those of the preceding heat treatment (e.g., contrast of process “A+B” and process “A,” the contrast of process “A+B+C,” and process “A+B”).

## 3. Results and discussion

### 3.1. Microstructure evolution of $\gamma'$ phase

Fig. 2 shows the morphology of the  $\gamma'$  precipitates in the disk forgings after the two heat-treatment processes, in addition to that of the as-preformed alloy. After HIP, the precipitates were mainly composed of a moderate amount of the primary  $\gamma'$  phase with a mean size of approximately 0.4–1.2  $\mu\text{m}$ , a relatively higher amount of the secondary  $\gamma'$  phase, with a mean size of approximately 0.2–0.4  $\mu\text{m}$ , and marginal amount of the fine tertiary  $\gamma'$  phase, with a

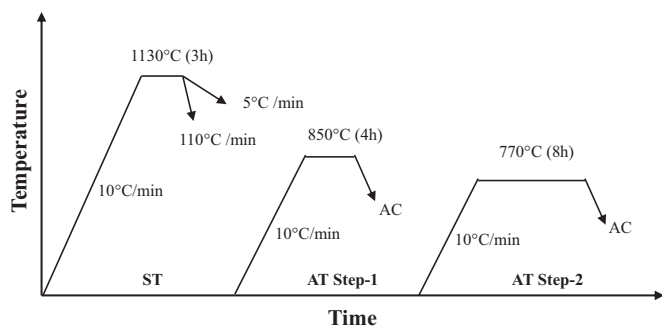


Fig. 1. Schematic of Process-A (ST cooling rate is 5 °C/min) and Process-B (ST cooling rate is 110 °C/min) (AC: air cooling).

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