



# Microstructural evolution and compositional homogenization of a low Re-bearing Ni-based single crystal superalloy during through progression of heat treatment

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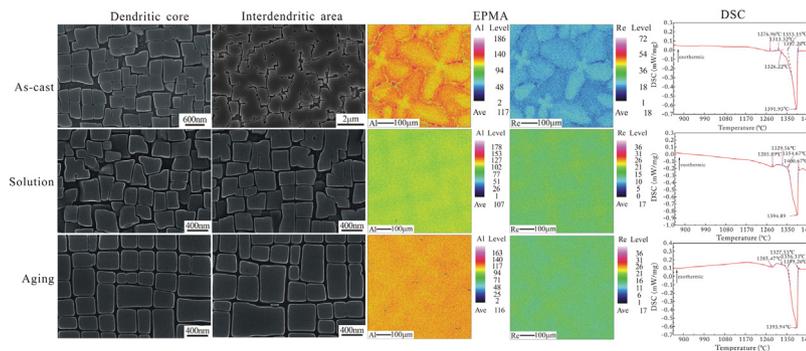
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## HIGHLIGHTS

- Microstructure formation and chemical segregation of as-cast DD6 alloy is studied.
- Microstructure evolution and compositional homogenization of DD6 alloy during heat treatment is investigated.
- Differential scanning calorimetry analysis and thermal-physical changes of DD6 alloy during heat treatment are evaluated.
- Correlations between microstructure evolution, compositional homogenization, and thermo-physical changes of DD6 alloy during heat treatment are established.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Microstructural and chemical heterogeneities may exacerbate the high-temperature mechanical properties of single crystal turbine blades. The evolution of microstructure and compositional homogeneity of an as-cast low Re-containing multicomponent DD6 alloy during through progression of heat treatment was detailedly studied. Using optical microscope, field emission gun scanning electron microscope, electron probe microanalysis, differential scanning calorimetry, and complementary thermodynamic calculations, we investigated the time-dependent size and volume fraction variations of  $\gamma/\gamma'$  eutectic during specific solution heat treatment steps, the evolution of  $\gamma'$  precipitates, carbides, and compositional homogeneity, and the thermo-physical changes of as-cast DD6 alloy during through progression of heat treatment. Based on our experimental and theoretical results, the mechanism underlying the microstructure formation and chemical segregation of as-cast DD6 alloy was clarified. The mechanism of microstructural evolution and compositional homogenization, and the diffusion mechanism of elements that occurs between dendritic and interdendritic regions of as-cast DD6 alloy during through progression of heat treatment were elucidated. The correlations between microstructural evolution, compositional homogenization, and thermo-physical changes of as-cast DD6 alloy during through progression of heat treatment were established.

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## 1. Introduction

Turbine blades, which must be capable of bearing aerodynamic loading, withstanding high centrifugal loading and vibratory stresses, and resisting environmental degradation by oxidation, corrosion, and erosion, are safety-critical rotating components of modern gas turbine engines and directly determine the engine thermodynamic efficiency and performance [1–5]. Ni-based superalloys have been the optimal materials employed exclusively for manufacturing turbine blades of aircraft engines and industrial gas turbines because of their excellent thermal and mechanical properties at elevated temperatures [1–4,6]. The superior mechanical properties of Ni-based superalloys at high temperatures originate principally from the precipitation of a comparatively high volume fraction of thermodynamically stable L1<sub>2</sub>-ordered  $\gamma'$  strengthening phase, which appear as cubes coherently embedded in the disordered face-centered cubic solid solution  $\gamma$  matrix to impede dislocation motion [4,7–14]. To achieve the increasingly demanding high-temperature capabilities and creep strength requirements of turbine blades [1–3,6,15], Ni-based superalloys have been continuously developed from conventionally cast alloys to the seventh generation single crystal (SX) alloys characterized by adding increasing amounts of dense refractory elements Re, Ru, and Ir to newer generations [16–22], and turbine blades have been successively evolved from globulitically solidified polycrystalline blades to directionally solidified columnar-grained and SX blades [1–3,6,23].

During investment casting to fabricate SX turbine blades, primary  $\gamma$  dendrite solidification is simultaneously accompanied by solute partitioning at solid-liquid interface [24–26]. Some solute elements preferentially diffuse to dendritic cores while other alloying elements preferentially segregate to the interdendritic liquid, which finally leads to the formation of non-equilibrium interdendritic constituents, including coarse, blocky, and incoherent  $\gamma'$  precipitates, a significant fraction of blocky  $\gamma/\gamma'$  eutectic, and minor primary carbides, and results in significant compositional inhomogeneities throughout the microstructure of SX turbine blades [26–29]. The microstructural and chemical heterogeneities directly influence the high-temperature mechanical properties of SX turbine blades [30–31]. The coarse and blocky  $\gamma'$  precipitates and incoherent  $\gamma-\gamma'$  interface are detrimental to the development of strength and creep resistance of SX turbine blades [28,29]. The undissolved  $\gamma/\gamma'$  eutectics are prone to serve as crack initiation sites and deteriorate the high-temperature stress rupture properties of SX turbine blades [32]. The enrichments of W and Re at dendritic cores may precipitate embrittling topologically close-packed (TCP) phases that exacerbate the high-temperature creep and fatigue properties of SX turbine blades, counteracting the original purpose of increasing the refractory levels [24,27,29,33–35]. The as-cast SX turbine blades therefore must be solution heat-treated to dissolve  $\gamma/\gamma'$  eutectic, minimize chemical heterogeneities, eliminate coring, and solutionize as-cast  $\gamma'$  precipitates for reprecipitation in an optimized size and morphology during subsequent aging heat treatments to confer the required mechanical properties at elevated temperatures.

To avoid incipient melting resulted from the severely segregated as-cast microstructure, stepwise solution heat treatment (SHT) schedules are carefully tailored to increasingly homogenize the SX turbine blades and progressively minimize the tendency for incipient melting. The SX turbine blades are commonly solutionized under Ar atmosphere in a vacuum furnace and immediately high-pressure gas furnace quenched, using a high purity forced argon flow, which provides an appropriate cooling rate at the final stage of SHT. After the completion of SHT, the residual  $\gamma/\gamma'$  eutectic and elemental microsegregation could still be observed across the microstructure of SX turbine blades. The SHT of SX turbine blades is largely controlled by the  $\gamma/\gamma'$  eutectic dissolution and compositional homogenization. Most previous studies were primarily focused on optimizing SHT process [25], designing homogenization-solution heat treatments [28,29,36–38], analyzing SHT response [26,27,39,

40], and investigating elemental sublimation [41], elemental vaporization and condensation [42], and microstructural instability [43] of Ni-based SX superalloys during SHT. Though the  $\gamma/\gamma'$  eutectic dissolution and compositional homogenization of Ni-based SX superalloys during SHT has been studied [26,27,40], the time-dependent size and volume fraction variations of  $\gamma/\gamma'$  eutectic during specific SHT steps have not been quantitatively understood. The evolution of compositional homogenization, the diffusion processes of elements that occur between dendritic and interdendritic regions, and the thermo-physical changes during through progression of heat treatment have not been fully investigated. The solution and reprecipitation behavior of as-cast  $\gamma'$  phase and the evolution of carbides during through progression of heat treatment need further investigation.

Numerical modeling approaches have also been proposed for simulating the microstructure and microsegregation evolution during solidification and subsequent homogenization-solution heat treatment of a five-component model Ni-based SX superalloy using one-dimensional (1D) front tracking and phase-field (PF) methods coupled to thermodynamic calculation and kinetic databases [44–48]. Nevertheless, a statistical treatment of data, and the local quasi-equilibrium approximation and multi-binary extrapolation methods have been introduced for 1D front tracking and PF simulation, respectively. Ni-based SX superalloys have complex compositions with as many as eleven different elements present in significant proportions. It is really challenging to obtain accurate thermodynamic and kinetic data for complex multicomponent multiphase Ni-based SX superalloys. Moreover, the simulation region is confined to one dendrite, and the simulation cannot simultaneously predict the solution and reprecipitation behavior of as-cast  $\gamma'$  precipitates, the carbide evolution, and the dissolution behavior of  $\gamma/\gamma'$  eutectic during homogenization-solution heat treatment. The microstructural evolution and elemental homogenization of as-cast Ni-based superalloy during SHT is strongly temperature-dependent. The microstructural evolution is not synchronized with the compositional uniformity, and the dendritic cores and interdendritic regions homogenize at different rates during SHT. For a complex multicomponent multiphase Ni-based SX superalloy, a detailed understanding of the solution and reprecipitation behavior of as-cast  $\gamma'$  precipitates, the dissolution behavior of  $\gamma/\gamma'$  eutectic, the evolution of compositional homogenization, the diffusion processes of elements that occur between dendritic and interdendritic regions, and the thermo-physical changes during through progression of heat treatment is exceedingly necessary for manipulating the microstructures and optimizing the mechanical properties for service applications.

A high content of costly Re in Ni-based SX superalloy could result in the complexity of SHT and precipitating TCP phases during service applications. Low-Re and Re-free SX compositions have accordingly been developed and increasingly emphasized [49–53]. DD6 alloy is a low Re-bearing Ni-based SX superalloy, which possesses superior tensile properties, creep rupture properties, oxidation resistance, and hot corrosion resistance compared with its counterparts PWA1484, CMSX-4, SC180, and René N5 [15,54]. Previous investigations have mainly been concentrated on the grain selection behavior during SX casting [55,56], low/high angle grain boundary [57–59], recrystallization [60–62], high-temperature tensile deformation behaviors [63–65], creep rupture, and fatigue properties [66–69] of DD6 alloy. However, the solution and reprecipitation behavior of as-cast  $\gamma'$  precipitates, the dissolution behavior of  $\gamma/\gamma'$  eutectic, the evolution of carbides and elemental homogenization, the diffusion processes of elements that occur between dendritic and interdendritic regions, and the thermo-physical changes during through progression of heat treatment are rarely reported in the literature. The objective of present research is therefore to detailedly investigate microstructural evolution and compositional homogenization of as-cast DD6 alloy during through progression of heat treatment.

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