

Role of Trace Element Hf in Nickel-Based Powder Superalloys: Postprint

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Abstract

The role of trace element Hf in nickel-based powder superalloys was systematically investigated using analytical techniques including FEG-SEM, TEM, AES, and EDS, as well as physicochemical phase analysis methods. The results indicate that Hf distributes in the interdendritic γ solid solution in a solid-solution state, which helps reduce the prior particle boundary (PPB) microstructure. Hf promotes morphological instability of the γ phase, leading to splitting of large cubic γ phases and a more rapid transition to the low-energy stable preferred morphology of the γ phase. Hf is mainly distributed in the γ phase and MC-type carbides, altering the redistribution of alloying elements among the γ phase, MC-type carbides, and γ solid solution, which is beneficial for eliminating the alloy's notch sensitivity and improving its comprehensive mechanical properties.

Full Text

Preamble

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Title

Function of Microelement Hf in Powder Metallurgy Nickel-Based Superalloys

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Abstract

Hafnium (Hf) is one of the most important microelements in powder metallurgy (P/M) superalloys. Hf modifies the microstructure and drastically improves mechanical properties in P/M superalloys. The effect of Hf in a nickel-based P/M superalloy was systematically studied by means of FEG-SEM, TEM, AES, EDS and physical and chemical phase analysis. Hf mainly distributes in the interdendritic region of the solidification powder in the form of solid solution, which is helpful to reduce prior particle boundary (PPB). Hf facilitates morphology of γ phase to become unstable and enhances the large cubic γ phase to split into smaller ones, so the γ phase turns into a stable state with lower energy faster. Hf is mainly distributed in γ phase and MC carbides, which changes the distribution of elements between the γ phase, MC and γ solid solution, which is beneficial to eliminate notch sensitivity and improves overall mechanical properties of the alloy.

Keywords: powder metallurgy superalloy, Hf, MC carbide, γ phase morphology stability, prior particle boundary (PPB)

Introduction

Reports on the role of Hf added to wrought superalloys first appeared in the late 1950s, but industrial applications did not follow. Until the late 1960s, foreign researchers began investigating the effects of Hf in cast superalloys, yielding numerous results. To date, Hf addition has been studied in at least 42 cast superalloys, with over 20 types currently used industrially.

However, research on adding microelement Hf to nickel-based powder superalloys started relatively late. It was not until the 1970s that Hf was added to powder superalloys such as PA101, AF115, MERL76, NASA II B-11, Astroloy, and EP741NP. In the 1990s, new high-performance powder superalloys including SR3, N18, N19, NR3, NR6, and RR1000 were developed with 0.2%-0.75% (mass fraction) Hf. Research results show that Hf addition can significantly improve high-temperature stress rupture ductility, help eliminate high-temperature notch sensitivity, increase γ phase volume fraction, enhance MC carbide stability, improve fatigue crack propagation resistance, and effectively

eliminate prior particle boundary (PPB) structures. Currently, the most widely used nickel-based powder superalloys are EP741NP, N18, and RR1000.

Nevertheless, studies on the fundamental mechanisms underlying Hf's beneficial effects in powder superalloys remain scarce. Particularly with advances in high-performance powder superalloy research, further investigation is needed on Hf's influence on precipitation kinetics, the relationship between composition, microstructure, and properties, and elemental partitioning behavior among phases.

The present work demonstrates that Hf can enter both γ phase and MC-type carbides, altering elemental partitioning in the γ solid solution, promoting morphological instability of γ phase, and enabling earlier attainment of low-energy stable preferred morphologies. Simultaneously, Hf enhances MC carbide stability, thereby inhibiting carbon diffusion in the γ solid solution and facilitating elimination of PPB structures formed during hot isostatic pressing. This contributes to a more comprehensive understanding of Hf's effects on the composition-microstructure-property relationships and corresponding phase transformation behaviors, providing new experimental foundations for developing high-performance, quality-stable FGH97 alloy turbine disks.

Experimental Materials and Methods

The experimental material was FGH97 nickel-based powder superalloy with varying Hf contents. The nominal composition of FGH97 alloy (mass fraction, %) is: C 0.02-0.06, Co 15.0-16.5, Cr 8.0-10.0, W 5.2-5.9, Mo 3.5-4.2, Al 4.8-5.3, Ti 1.6-2.0, Nb 2.4-2.8, Hf 0-0.89, trace B and Zr, balance Ni. Five Hf contents were used in this work: 0, 0.16%, 0.30%, 0.58%, and 0.89% (mass fraction). Powders with particle sizes of 50-150 nm were prepared by plasma rotating electrode process and consolidated by hot isostatic pressing (HIP) at 1200 °C.

The as-HIPed FGH97 alloy specimens were subjected to standard heat treatment: solution treatment at 1180-1220 °C for 2-4 h followed by air cooling, then three-stage aging treatment with final aging at 700 °C for 15-20 h and air cooling. Additional aging treatments were performed on three Hf-containing alloys (0, 0.30%, and 0.89%) at 750, 800, 850, and 900 °C for 100, 200, 500, and 1000 h followed by air cooling. Solution cooling rate experiments were conducted on a Gleeble-1500 thermal simulator using specimens of 8 mm diameter \times 8 mm. Standard heat-treated specimens were heated to 1200 °C, held for 2 h, then cooled to 500 °C at four cooling rates (10, 3, 0.1, and 0.01 °C/s), and finally water quenched.

Physical and chemical phase analysis was employed to determine the content and composition of γ phase, MC-type carbides, and M_3B_2 -type borides in the five FGH97 alloys with different Hf contents. A JEM-2100 transmission electron microscope (TEM) with energy-dispersive spectroscopy (EDS) was used to identify carbide types and compositions in powder particles; secondary car-

bon extraction replicas were prepared for TEM examination. Microstructure, γ phase morphology, and fracture surfaces were observed using a MEF4A optical microscope (OM), SUPRA 55 thermal field emission scanning electron microscope (FEG-SEM), and JSM-6480LV scanning electron microscope (SEM). γ phase sizes were measured and analyzed using Image-pro Plus 6.0 software.

Standard tensile specimens with a 5 mm gauge diameter were machined to measure tensile properties at 650 °C, stress rupture properties at 650 °C (both smooth and notched specimens with notch radius 0.15 mm), and creep properties at 750 °C for the five FGH97 alloys with different Hf contents. Compact tension (CT) specimens (25 mm \times 25 mm \times 10 mm) were used to measure fatigue crack propagation rates at 650 °C under stress ratio of 0.05, no hold time, and loading frequency of 10–30 cycles/min.

2.1 Metastable MC -Type Carbides in Rapidly Solidified Powder Particles with Hf

[Figure 1: see original paper] shows TEM images, selected area electron diffraction (SAED) patterns, and EDS analysis results of extracted carbides within powder particles of FGH97 alloy containing 0.30% Hf. The SAED pattern (inset in Fig. 1a) identifies the carbide as MC-type. EDS results (Fig. 1b) reveal that the MC-type carbide contains Nb, Ti, Mo, W, Cr, and other elements. Therefore, the MC-type carbides within powder particles are referred to as metastable MC -type carbides.

In FGH97 alloys with Hf contents of 0.16%, 0.30%, and 0.58%, no Hf was detected in MC -type carbides. Only in the alloy containing 0.89% Hf was Hf found in interdendritic MC -type carbides within powder particles, indicating that Hf incorporation into MC -type carbides during rapid solidification is very limited. In rapidly solidified powder particles, γ phase precipitation is almost completely suppressed, and no γ phase was observed. Thus, Hf exists in solid solution in the interdendritic regions of powder particles.

2.2 PPB Structures

PPB structures frequently appear in powder superalloys during HIP. [Figure 2: see original paper] shows OM images of microstructures in as-HIPed FGH97 alloys without Hf and with 0.30% Hf. The Hf-free alloy exhibits distinct PPB structures, while the alloy containing 0.30% Hf shows elimination of PPB structures. No PPB structures were observed in the other three alloys with different Hf additions.

TEM and EDS analysis of precipitates on PPBs in as-HIPed Hf-free FGH97 alloy are shown in [Figure 3: see original paper]. The SAED pattern (inset in Fig. 3a) identifies the precipitates as MC-type carbides, and EDS results (Fig. 3b) indicate the carbides are (Nb, Ti)C.

2.3 Interphase Partitioning Behavior of Hf in the Alloy

Tables 1 and 2 present the partitioning amounts of Hf in various phases of standard heat-treated FGH97 alloys with different Hf contents (mass fraction of Hf in each phase relative to the alloy mass) and the partition ratios: R_1 (Hf content in γ phase to that in MC carbide) and R_2 (Hf content in γ phase to that in γ phase). With increasing alloy Hf content, the relative amount of Hf entering γ phase remains essentially constant, while the proportion entering MC phase gradually increases and that entering γ phase decreases. Consequently, R_1 gradually decreases while R_2 gradually increases. On average, 85.5% of Hf enters γ phase, 8.5% enters MC phase, only 4.5% enters γ phase, and the remaining 1.5% enters M_3B_2 phase. The average values of R_1 and R_2 are 1:0.1 and 1:0.05, respectively, indicating that γ phase is the primary precipitate phase accommodating Hf.

Physical and chemical phase analysis results show that with increasing Hf addition, γ phase content slightly increases (from 61.9% to 62.7%), Hf in γ phase gradually increases while Al decreases, with Hf primarily substituting for Al in γ phase. MC-type carbide content increases (from 0.264% to 0.338%), Hf content in MC carbides increases while Nb and Ti contents decrease, with Hf substituting for Nb and Ti in MC carbides. For example, in FGH97 alloy containing 0.89% Hf, approximately 17% of Ti and Nb are substituted by Hf. These results demonstrate that Hf addition strongly induces redistribution of alloying elements among γ phase, MC carbides, and γ solid solution.

2.4 Morphological Instability of γ Phase

[Figure 4: see original paper] shows SEM images of γ phase morphology in standard heat-treated FGH97 alloys with different Hf contents. Hf content significantly affects γ phase size and morphology. In Hf-free and 0.16% Hf alloys, γ phase is primarily cubic with similar sizes (Figs. 4a and b). With increasing Hf content, γ phase size increases. In the 0.30% Hf alloy, γ phase begins to split into octet small cubes (i.e., a single γ particle composed of 8 cubes) (Fig. 4c). In the 0.58% Hf alloy, γ phase splitting is mostly complete, showing octet small cubic morphology (Fig. 4d). In the 0.89% Hf alloy, γ phase splitting is complete, with significantly reduced size and typical preferred cubic morphology (Fig. 4e). Notably, before splitting, depressions appear at the centers of cubic γ phase edges along $\langle 100 \rangle$ directions (indicated by arrows in Figs. 4c and d), and γ phase size tends to increase with Hf content.

At a cooling rate of 10 °C/s, γ phase in all FGH97 alloys with different Hf contents is cubic, with size increasing significantly as Hf content increases. At 0.1 °C/s, γ phase also remains cubic in all alloys, with no obvious size change with Hf content. [Figure 5: see original paper] shows SEM images of γ phase morphology at cooling rates of 3 and 0.01 °C/s. At 3 °C/s, γ phase morphology shows no significant change among Hf-free, 0.30% Hf, and 0.89% Hf alloys, all being cubic with slight size increase with Hf content (Figs. 5a, c, and e). At

0.01 °C/s, γ phase morphology changes dramatically (Figs. 5b, d, and f), with notches appearing at edges of irregular γ particles, indicating splitting has occurred. Compared with the Hf-free alloy (Fig. 5b), splitting is much more pronounced in the 0.30% Hf alloy (Fig. 5d). Due to interference from tertiary γ phase (fine spherical γ precipitated during aging) growth during splitting, the splitting initiation sites are irregular, but splitting clearly begins at the four edges of cubic γ phase $\{100\}$ planes. In the 0.89% Hf alloy, large γ phase has completed splitting, showing octet small cubic configurations (Fig. 5f). Thus, at low cooling rates, increased Hf content promotes splitting of cubic γ phase particles, enabling earlier formation of the preferred octet small cubic morphology.

[Figure 6: see original paper] shows SEM images of γ phase morphology during aging at 750 °C in FGH97 alloy containing 0.30% Hf. After 100 h aging, some γ phase reaches the critical splitting size and begins to split. After 500 h, most γ phase reaches critical size and undergoes splitting. After 1000 h, splitting is complete with reduced size, indicating stable morphology after splitting during aging.

[Figure 7: see original paper] shows SEM images of γ phase morphology after 100 h aging at different temperatures in FGH97 alloy containing 0.30% Hf. After 100 h at 750 °C, some γ phase reaches critical splitting size and begins to split. After 100 h at 800 °C, splitting is complete. After 100 h at 900 °C, γ phase shows stable preferred morphology after splitting. During aging, the morphological instability of γ phase causes its coarsening rate to deviate from classical LSW (Lifshitz-Slyozov-Wagner) ripening theory.

2.5 Effect of Hf Content on High-Temperature Mechanical Properties of FGH97 Alloy

[Figure 8: see original paper] shows tensile properties at 650 °C of standard heat-treated FGH97 alloys with different Hf contents. Hf content has no obvious effect on high-temperature tensile strength but significantly affects tensile ductility. The 0.30% Hf alloy shows markedly improved plasticity, with elongation and reduction of area increasing by 31% and 18%, respectively, compared with the Hf-free alloy.

[Figure 9: see original paper] presents stress rupture properties at 650 °C and 1020 MPa for smooth and notched specimens of standard heat-treated FGH97 alloys with different Hf contents. With increasing Hf content, notched specimen rupture life shows an increasing trend. The Hf-free alloy exhibits lower notched specimen life than smooth specimens, indicating notch sensitivity. When Hf content exceeds 0.16%, notched specimen life exceeds that of smooth specimens, eliminating notch sensitivity. The 0.30% Hf alloy shows the longest notched specimen rupture life.

[Figure 10: see original paper] shows creep elongation after different times at 750 °C and 460 MPa for standard heat-treated FGH97 alloys with different Hf

contents. With increasing Hf content, creep elongation decreases for both 100 h and 500 h exposures. Alloys with Hf content above 0.30% exhibit good creep resistance.

[Figure 11: see original paper] shows the relationship between fatigue crack propagation rate da/dN and stress intensity factor range ΔK at 650 °C under no-hold conditions for standard heat-treated FGH97 alloys with different Hf contents. With increasing Hf content, fatigue crack propagation rate first decreases then increases, with the 0.30% Hf alloy showing the lowest rate. At $\Delta K = 30 \text{ MPa} \cdot \text{m}^{1/2}$, the fatigue crack propagation rate of the 0.30% Hf alloy is one-fifth that of the Hf-free alloy.

[Figure 12: see original paper] shows fracture morphologies in the initiation and propagation regions of fatigue crack growth at 650 °C for standard heat-treated FGH97 alloy containing 0.30% Hf. The initial crack propagation facets are relatively long, with fewer secondary cracks in the propagation region and slow crack growth, exhibiting mixed transgranular and intergranular fracture.

Discussion

3.1 Hf Effectively Eliminates PPB Structures

Different conclusions and opinions remain regarding whether Hf addition can clearly eliminate PPB structures in powder superalloys. Reported experimental results mainly suffer from unclear identification of Hf location and state in the alloy, leading to inconsistent conclusions.

This work employed secondary carbon extraction replica techniques combined with EDS analysis to identify a metastable MC -type carbide in rapidly solidified powder particle structures, rich in Nb and Ti with certain amounts of low-diffusivity transition elements such as W and Mo. During HIP, interdendritic metastable MC -type carbides within rapidly solidified powder particles can decompose, with carbide-forming elements Ti and Nb diffusing to form stable (Nb, Ti)C carbides while weak carbide-forming elements W and Mo diffuse into the γ solid solution.

Experiments also revealed no Hf segregation layer on powder particle surfaces. Hf content in interdendritic MC -type carbides within powder particles is very low, while the interdendritic γ solid solution is Hf-rich. Therefore, when MC -type carbides decompose and release carbon, it combines with Hf in the supersaturated interdendritic γ solid solution to preferentially form HfC carbides. This in-situ selective chemical reaction $\gamma(\text{Hf}) + \text{C} \rightarrow \text{HfC}$ within powder particles to form stable carbides is termed the “internal reaction effect.” Consequently, during HIP, Hf in interdendritic regions undergoes internal reaction, capturing carbon released from MC -type carbide decomposition and reacting with Hf in the interdendritic γ solid solution to form stable carbides, reducing carbon diffusion to active powder particle surfaces and effectively eliminating PPB structures.

3.2 Hf Promotes γ Phase Morphological Instability and Early Transition to Low-Energy Preferred Stable State

Doi et al. and Miyazaki et al. noted that with precipitate growth in alloys, morphological evolution involves a process of preferred morphology formation. This work found that increasing Hf content in FGH97 alloys promotes earlier formation of γ phase preferred morphology. For example, after standard heat treatment, γ phase in 0.16% Hf FGH97 alloy maintains coherency with the matrix with low elastic strain energy, remaining cubic (Fig. 4b). When Hf content increases to 0.30%, large cubic γ phase grows to the critical splitting size and begins to split, forming octet small cubic configurations (Fig. 4c). In 0.58% Hf and 0.89% Hf alloys, γ phase splitting is mostly complete or fully completed, forming preferred cubic γ phase configurations (Figs. 4d and e).

Splitting of cubic γ phase is a common phenomenon of morphological instability. Qiu and Kaufman et al. demonstrated using convergent beam electron diffraction and TEM techniques that at the centers of the four edges of large cubic γ phase $\{100\}$ planes, elastic strain fields and their interaction with enriched solute atoms are strongest, becoming splitting initiation sites. The chemical driving force is larger along the preferred $\langle 110 \rangle$ orientation, where growth velocity is faster than along $\langle 100 \rangle$ directions, causing depressions at the centers of cubic γ phase edges along $\langle 100 \rangle$ directions (indicated by arrows in Fig. 4) and creating negative curvature radius at $\{100\}$ planes.

With increasing Hf content, γ phase lattice parameter increases, enhancing lattice distortion stress fields and altering the competition and coordination between surface energy and elastic strain energy during cubic γ phase growth. This creates elastic stress concentration around γ phase depressions, accelerating solute atom trapping, increasing depression curvature radius and deepening depressions, leading to splitting into 2, 4, or 8 cubic configurations. This is the primary mechanism by which microelement Hf addition promotes cubic γ phase splitting and enables faster transition to low-energy preferred stable morphology.

3.3 Hf Significantly Improves High-Temperature Notch Sensitivity

As shown in Fig. 12, the widening of the main crack propagation region and slow secondary crack growth on fatigue fracture surfaces indicate that Hf-added alloys possess higher plasticity, particularly the 0.30% Hf alloy, which shows significantly improved high-temperature mechanical properties.

Miner noted that in multicomponent multiphase alloys, microstructural effects on properties should consider interactions among various components. Experimental results show that since Hf is a strong carbide-forming element, added Hf in FGH97 alloy enters not only MC-type carbides but also γ phase, altering elemental compositions in γ phase, MC phase, and γ phase, and causing redistribution of Nb, Ti, Cr, Mo, W, and other elements among these phases. The 0.30% Hf addition can effectively substitute Nb and Ti in (Nb, Ti)C to

form (Nb, Ti, Hf)C. After strong carbide-forming elements Nb and Ti enter the γ solid solution, they preferentially form NbC and TiC, reducing carbon concentration in γ solid solution and weakening high-temperature strength of γ solid solution, which helps eliminate high-temperature stress rupture notch sensitivity and achieve good strength-ductility balance. If Hf addition is insufficient, less Nb and Ti in MC phase are substituted, and the weakening effect on γ solid solution strength is not obvious. Excessive Hf addition directly forms large amounts of HfC and HfO₂ and enters MC -type carbides, which is not conducive to effective substitution of Nb, Ti, and other elements in MC phase. Moreover, excessive Hf significantly lowers the solidus temperature and raises the γ phase complete dissolution temperature, making solid solution heat treatment process control difficult. Therefore, an optimal Hf content should be maintained in the alloy.

Conclusions

- (1) In as-HIPed FGH97 alloy powder particles, Hf dissolved in interdendritic regions undergoes in-situ internal reaction to form stable MC-type carbides HfC and (Nb, Ti, Hf)C, consuming carbon in the γ solid solution and reducing carbide formation at prior particle boundaries, effectively eliminating PPB structures.
- (2) In Hf-containing FGH97 alloys, Hf enters γ phase, altering elastic strain energy distribution during γ phase growth, promoting splitting of cubic γ phase into octet small cubes, enabling faster transition to low-energy stable cubic preferred configurations.
- (3) Appropriate Hf addition in FGH97 alloy effectively changes elemental redistribution between precipitates and γ solid solution, beneficial for improving high-temperature stress rupture ductility.

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