

Effect of (W+Mo)/Cr Ratio on the Aging Microstructure and Stress-Rupture Properties of Cast Nickel-Based Superalloys (Postprint)

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Abstract

Cast nickel-based superalloys with different (W+Mo)/Cr ratios (mass ratios) were prepared by vacuum melting. The microstructures of the alloy specimens were observed using OM, SEM, and TEM, and the influence of the (W+Mo)/Cr ratio on microstructural evolution and stress-rupture properties was investigated. The results indicate that the (W+Mo)/Cr ratio has no significant effect on the heat-treated microstructure, with the main constituent phases being the γ matrix, γ' phase, primary MC, and grain boundary secondary carbides. During long-term aging, the microstructural evolution of the alloy specimens primarily includes γ' phase coarsening, topologically close-packed (TCP) phase precipitation, MC decomposition, and grain boundary coarsening. With decreasing (W+Mo)/Cr ratio, the thermal stability of MC decreases significantly, the degree of grain boundary coarsening increases, and the grain boundary carbides undergo a transformation from $M_6C \rightarrow M_6C + M_{23}C_6 \rightarrow M_{23}C_6$. Simultaneously, the amount of TCP phase precipitation decreases markedly; when the (W+Mo)/Cr ratio is 0.22, no TCP phase precipitates. Additionally, when the (W+Mo)/Cr ratio decreases from above 0.55 to 0.37, the type of TCP phase transforms from the μ phase to the coexistence of μ and σ phases. γ' phase and grain boundary coarsening, along with TCP phase precipitation, are the main causes of degradation in the alloy's stress-rupture properties. Based on a comprehensive analysis of the effects of the (W+Mo)/Cr ratio on microstructural evolution and stress-rupture properties, it is concluded that the alloy exhibits optimal stress-rupture properties when the (W+Mo)/Cr ratio is approximately 0.37.

Full Text

Preamble

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Effects of (W+Mo)/Cr Ratio on Microstructural Evolution and Stress-Rupture Properties of Cast Nickel-Based Superalloys

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Abstract

Nickel-based superalloys are widely used as critical hot-section components in modern gas turbine engines due to their excellent high-temperature strength, fatigue and creep resistance, and hot-corrosion resistance. To enhance high-temperature capabilities, increasingly higher contents of refractory elements such as W and Mo have been added to these alloys while Cr content has gradually decreased. During long-term service, these alloys typically undergo various microstructural degradations, including γ' phase coarsening, formation of continuous grain boundary carbide networks, precipitation of topologically close-packed (TCP) phases, and degeneration of MC carbides. However, limited data are available regarding the effects of (W+Mo)/Cr ratios on the microstructural evolution of nickel-based superalloys. In this study, cast nickel-based superalloys with different (W+Mo)/Cr mass ratios were fabricated by vacuum induction melting. After standard heat treatment (1110 °C for 4.5 h, air cooling + 750 °C for 10.5 h, air cooling), the alloys were thermally exposed at 850 °C for various durations. Stress-rupture tests were conducted at 800 °C under 294 MPa. The effects of (W+Mo)/Cr ratios on microstructural evolution and mechanical properties were investigated using optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and stress-rupture testing. The results show that the (W+Mo)/Cr ratio has no significant influence on the standard heat-treated microstructure, which consists primarily of γ matrix, γ' phase, MC carbides, and secondary carbides distributed at grain boundaries. During long-term thermal exposure, microstructural evolution occurs through γ' phase coarsening, TCP phase formation, MC degener-

ation, and grain boundary coarsening. The γ' phase coarsening behavior is not significantly affected by the (W+Mo)/Cr ratio. However, the amount of TCP phases decreases significantly with decreasing (W+Mo)/Cr ratio, and the type of TCP phases transforms from μ -phase to coexisting μ - and σ -phases when the (W+Mo)/Cr ratio decreases from 0.55 to 0.37. No TCP phases are observed in the sample with a (W+Mo)/Cr ratio of 0.22. The thermal stability of MC carbides is markedly reduced, and grain boundary coarsening becomes more severe as the (W+Mo)/Cr ratio decreases. The degradation of stress-rupture properties is attributed to γ' phase and grain boundary coarsening and TCP phase formation. Considering the combined effects of (W+Mo)/Cr ratio on solid solution strengthening, microstructural evolution, and stress-rupture properties, the optimum stress-rupture performance is obtained when the (W+Mo)/Cr ratio is approximately 0.37.

Keywords nickel-based superalloy, (W+Mo)/Cr ratio, long-term aging, microstructural evolution, mechanical properties

1. Introduction

Cast nickel-based superalloys are widely used as core hot-section components in gas turbines, where they experience high temperatures and significant stresses during service. Early-generation cast nickel-based superalloys were primarily Ni-Cr systems with high Cr contents to ensure hot-corrosion resistance but relatively low W and Mo contents. As gas turbine technology has advanced, higher temperature capabilities have been required, leading to the addition of increasing amounts of refractory elements such as W, Mo, Ta, and Nb. This trend has increased matrix saturation and reduced microstructural stability.

During service, cast nickel-based superalloys undergo a series of microstructural degradations, including γ' phase coarsening, MC carbide decomposition, topologically close-packed (TCP) phase precipitation, and grain boundary coarsening, all of which degrade alloy performance. W and Mo are important solid solution strengthening elements and primary constituents of the TCP μ -phase, while Cr is the main component of the TCP σ -phase. Additionally, the (W+Mo)/Cr mass ratio significantly influences the types of secondary carbides and the decomposition behavior of primary MC carbides. However, a comprehensive understanding of how the (W+Mo)/Cr ratio affects microstructural evolution and mechanical properties remains lacking. This study investigates the effects of (W+Mo)/Cr ratio on γ' phase coarsening, TCP phase precipitation, MC carbide decomposition, and grain boundary coarsening in experimental alloys, and examines the relationship between (W+Mo)/Cr ratio, microstructural evolution, and stress-rupture properties.

2. Experimental Procedures

Experimental alloys were melted in a 25 kg vacuum induction furnace and remelted into test bars. The chemical compositions are listed in . The alloy design maintained a constant total content of W, Mo, and Cr while decreasing W and Mo contents and increasing Cr content to reduce the (W+Mo)/Cr ratio. The (W+Mo)/Cr ratios were systematically decreased from 1.07 to 0.22 in alloys A1, A2, A3, and A4 to investigate their effects on the long-term thermal stability of cast nickel-based superalloys.

All alloy samples underwent the same heat treatment: solution treatment at 1110 °C for 4.5 h followed by air cooling, then aging at 750 °C for 10.5 h followed by air cooling. After heat treatment, samples were aged at 850 °C for 0.5×10^3 , 1×10^3 , 3×10^3 , 6×10^3 , and 1×10^4 h. Stress-rupture tests were performed at 800 °C under 294 MPa on samples aged for 1×10^3 h and 1×10^4 h.

For two-dimensional microstructural observation, aged samples were chemically etched using a solution of 4 g CuSO₄ + 10 mL HCl + 20 mL H₂O. To observe the three-dimensional morphology of the primary strengthening γ' phase, electrolytic deep etching was performed using a solution of 10 g (NH₄)₂SO₄ + 30 g C₆H₈O₇ + 1000 mL H₂O.

Microstructural characterization was conducted using a GX51 optical microscope (OM), a JEM-6340 field emission scanning electron microscope (SEM), and a TECNAI F20 transmission electron microscope (TEM). Phase identification was performed using TEM energy-dispersive spectroscopy (EDS) and selected-area electron diffraction (SAED). The γ' phase size, volume fraction, and volume fractions of primary MC and TCP phases were quantified using Image-Pro Plus 6.0 software, with results averaged from at least 20 images.

3. Results and Discussion

3.1 Effects of (W+Mo)/Cr Ratio on Microstructure During Long-Term Aging

After standard heat treatment, all four alloy samples exhibited similar microstructures. The typical microstructure of alloy A1 is shown in [Figure 1: see original paper]. The primary constituent phases include the γ matrix, γ' phase, primary MC carbides [Figure 1a: see original paper], and secondary grain boundary carbides [Figure 1c: see original paper]. The γ' phase appears as fine spherical particles uniformly distributed in the matrix with an average diameter of approximately 30 nm [Figure 1b: see original paper]. Primary MC carbides are mainly distributed in interdendritic regions and at grain boundaries as irregular blocky particles, identified by EDS as TiC-type carbides. In addition to large blocky MC carbides, fine secondary carbides are dispersedly distributed at grain boundaries [Figure 1c: see original paper]. After heat treatment, the primary MC carbides remained stable without decomposition.

3.1.1 γ' Phase Coarsening [Figure 2: see original paper] shows SEM images of γ' phase in the alloy samples after aging at 850 °C for various times. The γ' phase coarsening behavior was similar across all four alloys; therefore, representative SEM images are presented for each alloy after specific aging durations. The γ' phase coarsened significantly, particularly within the first 3×10^3 h, where the particle diameter increased rapidly from approximately 30 nm in the heat-treated condition to about 450 nm, after which the growth rate slowed. Additionally, the γ' phase morphology transformed from spherical to cuboidal and back to spherical, with an increasing tendency for directional alignment as aging time progressed. γ' phase coarsening is primarily controlled by diffusion of γ' -forming elements Al, Ti, and Nb in the matrix, with an activation energy similar to that for diffusion of these elements. Although the four alloys have different (W+Mo)/Cr ratios, their contents of γ' -forming elements Al, Ti, and Nb are similar; consequently, the γ' phase coarsening behavior is comparable across all samples.

3.1.2 TCP Phase Precipitation [Figure 3: see original paper] presents the microstructures of the four alloy samples after aging at 850 °C for 0.5×10^3 h. In alloy A1 with the highest (W+Mo)/Cr ratio, a large quantity of needle-like, rod-like, and blocky TCP phases precipitated [Figure 3a: see original paper], identified as μ -phase through TEM imaging and SAED analysis. As the (W+Mo)/Cr ratio decreased, the amount of TCP phases decreased significantly, most notably when the ratio decreased from 1.07 to 0.55. No TCP phases were observed in alloy A4 with the lowest (W+Mo)/Cr ratio of 0.22 [Figure 3d: see original paper]. TEM observation and SAED confirmed that μ -phase precipitated in alloy A2 with a (W+Mo)/Cr ratio of 0.55 [Figure 3b: see original paper], while both μ - and σ -phases coexisted in alloy A3 with a (W+Mo)/Cr ratio of 0.37 [Figure 3c: see original paper]. The μ -phase appeared as small blocky particles, whereas the σ -phase exhibited needle-like or rod-like morphologies. The two phases shared common interfaces but showed no specific orientation relationship.

After extended aging to 1×10^4 h, the amount of TCP phases increased significantly in alloys A1, A2, and A3 with (W+Mo)/Cr ratios above 0.37 [FIGURE:4a-c]. In contrast, alloy A4 with a (W+Mo)/Cr ratio of 0.22 demonstrated good microstructural stability, with no TCP phase precipitation even after 1×10^4 h [Figure 4d: see original paper]. Compared with [Figure 3: see original paper], the morphology of μ -phase in alloys A1 and A2 transformed from mixed needle/rod/blocky shapes to predominantly blocky shapes. Qin et al. [?] investigated this phenomenon in detail and attributed it to dissolution of μ -phase during long-term aging and subsequent transformation to blocky shapes with lower surface energy.

W, Mo, and Cr are the primary constituent elements of TCP phases. W and Mo are the main forming elements for μ -phase, while Cr enriches in σ -phase. When the mass fraction of W+Mo exceeds 6%, μ -phase precipitates, which is

consistent with the extensive μ -phase formation in alloys A1 and A2. σ -phase is the most common TCP phase in cast superalloys and can be predicted using the electron vacancy number (Nv). σ -phase precipitates when Nv exceeds 2.45–2.50 [?]. Comparing the Nv values of alloys A3 and A4 (Table 1) reveals that alloy A4 with a higher Nv (2.44) shows no σ -phase [Figure 4a: see original paper], whereas alloy A3 with a lower Nv (2.41) exhibits σ -phase precipitation. This indicates that σ -phase precipitation depends not only on Nv but also on the (W+Mo)/Cr ratio.

3.1.3 MC Carbide Decomposition During long-term aging, MC carbides become metastable and gradually transform into other secondary carbides through a series of reactions [?]. [Figure 5: see original paper] shows the decomposition morphologies of MC carbides in the four alloys after aging for 1×10^3 h. All MC carbides underwent slight decomposition, forming distinct reaction zones around them. Further observation revealed that in alloys A1 and A2 with (W+Mo)/Cr ratios above 0.55, the secondary carbides formed from MC decomposition were M_6C [FIGURE:5a,b]. At a (W+Mo)/Cr ratio of 0.37, both M_6C and $M_{23}C_6$ carbides coexisted around MC carbides [Figure 5c: see original paper]. When the (W+Mo)/Cr ratio further decreased to 0.22, only $M_{23}C_6$ was present [Figure 5d: see original paper]. Formation of secondary carbides consumes W, Mo, and Cr from the matrix surrounding MC carbides, enriching the region in γ' -forming elements Al, Ti, and Nb and resulting in coexistence of γ' phase layers and secondary carbides in the MC decomposition zone [?, ?]. EDS analysis shows that W and Mo are the primary forming elements for M_6C , while Cr is the main element in $M_{23}C_6$. Therefore, the type of secondary carbide is closely related to the (W+Mo)/Cr ratio. During MC decomposition, carbon diffuses from MC into the matrix and combines with W, Mo, and Cr to form secondary carbides [?, ?]. At high (W+Mo)/Cr ratios (alloys A1 and A2), carbon preferentially combines with W and Mo to form M_6C . At low (W+Mo)/Cr ratios (alloy A4), Cr-rich $M_{23}C_6$ forms readily. At intermediate ratios (alloy A3), carbon shows comparable affinity for W, Mo, and Cr, resulting in coexistence of M_6C and $M_{23}C_6$ in the MC decomposition region.

After aging for 1×10^4 h, the MC decomposition morphologies are shown in [Figure 6: see original paper]. Compared with [Figure 5a: see original paper], MC decomposition in alloy A1 changed little during aging, with decomposition products remaining as M_6C and γ' phase [Figure 6a: see original paper]. In alloy A2, the MC decomposition reaction zone expanded, and in addition to M_6C and γ' phase, small amounts of $M_{23}C_6$ and η -phase formed [Figure 6b: see original paper]. The precipitation of $M_{23}C_6$ can be attributed to: (1) additional carbon released from MC decomposition, and (2) consumption of W and Mo by M_6C formation around MC, leading to Cr enrichment near MC and promoting formation of Cr-rich $M_{23}C_6$. η -phase formation is related to hindered elemental diffusion between MC and the matrix [?]. As MC decomposition progresses, the γ' phase layer around MC thickens, impeding diffusion of Ti and Nb from MC

into the matrix. This increases the (Ti+Nb)/Al mass ratio at the MC/ γ' interface, promoting η -phase precipitation. η -phase formation consumes γ' -forming elements Ti and Nb, thereby inhibiting further growth of the γ' phase layer [?]. Alloys A3 and A4 exhibited similar MC decomposition reactions [FIGURE:6c,d], though MC decomposition was more severe in these two alloys. After aging for 1×10^4 h, MC carbides were almost completely transformed into η -phase, $M_{23}C_6$, M_6C , and γ' phase.

The degree of MC decomposition in the four alloys is quantified in [Figure 7: see original paper]. During long-term aging, MC decomposition increases significantly with decreasing (W+Mo)/Cr ratio, and this trend becomes more pronounced with longer aging times, indicating that MC thermal stability decreases as the (W+Mo)/Cr ratio decreases. However, when the (W+Mo)/Cr ratio falls below 0.37 (alloy A3), the degree of MC decomposition no longer changes significantly. MC thermal stability is closely related to its composition; Nb and Ti enhance MC stability, while W and Mo reduce it [?]. The Nb/Ti and (Nb+Ti)/(W+Mo) ratios are parameters for MC thermal stability—higher values indicate greater stability [?, ?]. As shown in , as the (W+Mo)/Cr ratio decreases, the Nb/Ti ratio remains constant while the (Nb+Ti)/(W+Mo) ratio generally increases. However, MC thermal stability decreases significantly, demonstrating that in addition to these stability parameters, the bulk alloy (W+Mo)/Cr ratio is also an important factor affecting MC stability. MC decomposition involves interdiffusion of elements between MC and the matrix, with carbon diffusing from MC to the matrix and W, Mo, and Cr diffusing from the matrix to MC [?, ?]. Compared with Cr, W and Mo have slower diffusion rates. Therefore, a high (W+Mo)/Cr ratio slows the interdiffusion process, suppressing MC decomposition and enhancing its thermal stability. Conversely, a low (W+Mo)/Cr ratio accelerates MC decomposition and reduces its stability.

MC thermal stability significantly affects TCP phase precipitation [?]. When MC thermal stability is low, extensive carbon is released during long-term aging, forming secondary carbides M_6C or $M_{23}C_6$. Studies [?, ?] suggest that formation of secondary carbides consumes TCP-forming elements, thereby suppressing TCP phase precipitation. When MC thermal stability is high, limited carbon release has no significant effect on TCP phases. Therefore, the decreased MC thermal stability with decreasing (W+Mo)/Cr ratio is an important reason for the different amounts of TCP phases observed in the four alloys [FIGURE:3 and 4].

The grain boundary morphologies of the four alloys after aging for 1×10^4 h are shown in [Figure 8: see original paper]. Compared with the standard heat-treated condition [Figure 1c: see original paper], significant grain boundary coarsening occurred, with the degree of coarsening increasing as the (W+Mo)/Cr ratio decreased. MC decomposition at or near grain boundaries is an important cause of grain boundary coarsening [?]. The more severe grain boundary coarsening at lower (W+Mo)/Cr ratios is fundamentally caused by the significantly increased MC decomposition [Figure 7: see original paper].

In alloy A1, grain boundary carbides were distributed in a semi-continuous pattern [Figure 8a: see original paper], whereas in the other three alloys, grain boundary carbides formed continuous networks [FIGURE:8b-c]. The figure also clearly shows that as the (W+Mo)/Cr ratio decreased, the grain boundary carbides underwent transformation from M_6C to $M_{23}C_6$, which is caused by the different formation tendencies of M_6C and $M_{23}C_6$ at different (W+Mo)/Cr ratios.

Grain boundary morphology and carbide distribution significantly affect stress-rupture properties [?]. When grain boundaries are severely coarsened and grain boundary carbides form continuous networks, the grain boundaries become brittle and facilitate crack propagation, substantially reducing rupture life. In contrast, when grain boundary carbides are dispersed or semi-continuously distributed, they can inhibit grain boundary sliding and nucleation and growth of grain boundary voids, significantly improving rupture life and ductility [?]. Therefore, optimizing the (W+Mo)/Cr ratio to control MC thermal stability and achieve optimal grain boundary structure can significantly enhance stress-rupture performance.

3.2 Effects of (W+Mo)/Cr Ratio on Stress-Rupture Properties

[Figure 9: see original paper] shows the stress-rupture lives of the four alloys after aging for 1×10^3 h and 1×10^4 h, tested at 800 °C under 294 MPa. After aging for 1×10^3 h, rupture life initially increased then decreased with decreasing (W+Mo)/Cr ratio, reaching a peak at a (W+Mo)/Cr ratio of 0.55. Alloy A1 exhibited lower rupture life due to the high (W+Mo)/Cr ratio and the precipitation of large amounts of needle-like μ -phase. When the (W+Mo)/Cr ratio decreased to 0.22, the contents of solid solution strengthening elements W and Mo were too low, resulting in poor solid solution strengthening and inferior rupture life. Alloys with (W+Mo)/Cr ratios of 0.37 (A3) and 0.55 (A2) exhibited good solid solution strengthening effects and only small amounts of TCP phases, which had minimal impact on stress-rupture properties; consequently, these alloys showed higher rupture lives.

After aging for 1×10^4 h, rupture life decreased significantly compared with the 1×10^3 h condition due to γ' phase coarsening, increased TCP phase precipitation, and grain boundary coarsening. Alloy A1 showed the smallest reduction in rupture life, primarily due to the semi-continuous distribution of grain boundary carbides [Figure 8a: see original paper] and the blocky morphology of μ -phase [Figure 4a: see original paper], which can impede dislocation motion and strengthen the alloy, benefiting stress-rupture properties. In alloy A4, no TCP phases precipitated [Figure 4d: see original paper], and alloy A3 contained only small amounts of TCP phases [Figure 4c: see original paper]; therefore, γ' phase and grain boundary coarsening were the main causes of rupture life reduction. Alloy A2 exhibited the most significant decrease in rupture life, from 106 h after 1×10^3 h aging to 37 h after 1×10^4 h. In addition to γ' phase and grain boundary coarsening, the increased amount of TCP

phases was an important factor. Studies [?, ?] have shown that when the volume fraction of TCP phases exceeds 0.85%, high-temperature rupture life decreases substantially. After aging for 1×10^4 h, the volume fraction of μ -phase in alloy A2 increased from 0.74% (after 1×10^3 h) to 3.21%, severely reducing rupture life. Based on this comprehensive analysis, alloy A3 with a (W+Mo)/Cr ratio of 0.37 exhibits the optimal stress-rupture performance.

The longitudinal cross-section microstructures of the four alloys after rupture testing at 800 °C and 294 MPa (following aging at 850 °C for 1×10^4 h) are shown in [Figure 10: see original paper]. Cracks initiated and propagated along grain boundaries perpendicular to the stress axis in all four alloys. As seen in [Figure 10a: see original paper] and [b], cracks did not initiate at TCP phase/matrix interfaces, indicating good interfacial strength between TCP phases and the matrix. Additionally, needle-like TCP phases underwent internal fracture. These observations demonstrate that the (W+Mo)/Cr ratio did not significantly affect the fracture mode of the alloys.

4. Conclusions

1. After standard heat treatment, the alloys consist primarily of γ matrix, γ' phase, primary MC carbides, and secondary grain boundary carbides. The (W+Mo)/Cr ratio has no significant effect on the heat-treated microstructure.
2. During long-term aging, microstructural evolution includes coarsening of γ' phase and grain boundaries, TCP phase precipitation, and MC carbide decomposition. As the (W+Mo)/Cr ratio decreases, MC thermal stability decreases significantly and grain boundary coarsening becomes more severe. Simultaneously, the amount of TCP phases decreases markedly; no TCP phases precipitate when the (W+Mo)/Cr ratio is 0.22. Additionally, when the (W+Mo)/Cr ratio decreases from above 0.55 to 0.37, the type of TCP phases transforms from μ -phase to coexisting μ - and σ -phases.
3. The reduction in alloy stress-rupture life is primarily caused by γ' phase and grain boundary coarsening and TCP phase precipitation. The alloy with a (W+Mo)/Cr ratio of 0.37 exhibits the optimal stress-rupture life.

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