

Effect of Ti/Al Ratio on γ Precipitate Coarsening Behavior and Tensile Properties of GH984G Alloy During Long-Term Aging Postprint

Authors: Tan Meilin, Wang Changshuai, Guo Yong'an, Guo Jianting, Zhou Lanzhan

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Abstract

This study investigates the effects of two Ti/Al ratios on the coarsening behavior of γ precipitates and the mechanical properties of a novel Ni-Fe-Cr based alloy GH984G during high-temperature aging for up to ten thousand hours. The results indicate that as the aging temperature increases from 700°C to 800°C, the coarsening rate of spherical γ precipitates increases significantly. During long-term aging at 700°C and 750°C, the coarsening behavior of γ precipitates in both high Ti/Al ratio and low Ti/Al ratio alloys conforms to the Lifshitz-Slyozof-Wagner (LSW) theory and is controlled by diffusion processes, with the coarsening rate of γ precipitates being higher in the high Ti/Al ratio alloy. However, during long-term aging at 800°C, the coarsening behavior of γ precipitates in alloys with both Ti/Al ratios deviates from the LSW theory; furthermore, when the aging time is less than 3000 h, the γ precipitates in the high Ti/Al alloy grow faster, while with further extension of aging time, the γ precipitates in the low Ti/Al ratio alloy exhibit a faster growth rate. The Ti/Al ratio has no significant effect on the tensile properties at 700°C of the alloy in the standard heat-treated condition and after ten thousand hours of aging at 700-800°C. By selecting an appropriate Ti/Al ratio, the coarsening behavior of γ precipitates can be controlled and the microstructural stability of the alloy can be enhanced.

Full Text

Preamble

Influence of Ti/Al Ratio on γ Precipitate Coarsening Behavior and Tensile Properties of GH984G Alloy During Long-Term Thermal Exposure

Tan Meilin, Wang Changshuai, Guo Yong'an, Guo Jianting, Zhou Lanzhang*

Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016

Abstract

This study investigated the effects of two Ti/Al ratios on the coarsening behavior of γ' precipitates and tensile properties of a novel Ni-Fe-Cr based alloy GH984G during long-term high-temperature aging for up to ten thousand hours. The results demonstrate that the coarsening rate of spherical γ' precipitates increases significantly as the aging temperature rises from 700°C to 800°C. During long-term aging at 700°C and 750°C, the coarsening behavior of γ' precipitates in both high and low Ti/Al ratio alloys conforms to the Lifshitz-Slyozov-Wagner (LSW) theory and is controlled by diffusion processes, with the high Ti/Al ratio alloy exhibiting a higher coarsening rate. At 800°C, the coarsening behavior deviates from LSW theory for both alloys. Additionally, when the aging time is less than 3000 h, the high Ti/Al alloy shows faster γ' precipitate growth; however, with further extension of aging time, the low Ti/Al ratio alloy exhibits a faster growth rate. The Ti/Al ratio has no significant effect on the 700°C tensile properties of the alloy in either the standard heat-treated condition or after aging at 700–800°C for 1×10^4 h. By selecting an appropriate Ti/Al ratio, the coarsening behavior of γ' precipitates can be controlled and microstructural stability can be enhanced.

Keywords: Ni-Fe-Cr based alloy, long-term aging, γ' precipitate, coarsening behavior, tensile properties

Introduction

Electricity is the primary energy source in modern society and a critical guarantee for rapid economic development. With economic growth, China's thermal power generation capacity increased from 391 GW in 2005 to 796 GW in 2012. However, this expansion has placed tremendous pressure on national resources and the environment, with resource depletion and environmental degradation becoming major constraints on economic development. Improving power plant efficiency and reducing coal consumption are therefore of great significance for energy conservation, cost reduction, and emission control. The efficiency of thermal power units depends on steam temperature and pressure; increasing these parameters can significantly enhance efficiency. Consequently, countries worldwide have proposed advanced ultra-supercritical (A-USC) coal-fired power units with steam temperatures of 700–760°C and pressures of 35–37.5 MPa [1–3]. In these power units, boiler superheater and reheater tubes operate in the most severe environments. Traditional ferritic and austenitic steels used in ultra-supercritical boilers can no longer meet the requirements, making the development of materials suitable for 700°C A-USC superheater and reheater tubes a current research focus [1,4,5].

GH984G alloy is a Ni-Fe-Cr based alloy developed from GH984 alloy [6]. Compared with alloys such as CCA617 [6,8] and IN740 [1,9] currently under de-

velopment for 700°C A-USC boiler superheater and reheater tubes, GH984G is more economical due to the elimination of expensive Co and the addition of more than 20% Fe (by mass fraction). Compared with Sanicro 25 [9] and HR6W [10,11] alloys, GH984G exhibits higher creep strength and microstructural stability. Furthermore, the alloy inherits the high thermal conductivity of GH984 alloy, which benefits boiler heat transfer efficiency. GH984G alloy achieves excellent high-temperature creep strength through the precipitation of Ni (Al,Ti)-type γ' intermetallic compounds with L1 ordered structure in the matrix. The superior high-temperature mechanical properties are closely related to the compositional characteristics, volume fraction, size, morphology, and distribution of γ' precipitates [12-14]. The coarsening behavior of γ' precipitates during long-term high-temperature aging significantly affects the deformation mechanisms and mechanical properties of the alloy. The coarsening process of γ' precipitates in superalloys generally follows LSW theory [15,16]. However, LSW theory neglects interactions between particles and changes in solute diffusion fields, leading to deviations from many experimental results. Although subsequent modifications to LSW theory have been made, they still cannot adequately describe the coarsening behavior in many alloys [17-20]. Traditional superalloys are mainly used in aero-engines and gas turbines with relatively short service times, and few studies have investigated γ' precipitate evolution and mechanical properties during aging for tens of thousands of hours. Clarifying the coarsening behavior of γ' precipitates and the evolution of mechanical properties during such extended high-temperature aging, along with their influencing factors, is crucial for understanding coarsening mechanisms, optimizing alloy performance, and predicting service life.

The compositional characteristics, volume fraction, γ' lattice misfit, and mechanical properties of γ' precipitates are closely related to the Ti/Al ratio [12-14]. Therefore, this work aims to provide an experimental and theoretical foundation for alloy composition optimization and life prediction by elucidating the influence of Ti/Al ratio on the coarsening behavior of γ' precipitates and its effect on mechanical properties during long-term high-temperature aging.

Experimental Procedures

Two GH984G alloy ingots with different compositions were prepared by vacuum induction melting and casting in a 25 kg furnace. While maintaining a constant total (Ti+Al) content, the alloy compositions were configured according to Ti/Al mass ratios of 1.0 and 1.5. The measured chemical compositions are listed in Table 1. The alloy ingots were subjected to homogenization heat treatment, then forged into 30 mm × 30 mm billets, and subsequently hot-rolled into bars with a diameter of 16 mm. The heating temperature for both forging and rolling was 1150°C.

The as-rolled bars were subjected to standard heat treatment (1100°C for 1 h, air-cooled, then 750°C for 8 h, air-cooled) followed by long-term high-temperature aging experiments at temperatures of 700°C, 750°C, and 800°C for durations of

1000 h, 3000 h, 5000 h, and 10000 h.

Samples in the standard heat-treated and long-term aged conditions were machined into standard tensile specimens with a gauge dimension of 5 mm diameter \times 25 mm length. Instantaneous tensile tests at 700°C were conducted on a DCS-25J SHIMADZU AG-250KNE electronic tensile testing machine.

Metallographic samples were etched chemically using a reagent of 5 g FeCl + 50 mL HCl + 100 mL H₂O. Observation of δ precipitates was performed using electrolytic etching with 10% H₂CrO₄ (by mass fraction) at 18 V and approximately 1.0 A. The etched metallographic samples were examined using a JEOL 6340 field-emission scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS) for micro-area compositional analysis and microstructural observation. The average particle size of δ precipitates was measured using Image J software, with each data point representing the statistical average of at least 10 randomly selected SEM images.

Transmission electron microscopy (TEM) samples were first cut into 0.5 mm thick slices using wire electrical discharge machining, ground to approximately 50 μ m, punched into discs, and thinned by chemical twin-jet polishing using a solution of 10% HClO₄ + 90% C₂H₅OH (by volume fraction) at approximately -15°C. TEM observations were performed on a JEOL 2100FX TEM equipped with EDS. Bright-field (BF) imaging was used to characterize the microstructural features of precipitates, and selected-area electron diffraction patterns (SAEDP) combined with EDS were used to identify phases and orientation relationships.

Room-temperature lattice parameters of the δ matrix and δ precipitates were determined by X-ray diffraction. The δ matrix lattice parameter (a_{δ}) was measured using bulk samples, while the δ precipitate lattice parameter ($a_{\delta'}$) was determined using extracted powders with 10% H₂CrO₄ (by mass fraction) as the extraction solution. Both the δ matrix and δ precipitates used the (331) diffraction peak to calculate lattice parameters, with Si serving as a calibration standard. The lattice misfit ϵ is defined as:

$$\epsilon = (a_{\delta'} - a_{\delta}) / a_{\delta} \times 100\%$$

2.1 Microstructure in Standard Heat-Treated Condition

The microstructural characteristics of GH984G alloys with different Ti/Al ratios in the standard heat-treated condition are shown in Figure 1 [Figure 1: see original paper]. The alloy exhibits a bimodal grain size distribution ranging from 20 to 110 μ m. This microstructure forms because during hot rolling, the stress distribution is non-uniform; the deformation near original grain boundaries is greater than within the grains, resulting in higher stress and dislocation density near grain boundaries. During subsequent annealing, the regions near grain boundaries have numerous substructures, leading to smaller recrystallized grains, while the central regions of original grains experience less deformation

and retain larger grain sizes [21]. Additionally, numerous annealing twins exist within the grains, which are beneficial for improving alloy strength.

Figure 2 [Figure 2: see original paper] shows the microstructure of precipitates and their EDS analysis. Two types of white particulate precipitates with different sizes are distributed in the matrix. Larger white particles are randomly distributed within grains and at grain boundaries, identified by EDS as Ti- and Nb-rich MC-type carbides and Ti-rich cubic Ti(C,N) carbonitrides. Smaller white particles are primarily distributed at grain boundaries, and EDS analysis reveals these discontinuously distributed granular precipitates at grain boundaries to be Cr-rich M₂C-type carbides. Ni (Al,Ti,Nb)-type γ' precipitates appear as spherical particles randomly distributed within the matrix of grains, with an average size of approximately 20 nm and a volume fraction of about 6% (Figure 2e) [6]. The standard heat-treated microstructures of low and high Ti/Al ratio alloys are similar, indicating that Ti/Al ratio has no obvious effect on the microstructure. However, measurements of γ' lattice misfit show that the high Ti/Al ratio alloy has a significantly larger misfit than the low Ti/Al ratio alloy, consistent with results from a 35Ni-15Cr type iron-based superalloy [22], demonstrating that while Ti/Al ratio does not significantly affect the volume fraction and size of γ' precipitates, it markedly influences the lattice misfit.

2.2 Coarsening Behavior of γ' Precipitates

The morphological evolution of γ' precipitates in the high Ti/Al ratio alloy during long-term aging at 750°C is shown in Figure 3 [Figure 3: see original paper]. The γ' precipitates gradually coarsen with increasing aging time but remain spherical, consistent with the evolution observed in K452 and K446 alloys [23]. However, studies on K44 alloy found that larger cubic γ' precipitates degenerated while smaller ones dissolved and split during long-term aging [24]. Research on AM1 alloy revealed that γ' precipitate morphology transitions from spherical \rightarrow cubic \rightarrow octocube dendritic with increasing size. From an energy minimization perspective, this morphological evolution results from competition between interfacial energy and strain energy induced by lattice misfit. The relative magnitude of these energies determines γ' precipitate morphology: when γ' precipitates are small, interfacial energy dominates and they remain spherical; as size increases, strain energy gradually becomes dominant, leading to cubic, dendritic, and irregular shapes [25]. For the high Ti/Al ratio alloy, even after aging at 750°C for 10000 h, the γ' precipitate size is only about 180 nm, whereas γ' precipitates in K452 and K446 alloys remain spherical at sizes around 300 nm. Moreover, the high Ti/Al ratio alloy has lower lattice misfit than K452 and K446 alloys, resulting in lower strain energy. Therefore, after aging at 750°C for 10000 h, the γ' precipitate size in the high Ti/Al ratio alloy remains below the critical size, and the morphology stays spherical without significant change. The morphological evolution of γ' precipitates at 700°C and 800°C in both low and high Ti/Al ratio alloys follows the same pattern as observed at 750°C in

the high Ti/Al ratio alloy.

Figure 4 [Figure 4: see original paper] shows the variation of γ' precipitate size with aging time and temperature for the two Ti/Al ratio alloys. When aging time is less than 3000 h, the growth rate of γ' precipitates increases with aging temperature due to enhanced diffusion rates of alloying elements at higher temperatures. With prolonged aging time, the growth of γ' precipitates consumes γ' -forming elements in the surrounding matrix, creating a depletion zone near the precipitates and consequently reducing the growth rate when aging time exceeds 3000 h.

Although the two alloys have similar γ' precipitate sizes in the standard heat-treated condition, during aging at 700°C and 750°C, the high Ti/Al ratio alloy exhibits faster γ' precipitate growth. At 800°C, when aging time is less than 3000 h, the high Ti/Al ratio alloy shows more rapid growth; however, with extended aging time, the low Ti/Al ratio alloy's growth rate gradually accelerates, resulting in nearly equivalent γ' precipitate sizes after 10000 h of aging.

Fleetwood's research [26] indicates that γ' precipitate coarsening rate is closely related to coherency strain, γ'/γ interfacial free energy, solute concentration of γ' -forming elements in the matrix, and diffusion coefficients. The Ti/Al ratio in Ni-Fe based superalloys significantly affects the γ'/γ lattice misfit. Figure 5 [Figure 5: see original paper] shows the evolution of lattice misfit with aging time at 750°C for the two alloys. The high Ti/Al ratio alloy exhibits significantly higher misfit than the low Ti/Al ratio alloy, with misfit values stabilizing over aging time. Increased coherency strain resulting from higher misfit accelerates γ' precipitate coarsening, leading to higher coarsening rates in the high Ti/Al ratio alloy. Fell's experimental results also demonstrate that increased Ti/Al ratio enhances coherency strain and accelerates γ' precipitate coarsening [27].

Furthermore, the diffusion coefficient D of alloying elements can be expressed as:

$$D = D_0 \exp(-Q/RT)$$

where D_0 is the diffusion constant, R is the gas constant, Q is the diffusion activation energy, and T is the thermodynamic temperature. The diffusion coefficients of Al and Ti in pure Ni are 1.84×10^{-12} m²/s (1372-1553 K) and 4.1×10^{-12} m²/s (1323-1648 K), respectively [28], with diffusion activation energies in the matrix of 284 kJ/mol for Al and 257 kJ/mol for Ti [29]. Calculated diffusion coefficients of Al and Ti in the matrix at various temperatures are listed in Table 2, showing that Ti has higher diffusion coefficients than Al at 700°C, 750°C, and 800°C. Since γ' precipitate growth is controlled by diffusion of γ' -forming elements, the higher Ti content in the high Ti/Al ratio alloy results in faster growth rates. The similar γ' precipitate sizes after long-term aging at 800°C likely result from the identical total Ti+Al content in both alloys, leading to essentially balanced γ' -forming element concentrations after extended aging.

Figure 6 [Figure 6: see original paper] shows the relationship between γ' precip-

itate size variation ($d^3 - d_0^3$) and aging time. The coarsening behavior follows LSW theory during long-term aging at 700°C and 750°C, but deviates from LSW theory at 800°C. Ges et al. [30] also observed deviations from LSW theory at longer aging times but did not analyze the reasons. Berahmand [31] proposed that γ precipitate coarsening involves two stages: interface-controlled and strain-controlled. When γ precipitates are small, growth is interface-controlled (surface energy dominated) and follows LSW theory; when large, growth is primarily controlled by elastic energy. The total free energy change driving γ precipitate growth, considering elastic interaction energy, can be expressed as [31]:

$$\Delta E = \Delta E_{\text{sur}} + \Delta E_{\text{int}} = \sigma A - B d V = \sigma d - B V$$

where σ is surface energy, ΔE_{int} is elastic interaction energy, A is the surface area of γ precipitates, V is the volume fraction, B is a constant, and d is surface tension. As γ precipitate size increases, the contribution of surface energy decreases while elastic interaction energy increases, gradually reducing the driving force for growth and consequently decreasing the growth rate. Therefore, the high Ti/Al ratio alloy shows reduced growth rates and deviation from LSW predictions during long-term aging at 800°C due to significantly increased γ precipitate size.

Additionally, classical LSW theory assumes constant interfacial energy σ , molar volume V_m , and diffusion coefficient D , expressed as:

$$d^3 - d_0^3 = k \cdot t = (8 V_m D C_e) / (9RT) \cdot t$$

where d_0 is the initial γ precipitate size, C_e is the solute equilibrium coefficient, k is the rate constant, and t is time. However, the theory does not account for changes in diffusion coefficient D during growth. The effective diffusion coefficient decreases as γ -forming element depletion zones develop near precipitates, reducing growth rates.

2.3 Tensile Properties at 700°C

The effects of long-term aging on alloy tensile properties at 700°C are summarized in Table 3. Compared with the standard heat-treated condition, the yield strength of both alloys increases significantly after aging at 700°C for 10480 h, but decreases markedly after aging at 750°C and 800°C for 10480 h. Yield strength also decreases noticeably as aging temperature increases from 700°C to 800°C. Elongation shows the opposite trend to strength. The Ti/Al ratio has no significant effect on strength or ductility in either the standard heat-treated condition or after identical aging conditions at 700°C.

Figure 7 [Figure 7: see original paper] shows the fracture morphology and dislocation configurations of high Ti/Al ratio alloy tensile specimens tested at 700°C after long-term aging. The fracture surfaces exhibit complex features with co-existing facets, dimples, and tear ridges connected by tear ridges, showing both

transgranular and intergranular fracture characteristics—typical of mixed fracture mode. Compared with specimens aged at 700°C and 750°C, those aged at 800°C show more numerous and deeper dimples with significantly fewer facets, explaining the substantially increased elongation after aging at 800°C. TEM observations of dislocation configurations (Figure 7d) reveal a typical shearing mechanism, indicating that the effect of γ' precipitate coarsening on tensile properties can be considered solely in terms of the shearing mechanism [32].

In superalloys, the shearing mechanism can be described by two models: (1) weakly-coupled dislocation pair shearing [33], with critical resolved shear stress:

$$\Delta = \frac{\gamma_{\text{APB}}}{2b} \cdot (3fV/d)^{1/2} \cdot (1.72wT/(\gamma_{\text{APB}} \cdot d) - 1)$$

and (2) strongly-coupled dislocation pair shearing [34], with:

$$\Delta = (\gamma_{\text{APB}}/2b) \cdot (3fV/d)^{1/2} \cdot (1.28Gb/(\gamma_{\text{APB}} \cdot d) - 1)^{1/2}$$

where γ_{APB} is the anti-phase boundary (APB) energy, b is the Burgers vector (0.254 nm), d is the γ' precipitate size, V is the volume fraction (6%), T is the dislocation line tension, m is a particle shape factor (0.72 for spherical particles [35]), w is a constant (2.8) [41], and G is the shear modulus (60 GPa). The APB energy varies little at 700°C, 750°C, and 800°C, so a constant line tension approximation is used, estimated as 0.28 J/m². Both models assume small coherency stresses, which is valid for the experimental alloys.

For cast nickel-based superalloys with high γ' volume fractions, a Labusch-Schwarz correction is required:

$$\Delta_{\text{LS}} = 0.95(1 + C) \Delta = (V/d)^{1/2} \cdot$$

where C is the L-S parameter. However, since the γ' precipitate content in this alloy is relatively low, L-S correction is unnecessary.

Figure 8 [Figure 8: see original paper] shows the relationship between critical resolved shear stress and γ' precipitate size for the two alloys. For small γ' precipitates, the weakly-coupled dislocation pair shearing mechanism dominates. As precipitate size increases, the mechanism transitions to strongly-coupled dislocation pair shearing, with maximum critical resolved shear stress occurring at the transition point. Calculations indicate this transition occurs at a γ' precipitate size of 48 nm. The critical resolved shear stress increases initially then decreases with increasing γ' precipitate size. The increased yield strength after aging at 700°C may result from the initial small γ' precipitate size in the standard heat-treated condition; aging increases precipitate size and thus critical resolved shear stress. However, after aging at 750°C and 800°C, γ' precipitate size increases significantly, strongly-coupled interactions dominate, and critical resolved shear stress decreases with further size increase, leading to reduced yield strength.

3 Conclusions

- (1) The Ti/Al ratio has no significant effect on microstructural characteristics in the standard heat-treated condition, though the high Ti/Al ratio alloy exhibits higher γ' lattice misfit.
- (2) γ' precipitates remain spherical and gradually coarsen with increasing aging temperature and time, with Ti/Al ratio showing no obvious effect on morphology. During long-term aging at 700°C and 750°C, γ' precipitate growth follows LSW theory, with higher growth rates in the high Ti/Al ratio alloy. At 800°C, reduced driving force for growth and depletion of γ' -forming elements cause deviation from LSW theory, with significantly decreased growth rates after long-term aging.
- (3) Yield strength increases markedly after aging at 700°C for 10480 h, but decreases significantly after aging at 750°C and 800°C for 10480 h, while elongation increases substantially. The strength variation is attributed to the effect of γ' precipitate coarsening on critical resolved shear stress. Small variations in Ti/Al ratio have no significant effect on 700°C tensile properties.

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