

Microstructural Damage and Mechanical Properties of GH4033 Alloy After Short-term Overheating: Post-print

Authors: Tong Jinyan, Feng Wei, Fu Chao, Zheng Yunrong, Feng Qiang

Date: 2023-03-19T00:00:00+00:00

Abstract

Heat treatments involving short-term overheating at 900–1100 °C for 3 min were performed on the dovetail sections of in-service γ H4033 alloy second-stage turbine blades from aeroengines, followed by microstructural characterization and mechanical property testing, to investigate the microstructural damage during short-term overheating and its influence on room-temperature hardness and stress rupture life at 700 °C and 430 MPa. The results indicate that γ' phase particles in the γ H4033 alloy undergo coarsening and dissolution during short-term overheating; when the temperature reaches 980 °C and above, the γ' phase completely dissolves after holding for 3 min; with increasing overheating temperature, grain boundary carbides gradually dissolve, completely dissolving at 1100 °C and leading to initiation of grain growth. The room-temperature hardness of the blade alloy after short-term overheating decreases sharply with dissolution of the γ' phase, dropping to approximately 170 HV when the γ' phase is completely dissolved. The stress rupture life of the alloy at 700 °C and 430 MPa shows an initial increase followed by a sharp decrease with increasing short-term overheating temperature, which is primarily influenced by the dissolution and re-precipitation of the γ' phase and the dissolution of grain boundary carbides.

Full Text

MICROSTRUCTURAL DEGRADATION AND MECHANICAL PROPERTIES OF GH4033 ALLOY AFTER SHORT-TIME OVERHEATING

TONG Jinyan^{1,2}), FENG Wei^{1,3}), FU Chao²), ZHENG Yunrong²),
FENG Qiang^{1,2})

¹) National Centre for Materials Service Safety, University of Science and Technology Beijing, Beijing 100083

²⁾ State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083

³⁾ Casting Center, Beijing Institute of Aeronautical Materials, Beijing 100095

Correspondent: FENG Qiang, professor, Tel: (010)82375850, E-mail: qfeng@skl.ustb.edu.cn

Supported by National High Technology Research and Development Program of China (No.2012-AA03A513) and Key Project of Chinese Ministry of Education (No.625010337)

ABSTRACT

Turbine blades in aircraft engines are subjected to long-term service under high-temperature, high-pressure gas environments and complex stress conditions, with creep being one of the primary deformation modes. Consequently, turbine blades are typically manufactured from nickel-based superalloys with excellent high-temperature creep resistance. However, microstructural damage occurs in blade alloys during normal or overheating service, leading to property degradation and even blade failure.

This study investigated the microstructural degradation and its effects on mechanical properties of GH4033 alloy sections taken from the shank region of a serviced second-stage turbine blade. Samples were subjected to short-time overheating treatments at 900-1100 °C for 3 minutes, followed by microstructural characterization and mechanical property testing. The effects of overheating on room-temperature hardness and stress rupture life at 700 °C/430 MPa were systematically analyzed.

Results show that γ' precipitates in GH4033 alloy coarsened and dissolved during short-time overheating. Complete dissolution of γ' phase occurred after 3 minutes at 980 °C and above. Grain boundary carbides gradually dissolved with increasing temperature, disappearing entirely at 1100 °C, which triggered grain growth. Room-temperature hardness decreased sharply with γ' phase dissolution, dropping to approximately 170 HV upon complete dissolution. Stress rupture life at 700 °C/430 MPa initially increased then decreased dramatically with overheating temperature, primarily due to the competing effects of γ' phase dissolution/re-precipitation and grain boundary carbide dissolution.

KEY WORDS GH4033 wrought alloy, turbine blade, overheating, microstructure, rupture property

1. Introduction

Aircraft engine turbine blades operate under high-temperature, high-pressure gas and complex stress conditions for extended periods, with creep represent-

ing a primary deformation mode. Consequently, turbine blades are typically fabricated from nickel-based superalloys exhibiting excellent high-temperature creep performance. However, microstructural damage accumulates in blade alloys during normal or overheating service, resulting in property degradation and potential blade failure.

Significant temperature and stress fluctuations occur under different operating conditions, such as flame shift due to incomplete fuel combustion, cooling hole blockage, and sudden load increases. These abnormal engine operating states can lead to overheating service. Overheating service of turbine blades is generally classified into two categories based on service temperature: burning (when temperature exceeds the alloy solidus) and overheating (when temperature exceeds normal service temperature but remains below the solidus). For wrought superalloy turbine blades, overheating is the typical manifestation.

Overheating events usually last only a few minutes, yet can cause rapid microstructural damage and mechanical property degradation, potentially resulting in catastrophic consequences. Therefore, investigating the microstructural damage mechanisms during short-time overheating and their influence on properties is essential for ensuring engine service safety.

Previous studies have shown that: (1) γ' precipitates in superalloy turbine blades undergo progressive coarsening, partial dissolution, extensive dissolution, and complete dissolution with increasing overheating temperature and time, leading to reduced volume fraction and rapid creep elongation failure; (2) short-time overheating also causes degradation of grain boundary carbides, which sequentially exhibit thickness increase, formation of continuous carbide films, and dissolution. Dissolved carbides can re-precipitate in cellular morphology during subsequent cooling and service, creating Cr-depleted and γ' -depleted zones that drastically reduce oxidation resistance and high-temperature strength, promoting crack formation.

Despite recognized hazards, existing literature focuses primarily on failure analysis. Systematic investigations of microstructural damage characteristics during short-time overheating and their effects on properties remain limited. Moreover, due to the complexity and heterogeneity of superalloy microstructures, the independent and interactive effects of various damage types on mechanical properties are not fully understood.

To address these challenges, this work utilized serviced GH4033 alloy second-stage turbine blades with low alloying and low γ' phase content. The study characterized microstructural damage patterns under different overheating conditions and investigated degradation of room-temperature hardness and stress rupture life at 700 °C/430 MPa. The influence of microstructural damage on property degradation and its implications for overheating inspection of this blade type were analyzed to establish a foundation for evaluating overheating service damage in wrought superalloy turbine blades.

2. Experimental

2.1 Materials and Overheating Treatment

The experimental material consisted of GH4033 alloy second-stage turbine blades from an aircraft engine after approximately 1600 h of service. The chemical composition (wt.%) was: Cr 20.57, Al 0.84, Ti 2.62, Fe 0.69, C 0.05, Si 0.45, Ni balance. [Figure 1: see original paper] shows the blade profile, indicating a thin trailing edge. Finite element analysis of the blade under maximum operating conditions within normal service range revealed that the temperature near the blade tip reached approximately 768 °C, while the shank region experienced the lowest temperature of about 502 °C—below the design service temperature of GH4033 alloy. Therefore, the microstructure in the shank region was considered representative of the original pre-service condition.

A STA440C differential scanning calorimeter (DSC) was used to measure phase transformation behavior and temperatures. DSC samples were taken from the shank region, tested from room temperature to 1400 °C at a heating rate of 10 °C/min. The γ' phase dissolution temperature range was defined as the temperature corresponding to the intersection points between the baseline tangent and the maximum slope tangents at the onset and end of the dissolution endothermic peak.

Overheating treatment samples (5 mm thick) were also taken from the shank region. Overheating temperatures were 900, 950, 980, 1050, and 1100 °C, with a holding time of 3 minutes. Treatments were performed in a tube furnace under air atmosphere. Samples were inserted when the furnace reached the target temperature and stabilized; timing began upon reheating to the target temperature, followed by water quenching after 3 minutes.

2.2 Mechanical Testing

Vickers hardness was measured using a VMHT 30M microhardness tester with a 3 kg load; the average of six measurements was reported. For stress rupture testing, non-standard plate-type specimens were used due to the thin trailing edge, with dimensions shown in [Figure 2: see original paper]. Following technical requirements per HB/91-1985 for GH4033 alloy turbine blades, testing conditions were set at 700 °C/430 MPa. Tests were conducted on an RDJ50 mechanical creep rupture machine according to GB/T 2039-2009. All specimens were held at test temperature for 2 h before loading, then furnace-cooled after fracture. Two specimens were tested for each condition, with results averaged.

Since the test temperature coincided with the aging heat treatment temperature for GH4033 alloy, microstructural evolution could occur during the holding period. Therefore, some overheated samples were subjected to 700 °C/2 h heat treatment and water quenched to characterize the microstructure prior to loading.

2.3 Microstructural Characterization

Samples were prepared using standard metallographic procedures. Grain structure was revealed by electrolytic etching for 15–20 s at 3 V in a $\text{H}_2\text{C}_2\text{O}_4:\text{H}_2\text{O} = 1:9$ solution and observed with a 4XC optical microscope (OM). Grain boundary carbides and γ' phase were revealed by electrolytic etching for 1–2 s at 2.5 V in a $\text{H}_3\text{PO}_4:\text{HNO}_3:\text{H}_2\text{SO}_4 = 1:3:5$ solution. For samples overheated at 1050 and 1100 °C, a Murakami solution (10 g $\text{K}_3[\text{Fe}(\text{CN})_6]$ + 10 g KOH + 100 mL H_2O) was used for ~20 s chemical etching to reveal grain boundary carbide morphology. A SUPRA 55 field-emission scanning electron microscope (SEM) in secondary electron mode was used to observe grain boundary carbides and intragranular γ' phase morphology, with EDS for qualitative chemical analysis.

Average grain size was measured using Image Tool software, averaging over 100 grains from five different fields. γ' phase average particle size and volume fraction were quantified from 100,000 \times SEM images. To ensure measurement of only surface-layer γ' precipitates, Photoshop was used to select γ' particles with uniform brightness. Volume fraction was determined by grid method, and particle size was measured as the average of 200 randomly selected γ' particles using Image Pro software.

3. Results

3.1 Original Microstructure

[Figure 3: see original paper] shows the original microstructure of GH4033 alloy. Black, gray, and white contrast carbide particles were observed within grains and along grain boundaries, with fine γ' precipitates dispersed in the intragranular matrix. Based on GH4033 alloy characteristics and EDS analysis, large black particles were identified as Ti-rich MC carbides; smaller gray and white intragranular particles were Cr-rich Cr_7C_3 carbides; fine semi-continuous and discontinuous grain boundary particles were Cr-rich M_{23}C_6 carbides. Intragranular carbide volume fraction was low (<0.5%). The matrix contained ~14.0% γ' phase with a particle size of (26 \pm 3) nm. The low intragranular carbide volume fraction did not significantly affect properties.

[Figure 4: see original paper] presents the DSC heating curve. Distinct endothermic peaks appeared at 855 and 1335 °C. The peak at 855 °C corresponded to γ' phase dissolution in the matrix, with complete dissolution occurring at 979 °C. The 1335 °C peak represented the alloy solidus temperature.

3.2 Microstructural Evolution After Overheating

3.2.1 Grain Structure Grain morphology and size significantly affect creep-rupture properties of wrought superalloys, with potential for abnormal grain growth at high temperatures. [Figure 5: see original paper]a shows the original

GH4033 alloy OM image, revealing equiaxed grains of alternating sizes with an average grain size of $(250 \pm 25) \mu\text{m}$. After overheating at 900, 950, 980, and 1050 °C for 3 minutes and water quenching (see original paper [b]), with average grain size increasing to $(279 \pm 25) \mu\text{m}$.

3.2.2 Grain Boundary Carbide Distribution [Figure 6: see original paper] shows grain boundary carbide distribution in the original alloy and after overheating. Original carbides were primarily fine semi-continuous films. After 900 °C/3 min, distribution remained similar. At 950 and 980 °C, carbide particle density decreased, becoming predominantly fine discontinuous particles with local semi-continuous films. Above 1050 °C, extensive carbide dissolution occurred. Murakami etchant (which removes carbides) created dark contrast holes marking former carbide positions. [Figure 6: see original paper]e shows only sparse discontinuous carbide particles remained after 1050 °C/3 min, while complete dissolution occurred at 1100 °C ([Figure 6: see original paper]f).

3.2.3 γ' Phase Morphology [Figure 7: see original paper] shows γ' phase morphology after overheating, with quantitative data in . Both coarsening and dissolution occurred simultaneously during short-time overheating. As temperature increased, coarsening accelerated while volume fraction decreased. At 900 °C/3 min, γ' particles grew to ~ 32 nm with volume fraction decreasing from 14.0% to 12.0%. At 950 °C, particles reached ~ 37 nm with volume fraction dropping to $\sim 8.7\%$. Complete dissolution occurred at 980 °C and above.

3.3 Hardness After Overheating

[Figure 8: see original paper] shows room-temperature Vickers hardness after overheating. Hardness rapidly decreased from the original $(320 \pm 10) \text{HV}$ below the standard requirement range, with more significant reduction at high temperatures. After 900 °C/3 min, hardness was ~ 170 HV, respectively. No further decrease occurred at 1050 and 1100 °C, remaining ~ 170 HV. This correlates with γ' volume fraction changes, indicating a direct relationship.

3.4 Stress Rupture Properties

3.4.1 Rupture Life [Figure 9: see original paper] shows stress rupture life at 700 °C/430 MPa after overheating. Rupture life initially increased then decreased sharply with temperature: 900 °C/3 min yielded $(125.0 \pm 6.0) \text{h}$, similar to the original $(130.0 \pm 5.0) \text{h}$; 950 °C/3 min showed significant improvement to $(169.0 \pm 10.0) \text{h}$; at 1100 °C, specimens failed in only 0.5 h.

3.4.2 Pre-Loading Microstructure To explain the life variation, microstructures before loading were examined for samples overheated at 950 and 980 °C. Since GH4033 alloy does not exhibit grain structure changes during long-term aging at 700 °C, only grain boundary carbides and γ' phase were analyzed. [Figure 10: see original paper] shows that after 950 °C/3 min + 700 °C/2 h, grain boundary carbides transformed from discontinuous particles to

semi-continuous films, and finer γ' precipitates reappeared between original particles, increasing volume fraction to 13.5%—similar to the original state. In contrast, after 980 °C/3 min + 700 °C/2 h, grain boundary carbides remained unchanged and no γ' re-precipitation was observed.

3.4.3 Post-Rupture Microstructure [Figure 11: see original paper] shows microstructures near fracture surfaces after rupture testing. Grain boundary carbide distributions remained similar to pre-loading states. Both conditions exhibited dispersed γ' particles. For 950 °C/3 min specimens, post-rupture γ' volume fraction was 13.4% (similar to pre-loading) with particle size ~40 nm. For 980 °C/3 min specimens, γ' re-precipitated during testing and cooling, with size ~9 nm.

4. Discussion

Grain size, grain boundary carbide distribution, and γ' strengthening phase volume fraction and size significantly influence wrought superalloy performance. Large grains reduce grain boundary area and sliding, improving properties. Grain boundary carbides prevent abnormal grain growth, and fine discontinuous carbides effectively hinder grain boundary sliding during creep, enhancing rupture life. During short-time overheating, these microstructures degrade differently, affecting mechanical properties.

4.1 Effect of Overheating Temperature on Microstructural Damage

GH4033 alloy turbine blades operate below 700 °C. Abnormal engine operation can cause overheating, drastically reducing properties and potentially causing premature failure. Overheating duration must be strictly limited. Before installation, GH4033 blades receive standard heat treatment (1080 °C/8 h/air cool + 700 °C/16 h/air cool), producing ~8.0% γ' phase (20 nm) and 0.25-0.32% carbides (Cr_{23}C_6 , Cr_7C_3 , TiC/Ti(C,N)), with Cr_{23}C_6 as fine discontinuous grain boundary particles.

After 900-1100 °C/3 min overheating, microstructural damage manifested as γ' dissolution, grain boundary carbide dissolution, and grain growth. γ' volume fraction decreased from 14.0% to 8.7% at 950 °C, with complete dissolution above 980 °C. Grain boundary carbides dissolved significantly at 1050 °C and completely at 1100 °C, accompanied by grain growth.

4.2 Effect of γ' Phase on Rupture Performance

As the primary strengthening phase, γ' volume fraction and size critically affect creep-rupture properties. Higher volume fraction and smaller particle size improve performance. After 950 °C/3 min, γ' volume fraction decreased to 8.7% while particles coarsened to ~37 nm, yet rupture life increased. This resulted

from re-precipitation during the 2 h pre-loading hold at 700 °C. Partial dissolution created supersaturation and retained ultrafine γ' particles that served as nucleation sites, enabling rapid re-precipitation of finer γ' particles and volume fraction recovery to near-original levels, thereby strengthening the matrix and increasing life.

Conversely, after 980 °C/3 min, complete dissolution eliminated nucleation sites, preventing re-precipitation during the 2 h hold. Consequently, the alloy lacked γ' strengthening during initial loading, facilitating dislocation slip and causing drastic life reduction.

4.3 Room-Temperature Hardness for Overheating Inspection

While creep-rupture properties are critical, testing is time-consuming and costly for maintenance inspection. For low-alloy, low- γ' content superalloys like GH4033, γ' dissolution is highly temperature-sensitive and cannot re-precipitate during rapid cooling, causing hardness degradation. Room-temperature hardness measurement provides a simple, effective overheating inspection method.

Vickers hardness indents were <100 μm , smaller than average grain sizes. Intragranular carbide volume fraction was $<0.5\%$ with large interparticle spacing, allowing negligible effects on hardness. Thus, hardness was primarily governed by γ' phase and grain boundary carbides. Hardness decreased progressively with γ' dissolution, stabilizing at ~ 170 HV after complete dissolution, regardless of further carbide dissolution.

Industry practice uses room-temperature hardness to assess overheating in low-alloy wrought superalloy blades. However, this study reveals hardness alone cannot determine serviceability. Per aerospace standard HB/Z 91-1985, GH4033 blade forgings require Vickers hardness of 268-339 HV and rupture life ≥ 60 h at 700 °C/430 MPa. All overheated samples (900-1100 °C/3 min) exhibited hardness below standard. Yet rupture testing showed specimens overheated at 900 and 950 °C still met requirements (125.0 and 169.0 h), while those at ≥ 980 °C failed. A hardness of ~ 170 HV serves as an indirect indicator of complete γ' dissolution and non-compliant rupture performance. For hardness values between 170 HV and the standard minimum, supplementary rupture testing is necessary for accurate damage assessment.

Notably, damage severity varies with blade type, alloy grade, and overheating condition. Overheating detection does not automatically warrant blade retirement; retirement criteria must combine mechanical performance under overheating conditions with field experience. Turbine blade overheating is complex, requiring systematic material-specific studies of damage mechanisms, property effects, and restoration heat treatments to establish appropriate retirement standards and optimize economics.

5. Conclusions

1. Microstructural damage in GH4033 alloy after 900–1100 °C/3 min overheating included γ' phase dissolution, grain boundary carbide dissolution, and grain growth. γ' volume fraction decreased from 14.0% to 8.7% at 950 °C, with complete dissolution above 980 °C. Grain boundary carbides dissolved significantly at 1050 °C and completely at 1100 °C, initiating grain growth.
2. After overheating damage, stress rupture life at 700 °C/430 MPa decreased drastically from 130.0 h in the original state to ~3.7 h after 980 °C/3 min upon complete γ' dissolution. Grain boundary carbide distribution did not significantly affect life until complete dissolution, after which life further decreased to ~0.5 h.
3. Room-temperature Vickers hardness after overheating above 900 °C fell below the aerospace standard (268–339 HV), decreasing with γ' volume fraction. A hardness of ~170 HV serves as an indirect criterion for complete γ' dissolution and non-compliant rupture performance. Hardness values above 170 HV require supplementary rupture testing to evaluate overheating damage severity.

References

- [1] Reed R C. *The Superalloys Fundamentals and Applications*. Cambridge, UK: Cambridge University Press, 2006: 18
- [2] Guo J T. *Materials Science and Engineering for Superalloys*. Vol.3, Beijing: Science Press, 2010: 508
- [3] Feng Q, Tong J Y, Zheng Y R, Wang M L, Wei W J, Zhao H L, Yuan X F, Ding X F. *Mater China*, 2012; 31(12): 21
- [4] Koul A, Wallace W. *Met Mater Trans*, 1983; 14A: 183
- [5] Liburdi J, Lowden P, Nagy D, De Priamus T R, Shaw S. *Proc ASME Turbo Expo*, Orlando: International Gas Turbine Institute, 2009: 819
- [6] Yoo K B, Lee H S. *Mater Sci Forum*, 2010; 654-656: 2523
- [7] Liu Q Q. *Manufacture Technologies and Failure Analyses of Blades in Aircraft Engines*. Beijing: Aviation Industry Press, 2011: 98
- [8] Tao C H. *Failure Analysis and Prevention for Rotor in Aero-Engine*. Beijing: National Defense Industry Press, 2008: 1
- [9] Zhao W X, Li Y, Fan Y W, Zheng Y R. *J Mater Eng*, 2012; (8): 39
- [10] Tawancy H M, Al-Hdhrami L. *Eng Fail Anal*, 2008; 15: 1027
- [11] Cai Y L, Zheng Y R. *Metallographic Research of Superalloys*. Beijing: National Defense Industry Press, 1986: 228
- [12] Sun S Z, Li S Y, Zheng Y R. *J Mater Eng*, 1990; (3): 45
- [13] Li Y, Hou X Q, Tao C H, Jiang T. *J Iron Steel Res*, 2011; 23(suppl 2): 452
- [14] Liu D L, Zhang W F, Li C G, Miao H B. *Heat Treat Met*, 2007; 32(1): 71
- [15] Gao Y. *New Manufacture Technologies, Metallograph Atlas and Manual for*

- Data of Superalloys*. Beijing: Science and Technology of China Press, 2006: 57
- [16] Tong J Y, Ding X F, Wang M L, Zheng Y R, Yagi K, Feng Q. *Mater Sci Eng*, 2014; A618: 605
- [17] Xu Y L, Jin Q M, Xiao X S, Cao X L, Jia G Q, Zhu Y M, Yin H J. *Mater Sci Eng*, 2011; A528: 4600
- [18] Zhao Y, Gai X Y, Song G H. *Phys Test Chem Anal: Phys Test*, 2007; 43A: 498
- [19] Bridges P J, White C H, Durber G L R. *The Nimonic Alloys*. Bristol: Edward Arnold Ltd, 1974: 33
- [20] Richards E. *J Inst Met*, 1968; 96: 365
- [21] Carter T J. *Eng Fail Anal*, 2005; 12: 237
- [22] Zhang L H. *Heat Treat*, 2003; 18(3): 26
- [23] Ge T T. Master Thesis, University of Science and Technology Beijing, 2006
- [24] Voice W E, Faulkner R G. *Met Mater Trans*, 1985; 16A: 511
- [25] Furillo F T, Davidson J M, Tien J K, Jackman L A. *Mater Sci Eng*, 1979; A39: 267
- [26] Iwashita C H. PhD Dissertation, Lehigh University, Bethlehem, 1979
- [27] Bhowal P, Wright E, Raymond E. *Met Trans*, 1990; 21A: 1709
- [28] Locq D, Caron P, Raujol S, Pettinari-Sturmel F, Coujou A, Clement N. In: Green K A, Pollock T M, Harada H, Howson T E, Reed R C, Schirra J J, Walston S eds., *Superalloys 2004*, Pennsylvania: TMS, 2004: 179
- [29] Sun K J, Gai X Y, Li C X. *Phys Test Chem Anal: Phys Test*, 2009; 7A: 393
- [30] Osada T, Nagashima N, Gu Y F, Yuan Y, Yokokawa T, Harada H. *Scr Mater*, 2011; 64: 892

Note: Figure translations are in progress. See original paper for figures.

Source: ChinaXiv – Machine translation. Verify with original.