

Influence of carbon on microstructural stability and stress rupture properties of an additively manufactured superalloy

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Abstract: The effects of carbon content (0.05, 0.1, and 0.15, in wt.%) on the microstructural evolution and stress rupture behavior of an additively manufactured Ni-based superalloy (ZGH451) were comprehensively investigated. Long-term thermal exposure tests at 1,000 °C reveal that γ' precipitates follow a diffusion-controlled coarsening process consistent with the Lifshitz-Slyozov-Wagner (LSW) model, while higher carbon contents increase the effective diffusion coefficient and accelerate coarsening. The evolution of carbides displays a strong dependence on carbon level: low-carbon alloys maintain discrete, fine carbides that stabilize grain boundaries, whereas excessive carbon (>1wt.%) promotes the formation of coarse and continuous carbide films along grain boundaries, serving as preferential crack initiation sites. Stress rupture tests conducted at 760 °C/780 MPa and 980 °C/260 MPa show a pronounced reduction in rupture life with increasing carbon content due to intergranular cracking, carbide-induced stress concentration, and carbon-assisted rafting of γ' precipitates. Overall, an optimal carbon content near 0.05wt.% provides a balance between microstructural stability and high-temperature strength. These findings provide guidances for the compositional optimization of additively manufactured superalloys intended for long-term high-temperature service.

Keywords: additive manufacturing; superalloy; carbon; stress rupture; microstructural stability

CLC numbers: TG146.1+5

Document code: A

Article ID: 1672-6421(2026)05-660-13

1 Introduction

Superalloys are designed to perform at elevated temperatures, offering excellent mechanical strength, oxidation resistance, and creep resistance. These materials are critical in industries such as aerospace, power generation, and automotive, where components endure extreme thermal and mechanical stresses^[1-3]. The emergence of additive manufacturing (AM)

has significantly accelerated the development of superalloys, as AM enables the production of complex geometries, reduces material waste, and enhances both performance and design flexibility^[4, 5].

A key strategy for improving high-temperature performance is the incorporation of grain boundary strengthening elements^[6, 7]. These elements enhance resistance to deformation and failure through strengthening the grain boundaries, which are critical in determining the high-temperature strength and structural stability. Elements such as carbon, boron, zirconium, and hafnium are commonly employed to promote the formation of stable phase at grain boundaries or to improve grain boundary cohesion, thereby enhancing the resistance to both thermal and mechanical degradation^[8-10].

Carbon serves as a critical grain boundary strengthening element in superalloys by forming carbides that enhance strength and resistance to high-temperature

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Received: 2025-01-30; Revised: 2025-06-26; Accepted: 2025-10-20

degradation^[11, 12]. In addition to mechanical enhancement, carbon helps prevent grain boundary sliding and maintains structural stability under thermal stresses, thereby improving creep resistance and oxidation performance^[13-15]. Although these roles of carbon are well-established in conventional cast superalloys, its behavior within AM systems remains relatively unexplored.

Recent research has demonstrated carbon's dual advantages in AM superalloys: reducing solidification cracking while enhancing alloy strength^[16, 17]. However, considerable uncertainties remain regarding its influence on stress rupture life, a critical parameter for material durability, during prolonged high-temperature service. This uncertainty arises from the fundamental differences in microstructural evolution between AM and conventional casting processes. The characteristically rapid cooling rates of AM processes (approximately 10^3 – 10^4 K·s⁻¹) result in distinctive microstructures featuring refined grains, minimized elemental segregation, and nanoscale precipitates, which are markedly different from those formed in conventional casting^[16, 17]. These differences may modify the mechanisms by which carbon exerts its effects, such as promoting solute trapping rather than the formation of large blocky carbides. Consequently, existing models that elucidate carbon's role in cast superalloys^[18, 19] may not be directly transferable to AM materials.

To address this research gap, a systematic investigation of carbon-containing nickel-based AM superalloy ZGH451 was carried out, focusing on the interplay among carbon content, microstructural evolution, and stress rupture resistance. By combining service-condition simulations with advanced microstructural characterization, this study elucidates the

influence of carbon on the long-term performance of AM superalloys. These findings highlight two fundamental aspects of carbon's role in AM superalloys: (1) its influence on microstructural stability, and (2) the governing mechanisms behind its impact on stress rupture performance during rapid solidification processes. These results are expected to provide actionable insights for optimizing both composition and processing parameters of AM superalloys.

2 Experimental procedure

The experimental alloy used in this study was ZGH451, a superalloy specifically designed for additive manufacturing, offering excellent high-temperature mechanical properties. The alloy ingots with different carbon contents (0.05wt.%, 0.1wt.%, and 0.15wt.%), produced by vacuum induction melting, were processed into powders via vacuum gas atomization, resulting in powders with varying carbon contents. The corresponding alloys were designated as 0.05C, 0.10C, and 0.15C, respectively, based on their carbon content. The powders' compositions are listed in Table 1. The powders were sieved to achieve the target particle sizes ranging from 80 to 325 mesh. Test samples were fabricated using the direct energy deposition (DED) method with an LDM8060 coaxial powder feeder. Since printing parameters can influence microstructure and defect formation, all samples were produced under identical conditions: laser power of 1,800 W, scan speed of 720 mm·min⁻¹, overlap rate of 40%, layer thickness of 0.3 mm, and powder feed rate of 338 g·h⁻¹. The building process is shown in Fig. 1(a). The final samples were cubic columns with a height of 60 mm and a base of 20 mm×20 mm.

Table 1: Measured ZGH451 alloy composition for current study in wt.%

Alloy	Al+Ta+Ti	Co+Cr+Mo+W	C	B	Ni
0.05C	11.45	25.95	0.046	0.016	Bal.
0.1C	11.49	25.91	0.087	0.015	Bal.
0.15C	11.49	25.94	0.14	0.013	Bal.

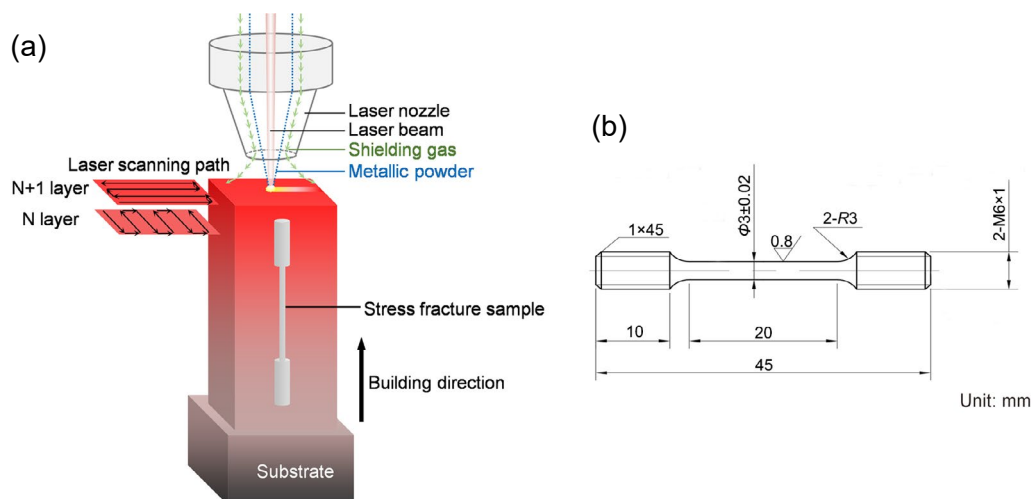


Fig. 1: Schematic diagrams of building process (a) and tensile specimen (b)

A series of thermal exposure tests at 1,000 °C for different times (100, 200, 500, and 1,000 h) were performed to study the influence of carbon on the alloy's microstructural stability. Stress rupture specimens with dimensions of M6×Φ3 (mm) were cut from the middle section of the deposited cubic columns, parallel to the building direction, to avoid potential microcrack regions at the bottom and the equiaxed grain zone at the top, as shown in Fig. 1(b). According to the Chinese standard GB/T 2039-2012, stress rupture tests were carried out on an RDL50 high-temperature stress-rupture testing machine at 760 °C/780 MPa and 980 °C/260 MPa, respectively.

The as-deposited microstructure was observed from samples cut at the same height of the as-deposited columns at about 30 mm from the substrate. Microstructural analysis was conducted following a combination of electrochemical etching and chemical corrosion. Electrochemical etching was performed in a solution containing 170 mL H₃PO₄, 10 mL H₂SO₄, and 15 g Cr₂O₃ at 8 V for 5 s. Chemical etching was performed in a solution consisting of 10 g CuSO₄, 50 mL HCl, and 100 mL H₂O for 10 s.

To prevent contamination, stress rupture specimens were ultrasonically cleaned in ethanol after testing. Vibratory polishing was then performed for electron backscatter diffraction (EBSD) analysis using a Tescan Mira3 field emission scanning electron microscope (SEM) equipped with a Nordlys Max3 EBSD system. The deformation mechanisms of the alloy were studied using a Talos F200X transmission electron microscope (TEM). TEM samples were collected 5 mm from the fracture surface, cut perpendicular to the building

direction with a thickness of 500 μm, and then mechanically thinned to approximately 50 μm. Final electron transparency was achieved via twin-jet electrolytic polishing performed at -25 °C. The electrolyte used for polishing consisted of 10% perchloric acid and 90% ethanol. The equilibrium phase diagrams were calculated using Thermo-Calc software with the TCNI10 database to assess the impact of carbon on precipitation.

3 Results

3.1 As-built microstructure

Figure 2 illustrates the the microstructural evolution of as-deposited alloys with different carbon contents. Table 2 summarizes the statistically quantified area fractions and dimensions of both γ' precipitates and carbides. As a non-γ'-forming element, carbon has minimal impact on the volume fraction, morphology, and size of γ' precipitates. Nevertheless, the carbide volume fraction increases significantly with an increase in carbon content. The characteristic rapid solidification inherent to additive manufacturing processes significantly inhibits carbide coarsening. This effect is primarily attributed to the substantial thermal undercooling (Δ*T*) generated during accelerated solidification. The elevated Δ*T* not only promotes the formation of high-density nanoscale carbide nuclei during precipitation but also restricts solute diffusion^[20, 21]. As the carbon content increases from 0.05wt.% to 0.15wt.%, the average carbide size increases by only about 1 μm, indicating a relatively weak effect of carbon on carbide size, consistent

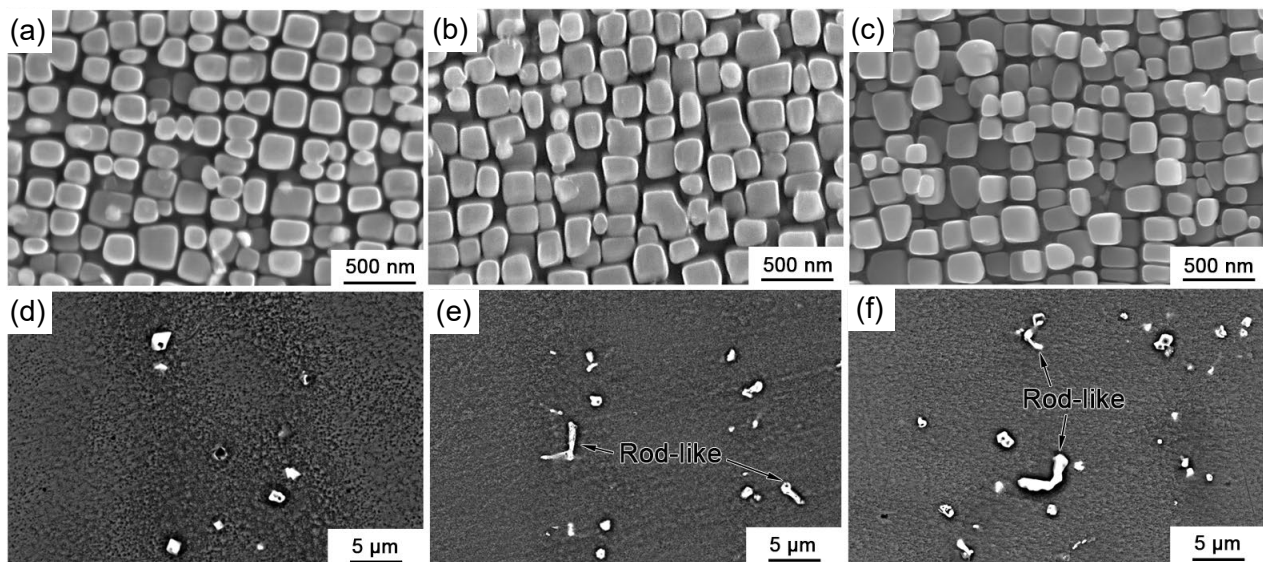


Fig. 2: Morphologies of γ' precipitates and carbides in 0.05C (a, d), 0.1C (b, e), and 0.15C (c, f) alloys

Table 2: Statistic results of precipitates and carbides

Alloy	γ' size (nm)	γ' area fraction (%)	Carbide size (μm)	Carbide area fraction (%)
0.05C	254.29±155.21	61.79±4.23	0.97±0.54	0.03±0.23
0.1C	230.74±142.59	59.89±3.18	1.53±0.89	0.16±0.10
0.15C	262.85±112.83	60.61±5.62	1.98±1.18	0.27±0.18

with the behavior observed in cast superalloys^[19]. Notably, the 0.1C alloy exhibits sparsely distributed larger rod-shaped carbides (~5 μm), whereas in the 0.15C alloy, both the density and dimensions of these rod-like carbides progressively increase.

3.2 Microstructure after thermal exposure

Figure 3 illustrates the morphological changes of γ' precipitates in the alloys subjected to prolonged thermal exposure. Remarkably, topologically close-packed (TCP) phases emerge exclusively after 1,000 h of exposure, as evidenced by the white needle-shaped precipitates visible in the insets of Figs. 3(d), (h), and (l). This finding demonstrates the superior microstructural stability of AM superalloys, which can be attributed to reduced elemental segregation^[22, 23]. Progressive coarsening of γ' precipitates is observed in all alloys with extended exposure duration. Moreover, at equivalent exposure times, the γ' volume fraction exhibits a gradual decrease with increasing carbon content.

To quantify γ' coarsening kinetics, precipitate dimensions were systematically measured across various exposure durations. Notably, γ' characteristics differ between dendritic

core and interdendritic regions due to elevated Al/Ti concentrations in the interdendritic regions. Consequently, measurements were specifically conducted on γ' precipitates located in the dendritic cores. According to the well-established Ostwald ripening theory, the γ' evolution conforms to the LSW matrix diffusion model^[24] and follows the relationship of $(r^3 - r_0^3)$ with t , where r_0 and r are the sizes of γ' before and after thermal exposure, and t is the duration of thermal exposure. As depicted in Fig. 4, linear regression of 1,000 °C exposure data reveals strong correlation coefficients, confirming diffusion-dominated coarsening mechanisms during extended aging. Intriguingly, the γ' coarsening rate constant (K) exhibits carbon-dependent enhancement, suggesting carbon's potential influence on atomic diffusion processes, a phenomenon will be thoroughly analyzed in subsequent sections.

Figure 5 reveals the morphological evolution of carbides (the white precipitated phase, partially indicated by the yellow arrows) during thermal exposure, exhibiting multifaceted evolution patterns distinct from γ' precipitates. To ensure statistical rigor, five representative fields were analyzed at identical magnifications for each condition. Notably, grain boundary carbides demonstrating abnormal coarsening

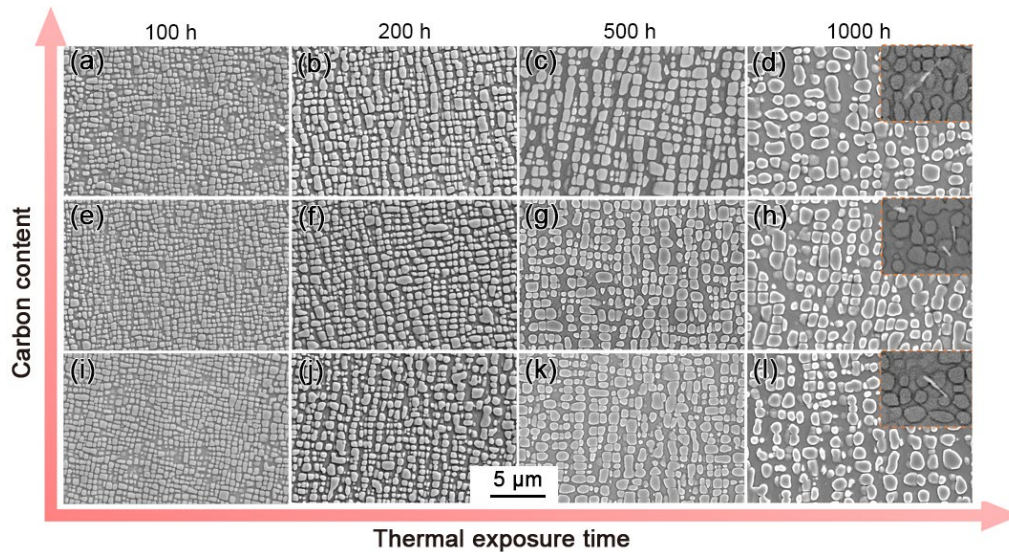


Fig. 3: Morphologies of γ' precipitates of three alloys after thermal exposure for 100 h (a, e, i), 200 h (b, f, j), 500 h (c, g, k), and 1,000 h (d, h, l), respectively: (a-d) 0.05C alloy; (e-h) 0.1C alloy; (i-l) 0.15C alloy

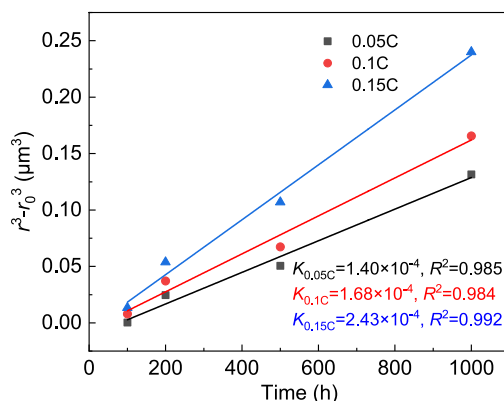


Fig. 4: Relationship between thermal exposure time and size difference of γ' precipitates before and after thermal exposure

were systematically omitted from quantification to avoid skewing results. Divergent evolutionary trajectories emerged across carbon levels: In the 0.05C alloy, carbide dimensions initially expand before subsequent refinement with prolonged exposure. In the 0.05C alloy, the carbide perimeter exhibits a non-monotonic trend, initially increasing before decreasing after 200 h. Despite this reduction in average carbide size at later stages, the total carbide area fraction remains remarkably stable. This indicates that the reduction in average carbide size is effectively offset by the precipitation of fine carbides, preventing a drop in the overall phase fraction. This stabilization mechanism contrasts sharply with that observed in the other two high-carbon alloys, where both carbide size and area fraction exhibit sustained increases under prolonged thermal exposure, as shown in Fig. 6.

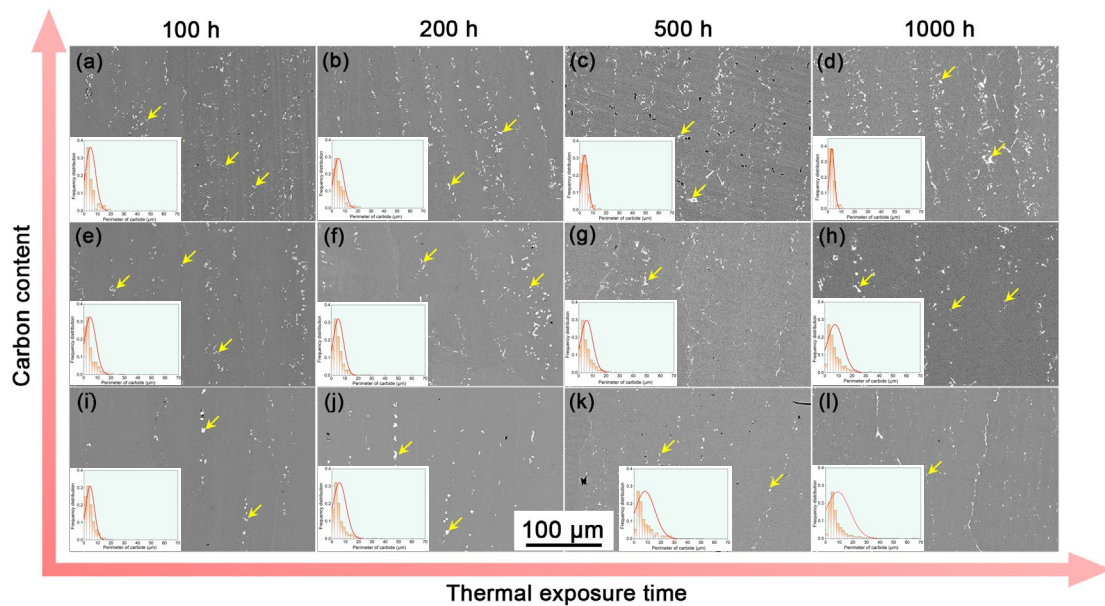


Fig. 5: Morphology of carbides of three alloys after thermal exposure for 100 h, 200 h, 500 h, and 1,000 h, respectively: (a–d) 0.05C; (e–h) 0.1C; (i–l) 0.15C

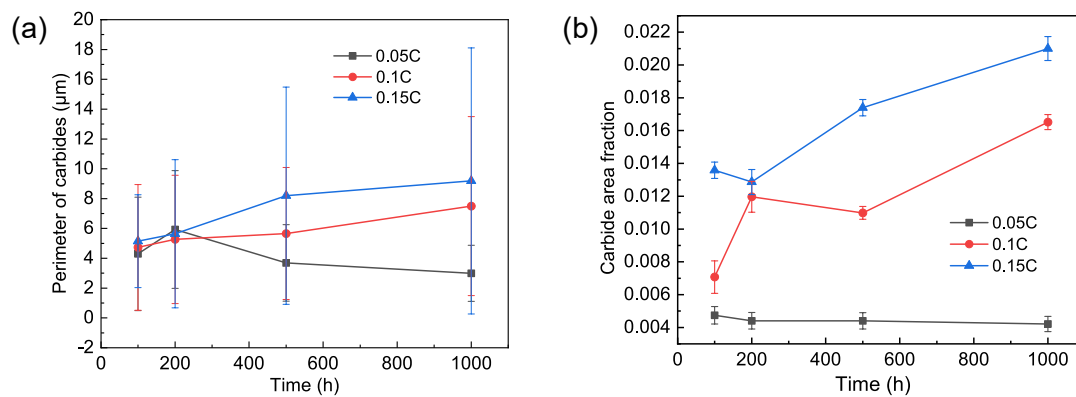


Fig. 6: Relationships between thermal exposure time and size (a) as well as the area fraction of carbides (b)

3.3 Stress rupture properties

Figure 7 compares the stress-rupture life with strain and strain rate responses of ZGH451 alloys with graded carbon contents under 760 °C/780 MPa and 980 °C/260 MPa conditions. Three characteristic behaviors emerge from the analysis: (i) Temperature sensitivity: All alloys exhibit a dramatic reduction in rupture life with increasing temperature, regardless of carbon content. For instance, when the temperature is raised from 760 °C to 980 °C, accompanied by a decrease in stress, the 0.05C alloy exhibits a marked decrease in fracture life from 115.08 h to 34 h, as shown in Figs. 7(a) and (b). (ii) Deformation stage evolution: Following creep mechanisms, the stress-rupture process progresses through three strain rate dependent stages: Primary creep, during which the strain rate decreases; secondary creep, characterized by a relatively constant strain rate; and tertiary creep, in which the strain rate accelerates until rupture occurs. At 760 °C/780 MPa, all alloys progress through these three stages regardless of carbon levels. Strikingly, under conditions of 760 °C/780 MPa, increasing carbon content severely compromises rupture life from 115.08 h (0.05C) to 21.42 h (0.15C), representing an 81.4% performance

degradation. Concurrently, minimum strain rate increases with carbon addition: $1.49 \times 10^{-4} \text{ s}^{-1}$ (0.05C), $6.07 \times 10^{-4} \text{ s}^{-1}$ (0.1C), and $8.43 \times 10^{-4} \text{ s}^{-1}$ (0.15C). (iii) High-temperature anomaly: At 980 °C/260 MPa, the 0.05C and 0.1C alloys retain their three-stage deformation characteristics. Notably, the 0.15C alloy exhibits divergent behavior: Its strain rate oscillates within a narrow range without abrupt transitions, resulting in a time-strain curve that lacks clearly defined primary, secondary, and tertiary stages.

3.4 Stress rupture characteristics

Figure 8 presents the macroscopic fracture morphology of the specimens after stress rupture testing at 760 °C/780 MPa. The fracture surface consists of two distinct zones with contrasting failure features: (i) a smooth region dominated by large cleavage planes, characteristic of brittle fracture; and (ii) a rough area containing ductile dimples, indicative of ductile fracture. This indicates a mixed fracture mode under the test conditions. Detailed analysis reveals that both 0.05C and 0.1C alloys exhibit abundant micropores (red arrows) and carbide particles (yellow arrows), suggesting these features act as

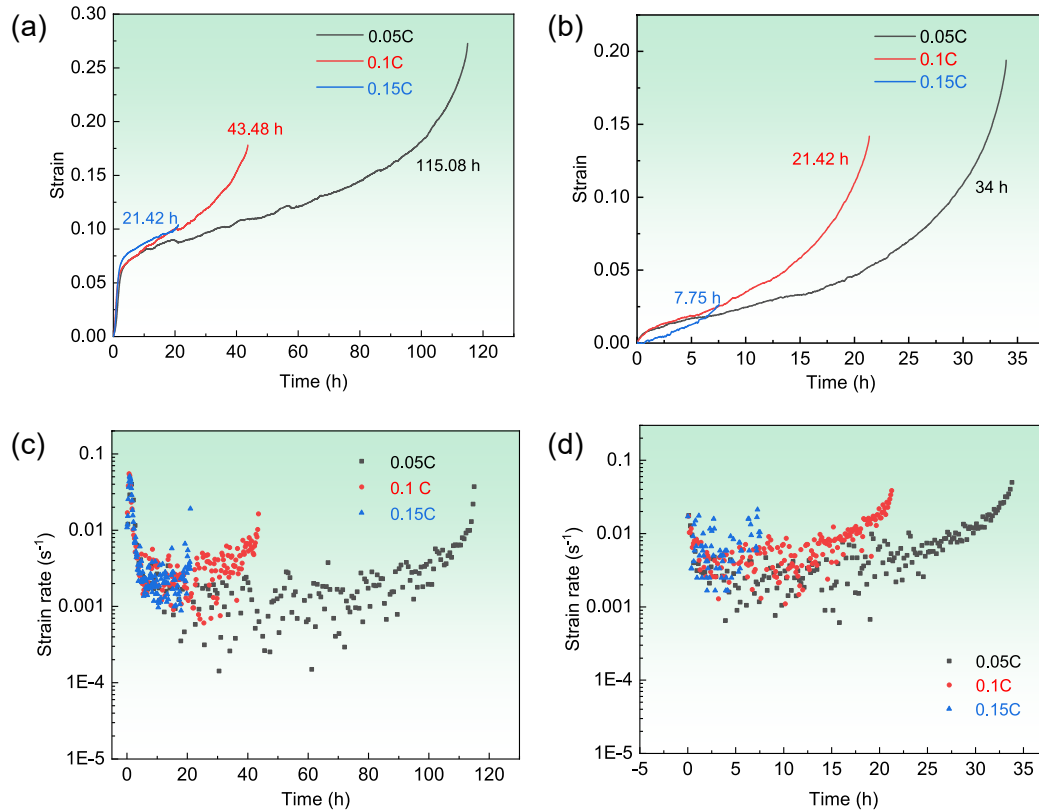


Fig. 7: Stress-rupture of the alloys at 760 °C/780 MPa (a) and 980 °C/260 MPa (b), and stress-rupture strain rate-life curve of alloys at 760 °C/780 MPa (c) and 980 °C/260 MPa (d)

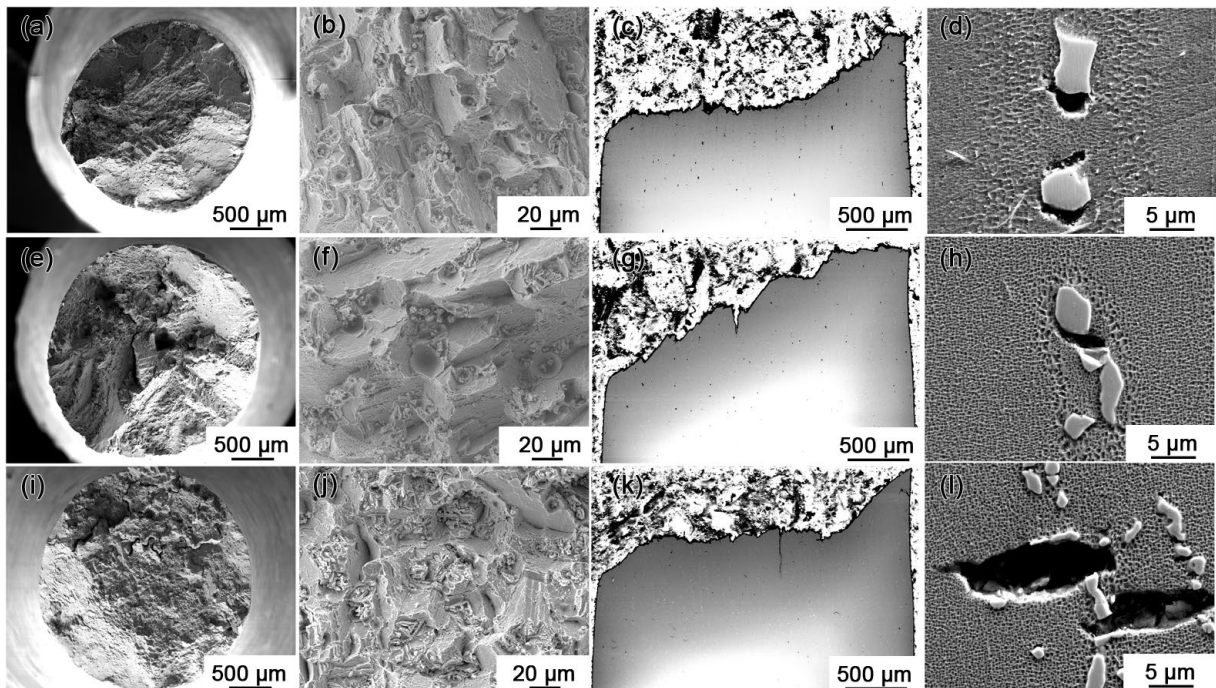


Fig. 8: Fracture morphologies at 760 °C/780 MPa: cross-sections of 0.05C (a, b), 0.1C (e, f), and 0.15C (i, j) alloys; and longitudinal sections of 0.05C (c, d), 0.1C (g, h), and 0.15C (k, l) alloys

primary crack initiation sites. In contrast, the 0.15C alloy displays substantially fewer micropores while maintaining a high carbide density, indicating that carbon content plays a key role in modulating the origin of failure. This observation is further supported by longitudinal section analysis, which identifies two microcrack types: carbide-associated cracks and

non-carbide cracks. Notably, the 0.05C alloy demonstrates transgranular fracture characteristics, whereas both the 0.1C and 0.15C alloys exhibit intergranular cracking (yellow circles) in their longitudinal sections. This transition implies reduced grain boundary strength, which may explain the decreased stress rupture life in alloys containing a higher carbon content.

Figure 9 illustrates the macroscopic fracture morphology of the specimen subjected to rupture testing at 980 °C/260 MPa. The fracture surface exhibits a uniform fracture pattern characterized by rough transgranular facets and numerous ductile dimples, demonstrating typical ductile fracture behavior. This morphology implies a micropore aggregation fracture mechanism, accompanied by significant plastic deformation through necking near the fracture zone. Microstructural analysis of longitudinal sections (980 °C/260 MPa) confirms that the crack initiation mechanisms are consistent with those observed at 760 °C/780 MPa. High-temperature microcracks predominantly nucleate at carbide interfaces (carbide-associated cracks) or form independent carbides.

Notably, carbide particles serve as critical nucleation sites for microcracks under elevated temperature conditions. In the 0.15C alloy, rod-shaped carbides (>5 µm as deposited) coarsen to over 10 µm under thermomechanical loading, Fig. 9(l). Stress concentration at these enlarged carbide interfaces induces the formation of microcracks perpendicular to the loading direction, as shown in Fig. 9(l), with subsequent crack linkage leading to final fracture. Furthermore, rafting morphology of γ' precipitates is observed perpendicular to the applied stress direction during high-temperature exposure. The relationship between carbon content variations and the impact of the rafting microstructure on mechanical performance will be comprehensively analyzed in the subsequent sections.

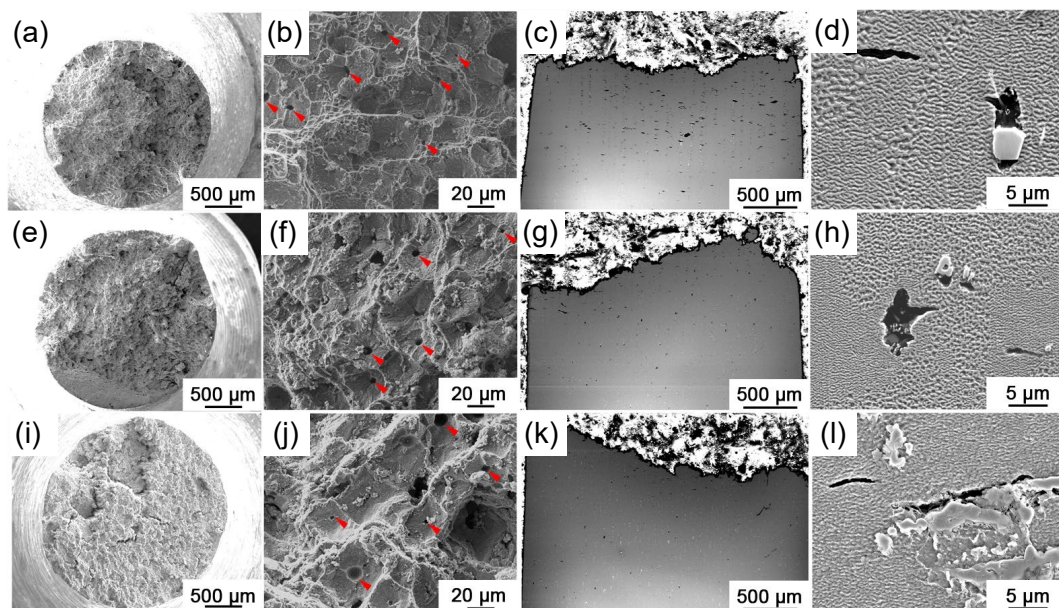


Fig. 9: Fracture morphologies at 980 °C/260 MPa: cross-sections of the 0.05C (a, b), 0.1C (e, f), and 0.15C (i, j) alloys; and longitudinal sections of 0.05C (c, d), 0.1C (g, h), and 0.15C (k, l) alloys

3.5 Deformation mechanism

Figure 10 elucidates the deformation mechanisms of the investigated alloys under various testing conditions. At 760 °C/260 MPa, the stacking fault energy (SFE) exhibits a marked reduction compared to antiphase boundary (APB) energy, promoting extensive stacking faults (SFs) formation within γ' precipitates (indicated by red arrows). In agreement with prior studies^[25], an increase in carbon content enhances the density of SFs across all alloys. The formation of Lomer-Cottrell (L-C) lock structures (highlighted by yellow circles) is observed in all alloys. These locks arise when two sets of SFs glide on different slip planes and interact via leading Shockley partial dislocations, resulting in a complex dislocation network. These L-C locks effectively enhance dislocation obstruction, thereby establishing a deformation mechanism dominated by matrix dislocation slip in conjunction with SF-mediated γ' precipitate shearing.

Under 980 °C/260 MPa conditions, thermal activation induces three critical changes: (i) an elevation in SFE concurrent with a reduction in APB energy; (ii) replacement of SFs by superdislocations (blue arrows) in γ' precipitates; and (iii) the

formation of interfacial dislocation networks at γ/γ' interfaces (red circles), driven by lattice misfit stresses. These networks exhibit lower density than conventional cast superalloys, which can be attributed to the reduced lattice misfit, as evidenced by the intermediate γ' morphology between spherical and cubic configurations (Fig. 1). Superdislocation analysis reveals that short $a\langle 110 \rangle$ configurations are the predominant type within the γ' precipitates. Coarsening of γ' precipitates under thermal exposure widens matrix channels while promoting two concurrent deformation pathways: matrix dislocation slip/climb and superdislocation-mediated γ' precipitate interactions via bypassing and shearing mechanisms.

4 Discussion

4.1 Evolution of carbides

Figure 11 illustrates equilibrium phase diagram calculations predicting the evolution of phase volume fractions across the temperature range of 500–1,500 °C. Notably, in the high-temperature equilibrium regime (1,100–1,300 °C), primary MC carbides in the 0.05C alloy are completely dissolved,

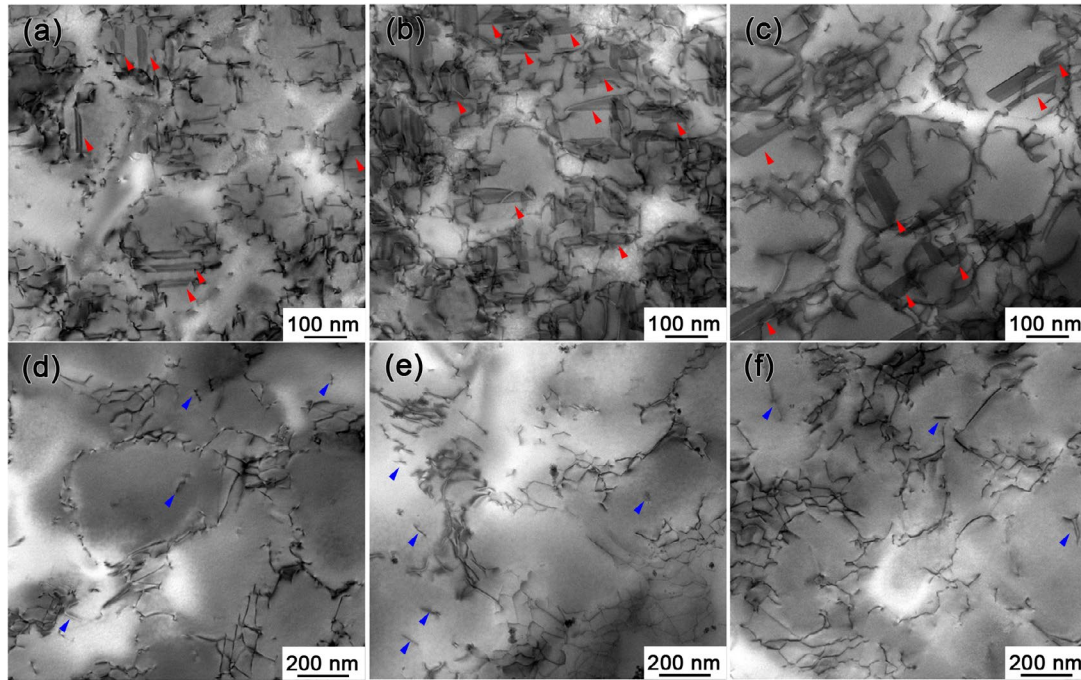


Fig. 10: TEM images of deformed microstructures of the alloys after fracture under different stress rupture conditions: (a–c) 0.05C, 0.1C, and 0.15C alloys under 760 °C/780 MPa, respectively; (d–f) 0.05C, 0.1C, and 0.15C alloys under 980 °C/260 MPa, respectively

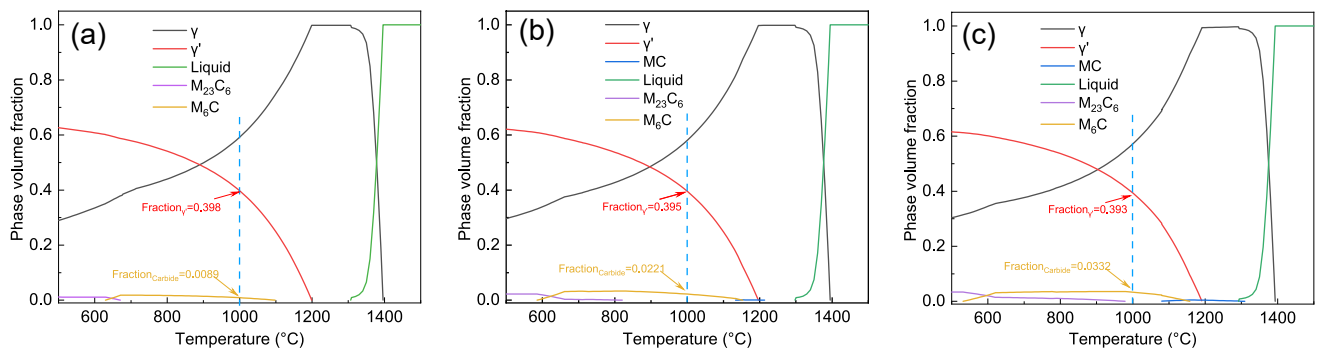


Fig. 11: Calculation results of phase diagram for 0.05C (a), 0.1C (b), and 0.15C (c) alloys

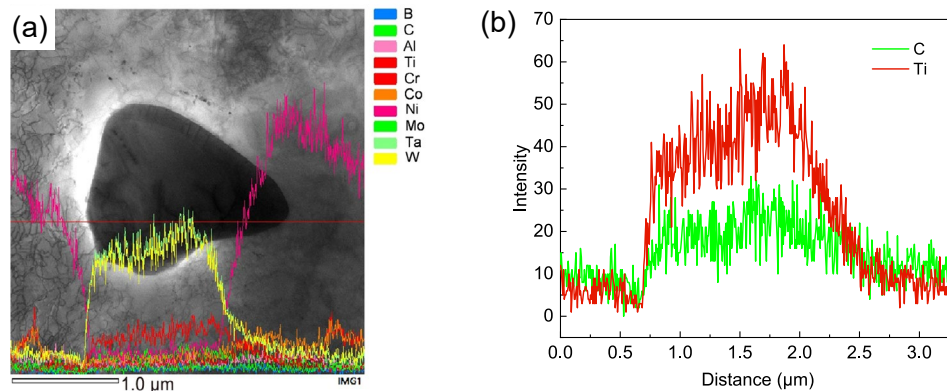
while 0.1C and 0.15C alloys retain residual MC carbides and delay secondary carbide precipitation. At 1,000 °C, all alloys demonstrate a carbon content-dependent increase in carbide volume fraction, as confirmed by experimental measurements. Integrated experimental and thermodynamic analyses reveal a two-stage carbide evolution process: initial coarsening followed by dissolution. After 100 h of thermal exposure, the limited carbon supply in the 0.05C alloy (dominance of Stage 2) results in carbide refinement through dissolution. In contrast, the 0.1C and 0.15C alloys exhibit sustained coarsening via solute capture (W, Mo, Cr) from the matrix during Stage 1, enabled by sufficient carbon availability.

However, determining the source of carbon during the sustained growth of carbides remains a challenge. Although rapid solidification, characteristic of additive manufacturing, has not been reported to significantly enhance the solid solubility of superalloys, extensive research has been conducted on its effects in other alloy systems^[26, 27]. Rapid solidification not only suppresses phase precipitation but also enhances the solid solubility of the alloy. Consequently, it is assumed

that the carbon required for continued carbide growth in 0.1C and 0.15C alloys originates from the matrix following extended solid solubility. Based on this hypothesis, thermodynamic calculations were performed to compare carbon content in the matrix under equilibrium and non-equilibrium conditions. The results demonstrate enhanced carbon dissolution in the matrix under non-equilibrium states, although the absolute values remain relatively low (Table 3). Further investigation through TEM analysis of the long-term thermal exposed 0.15C alloy after 500 h reveals a gradual carbon concentration gradient between carbides and matrix, as evidenced by EDX line scanning (Fig. 12), indicating potential carbon diffusion. Notably, the significantly higher carbon content in carbides implies an insufficient carbon supply solely from the matrix to sustain carbide growth during thermal exposure. Analysis of carbide size distribution (inset in Fig. 5) shows progressive decrease in small carbides (0–4 μm perimeter) and continuous increase in large carbides (≥ 10 μm perimeter) with extended exposure time. This aligns with Ostwald ripening behavior, where large carbides grow through element diffusion from

Table 3: Comparison of matrix element content (at.%) at solid fraction of 0.99 under equilibrium state and non-equilibrium rapid solidification calculated by Scheil model

Elements	0.05C Non-equilibrium/equilibrium	0.1C Non-equilibrium/equilibrium	0.15C Non-equilibrium/equilibrium
Cr	0.1169/0.1980	0.1166/0.1879	0.1462/0.1682
Ti	0.1722/0.0004	0.1737/0.0004	0.0041/0.0003
Mo	0.0216/0.0153	0.0215/0.0134	0.0272/0.0100
Al	0.1034/0.0232	0.0920/0.0205	0.0990/0.0159
Co	0.0692/0.1741	0.0701/0.1816	0.0911/0.1914
C	0.0040/0.00006	0.0063/0.00005	0.0083/0.00003
W	0.0053/0.0378	0.0074/0.0387	0.0098/0.0407
Ta	0.0396/0.0004	0.0400/0.0003	0.0473/0.0002
Ni	0.4666/0.5506	0.4706/0.5571	0.5643/0.5731

**Fig. 12: EDX line scanning results of carbides in 0.15C alloy after 500 h of thermal exposure, showing the scan path (a) and the corresponding magnified elemental profiles of C and Ti (b)**

dissolving small carbides via the matrix. This interfacial energy-driven process is particularly sustainable in high-carbon alloys, as carbides exhibit sufficient elemental diffusion to maintain stability and support continued growth.

In summary, the carbon required for carbide growth mainly comes from small-sized carbides dissolved nearby. Whether the increased solid solution carbon atoms in the matrix under non-equilibrium solidification will affect carbide growth requires further exploration.

4.2 Influence of carbon on diffusion

As mentioned earlier, carbon may have an impact on the diffusion of alloying elements, thereby affecting the coarsening rate of the γ' precipitates. To clarify the effect of carbon on the diffusion capability of elements, the effective diffusion coefficient (D^{eff}) of the three alloys was calculated^[28].

$$D_{\text{eff}} = D_0^{\text{eff}} \exp\left(-\frac{Q_{\text{eff}}}{RT}\right) \quad (1)$$

where D_0^{eff} and Q_{eff} represent the diffusion pre-factor and activation energy, respectively; T is the experimental temperature, and R is the ideal gas constant. The effective diffusion pre-factor D_0^{eff} of the alloy is related to the elemental

composition of the alloy, and their relationship is given by^[29]:

$$D_0^{\text{eff}} = \sum_m x_m D_0^{m, \text{Ni}} \quad (2)$$

where x_m represents the atomic concentration of element m in the γ matrix, and $D_0^{m, \text{Ni}}$ is the diffusion pre-factor of element m in pure nickel (listed in Table 4). The effective activation energy Q_{eff} is the activation energy of pure nickel plus the weighted average of the activation energies contributed by different solute elements, as follows^[29]:

$$Q_{\text{eff}} = Q_{\text{Ni}} + \sum_m x_m Q_{m, \text{Ni}} \quad (3)$$

where Q_{Ni} is the self-diffusion activation energy of nickel, and $Q_{m, \text{Ni}}$ is the activation energy for the mutual diffusion of solute element m in nickel. The atomic percentages of elements in the γ matrix under equilibrium at 1,000 °C were determined through Thermo-Calc simulations (Table 5). Substituting these values into the Eqs. (1–3) reveals that elevated carbon content enhances elemental diffusion, with the effective diffusion coefficients of the three alloy compositions showing sequential increases with carbon concentration. This correlation accounts for the accelerated γ' coarsening rates observed in high-carbon alloys during long-term thermal exposure, as the process is diffusion-controlled^[30].

Table 4: Parameters related to elemental diffusion^[29], and calculated effective diffusion coefficients of the three alloys

Element	D_0 ($\text{m}^2\cdot\text{s}^{-1}$)	$Q_{m, \text{Ni}}$ ($\text{kJ}\cdot\text{mol}^{-1}$)
Ni	1.9×10^{-4}	284
Cr	3×10^{-6}	170.7
Co	7.5×10^{-5}	285.1
W	8.0×10^{-6}	264
Al	7.52×10^{-4}	284
Ta	2.19×10^{-5}	251
Mo	1.15×10^{-4}	281.3
Ti	8.6×10^{-5}	257

Alloy	D_0^{eff} ($\text{m}^2\cdot\text{s}^{-1}$)	Q_{eff} ($\text{kJ}\cdot\text{mol}^{-1}$)	D_{eff} ($\text{m}^2\cdot\text{s}^{-1}$)
0.05C	1.78×10^{-4}	380.04	1.72×10^{-4}
0.1C	1.80×10^{-4}	378.79	1.74×10^{-4}
0.15C	1.81×10^{-4}	377.63	1.75×10^{-4}

Table 5: Elemental contents (at.%) in γ matrix at 1,000 °C equilibrium state calculated by Thermal-Calc

Alloy	Ni	Al	Co	Cr	Mo	W	Ta	Ti
0.05C	0.6004	0.0686	0.1255	0.1434	0.0175	0.0328	0.0062	0.0042
0.1C	0.6049	0.0699	0.1267	0.1423	0.0148	0.0287	0.0063	0.0043
0.15C	0.6089	0.0713	0.1277	0.1407	0.0124	0.0251	0.0065	0.0044

(such as carbides), particularly at grain boundaries. According to the classical pile-up theory, the local stress concentration, local, at the head of the pile-up can be expressed as^[32]:

$$\tau_{\text{local}} = N \times \tau_{\text{applied}} \quad (4)$$

where N represents the number of dislocations in the pile-up and applied is the applied resolved shear stress. In the 0.15C alloy, the presence of L-C locks inhibits the dynamic relaxation of dislocation sources, maintaining a high value of N along extended slip bands^[33]. Consequently, the local stress at the pile-up tip is amplified significantly.

This mechanism is directly supported by the Kernel Average Misorientation (KAM) analysis. Figure 13 presents the EBSD characterization of carbides located at grain boundary

4.3 Influence of carbon on stress fracture

Experimental results demonstrate that increasing the carbon content from 0.05wt.% to 0.15wt.% leads to a precipitous decline in stress rupture life at 760 °C/780 MPa, dropping from 115.08 h to 21.42 h. Concurrently, the fracture mode transitions from predominantly transgranular to significant intergranular fracture (Fig. 8). This shift signifies a fundamental change in the underlying failure mechanism.

As detailed in Section 3.5 (Fig. 10), elevated carbon levels significantly enhance the density of stacking faults (SFs) within the γ' precipitates. The intersection of these faults on varying slip planes promotes the formation of immobile L-C locks. These sessile configurations, combined with the high density of SFs, establish a rigid network of dislocation barriers within the grain interior. While this substructure effectively impedes dislocation motion, resulting in significant work hardening^[31], it concomitantly restricts the deformation to a planar slip mode. Unlike wavy slip, planar slip limits the ability of dislocations to cross-slip and relax local stresses. Under this planar slip regime, dislocation groups pile up against obstacles

cracks in the longitudinal section of the 0.15C alloy tested at 760 °C/780 MPa. The maps reveal that while the global stress level along the grain boundaries remains relatively low, extreme stress concentrations are localized at the interfaces of rod-shaped carbides. This observation provides compelling evidence of substantial dislocation accumulation (high N) at the carbide-matrix interfaces. The resulting local stress exceeds the interfacial bond strength, triggering premature intergranular crack initiation.

Unlike the stress rupture life degradation at 760 °C/780 MPa, which is controlled by the mechanical interaction between high applied stress and deformation substructures, the alloy at 980 °C/260 MPa is primarily affected by diffusion-controlled

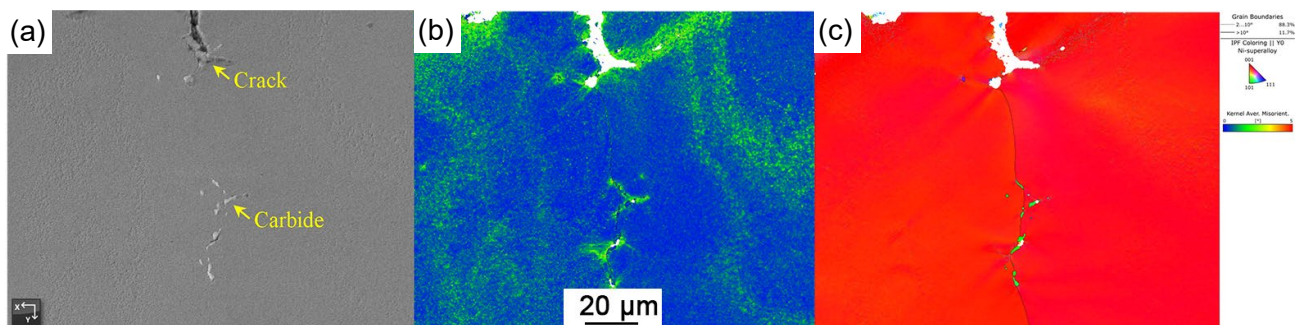


Fig. 13: EBSD analysis of fracture surface of 0.15C alloy under 760 °C/780 MPa: (a) band contrast mapping; (b) KAM mapping; (c) inverse pole figure (IPF) mapping

microstructural degradation. Fracture surface analysis reveals that secondary carbides preferentially precipitate and grow at grain boundaries, which can be attributed to accelerated elemental diffusion and elevated interfacial energy. In the 0.15C alloy, near-complete coverage of grain boundaries by a continuous film-like carbide structure is observed (Fig. 14). TEM characterization further identifies two distinct carbide morphologies: carbides distributed along the grain boundaries and those protruding perpendicularly into the matrix. These perpendicularly oriented carbides act as preferential crack nucleation sites under tensile loading, thereby facilitating grain boundary separation. The high solute concentration and enhanced diffusion kinetics in high-carbon alloys synergistically promote the formation of such continuous carbide networks at grain boundaries, which explains the grain boundary-dominated cracking evident in fracture surface along longitudinal sections.

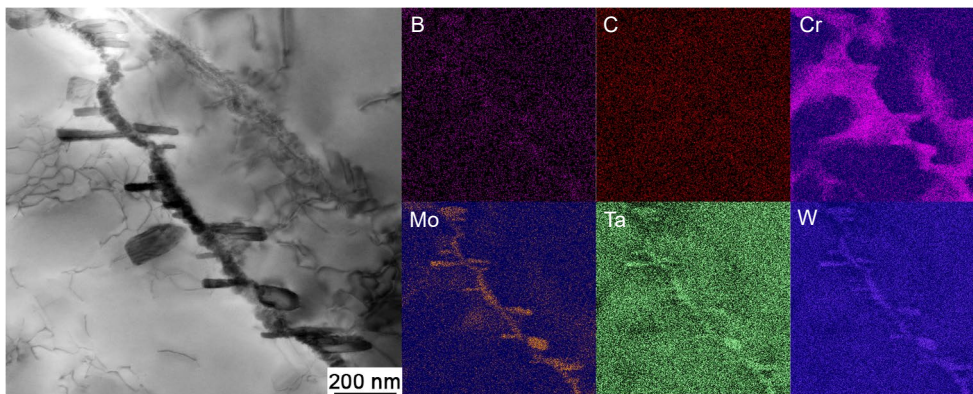


Fig. 14: TEM images of continuous film-like carbides at grain boundaries of 0.15C alloy and the corresponding elemental distributions

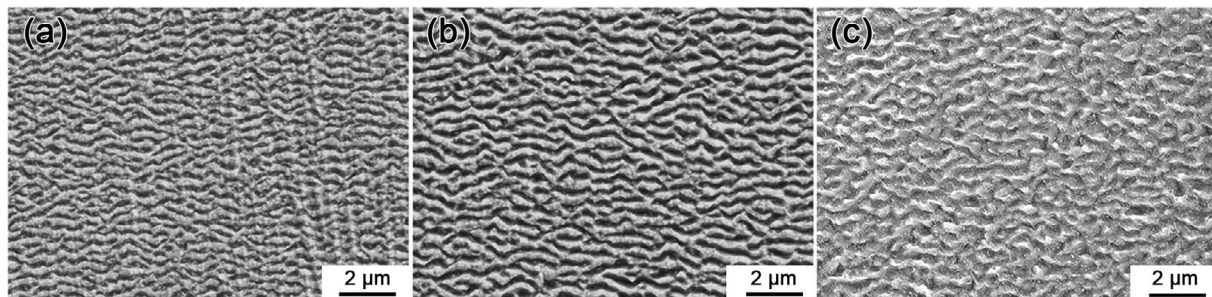


Fig. 15: Rafted structures of 0.05C (a), 0.1C (b), and 0.15C (c) alloys under 980 °C/260 MPa

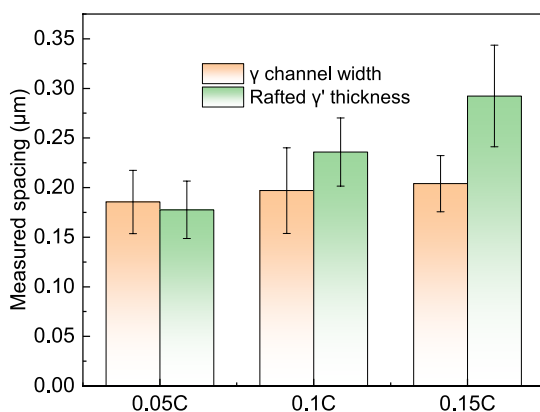


Fig. 16: Statistics of channel width of γ matrix and thickness of raft γ' precipitates

The rafted structure, characterized by directional coarsening of γ' precipitates under stress (referred to as N-type rafting, with the wide faces oriented perpendicular to the stress axis), is illustrated in Fig. 15. Quantitative measurements of γ matrix channel width and rafted γ' precipitate thickness (Fig. 16) demonstrate a progressive increase in γ channel width with higher carbon contents. As reported by Pollock and Argon^[34], dislocation bowing into γ channels requires applied stresses (τ_{or}) that exceed the Orowan resistance within these regions, which can be calculated by^[35]:

$$\tau_{or} = \sqrt{2/3} \mu b / l \quad (5)$$

where μ is the shear modulus, b is the Burgers vector, and l is the channel width of γ matrix. An increase in l due to rafting decreases τ_{or} , facilitating dislocation movement through the matrix channels. This increased dislocation mobility leads to

higher plastic strain in the γ channels and at the γ/γ' interfaces, which negatively impacts the alloy's creep resistance. Studies have shown that wide matrix channels promote crack propagation along the γ channels or γ/γ' interfaces, leading to a significant decrease in strength and accelerated failure^[35, 36].

Increased carbon content further promotes the widening of rafted γ' precipitates, a phenomenon that is likely attributable to carbon-enhanced diffusion kinetics, which accelerate the coarsening of the rafted microstructure^[37, 38]. The resultant γ' coarsening reduces the γ/γ' interface density per unit volume, critical barriers for dislocation climb, as these boundaries must be overcome during plastic deformation^[39]. The diminished presence of these interfaces in heavily rafted regions reduces resistance to dislocation climb, thereby facilitating deformation

and deteriorating stress rupture life^[40]. Although compact raft structures can enhance creep resistance by suppressing dislocation climb (e.g., N-type rafts under low stress^[41]), the observed morphology, characterized by expanded γ' rafts and widened γ channels, enables dislocations to bypass precipitates through mechanisms such as Orowan looping and dislocation climb. Furthermore, while dense dislocation networks initially inhibit γ' shearing in minimally rafted alloys^[42], their protective effect weakens with progressive rafting. This arises from rafting-induced misfit stress reduction at γ/γ' interfaces, rendering dislocation networks ineffective for blocking dislocation motion. Instead, these networks evolve into superdislocation sources that drive interfacial crack propagation. High-carbon alloys exacerbate this degradation by accelerating γ' coarsening and rafting through enhanced diffusion coefficients (D_{eff}), which progressively undermine the effectiveness of the interfacial network and ultimately reduce creep resistance.

In summary, the combined effect of widened γ matrix channels and thickened γ' rafts results in a reduced capacity of the alloy to resist deformation under the applied conditions of 980 °C/260 MPa. This is consistent with the observed reduction in stress rupture life with increasing carbon content.

5 Conclusions

This study systematically investigates the influence of carbon content (0.05wt.%–0.15wt.%) on the microstructural evolution and stress rupture properties of the additively manufactured Ni-based superalloy ZGH451. The main conclusions are summarized as follows:

(1) Carbon enhances the effective diffusion coefficient of the alloy, accelerating the coarsening kinetics of γ' precipitates during long-term thermal exposure at 1,000 °C. The coarsening behavior follows the diffusion-controlled LSW theory.

(2) Carbides act as critical sites for microcrack initiation during stress rupture. While small carbides contribute to grain boundary strengthening at low carbon levels (0.05wt.%), excessive carbon (0.15wt.%) leads to coarse and continuous carbide films along grain boundaries. These films serve as stress concentrators and facilitate intergranular crack propagation, significantly degrading rupture life.

(3) Carbon influences the rafting behavior of γ' precipitates under high-temperature stress. Increased carbon content promotes the thickening of γ' rafts and widening of γ matrix channels. This microstructural evolution reduces the Orowan stress and facilitates dislocation climb and bypass mechanisms, thereby weakening creep resistance.

In summary, while carbon can enhance certain aspects of grain boundary strength, its overall effect on the stress rupture life of this additively manufactured superalloy is detrimental beyond an optimal level (~0.05wt.%), due to accelerated γ' coarsening, carbide-induced cracking, and unfavorable rafting morphology. These insights provide critical guidance for optimizing carbon content in AM superalloys aimed for high-temperature applications.

Acknowledgments

This study was supported by the National Key R&D Program of China (Grant Nos. 2023YFB3712002, 2021YFB3702503), the Project of TaiHang Laboratory (Grant No. A3023), and the National Natural Science Foundation of China (Grant No. 52204383).

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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