Effect of Grain Size on the Stress-Rupture Properties of K417G Superalloy: Postprint

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Abstract

The influence of grain size on the stress-rupture properties of K417G superalloy was investigated under 760°C/645MPa, 900°C/315MPa, and 950°C/235MPa. The results indicate that the effect of grain size on the alloy's stress-rupture properties depends on the experimental conditions. At 760°C/645MPa, the stress-rupture properties improve with grain refinement, with the deformation mode being dominated by intragranular deformation; at 900°C/315MPa, the stress-rupture properties first increase then decrease with grain refinement, with the deformation mode involving competitive interaction between intragranular deformation and grain boundary sliding; at 950°C/235MPa, the stress-rupture properties degrade with grain refinement, with the deformation mode being dominated by grain boundary sliding. TEM observations reveal that at 760°C/645MPa, dislocations shear the phase and no dislocation networks are generated in the matrix channels; at 900°C/315MPa and 950°C/235MPa, dislocations bypass the phase via the Orowan mechanism, dislocation networks form in the matrix channels, and M23C6 precipitates intragranularly.

Full Text

Effects of Grain Refinement on Creep Properties of K417G Superalloy

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Abstract

Grain size is one of the most important parameters affecting the mechanical properties of cast polycrystalline superalloys. To investigate the effect of grain refinement on the creep behavior of K417G superalloy, creep tests were conducted on K417G specimens with four different grain sizes at 760 °C/645 MPa, 900 °C/315 MPa, and 950 °C/235 MPa. The longitudinal section of fracture surfaces, crack propagation paths, dislocation structures, and plastic deformation distribution near cracks were examined using SEM, TEM, and EBSD techniques to determine the deformation mechanisms and grain refinement effects on creep properties under different conditions.

The results showed that grain refinement effects on creep properties varied with temperature and stress. At 760 °C/645 MPa, grain refinement improved creep life and reduced the steady-state deformation rate, with creep deformation dominated by intragranular deformation. At 900 °C/315 MPa, as grain size decreased, creep life first increased then decreased, while the steady-state deformation rate first decreased then increased, showing competitive effects between intragranular deformation and grain boundary sliding. At 950 °C/235 MPa, creep life decreased and steady-state deformation rate increased with decreasing grain size, with grain boundary sliding as the main deformation mode. Grain refinement also refined dendrites and carbides, which slightly affected creep behavior.

TEM observations revealed that at 760 °C/645 MPa, dislocations interacted with particles through a shearing mechanism and no dislocation networks formed in the matrix. In contrast, at 900 °C/315 MPa and 950 °C/235 MPa, dislocations bypassed particles via the Orowan mechanism, dislocation networks formed in the matrix, and M23C6 precipitated within grains with a [001]M23C6//[001]Matrix orientation relationship.

Keywords: superalloy, grain size, creep

1. Introduction

K417G alloy is a -precipitation-strengthened cast nickel-based superalloy with low density, excellent high-temperature strength, and good creep resistance, widely used for hot-section components operating in medium-low temperature environments [1~3]. Under these conditions, the mechanical properties of the alloy are particularly sensitive to grain size. Superalloy castings produced by conventional investment casting generally have coarse grains, which is a key factor causing performance degradation and reduced application levels. Grain refinement can improve mechanical properties [4~10] and is an effective means to extend the service life and delay failure of polycrystalline castings.

Meanwhile, grain refinement also refines precipitates and carbides in the alloy [8], which is beneficial for strength improvement. However, grain refinement does not enhance material strength under all conditions. Studies [7,10,11] have shown that as the service temperature increases, the strengthening effect of grain refinement gradually diminishes, and can even lead to lower strength with finer grains. This occurs because both grain boundary and intragranular strengths decrease with increasing temperature, but grain boundary strength decreases more rapidly. At a certain temperature, grain boundary strength equals intragranular strength; this temperature is called the equicohesive temperature. When the service temperature exceeds this point, intragranular strength becomes greater than grain boundary strength, and grain refinement can degrade alloy performance. Numerous studies [7,11~14] have found that under high-temperature creep conditions, grain boundary sliding occurs, causing intergranular fracture. Under such conditions, grain refinement reduces alloy performance. Therefore, the effect of grain refinement on mechanical properties is closely related to test conditions, and the influence of grain size on superalloy properties has remained a research focus.

Researchers [5] calculated that grain size affects the magnitude of local irreversible plastic deformation in slip bands, thereby influencing crack initiation. In studies of nickel' s room-temperature fatigue performance [15], coarse grains promoted slip band formation, inducing fatigue cracks and resulting in longer fatigue life for fine-grained alloys. Fatigue cracks were more likely to initiate at large grains [4], and grain refinement reduced the probability of fatigue crack initiation. Research on fatigue crack propagation [16] found that crack growth rates decreased when approaching grain boundaries, proving that grain refinement reduces fatigue crack propagation rates. However, studies on grain size effects on creep performance showed that IN792 alloy' s creep properties at 650~760 °C improved with increasing grain size [17]. As grain size increased, U720LI alloy' s yield strength at 20, 600, and 760 °C decreased, while creep resistance at 760 °C/370 MPa increased and low-cycle fatigue strength at 600 °C slightly decreased [7]. Recent research on nickel-based superalloy NR6 with different grain sizes at 700 °C/700 MPa [13] found that coarse grains increased intragranular creep deformation inhomogeneity but reduced grain boundary creep deformation inhomogeneity and caused grain boundary sliding lag.

Many studies have also examined grain refinement effects on cast superalloy properties. Researchers [8] analyzed the relationship between grain refinement and room-temperature tensile strength of IN713-LC cast superalloy, finding that yield strength gradually increased with grain refinement, but tensile strength decreased when grains were refined to 25 m due to higher porosity content. Studies on K4169 alloy grain-refined with additives found that fine-grained structures significantly improved room-temperature and intermediate-temperature tensile properties, particularly increasing rupture life at 760 °C/480 MPa [9]. Quested and Osgerby [18] investigated grain refinement effects on IN939 cast superalloy creep properties, showing that minimum creep rate decreased with grain refinement at high stresses but increased at low stresses. Although many studies have addressed grain size relationships with superalloy properties, research on grain refinement effects on cast superalloy creep properties and deformation mechanisms remains incomplete. Therefore, this work studied the creep properties of K417G superalloy with four grain sizes under three different conditions, with detailed analysis of creep deformation mechanisms to understand grain refinement effects and provide guidance for engineering applications.

2. Experimental Procedures

The chemical composition of K417G alloy is (mass fraction, %): C 0.13~0.22, Cr 8.50~9.50, Al 4.80~5.70, Ti 4.10~4.70, Mo 2.50~3.5, Co 9.00~11.00, V 0.60~0.90, B 0.012~0.024, Zr 0.05~0.09, S 0.010, P 0.015, Ni balance. Four K417G alloys with different grain sizes were cast in a 10 kg vacuum induction furnace using different pouring temperatures. The alloys were not heat-treated. One creep specimen was taken from each grain size condition. Cylindrical specimens were prepared from the gauge sections of test bars, then ground, polished, and chemically etched with a solution of 20 g CuSO + 100 ml HCl + 100 ml H O. Grain size and microstructure were analyzed using an Axio Observer ZIm optical microscope (OM) and INSPECT F50 scanning electron microscope (SEM). An Oxford EDS detector attached to the SEM was used for compositional analysis of microstructures. Creep tests were performed using a CSS3905 creep testing machine. For each grain size, three specimens were tested in parallel, and results were averaged. SEM was used to observe longitudinal sections of creep fracture surfaces to analyze fracture mechanisms. Electron backscatter diffraction (EBSD) mapping was employed to examine plastic deformation distribution near fracture surfaces on longitudinal sections. Thin foil samples were cut approximately 5 mm from the fracture surface, ground, and twin-jet electropolished using a solution of 10% HClO + 90% C H OH for TEM observation. A JEOL2100 transmission electron microscope (TEM) was used to observe dislocation configurations and microstructural changes after creep fracture.

2.1 Grain Size and Microstructure Analysis

Specimens were labeled A, B, C, and D in order of decreasing grain size. The average grain sizes at the cross-section of specimen gauge sections were measured, with results shown in Table 1 . Grain size analysis revealed that specimen A had coarse grains, with many exceeding 3 mm. Specimen B grain sizes were mainly distributed between 0.7 and 3 mm. Specimen C grain sizes were mostly between 100 and 500 m. Specimen D grain sizes were predominantly in the 0-100 m range.

Microstructures of the different grain size specimens are shown in Figure 1 [Figure 1: see original paper]. All specimens exhibited dendritic morphologies within grains. As shown in Figures 1e~h, the intragranular dendritic structure gradually refined with decreasing grain size, and dendritic features became indistinct in the finest-grained specimen D. Carbides were dispersed as blocky and strip-shaped particles in interdendritic and grain boundary regions. Compositional analysis indicated that carbides primarily contained Ti, Mo, Cr, Co, and V. The total carbide area fraction in all four grain size specimens was approximately 1.7%~2.0%. Carbide average sizes were statistically measured for the four specimens, with results listed in Table 1. As shown in Figures 1i~l, carbide size decreased with grain size refinement. Eutectic structures were distributed as sunflower-shaped in interdendritic regions or chain-like at grain boundaries, with size also decreasing as grain size refined. The phase in dendrite cores was cubic-shaped, while interdendritic phase was irregular. As shown in Figures 1m~p, phase size gradually decreased with grain size refinement, and average phase sizes in the four specimens were statistically measured, with results listed in Table 1.

2.2 Creep Properties

The creep lives of specimens A~D under the three conditions of 760 °C/645 MPa, 900 °C/315 MPa, and 950 °C/235 MPa are shown in Table 2 .

At 760 °C/645 MPa, the creep life of K417G alloy gradually increased from 60.7 h to 105.4 h as grain size decreased. The finest-grained specimen D exhibited the best creep performance. At 900 °C/315 MPa, when average grain size decreased from 2.5 mm to 1.7 mm, creep life increased from 126.4 h to 166.2 h. However, when grain size further decreased from 1.7 mm to 0.075 mm, creep life gradually decreased from 166.2 h to 105.5 h. Under this condition, creep performance showed an initial increase then decrease trend with grain size refinement, with specimen B exhibiting optimal creep performance. At 950 °C/235 MPa, K417G alloy creep life decreased from 90.2 h to 54.7 h as grain size decreased from 2.5 mm to 0.075 mm. The coarsest-grained specimen A showed the best creep performance, while specimen D had the lowest creep life.

Figure 2 [Figure 2: see original paper] shows the creep deformation curves for specimens A~D under the three test conditions. The proportion of stage II in the creep deformation curves gradually decreased as test conditions changed from 760 °C/645 MPa to 900 °C/315 MPa and 950 °C/235 MPa.

The stage II creep deformation rate was approximately constant, denoted as K to represent the steady-state creep deformation rate. K values for specimens A~D under the three conditions were calculated, with results shown in Table 3 . At 760 °C/645 MPa, K values gradually decreased with grain size refinement, though specimen D showed an anomaly. The small differences in K values among the four grain size specimens indicated similar steady-state creep deformation rates. Additionally, the stage II portions of creep curves for specimens A~D were nearly parallel. At 900 °C/315 MPa, K values decreased as grain size decreased from 2.5 mm to 1.7 mm, then increased as grain size further decreased from

1.7 mm to 0.075 mm, with the specimen having 1.7 mm grain size showing the maximum K value. At 950 °C/235 MPa, K values gradually increased as grain size decreased from 2.5 mm to 0.075 mm, with the specimen having 0.075 mm grain size showing the maximum K value.

2.3 Longitudinal Fracture Surface Morphology

Longitudinal sections of creep fracture surfaces under the three conditions were examined, with results shown in Figure 3 [Figure 3: see original paper]. At 760 °C/645 MPa, phase in some regions near the fracture surface was elongated along the stress direction, while in other regions it retained its original morphology. Numerous fractured or debonded carbides were observed near the fracture surface, as shown in Figure 3a. Cracks propagated in a transgranular mode, as shown in Figure 3c, indicating that cracks formed through microcrack generation from carbide fracture or debonding, which then interconnected.

At 950 °C/235 MPa, cracks propagated along grain boundaries, with extensive grain boundary cracking observed near the fracture surface on longitudinal sections. phase rafting was more severe near the fracture surface, with rafting direction perpendicular to the stress direction, as shown in Figures 3e and f. No intragranular carbide fracture or debonding was observed. Creep fracture under this condition resulted from microcracks generated by carbide fracture or debonding at grain boundaries, which then interconnected along grain boundaries.

At 900 °C/315 MPa, cracks propagated in a mixed transgranular and intergranular mode. As shown in Figures 3b and d, both transgranular and intergranular cracks were present on specimen longitudinal sections. Specimens A and B with coarser grains showed more transgranular crack propagation. In contrast, specimens C and D with finer grains exhibited more secondary cracks propagating internally along grain boundaries, indicating dominant intergranular crack propagation. Slight phase rafting was observed near fracture surfaces, with phases interconnecting, as shown in Figures 3b and d. Some carbide fracture and debonding still occurred. In specimens A and B with coarser grains, grain boundary cracks were fewer than intragranular cracks, making transgranular fracture dominant. In specimens C and D with finer grains, grain boundary cracks were more numerous, making intergranular fracture dominant.

Under all three test conditions, no significant eutectic fracture was observed near fracture surfaces, indicating that eutectic structures had negligible influence on creep fracture.

2.4 EBSD Analysis of Longitudinal Fracture Sections

EBSD can investigate the interaction between microstructure and plastic deformation in materials, assessing changes in polycrystalline superalloys after plastic deformation [19~22]. Plastic deformation causes extensive dislocation motion within grains, generating orientation differences between different regions of the same grain. Since plastic deformation distribution is non-uniform, regions with larger plastic deformation exhibit larger local misorientation [21]. Therefore, local misorientation maps in EBSD images can reveal plastic deformation distribution. By correlating with Euler and grain boundary maps, the distribution of plastic deformation at grain boundaries and within grains in specimen D under different creep conditions can be visually demonstrated. EBSD phase maps near fracture surfaces of specimen D after creep tests at 760 °C/645 MPa, 900 °C/315 MPa, and 950 °C/235 MPa are shown in Figure 4 [Figure 4: see original paper].

At 760 °C/645 MPa, grain boundary maps showed white lines within grains (Figure 4b), indicating small-angle tilting caused by plastic deformation. Figure 4c shows that locations with large local misorientation were within grains, corresponding exactly to white line positions in grain boundary maps, demonstrating that plastic deformation near the fracture surface in specimen D concentrated in slip bands within grains, with limited deformation at grain boundaries.

At 900 °C/315 MPa and 950 °C/235 MPa, Figures 4e and h show numerous white lines distributed at grain boundaries, indicating small-angle tilting caused by plastic deformation near grain boundaries. Figures 4f and i show that locations with large local misorientation were at grain boundaries, indicating that plastic deformation in specimen D under these two conditions concentrated primarily at grain boundaries.

2.5 Dislocation Structure Observation

Figure 5 [Figure 5: see original paper] shows TEM images of dislocation configurations and microstructures after different test conditions. After creep testing at 760 °C/645 MPa, specimen D exhibited numerous stacking faults within phase (Figure 5d), indicating that dislocations sheared through phase via partial dislocation shearing mechanism. In contrast, no stacking faults were observed in specimens tested at 900 °C/315 MPa and 950 °C/235 MPa; dislocations primarily bypassed phase via the Orowan mechanism, as shown in Figure 5c.

After creep testing at 760 °C/645 MPa, dislocation density in matrix channels was relatively low, with most dislocations having similar morphology and parallel distribution (Figure 5d). However, after creep testing at 900 °C/315 MPa and 950 °C/235 MPa, dislocation density in matrix channels was higher and formed dislocation networks (Figures 5e and f). The dislocation network morphology was more regular in the specimen tested at 950 °C/235 MPa (Figure 5f).

As shown in Figure 5a, granular precipitates approximately 300 nm in size were observed in specimens after creep testing at 900 °C/315 MPa and 950 °C/235 MPa. EDS analysis indicated these precipitates contained substantial Cr and small amounts of Mo, Ni, and Co. Selected area diffraction results (Figure 5b) revealed an orientation relationship of [001]M23C6//[001]Matrix between the precipitates and matrix. Spot indexing identified these as M23C6 carbides with a lattice constant of approximately 1.058 nm. During loading, phase rafting expelled Cr and Mo elements into matrix channels, creating enriched regions of these elements. Additionally, high-density dislocation networks formed at / interfaces during creep, attracting C atoms to form C-enriched regions near dislocation networks. The enrichment of Cr, Mo, and C atoms during creep promoted M23C6 carbide formation and growth [23~25].

3. Analysis and Discussion

Polycrystalline alloy creep deformation includes intragranular deformation and grain boundary sliding [11,13], as schematically shown in Figure 6 [Figure 6: see original paper]. At 760 °C/645 MPa, fracture surface observations revealed transgranular crack propagation (Figure 3c), with plastic deformation concentrated in slip bands within grains (Figures 4a~c), indicating that grain boundary strength was higher than intragranular strength. Under these conditions, creep deformation was dominated by intragranular slip, i.e., dislocation motion concentrated in slip bands within grains. Finer grain sizes result in shorter slip band lengths, smaller local stress concentrations, more uniform intragranular deformation, and easier deformation coordination between grains, thereby reducing stress concentration and crack initiation probability [13]. Therefore, grain refinement improved creep life and reduced steady-state deformation rate K at 760 °C/645 MPa. However, due to the relatively low deformation temperature, steady-state deformation rates were small, leading to anomalies such as specimen D showing higher K values than specimen C.

At 950 °C/235 MPa, fracture surface observations showed intergranular crack propagation (Figure 3f), and EBSD mapping of specimen D revealed plastic deformation concentrated at grain boundaries (Figures 4g~i), indicating that creep deformation was controlled by grain boundary sliding. Grain refinement increases grain boundary content, promoting grain boundary sliding and crack formation. Additionally, Kevin et al. [13] found that coarse-grained superalloys exhibited more uniform plastic deformation distribution at grain boundaries, resulting in smaller strain concentration and reduced crack susceptibility. Therefore, at 950 °C/235 MPa, finer grain sizes led to shorter creep life and larger steady-state deformation rate K values.

At 900 °C/315 MPa, fracture surface observations showed mixed transgranular and intergranular crack propagation (Figures 3b and d), with creep deformation resulting from combined intragranular deformation and grain boundary sliding. For intragranular deformation, TEM observations showed dislocations bypassing phase via the Orowan mechanism. According to the formula:

$$\Delta\tau = \frac{Gb}{L} \quad (1)$$

where Δ is the resistance for dislocations bypassing phase via the Orowan mechanism, G is the alloy shear modulus, b is the magnitude of the Burgers vector, and L is the spacing between particles. Grain refinement refines phase and reduces L, increasing Δ and thus the resistance to dislocation bypassing via the Orowan mechanism [26]. For grain boundary sliding, grain refinement increases grain boundary content, promoting grain boundary sliding and crack formation, thereby reducing creep resistance [13]. Therefore, at 900 °C/315 MPa, grain size effects on creep life resulted from competition between intragranular deformation and grain boundary sliding mechanisms. Specimens A and B with coarser grains had less grain boundary content, making intragranular deformation dominant and thus creep life increased with grain size reduction. As grain size decreased from B to D, grain boundary content increased, making grain boundary sliding dominant and thus creep life decreased with further grain size reduction. Consequently, specimen B exhibited maximum creep life at 900 °C/315 MPa.

For intragranular deformation, many studies [27~30] have found that steady-state creep deformation rate is related to interfacial dislocation network formation. Interfacial dislocation networks form gradually as dislocations deposit at / interfaces during motion in matrix channels [26,27]. With increasing strain, dislocation density in matrix channels increases, and elastic stress field interactions between dislocations make it increasingly difficult for additional dislocations to enter matrix channels, causing creep deformation rate to gradually decrease to a minimum value, marking the transition to steady-state creep [26], while interfacial dislocation networks simultaneously form. After creep testing at 900 °C/315 MPa, dislocation configuration observations revealed dislocation networks at / interfaces (Figure 6c). Grain size reduction refined phase, narrowing matrix channels between particles and reducing the width of accommodatable dislocation networks. Therefore, as strain increased, dislocation entry into matrix channels became more difficult, reducing stage II deformation rate. For grain boundary sliding, grain size reduction increased grain boundary content, accelerating creep deformation rate. At 900 °C/315 MPa, specimens A and B still had relatively large grain sizes, making intragranular deformation dominant. Therefore, as grain size decreased from A to B, stage II creep deformation rate decreased and K values decreased. When grain size decreased from B to D, grain boundary sliding became dominant, causing stage II creep deformation rate to increase and K values to increase with further grain size reduction.

Additionally, grain refinement simultaneously refined dendrites, carbides, and eutectic structures. Under creep conditions, microcracks generated by carbide fracture or debonding led to creep crack formation. As grain size refined, carbide size gradually decreased, reducing microcrack generation capability and thus inhibiting creep crack formation. However, the carbide size reduction caused by grain refinement was not significant, so its effect on creep life was not obvious. Eutectic structure effects on creep life were negligible. For specimens A~D under 760 °C/645 MPa and specimens A and B under 900 °C/315 MPa, transgranular fracture was the dominant fracture mode. In transgranular fracture, cracks propagated along dendrites, and dendrite refinement with grain size reduction also hindered transgranular crack propagation.

4. Conclusions

(1) At 760 °C/645 MPa, grain refinement improved K417G alloy creep life; at 900 °C/315 MPa, grain refinement caused creep life to first increase then decrease; at 950 °C/235 MPa, grain refinement reduced creep life.

(2) At 760 °C/645 MPa, grain refinement reduced steady-state creep deformation rate; at 900 °C/315 MPa, grain refinement caused steady-state deformation rate to first decrease then increase; at 950 °C/235 MPa, grain refinement increased steady-state deformation rate.

(3) At 760 °C/645 MPa, creep deformation was dominated by intragranular deformation; at 900 °C/315 MPa, creep deformation resulted from competition between intragranular deformation and grain boundary sliding; at 950 °C/235 MPa, creep deformation was dominated by grain boundary sliding.

(4) Grain refinement simultaneously refined carbides, dendrites, and other microstructural features, which also affected alloy creep properties to some extent.

(5) After creep deformation at 900 °C/315 MPa and 950 °C/235 MPa, fine M23C6 carbides precipitated with a [001]M23C6//[001]Matrix orientation relationship with the matrix.

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