High-Temperature Creep Deformation Mechanism of BSTMUF601 Alloy Postprint

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Abstract

Tensile creep experiments were conducted on BSTMUF601 alloy under various temperature and stress conditions to investigate its high-temperature creep deformation behavior. Based on the experimental results, a novel modified q-mapping creep constitutive model was proposed, which accounts for the characteristics of all three creep stages. The model predictions show good agreement with experimental data, with an average relative error of 1.86%. Compared with the -mapping model that neglects the second stage and the modified q-mapping model that neglects the first stage, the relative errors are reduced by 0.10% and 6.02%, respectively, demonstrating that the model possesses strong applicability without compromising predictive accuracy. Microstructural analysis of the dislocation configurations and void evolution in creep and creep fracture specimens revealed that the creep stress exponent in the steady-state creep stage is approximately 5. The alloy deforms primarily via dislocation climb over the γ phase, with no stacking faults or microtwins observed in either the γ phase or the matrix, indicating that dislocation climb is the dominant creep deformation mechanism. Voids nucleate on grain boundaries, grow, and coalesce to form cracks. Under stress concentration, these cracks propagate along grain boundaries, ultimately leading to fracture, with grain boundary fracture being the primary creep fracture mechanism.

Full Text

High Temperature Creep Deformation Mechanism of BSTMUF601 Superalloy

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ABSTRACT

Muffle tubes are core components in large bright annealing muffle furnaces. After long-term service under high temperature, self-weight, and non-uniform temperature conditions, numerous defects develop in muffle tubes, among which creep deformation is particularly severe, directly affecting their service performance and lifespan. High-temperature creep and rupture properties are critical indicators for muffle tube materials, and BSTMUF601 nickel-based superalloy is commonly used for this application. Nickel-based superalloys have attracted significant attention due to their good oxidation resistance, high strength, and excellent creep resistance at elevated temperatures, particularly regarding their creep mechanisms. However, conclusions about creep mechanisms vary for different alloys or even for the same alloy under different conditions, necessitating individual studies for each alloy.

Creep tests of BSTMUF601 superalloy were conducted under various temperatures and stresses to investigate its high-temperature creep deformation characteristics. Based on the experimental results, a new modified projection creep constitutive model was proposed that considers all three stages of creep. The model predictions show good agreement with experimental data, with an average relative error of only 1.86%. Compared with the projection model that ignores the second stage and the modified projection model that ignores the first stage, the average relative error is reduced by 0.10% and 6.02%, respectively, demonstrating that the new model offers strong applicability without compromising prediction accuracy.

Microstructural analysis of dislocation configurations in creep specimens and void evolution in ruptured specimens reveals that the creep stress exponent during the steady-state creep stage is close to 5 under all test conditions. The alloy primarily deforms through dislocation climb over the γ' phase, with no stacking faults or microtwins observed in either the γ' phase or the matrix, confirming that dislocation climb is the dominant creep deformation mechanism. Voids nucleate at grain boundaries, grow, and coalesce to form cracks. Under stress concentration, these cracks propagate along grain boundaries, ultimately leading to fracture, indicating that grain boundary fracture is the primary creep rupture mechanism.

KEY WORDS BSTMUF601 alloy, creep deformation, steady-state creep rate, creep rupture

Introduction

Muffle tubes are the core components in large bright annealing muffle furnaces. Under long-term exposure to high temperatures (approximately 1050 °C), self-weight, and non-uniform temperature distributions, these tubes experience severe creep deformation that generates various defects, directly impacting their service performance and lifespan [1]. Consequently, high-temperature creep resistance and rupture properties are important indicators for evaluating muffle tube materials. BSTMUF601 nickel-based superalloy is a commonly used material for muffle tubes, and with the development of large bright annealing furnace technology, increasingly higher demands are being placed on the high-temperature performance of these materials. Nickel-based superalloys have garnered significant attention due to their excellent oxidation resistance, high strength, and good creep properties at elevated temperatures [2-4], particularly regarding their creep mechanisms which have attracted widespread interest from researchers both domestically and internationally [5-8].

Numerous studies have investigated the creep deformation mechanisms of nickel-based alloys. Xu et al. [9] studied the high-temperature creep behavior of a novel Ni-Co based superalloy and reported that at lower temperatures, the alloy deformed through dislocation slip cutting the γ' phase to form stacking faults; at higher temperatures, the creep deformation microstructure primarily consisted of stacking faults and microtwins penetrating the γ' phase; and at very high temperatures, the alloy deformed through dislocation bypassing the γ' phase. Liu et al. [10] observed in a nickel-based single crystal superalloy that at 760 °C and 780 MPa, dislocations cut the γ' phase as stacking faults during low strain stages, while during high strain stages, dislocations cut the γ' phase as dislocation pairs. At 982 °C and 248 MPa, matrix a/2 ⟨110⟩ dislocations (where a is the lattice constant) bowed out in the matrix and bypassed the γ' phase, forming dislocation networks through dislocation reactions. Yuan et al. [11] observed in U720Li alloy that under creep conditions of 725 °C and 630 MPa, at 0.1% strain, dislocations bypassed the γ' phase via the Orowan mechanism; at 5% strain, partial dislocations cut the γ' phase, forming stacking faults and microtwins; and at 27% strain, grain boundary sliding occurred due to increased stress and strain. Viswanathan et al. [12] observed in René 88 DT nickel-based alloy that at a creep temperature of 650 °C, microtwins formed by partial dislocations were the dominant deformation mechanism at low applied stresses, while 1/2 ⟨110⟩ dislocations cutting the matrix and bypassing the γ' phase via the Orowan mechanism dominated at high stresses. Since different researchers have reached different conclusions for different alloys or even for the same alloy under different conditions, individual studies of each alloy' s creep mechanism are necessary. As nickel-based superalloy components operate under high temperature and sustained loading for extended periods, undergoing creep deformation during service, predicting their creep performance becomes particularly important. Kim et al. [13] proposed that the creep process consists primarily of decelerating and accelerating creep stages, and employed the projection method to

predict the creep behavior of Hastelloy-X alloy. Liu et al. [14] used a modified projection method to predict the creep properties of Cr25Ni35Nb furnace tube steel, with their model considering only the second and third creep stages. Although these two models can reflect deformation behavior under specific conditions, actual creep processes often exhibit three distinct stages. Therefore, for broader applicability and practicality, an improved model must comprehensively consider the deformation characteristics of all three creep stages.

Referencing actual service conditions of muffle tubes and employing accelerated creep testing methods, this work conducted high-temperature tensile creep tests on BSTMUF601 alloy at various temperatures and stresses to obtain creep curves under different conditions. A constitutive model incorporating all three creep stages was established. Based on the creep curves, steady-state creep rates were determined to calculate the creep stress exponent. Microstructural analysis of dislocation configurations and void evolution in crept and ruptured specimens was performed to elucidate the high-temperature creep deformation and fracture mechanisms of this alloy, providing a theoretical basis for life prediction and creep deformation simulation of muffle tubes.

Experimental

The BSTMUF601 alloy used for muffle tubes is a Ni-Cr-Al series nickel-based superalloy with a chemical composition (mass fraction, %) of: Ni 58.0-63.0, Cr 21.0-25.0, Al 1.0-1.7, Mn 0.5, Cu 1.0, and balance Fe. The experimental material underwent vacuum induction melting, forging, multi-pass hot rolling, and solution heat treatment at 1080 °C. Intermediate temperature aging after solution treatment produced regular cubic strengthening phases that enhance creep performance. After heat treatment, plates were machined along the rolling direction into cylindrical creep specimens with a diameter of 10 mm and gauge length of 50 mm.

The actual service temperature of muffle tube materials is approximately 1050 °C, with only self-weight loading during normal operation, resulting in a maximum stress of 1.48 MPa. To investigate creep deformation behavior under various temperature and stress conditions, accelerated creep testing methods were employed. High-temperature tensile creep tests were conducted on an RJ-30 creep testing machine with temperature control within $±$3 °C, load fluctuation within $±$5 N, and deformation measurement resolution of 0.1 m. The test matrix included: at 870 °C, applied stresses of 20, 32, and 40 MPa; at 980 °C, applied stresses of 12.4, 14.4, and 16.4 MPa; and at 1095 °C, applied stresses of 5.7, 6.7, and 7.7 MPa. Most tests were stopped upon reaching the steady-state or acceleration stage, while some creep tests were continued to fracture under constant load before cooling.

To observe the microstructure during the steady-state creep stage, 5 mm long samples were sectioned longitudinally from crept specimens, then ground, polished, and etched for observation using an MR5000 inverted metallurgical micro-

scope (OM). The etching solution consisted of 1.5 g $CuSO_4$ + 20 mL C_2H_5OH + 40 mL HCl. To examine dislocation configurations after creep deformation, 0.5 mm thick slices were cut longitudinally from crept specimens, ground to 0.15 mm thickness, punched into 3 mm diameter discs, and further ground to 60 m thickness. These discs were then thinned using a TenuPol-5 twin-jet electropolishing unit with a 10% perchloric acid ethanol solution (mass fraction). The prepared thin foil samples were examined using a Tecnai F30 transmission electron microscope (TEM). To observe the microstructure after creep rupture, two 10 mm long samples were sectioned longitudinally from a pair of ruptured creep specimens. One sample was split longitudinally, ground, polished, and electrolytically etched for examination using a LEO-1450 scanning electron microscope (SEM) with a 10% oxalic acid solution (mass fraction).

2.1 Microstructure

Figure 1a [Figure 1: see original paper] shows the microstructure of BSTMUF601 alloy before creep testing. The alloy exhibits a lamellar and lath-like microstructure consisting of α+β phases, with irregular polygonal grain boundaries and precipitation of fine second-phase particles within grains. Figure 1b shows the microstructure after creep testing at 1095 °C under an applied stress of 5.7 MPa. The post-creep microstructure still consists of α+β phases with a typical lamellar structure within grains. The grain boundaries have undergone some deformation and exhibit segregation, with numerous fine second-phase particles precipitated along them. Grain growth is evident, and twins and subgrain boundaries appear within grains. Multiple sets of twins can be observed within the same grain, characterized by parallel features indicating identical twin orientation. Twins with different orientations also appear within larger grains.

2.2 Modified Projection Creep Constitutive Model

Figure 2 [Figure 2: see original paper] shows the creep curves of BSTMUF601 alloy under different temperatures and stresses. The creep curves exhibit different stages depending on conditions. At 1095 °C, the curves under 5.7 and 6.7 MPa consist primarily of the first and second stages, mainly because the lower stresses produce an extended secondary creep stage, preventing the development of a distinct tertiary stage before test termination. In contrast, the curve at 7.7 MPa shows complete three-stage behavior. At 980 °C and 870 °C, the creep curves consist mainly of the first and third stages because the higher stresses under these conditions result in no obvious secondary creep stage.

The first creep stage represents decelerating creep, where dislocations generated by instantaneous strain become piled up in matrix channels. Dislocations with different Burgers vectors meet in these channels and multiply through dislocation reactions, causing strain hardening and decreasing strain rate [15]. As creep progresses, piled-up dislocations leave the pile-up regions through thermal activation, resulting in strain softening. When hardening and softening reach relative equilibrium, creep enters the second stage—steady-state creep—where

the creep curve becomes approximately linear and the creep rate reaches its minimum value. Upon entering the third stage—accelerating creep—the creep rate increases significantly and strain grows rapidly until fracture occurs. At constant temperature, increasing stress accelerates creep curve evolution, shortens the secondary stage, and promotes early transition to the tertiary stage. Conversely, at lower stresses, the secondary stage is prolonged and may even lack a tertiary stage, demonstrating better ductility [16]. Temperature also significantly influences creep behavior [6].

Various methods exist for fitting creep curves. The projection method, which predicts long-term creep performance, conceptually divides the creep process into hardening during the first stage and softening during the third stage, without considering a second stage. Kim et al. [13] proposed that creep consists of decelerating and accelerating stages, with the second stage merely reflecting the balance between strain hardening and softening. They applied the projection method to fit creep curves of Hastelloy-X alloy at 950 °C under various stresses, expressed as:

$$= {}_1[1 - \exp(- {}_2t)] + {}_3[\exp({}_4t) - 1] \quad (1)$$

where is creep strain, t is time, and (i = 1, 2, 3, 4) are coefficients related to material, temperature, and stress, satisfying the following projection transformation:

$$= a\ \sigma\hat{}\{b\ \}\ \exp(-c\ /T) \quad (2)$$

where a , b , c are material constants related to temperature; T is thermodynamic temperature; and σ is stress.

Liu et al. [14] argued that creep curves measured under constant load typically show a short first stage and long second stage, which may lead to inaccurate results when using the projection method. They therefore proposed a modified projection method to fit creep curves, considering only the second and third stages:

$$= {}_0 + {}_1t + {}_2[1 - \exp(- {}_3t)] \quad (3)$$

where ${}_0$ is initial elastic strain; (i = 1, 2, 3) are parameters related to temperature and stress, also satisfying Eq. (2).

As shown in Figure 2, at 870 °C and 980 °C, the creep curves align with the model proposed in Eq. (1), consisting mainly of the first and third stages. At 1095 °C under 5.7 and 6.7 MPa, the curves exhibit primarily the first and second stages, while at 7.7 MPa, all three stages are clearly present. Since neither of the two existing models considers the complete three-stage creep process [13,14], this work proposes a new modified projection method to establish a creep constitutive model incorporating stages 1, 2, and 3:

$$= {}_0 + {}_1[1 - \exp(- {}_2t)] + {}_3t + {}_4[\exp({}_5t) - 1] \quad (4)$$

where ${}_0$ is initial elastic strain and ${}_3$ is the steady-state creep rate. The second

and fourth terms in Eq. (4) describe strain hardening and recovery softening processes, respectively, while the third term characterizes the steady-state creep stage. This model considers deformation features of all three creep stages and demonstrates strong applicability. Based on this constitutive model, creep curves at various temperatures and stresses were fitted, as shown in Figure 2.

To verify the accuracy of Eq. (4), creep data for BSTMUF601 alloy at 1095 °C and 7.7 MPa were fitted using all three models, as shown in Figure 3 [Figure 3: see original paper]. To quantify fitting accuracy, the concept of average relative error δ was introduced [17]:

$\delta = (1/N)\ \Sigma\_\{j=1\}\hat{}N\ |(E\_j - P\_j)/E\_j| \times 100\%$ (5)

where j is the data point index, E_j is the experimental strain value, P_j is the calculated strain value, and N is the number of data points (90 in this work).

The calculated average relative errors for the three projection methods are 1.96% for Kim et al.' s model [13] (Eq. (1)) and 7.88% for Liu et al.' s model [14] (Eq. (3)). In contrast, the new model proposed in this work achieves an average relative error of only 1.86%, representing reductions of 0.10% and 6.02% compared with the models that ignore the second stage [13] and first stage [14], respectively. This demonstrates that the new model offers strong applicability without compromising fitting precision.

2.3 Creep Deformation Mechanism

According to creep theory [18], the steady-state creep rate (minimum creep rate) is a crucial parameter for evaluating creep performance. The steady-state creep rate can be determined from creep curves. When the second stage is clearly defined, linear fitting of this stage yields the steady-state creep rate as the slope. When the second stage is not obvious, fitting and differentiating the creep curve identifies the minimum creep rate as the steady-state creep rate. Based on Figure 2, the steady-state creep rates of BSTMUF601 alloy under various temperatures and stresses were calculated and listed in Table 1 .

Table 1 shows that steady-state creep rate increases significantly with increasing creep temperature and stress. Higher temperatures increase the equilibrium vacancy concentration and mobility in the alloy, facilitating high-temperature deformation, while higher stresses markedly increase dislocation generation and mobility, thereby accelerating the creep rate [19].

To reveal the creep deformation mechanism of BSTMUF601 alloy, the creep stress exponent was calculated. At high temperatures, the steady-state creep rate $\dot{}$_s relates to stress σ and temperature T through the phenomenological equation [6]:

$\dot{}s = A\sigma\hat{}n\ exp(-Q\{app\}/RT)$ (6)

where n is the creep stress exponent, Q_{app} is the apparent creep activation energy, R is the universal gas constant, and A is a material constant. Taking

the natural logarithm and derivative of Eq. (6) yields:

n = (ln ˙_s)/ (ln σ) (7)

Based on the steady-state creep rates in Table 1, the relationship between ln ˙_s and ln σ was plotted, as shown in Figure 4 [Figure 4: see original paper]. From this figure, the stress exponent n was determined to be 4.9, 4.6, and 5.2 at 1095 °C, 980 °C, and 870 °C, respectively. The plot shows that the natural logarithm of steady-state creep rate is essentially linear with respect to the natural logarithm of stress. The value of the creep stress exponent n characterizes different creep mechanisms. When creep deformation is controlled primarily by dislocation climb, the creep stress exponent is approximately 5 [5,20,21]. The experimental results from this work, with n values all close to 5, indicate that creep deformation is mainly controlled by dislocation climb.

To further clarify the creep mechanism during the steady-state stage, dislocation configurations were examined. Figure 5 [Figure 5: see original paper] shows the microstructure of BSTMUF601 alloy during steady-state creep at 1095 °C and 5.7 MPa. Dislocations are clearly visible within the alloy structure after creep testing (Figure 5a). As creep progresses, dislocations with different Burgers vectors in the matrix channels undergo long-range cross-slip and multiply through reactions when they meet, gradually increasing the dislocation density in the matrix. Dislocation tangles and pile-ups appear near grain boundaries, γ' phase interfaces, or precipitated carbides, generating stress concentration (Figures 5b and c). This indicates that grain boundaries can effectively impede dislocation motion, thereby improving creep resistance [22]. When numerous dislocations in the matrix encounter γ' phase particles, they exhibit climb behavior through thermal activation, moving perpendicular to their slip planes (Figure 5d). Additionally, no stacking faults or microtwins were observed, indicating that under these creep conditions, dislocations cannot overcome γ' phase particles by cutting.

In BSTMUF601 alloy under low-temperature, high-stress conditions, the applied stress is sufficient for dislocations to cut γ' phase, making dislocation cutting the primary mechanism. However, under high-temperature, low-stress conditions, the applied stress is insufficient to overcome γ' phase, and dislocations can only bypass γ' phase through thermally activated climb. Portions or all of matrix dislocations undergo climb motion perpendicular to their slip planes via thermal activation (Figure 5d), eventually bypassing γ' phase [23,24]. During steady-state creep, dislocations in the matrix move to γ' phase interfaces under stress, where they pile up and react with dislocation networks. The decomposed dislocation components change their original movement direction, and dislocations climb through thermal activation, causing creep deformation. Throughout this process, dislocation climb controls creep deformation. Caron et al. [25] also concluded that creep deformation is primarily controlled by dislocation climb at 1050 °C. These analyses demonstrate that under high-temperature, low-stress conditions, the main deformation mechanism of BSTMUF601 alloy during steady-state creep is dislocation climb.

2.4 Creep Rupture Mechanism

Figure 6 [Figure 6: see original paper] shows the microstructure near the fracture surface of BSTMUF601 alloy after creep rupture at 870 °C and 32 MPa. Numerous honeycomb-like voids appear in the ruptured alloy, along with substantial carbides and second-phase particles precipitated along grain boundaries. As carbides and precipitates gradually increase and voids grow, the material becomes embrittled, the effective load-bearing cross-section decreases, and localized necking occurs, causing rapid creep acceleration and final fracture [26].

To further analyze the creep rupture mechanism, the microstructure near the fracture surface was examined. Figure 7 [Figure 7: see original paper] shows void and crack morphologies on longitudinal and cross-sections near the fracture surface of a creep specimen tested at 1095 °C and 7.7 MPa. Voids nucleate exclusively at grain boundaries, with no voids observed within grains (Figures 7a and c). This results from inconsistent deformation among grains, grain boundaries, and carbides. During creep, grain boundaries slide under applied stress while grain interiors cannot deform compatibly, causing strain in the matrix while carbides remain undeformed. Gradual decohesion between carbides and matrix creates void nuclei. Under applied stress, voids grow and coalesce to form cracks. As creep continues, dislocations continue to pile up near grain boundaries, generating stress concentration that drives crack propagation along grain boundaries and connection with adjacent cracks [22] (Figures 7b and d), ultimately leading to specimen fracture.

During creep, dislocation motion is blocked by grain boundaries or carbides, forming dislocation tangles and pile-ups that demonstrate the effectiveness of grain boundaries and carbides in impeding dislocation motion. As creep continues, numerous dislocations pile up near carbides at grain boundaries, creating stress concentration that provides favorable conditions for void nucleation at grain boundaries. Voids grow and coalesce to form microcracks that propagate continuously along grain boundaries under stress concentration. Although discontinuous precipitation of granular carbides in grain boundary regions can improve grain boundary strength, the fact that cracks initiate and propagate along grain boundaries indicates that under high-temperature creep conditions, grain boundaries remain the weak link causing creep fracture.

Conclusions

(1) A new modified projection creep constitutive model was proposed that comprehensively considers the deformation characteristics of all three creep stages. The fitting results agree well with experimental data, with an average relative error of only 1.86% under conditions of 1095 °C and 5.7 MPa. Compared with the projection model ignoring the second stage and the modified projection model ignoring the first stage, the average relative error is reduced by 0.10% and 6.02%, respectively, demonstrating that the new model offers strong applicability without compromising

fitting accuracy.

(2) Under high-temperature, low-stress conditions, BSTMUF601 alloy exhibits low steady-state creep rates, indicating good creep resistance. The natural logarithm of steady-state creep rate shows an approximately linear relationship with the natural logarithm of stress, yielding creep stress exponents close to 5 at different temperatures.

(3) During steady-state creep, BSTMUF601 alloy primarily deforms through dislocation climb over the γ' phase. No stacking faults or microtwins were observed in either the matrix or γ' phase, confirming that dislocation climb is the dominant creep deformation mechanism.

(4) Cracks originate from voids at grain boundaries and propagate continuously along grain boundaries under stress concentration, ultimately causing fracture. Under high-temperature creep conditions, grain boundaries are the weak link leading to creep rupture.

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