Experimental Study on the High-Temperature Creep Deformation Mechanism of BSTMUF601 Alloy (Postprint)

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Abstract

Tensile creep experiments were conducted on BSTMUF601 alloy under different temperature and stress conditions to obtain the high-temperature creep deformation behavior of the alloy. Based on this, a new modified -projection creep constitutive model was proposed, which considers the characteristics of all three creep stages with clear physical significance. The model predictions show good agreement with experimental results, with an average relative error of 1.86%, representing reductions of 0.1% and 6.02% compared to the -projection model without considering the second stage and the modified -projection model without considering the first stage, respectively, demonstrating that the model has strong applicability without compromising prediction accuracy. Microstructural analysis was performed on the dislocation configurations and void evolution of creep and creep fracture specimens. The results show that the creep stress exponent in the steady-state creep stage is close to 5, the alloy deforms primarily through dislocation climb over the ' phase, stacking faults and microtwins were not observed in the ' phase or matrix, and the creep deformation mechanism is primarily dislocation climb; voids nucleate on grain boundaries, grow and coalesce to form cracks, under stress concentration cracks propagate along grain boundaries, ultimately leading to fracture, and the creep fracture mechanism is primarily grain boundary fracture.

Full Text

Preamble

High Temperature Creep Deformation Mechanism of BSTMUF601 Superalloy

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Abstract

Muffle tube is the core component 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 usability and service life. High-temperature creep and rupture properties are critical indicators for muffle tube materials, with BSTMUF601 nickel-based superalloy being commonly used for this application. Nickel-based superalloys have attracted significant attention due to their excellent oxidation resistance, high strength, and good creep performance at elevated temperatures, particularly regarding their creep mechanisms. Since conclusions about creep mechanisms vary for different alloys or even for the same alloy under different conditions, individual investigation of each alloy is necessary.

This work conducted tensile creep experiments on BSTMUF601 alloy under various temperature and stress conditions to obtain its high-temperature creep deformation characteristics. Based on these results, a novel modified projection method creep constitutive model was proposed that considers all three stages of creep. The model predictions show good agreement with experimental results, with an average relative error of 1.86%. Compared with the projection method model that ignores the second stage and the modified projection method model that ignores the first stage, the relative error is reduced by 0.1% and 6.02%, respectively, demonstrating strong applicability without compromising prediction accuracy. Microstructural analysis of dislocation configurations in creep specimens and void evolution in creep rupture specimens revealed that the creep stress exponent during the steady-state creep stage is close to 5 under all conditions. The alloy primarily deforms via dislocation climb over the phase, with no stacking faults or microtwins observed in either the phase or the matrix. The main creep deformation mechanism is dislocation climb. Voids nucleate at grain boundaries, grow, and coalesce to form cracks. Under stress concentration, cracks propagate along grain boundaries, ultimately leading to

fracture, indicating that grain boundary fracture is the dominant creep rupture mechanism.

Keywords: BSTMUF601 superalloy, creep deformation, steady creep rate, creep rupture

Introduction

Muffle tubes are core components in large bright annealing muffle furnaces. Under prolonged exposure to high temperatures (~1050°C), self-weight, and temperature non-uniformity, muffle tubes develop various defects, with creep deformation being particularly severe and directly impacting performance and service life. Consequently, high-temperature creep resistance and rupture properties are critical metrics for muffle tube materials, with BSTMUF601 nickel-based superalloy being a common choice. Nickel-based superalloys have garnered significant attention due to their excellent high-temperature oxidation resistance, high strength, and superior creep performance, with particular focus on their creep mechanisms.

Extensive research has been conducted on creep deformation mechanisms in nickel-based alloys. Xu et al. studied a novel Ni-Co based superalloy and reported that at lower temperatures, deformation occurs via dislocation slip cutting through the phase to form stacking faults; at intermediate temperatures, the microstructure is dominated by stacking faults and microtwins traversing both the phase and matrix; and at very high temperatures, deformation occurs via dislocation bypassing of the phase. Liu et al. observed in a nickel-based single crystal superalloy that at 760°C and 780 MPa, dislocations cut into the phase via stacking faults at low strain stages and via dislocation pairs at high strain stages; whereas at 982°C and 248 MPa, matrix a/2<110> dislocations bow out in the matrix, bypass the phase, and form dislocation networks through dislocation reactions. Yuan et al. observed in U720Li alloy that under creep conditions of 725°C and 630 MPa, at 0.1% strain dislocations bypass the phase via the Orowan mechanism, at 5% strain partial dislocations cut the phase forming stacking faults and microtwins, and at 27% strain, grain boundary sliding occurs due to increased stress and strain. Viswanathan et al. observed in René 88 DT alloy that at 650°C, microtwins formed by partial dislocations dominate at low applied stresses, while 1/2<110> dislocations cutting the matrix and bypassing the phase via the Orowan mechanism dominate at high stresses. Since different researchers have reached different conclusions for different alloys or even the same alloy under different conditions, individual investigation of each alloy' s creep mechanism is necessary.

Given that nickel-based superalloy components operate long-term under high temperature and load, predicting their creep performance is essential. Kim et al. proposed that the creep process consists primarily of decelerating and accelerating creep stages, using the projection method to predict Hastelloy-X alloy

behavior. Liu et al. employed a modified projection method to predict creep performance of Cr25Ni35Nb furnace tube steel, considering only stages 2 and 3. While these models reflect deformation behavior under specific conditions, actual creep processes typically exhibit three distinct stages. For broader applicability and practicality, models must comprehensively consider deformation characteristics across all three creep stages.

This work references actual muffle tube service conditions, using accelerated creep testing at various temperatures and stresses to conduct high-temperature tensile creep experiments on BSTMUF601 alloy. The resulting creep curves were used to establish a constitutive model incorporating all three creep stages. The steady-state creep rate was determined from these curves 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, providing a theoretical basis for muffle tube life prediction and creep deformation simulation.

Experimental

The BSTMUF601 alloy used for muffle tubes is a Ni-Cr-Al series nickel-based alloy with chemical composition (mass fraction, %) of: Ni 58.0–63.0, Cr 21.0–25.0, Al 1.0–1.7, Mn 1.0, Cu 0.5, balance Fe. The experimental material was processed via vacuum induction melting, forging, multi-pass hot rolling, and solution heat treatment at 1080°C. Intermediate temperature aging after solution treatment produces 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 under 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 was employed. High-temperature tensile creep experiments 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 was: at 870°C, stresses of 20, 32, and 40 MPa; at 980°C, stresses of 12.4, 14.4, and 16.4 MPa; at 1095°C, stresses of 5.7, 6.7, and 7.7 MPa. Most tests were terminated upon reaching the steady-state or acceleration stage, while some were continued to fracture. Fractured specimens were cooled under load to room temperature for subsequent SEM and TEM observation.

To examine the microstructure during steady-state creep, 5 mm long samples were sectioned longitudinally from creep specimens, then ground, polished, and etched for observation using an MR5000 inverted metallurgical microscope (OM) with etchant of 1.5 g CuSO + 20 mL C H OH + 40 mL HCl. To observe dislo-

cation configurations after creep deformation, 0.5 mm thick slices were cut longitudinally, ground to 0.15 mm, punched into 3 mm diameter discs, and further ground to 60 m thickness. These were then thinned using a TenuPol-5 twin-jet electropolisher with 10% perchloric acid ethanol solution (mass fraction). The prepared foils were examined using a Tecnai F30 transmission electron microscope (TEM). To observe fracture morphology, two 10 mm long samples were sectioned longitudinally from each pair of ruptured creep specimens. One sample was split longitudinally, ground, polished, and electrolytically etched for observation using a LEO-1450 scanning electron microscope (SEM) with 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, exhibiting lamellar and lath-like morphologies composed of + phases with irregular polygonal grain boundaries and twinning within grains.

Figure 1b shows the microstructure after creep testing at 1095°C under 5.7 MPa. The alloy remains composed of + phases, with grain interiors displaying typical lamellar structures. Grain boundaries exhibit some deformation and segregation, with numerous fine second-phase particles precipitated along them. Grain growth is evident, with twins and subgrain boundaries appearing within grains. Multiple twin sets with parallel characteristics (identical twin orientation) are observed within the same grain, while larger grains contain twins of different orientations.

2.2 Modified Projection Method Creep Constitutive Model

Figure 2 [Figure 2: see original paper] shows creep curves for BSTMUF601 alloy under various temperatures and stresses. Different conditions exhibit distinct creep stages. At 1095°C and stresses of 5.7 and 6.7 MPa, creep curves consist primarily of stages 1 and 2, due to the relatively low stress producing an extended stage 2 without obvious stage 3 before test termination. At 7.7 MPa, the creep curve displays all three stages. At 980°C and 870°C, creep curves mainly comprise stages 1 and 3, as the higher stresses under these conditions suppress the distinct stage 2.

Stage 1 is the decelerating creep stage, where instantaneous strain produces dislocation pile-ups in matrix channels. Dislocations with different Burgers vectors meet and multiply through dislocation reactions, causing strain hardening and decreasing strain rate. Concurrently, as creep progresses, piled-up dislocations leave the pile-up region via thermal activation, causing strain softening. When hardening and softening reach equilibrium, creep enters stage 2—the steady-

state creep stage—where the creep curve becomes nearly linear and the creep rate reaches a minimum. During stage 3 (accelerating creep), the creep rate increases significantly, strain grows rapidly, and fracture eventually occurs. At constant temperature, increasing stress accelerates creep curve evolution, shortening stage 2 and rapidly initiating stage 3. At lower stresses, stage 2 is extended and stage 3 may be absent, demonstrating better ductility. Temperature also significantly influences creep behavior.

Various methods exist for fitting creep curves. The projection method conceptualizes creep as comprising hardening (stage 1) and softening (stage 3) processes without a distinct stage 2. Kim et al. proposed that creep consists of decelerating and accelerating stages, with stage 2 merely reflecting the balance between strain hardening and softening. They applied the projection method to Hastelloy-X alloy at 950°C under various stresses:

$$\varepsilon = \theta_1[1 - \exp(-\theta_2 t)] + \theta_3[\exp(\theta_4 t) - 1]$$

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

$$\lg \theta_i = a_i + b_i T + c_i \sigma + d_i T \sigma$$

where a_i, b_i, c_i, d_i are temperature-dependent material constants and T is thermodynamic temperature.

Liu et al. argued that in constant-load creep curves, stage 1 is short while stage 2 is long, potentially causing inaccuracies with the projection method. They proposed a modified projection method considering only stages 2 and 3:

$$\varepsilon = \varepsilon_0 + \theta_1' \exp(\theta_3' t) + \theta_2'[\exp(\theta_4' t) - 1]$$

where ε_0 is initial elastic strain and $\theta_1', \theta_2', \theta_3', \theta_4'$ are temperature- and stress-dependent parameters also satisfying Equation (2).

As shown in Figure 2, at 870°C and 980°C, creep curves align with Equation (1), comprising mainly stages 1 and 3. At 1095°C and stresses of 5.7 and 6.7 MPa, creep curves exhibit stages 1 and 2, while at 7.7 MPa all three stages are evident. Since neither previous model considers the complete three-stage creep process, this work proposes a new modified projection method incorporating stages 1, 2, and 3:

$$\varepsilon = \varepsilon_0 + \theta_1[1 - \exp(-\theta_2 t)] + \dot{\varepsilon}_s t + \theta_3[\exp(\theta_4 t) - 1]$$

where $\dot{\varepsilon}_s$ is the steady-state creep rate. The second and fourth terms describe strain hardening and recovery softening processes, respectively, while the third

term describes the steady-state creep stage. This model, considering deformation characteristics across all three creep stages, demonstrates strong applicability. Based on this constitutive model, creep curves at various temperatures and stresses were fitted, as shown in Figure 2.

To validate Equation (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 was introduced:

$$E = \frac{1}{N}\sum_{j=1}^{N}\frac{|E_j - P_j|}{E_j} \times 100\%$$

where E_j is experimental strain, P_j is calculated strain, and N is the number of data points (90 in this work). The average relative errors are: Kim' s model (Equation (1)) = 1.96%, Liu' s model (Equation (3)) = 7.88%, and this work' s model (Equation (4)) = 1.86%. The new model reduces error by 0.1% and 6.02% compared to models ignoring stage 2 and stage 1, respectively, demonstrating strong applicability without compromising fitting precision.

2.3 Creep Deformation Mechanism

According to creep theory, the steady-state creep rate (minimum creep rate) is a critical parameter for evaluating creep performance. The steady-state creep rate can be determined from creep curves: when stage 2 is distinct, linear fitting of this stage yields the rate as its slope; when stage 2 is not obvious, fitting and differentiating the creep curve identifies the minimum rate as the steady-state value. Figure 1 enables calculation of steady-state creep rates for BSTMUF601 alloy under various conditions, as listed in Table 1 .

Table 1 shows that steady-state creep rate increases significantly with both temperature and stress. Elevated temperature increases equilibrium vacancy concentration and mobility, facilitating high-temperature deformation, while increased stress enhances dislocation generation and mobility, accelerating creep rate.

To reveal the creep deformation mechanism, the creep stress exponent during steady-state creep was calculated. At high temperatures, the relationship between steady-state creep rate $\dot{\varepsilon}_s$, stress σ, and temperature T can be expressed macroscopically as:

$$\dot{\varepsilon}_s = A\sigma^n \exp\left(-\frac{Q_{app}}{RT}\right)$$

where σ is applied stress, n is creep stress exponent, Q_{app} is apparent creep activation energy, R is the universal gas constant, and A is a material constant. Taking the natural logarithm and differentiating yields n:

$$n = \left(\frac{\partial \ln \dot{\varepsilon}_s}{\partial \ln \sigma} \right)_T$$

Using the steady-state creep rates from Table 1, $\ln \dot{\varepsilon}_s - \ln \sigma$ relationships were plotted, as shown in Figure 4 [Figure 4: see original paper]. The stress exponents obtained are 4.9 at 1095°C, 4.6 at 980°C, and 5.2 at 870°C.

The creep stress exponent characterizes different creep mechanisms. When creep deformation is controlled primarily by dislocation climb, the exponent is approximately 5. The experimental results, with exponents near 5, indicate that creep deformation is mainly controlled by dislocation climb.

To further clarify the creep mechanism during steady-state creep, dislocation configurations were examined. Figure 5 [Figure 5: see original paper] shows the microstructure during steady-state creep at 1095°C and 5.7 MPa.

Significant dislocations are observed after creep testing (Figure 5a). As creep progresses, dislocations with different Burgers vectors in matrix channels undergo long-range cross-slip, meet, and multiply through reactions, gradually increasing dislocation density. Dislocation tangles and pile-ups appear near grain boundaries, / interfaces, and carbide precipitates, creating stress concentrations (Figures 5b and c). This indicates that grain boundaries effectively impede dislocation motion, enhancing creep resistance. When numerous matrix dislocations encounter precipitates, they move perpendicular to the slip plane at the / interface, exhibiting climb behavior (Figure 5d). No stacking faults or microtwins are observed, indicating that dislocations cannot cross the / interface into the phase under these conditions.

At low temperatures and high stresses, applied stress is sufficient for dislocations to cut into the phase, making dislocation cutting the primary mechanism. However, at high temperatures and low stresses, applied stress is insufficient for dislocation cutting, and dislocations can only bypass the phase via thermally activated climb. Matrix dislocations partially or completely climb perpendicular to the slip plane through thermal activation (Figure 5d), eventually bypassing the phase. During steady-state creep, dislocations moving in the matrix under stress pile up at / interfaces, meet dislocation networks, react, and change direction, climbing via thermal activation to cause creep deformation. This process is controlled by dislocation climb. Caron et al. also concluded that at 1050°C, creep deformation is primarily controlled by dislocation climb. These findings indicate that under high-temperature, low-stress conditions, the main deformation mechanism during steady-state creep of BSTMUF601 alloy is dislocation climb.

2.4 Creep Rupture Mechanism

Figure 6 [Figure 6: see original paper] shows the microstructure near the fracture surface after creep rupture at 870°C and 32 MPa. Numerous honeycomb-like voids and extensive carbide and second-phase particle precipitation along grain boundaries are observed. As carbides and precipitates increase and voids grow, the material embrittles, the effective load-bearing area decreases, and localized necking occurs, leading to rapid creep acceleration and eventual fracture.

To further analyze the creep rupture mechanism, microstructures near fracture surfaces were examined. Figure 7 [Figure 7: see original paper] shows void and crack morphologies on longitudinal and cross sections near the fracture surface after creep at 1095°C and 7.7 MPa. Voids nucleate exclusively at grain boundaries, with none observed within grains (Figures 7a and c), due to inconsistent deformation between grains, grain boundaries, and carbides. During creep, grain boundaries slide under applied stress while grain interiors cannot deform compatibly, straining the matrix while carbides remain undeformed, gradually debonding from the matrix to form void nuclei. Under applied stress, voids grow and coalesce to form cracks. As creep continues, dislocations continue to pile up near grain boundaries, creating stress concentrations that drive crack propagation along grain boundaries and connection with adjacent cracks (Figures 7b and d), ultimately causing fracture.

During creep, dislocation motion is impeded by grain boundaries and carbides, forming dislocation tangles and pile-ups that demonstrate the effectiveness of grain boundaries and carbides in blocking dislocation motion. As creep progresses, numerous dislocations pile up near carbides at grain boundaries, creating stress concentrations that facilitate void nucleation. Voids grow and connect to form microcracks, which propagate continuously along grain boundaries under stress concentration. Although granular carbides precipitated discontinuously along grain boundaries can enhance boundary cohesion, the fact that cracks nucleate and propagate along grain boundaries indicates that grain boundaries remain the weak link causing creep fracture under high-temperature creep conditions.

Conclusions

(1) A novel modified projection method creep constitutive model was proposed that comprehensively considers deformation characteristics across all three creep stages. The fitted results agree well with experimental data, with an average relative error of only 1.86% at 1095°C and 5.7 MPa. This represents error reductions of 0.1% and 6.02% compared to the projection method model ignoring stage 2 and the modified projection method model ignoring stage 1, respectively, demonstrating strong applicability without compromising fitting precision.

(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 versus stress shows an approximately linear relationship, with creep stress exponents near 5 at different temperatures.

(3) During steady-state creep, BSTMUF601 alloy primarily deforms via dislocation climb over the phase. The absence of stacking faults and microtwins in both the matrix and phase confirms that dislocation climb is the dominant creep deformation mechanism.

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

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