Microstructure evolution and stress rupture properties of A286 superalloy in the 600 to 750 °C temperature range

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Abstract
To study the stress rupture properties of the A286 alloy in high temperature service environment, the stress rupture tests of A286 alloy at different temperature ranges and stress levels were carried out. Moreover, the Larson-Miller Parameter (LMP) for predicting the rupture life was established as LMP = (T + 273)(log tr + C) × 10−3, C = 22.3. The fracture morphology and the microstructure evolution of the stress rupture specimens were observed by scanning electron microscopy. The results show that for the stress rupture tests at 700 °C–750 °C/150–400 MPa, the precipitation of the closely arranged cellular η phase and the coarse MC at the grain boundary acted as the crack source. For the stress rupture test at 600 °C–700 °C/400–700 MPa, the coarse MC at the grain boundary led to stress concentration to give rise to crack initiation and propagation during deformation. With decreasing of temperature and increasing of stress, the fracture mode of the A286 alloy changed from the ductile fracture mode of microvoids to the brittle fracture mode of intergranular. The deformation mechanisms of A286 alloy at different temperatures and stress levels also have been systematically analyzed by transmission electron microscopy. The deformation mechanisms of A286 at different temperatures and stress levels were Orowan looping, microtwins and dislocation pairs shearing with Orowan looping and microtwins as the dominant deformation mechanisms. The critical transition radius of γ’ phase during the transition from dislocation shearing mechanism to Orowan looping mechanism was calculated.

1. Introduction

A286 alloy is an austenitic precipitation-hardening stainless steel that is mainly strengthened by the precipitation of a large amount of fine and dispersed γ’-Ni3(Ti, Al) phase in the matrix during aging [1–3]. Due to its high yield strength and excellent stress rupture properties below 650 °C, this alloy is widely used in high-temperature aero-engine bearings for parts that operate at temperature below 650 °C for a long time, such as aviation turbine disks, blades, fasteners and other high-temperature parts [4, 5]. Generally, high-temperature stress rupture behavior of materials plays a critical role for these components exposed to high temperatures. Previous studies have shown that long-term high-temperature service will cause changes in the material microstructure such as agglomeration, coarsening of second phase particles, and precipitation of unfavorable phases on the grain boundaries, thus affecting the stress rupture properties of materials [6–8]. Therefore, it is particularly important to study the deformation behavior of the A286 alloy at elevated temperatures. Pirali et al studied the high-temperature creep behavior of A286 alloy using CT samples, and found that overaging of the alloy occurs when the test time was longer than 170 h at 650 °C, leading to the deterioration of mechanical properties [2]. DeCicco et al investigated the creep behavior of the A286 alloy in different temperature ranges and stress levels and found that all creep tests showed intergranular fracture that was closely related to the η phase precipitated at grain boundaries.
A286 alloy were analyzed in detail. The optical microscopy interaction between microstructure in the process of stress rupture were observed, the initiation and propagation of crack and the temperature and stress was established to predict the stress-rupture life. Meanwhile, the fracture and microstructure in the process of stress rupture were observed, the initiation and propagation of crack and the interaction between γ phase and dislocation were examined, and the fracture and deformation mechanisms of A286 alloy were analyzed in detail.

2. Material and experimental

A286 ingots were prepared via electric arc furnace smelting (EAF) + ladle refining (LF) + electroslag remelting (ESR) and then hot-rolled and cold-drawn into bars. The corresponding compositions are given in table 1. Since the A286 alloy is usually used after solid solution and aging treatment, the same heat treatment regime was used for all of the bars, namely solid solution at 980 °C for 1 h, followed by water cooling, and then by aging at 720 °C for 16 h and air cooling. After the heat treatment, according to the dimensions of GB/T 2039–2012, these bars were machined into stress rupture samples with a gauge diameter and length of 5 mm and 25 mm, respectively. The stress rupture tests were performed at 600, 650, 700 and 750 °C for three stress levels using a GNCJ-30 creep rupture strength testing machine. During the stress rupture test, the stress rupture properties such as stress rupture time (h), elongation (σ) and percentage reduction of the area (Ψ) were measured.

The optical micrographs of the samples after heat treatment were observed using an Axiovert A1 digital metallographic microscope. The corrosive agent was KMnO₄ (1 g) dissolved in a mixture solution of H₂SO₄ (7.5 ml) and H₂O (100 ml). The fracture morphology of the stress rupture samples was observed using a Quanta 60FEG scanning electron microscope, and then cut along the axial direction of the sample to observe the microstructure near the fracture. Thin foils specimens for transmission electron microscopy (TEM) observation were prepared using a Gatan 691 ion bombardment. The dislocation configurations of the specimens were observed using a Tecnai G2 F20 transmission electron microscope.

3. Results and discussion

3.1. Grain size and microstructure

The optical microscopy (OM) photograph of the A286 alloy after solution treatment at 980 °C for 1 h and aging treatment at 720 °C for 16 h is shown in figure 1(a). It is observed that the grains are equiaxed and there are many twins in the grains. According to GB/T6394-2017 method for the determination of the grain size of steel, the grain size grade of A286 alloy is 7. The grain size of the A286 alloy is distributed between 20 μm–70 μm, the average grain size is approximately 27 μm. Figure 1(b) shows the SEM image of the A286 alloy. It is observed that fine granular M₂₃C₆ and massive MC precipitated at the grain boundary. Previous studies have determined that the MC is TiC formed during solidification, and MC₂₃C₆ is the fine Cr-rich carbide [15, 16].

3.2. Stress rupture properties

The stress rupture tests were carried out at 600, 650, 700 and 750 °C, and three stress levels were examined at each temperature. Figure 2(a) shows the relationship between stress rupture life and stress. It is observed that the stress rupture life clearly decreased with the increasing stress. The elongation of the A286 alloy increased with increasing of temperature, as shown in figure 2(b). It is inferred that temperature and stress play an important role in the stress rupture process of the A286 alloy.

The stress rupture strength required in engineering design generally corresponds to a life of thousands to hundreds of thousands of hours, so that it is extremely difficult to obtain such long-term stress rupture data

| C   | Si  | P   | B   | Cr | Ni | Mo | Mn | V  | Ti | Al | Fe |
|-----|-----|-----|-----|----|----|----|----|----|----|----|----|
| 0.043 | 0.16 | 0.013 | 0.0089 | 14.87 | 25.58 | 1.21 | 1.38 | 0.28 | 2.16 | 0.13 | residue |

Table 1. Chemical compositions of the A286 alloy in weight percent.
experimentally. To simplify the estimation of stress rupture life, the Larsen–Miller Parameter (LMP) was used to predict the long-term stress rupture life of materials. LMP is a comprehensive parameter that is a function of the time and temperature that can be expressed as follows [17]:

\[
LMP = (T + 273)(\log t_r + C) \times 10^{-3}
\]  

(1)

where \(T\) is the test temperature (°C), \(t_r\) is the stress rupture life (h), \(C\) is the constant related to the temperature, and the value of \(C\) is generally set to approximately 20. However, previous studies have found that the value of \(C\) is not 20 and in fact can fluctuate over a wide range [18, 19]. The determination of the \(C\) value referred to the work of Zhu et al [20]. Based on the stress rupture test data, the value of \(C\) was obtained by using the surface fitting tool in the MATLAB software, as shown in figure 3. The specific surface fitting results and fitting parameters are listed in table 2, where the values of \(R^2\) and Adjusted \(R^2\) are closer to 1 and the values of SSE and RMSE are closer to 0, indicating the high quality of the fitting results.

The scatter diagram of the relationship between stress and LMP was drawn using the Origin software, and linear regression analysis was carried out, as shown in figure 4. It is observed that there exists a close linear relationship between stress (\(\sigma_r\)) and LMP, and LMP decreases with the increasing stress. The linear regression equation was: \(\sigma_r = 2872.8 - 107.36 \times 10^{-3} \times \text{LMP}\), and the linear correlation coefficient \(R^2 = 0.99\). According to the fitting results, the stress rupture life of the A286 alloy under given stress and temperature can be easily predicted.

### 3.3. Stress rupture fracture surfaces

Figure 5(a) shows the macroscopic fracture morphology of the stress rupture at 750 °C/150 MPa. It is observed that the macroscopic fracture surface was uneven and granular, with large fiber area, no clear radiation area, and
slight necking phenomenon on the fracture surface. The microscopic fracture analysis shows that the fiber zone consisted of equiaxed dimples of different sizes, corresponding to a typical microporous condensation fracture and belonged to the plastic fracture pattern, as shown in figure 5(b). When the loading stress was increased to 250 MPa, the specimen showed no obvious necking phenomenon and the macroscopic fracture surface was granular. The number of holes in the microscopic fracture clearly decreased, and the fiber zone was ductile intergranular fracture, accompanied by a large number of tearing dimple bands, as shown in figure 5(d).

Figure 6(a) shows the macroscopic fracture morphology of the stress rupture at 600 °C/600 MPa. The macroscopic fracture shows that the fracture region was mainly composed of shear lip region and fiber region, while the microscopic fracture shows that the fiber region was mainly intergranular fracture, and many secondary cracks have been observed. The macroscopic fracture surface was very flat, and there was a discontinuous intergranular fracture zone at the edge of the fracture, as shown in the area of the yellow coil in figure 6(c), it is inferred that the sample begins to crack from the edge. After the cracks grew to a certain extent, the remaining cross-sectional area was not sufficient to withstand the loaded stress, and the sample exhibited rapid fracture.
Figure 5. Fractographs of the A286 alloy after it was stress-ruptured at 750 °C: (a), (b) 150 MPa; (c), (d) 250 MPa.

Figure 6. Fractographs of the A286 alloy after it was stress-ruptured at 600 °C: (a), (b) 600 MPa; (c), (d) 700 MPa.
According to the above fracture morphology characteristics, it was found that with the decrease in the temperature and the increase in the stress, the stress fracture mode of the A286 alloy changes from the ductile fracture mode of the microvoids to the brittle fracture mode of intergranular.

Figure 7 shows the microstructure near the fracture of the stress rupture specimens at 750 °C, 150 MPa / 250 MPa. It is observed that a large number of secondary cracks and holes were present near the fracture, which may be the main reason for the fracture of the specimens. Further observation shows that the cracks mainly existed at the grain boundary, and it was found that a large number of cellular η phases precipitated along the grain boundaries and continuously grew into the grain and M23C6 on the grain boundaries coarsened, as shown in figure 7(b). The precipitation of coarse precipitates on the grain boundary will destroy the continuity of the grain boundary and decreased the grain boundary strength to values lower than that of the grain. At the same time, microcracks also were initiated at MC. Under the conditions of stress and high temperature, the cracks nucleated and propagated at the η phase and MC, and finally broke under the action of stress.

No lamellar η phase was found under the stresses of 600 MPa and 700 MPa at 600 °C, and no coarsening of M23C6 occurred. The secondary cracks were mainly located at the position of the large MC, as shown in figures 8(b), (d). It was clear that due to the lack of coordination between MC and the matrix, the secondary crack was easily initiated and propagated rapidly at the MC position, leading to fracture. Since the η phase mainly precipitated during the long-term aging at higher temperature, and the MC in A286 alloy was primitive TiC in the solidification process, its morphology generally did not change during the subsequent heat treatment [21]. Therefore, when the test condition satisfied the critical precipitation condition of the cellular η phase, the precipitation of the closely arranged cellular η phase and the coarse MC at the grain boundary acted as the crack source, while when the η phase did not precipitate in the sample, the coarse MC at the grain boundary gave rise to the stress concentration in deformation, leading to crack initiation and propagation.

3.4. Precipitation behavior of η phase
The ordered fcc γ'-Ni3(Ti, Al) phase is a metastable phase, and its stable structure is the hcp η-(Ni3Ti) phase [12]. It was found that the η phase and γ' phases are based on the same Ni3Ti composition. It was reported that the η
Figure 8. SEM micrographs near the fracture of A286 alloy under 600 °C, (a), (b) 600 MPa and (c), (d) 700 MPa.

Figure 9. Different morphologies of the η phase at 750 °C/150 MPa: (a) Cellular η phase; (b) Widmanstatten η phase. (c), (d) and (e) TEM micrograph of the η phase and its EDS and SAED patterns.
phase tends to precipitate at the grain boundary, and the morphology of the \( \eta \) phase can transform from cellular to Widmanstatten type during long-term aging at high temperature, further degrading the mechanical properties of the alloy [1, 9, 11]. It is observed that at 750 °C and 150 MPa, a large amount of the cellular \( \eta \) phase was precipitated, and a small amount of the cellular \( \eta \) phase has been transformed into the Widmanstatten \( \eta \), as shown in figure 9(b). The \( \eta \) phase had a coherent orientation relationship with the matrix as follows: \( \{001\}\eta// \{100\}\gamma \), \( \langle 100 \rangle \eta// \langle 110 \rangle \gamma \). Figure 10 shows the evolution of the grain boundary precipitates at different temperature ranges and stress levels. It was found that the \( \eta \) phase did not precipitate below 700 °C, while the amount of the \( \eta \) phase precipitated along the grain boundaries with increasing of stress rupture life above 700 °C. Meanwhile, the size of \( \gamma' \) phase at 750 °C, 150 MPa and 250 MPa stress levels were measured by Image-Pro Plus software, as shown in figure 11. The results show that the radius of \( \gamma' \) phase is mostly from 25 nm to 30 nm and no coarsening of \( \gamma' \) phase occurred. Therefore, it can be inferred that the continuous precipitation of \( \eta \) phase was the main reason for the decrease of stress rupture life of the A286 alloy at elevated temperature.

The formation of the \( \eta \) phase has been intensely investigated in previous work. To date, it has been generally believed that there were three sources of the \( \eta \) phase. The first point of view is that the precipitation of the \( \eta \) phase occurs at the expense of \( \gamma' \) phase: \( \gamma' \rightarrow \eta \), forming \( \gamma' \) precipitation free-zones [21–24]. Figure 9(a) also confirmed this view. \( \gamma' \) phase is the most important strengthening phase in the A286 alloy, so that the formation of \( \eta \) at the expense of \( \gamma' \) will certainly reduce the strength of the matrix. The second point of view is that in the superalloy with high Ti/Al ratio, the \( \eta \) phase can also be degraded by MC during long aging, and \( \text{M}_{23}\text{C}_6 \) can be formed during the transformation process: \( \text{MC} \rightarrow \gamma' \rightarrow \text{M}_{23}\text{C}_6 \rightarrow \eta \) [25–28]. Additionally, it has also been suggested that the \( \eta \) phase may nucleate directly in the austenite matrix at higher temperatures [23].

\[ \text{MC} \rightarrow \gamma' \rightarrow \text{M}_{23}\text{C}_6 \rightarrow \eta \]
3.5. Deformation mechanism

To further elucidate the high-temperature deformation mechanism of A286, the deformation microstructure of typical samples was observed by TEM. Figure 12 shows the microstructure of stress rupture at 750 °C/150 MPa. It is observed that after the stress rupture test, a large number of dislocations increase the dislocation density, and the dislocations were entangled to form dislocation walls, as shown in the orange arrow of figure 12(a). The dispersed γ′ phase is an effective obstacle to the movement of dislocations, and with increasing degree of deformation, some dislocations bypass γ′, leaving a large number of Orowan loops, as shown in figures 12(b), (c). The existence of Orowan loops reduced the distance between effective particles, hindering the subsequent dislocation movement. Meanwhile, several dislocation pairs were observed in figure 12(c). Dislocation pair is generally considered to be an important precipitation hardening effect. When the first dislocation cut through the ordered phase γ′, an anti-phase boundary (APB) can be produced, and then the second a/2〈110〉 dislocation matrix will eliminate APB and restore the order of the γ′ phase [27, 29–31]. In addition, SAED results show the existence of microtwins formed by the two η phases, as shown in figure 12(d).

Figure 13 shows the microstructure after the stress rupture test at 750 °C/250 MPa. It was found that the dislocations were mainly concentrated near the grain boundary, and dislocation tangles became more pronounced with increasing proximity to the grain boundary (figure 13(a)). SAED results in the yellow dotted line region shows the existence of microtwins (figure 13(a)), and it was that microtwins appeared at the boundary of annealing twins. Viswanathan et al also observed the initiation of microscopic twins at the boundary of annealing twins by preparing creep specimens TEM foil [32]. The existence of microtwins hindered the movement of dislocations, leading to an accumulation of a large number of dislocations between the microtwins and grain boundaries that then hindered the movement of dislocations more difficult. Figure 13(b) shows the interaction between the γ′ phase and dislocations, displaying dislocation loops, and some longer slip dislocations form relatively large loops that hinder dislocations movement. Meanwhile, with increasing stress, the probability of dislocation pairs increased (figure 13(c)). Figure 13(d) shows the existence of M₆C at the grain boundary. For superalloys, MC carbides rich in Mo, Ti, Nb and W decompose into M₆C under long-term high-

![Figure 11. The size distribution of γ′ phase at 750 °C with different stress: (a) 150 MPa; (b) 250 MPa.](image-url)
temperature conditions. This is because during the decomposition of MC carbides, C diffuses from the MC carbide at the grain boundary to the grain boundary, while Ti, Cr, Mo, and other elements diffuse to the grain boundary, resulting in the formation of low-carbon M6C carbide. The M6C at the grain boundary prevented the dislocation from crossing the grain boundary, as shown in figure 13(d).

Figure 14 shows the microstructure after stress rupture at 600 °C/600 MPa. It is observed that a large number of dislocations formed in the matrix, and the interaction between the dislocations and the γ′ phase formed Orowan loops, as shown in figures 14(a), (b). Dislocation pairs were also observed in the sample, as shown in figure 14(c). Comparison of the position of Orowan loops and dislocation pairs showed that the position of the dislocation tangles were mainly dislocation loops, while the dislocation pairs were mainly found at the locations with relatively few dislocations. In addition, microtwins were also found in the specimen, as shown in figure 14(d), and a large number of dislocations were found to be entangled near the grain boundary.

Figure 15 shows the microstructure after the stress rupture at 600 °C/700 MPa, and figures 15(a), (b) show the bright and dark field phases of γ′ phase and dislocations, respectively. It is observed that the dislocation density in the matrix was high and there were several dislocation loops. The dislocations were hindered at the γ/γ′ phase interface to form the dislocation network that counteracted the lattice mismatch caused by the cutting of the γ′ phases by the dislocations, reduced the degree of stress concentration, and promoted the stability of the γ/γ′ interface. With increasing stress, the failure process of the dislocation network was accelerated, and then a notch was formed at the phase interface to guide the moving dislocation in the matrix into the γ′ phase. When the density of the interface defect was high, the defects interface formed an external stacking fault that was represented by the increased stacking fault probability, as shown in figure 15(c). This may be attributed to the
increase in the stress that provides sufficient energy for the dislocation to cut through the $\gamma’$ phase. However, the strengthening effect of the $\gamma’$ phase inside the stacking fault was poor, reducing the stress rupture life of the alloy [34]. At the same time, it is observed that microtwins existed near the grain boundary, a large number of dislocations gathered at the position of the microtwins, and slip bands were observed to be blocked by the microtwins, as shown in figure 15(d).

TEM results show that there exist two different strengthening mechanisms in the interaction of $\gamma’$ phase and dislocation, involving dislocation shearing and Orowan looping mechanisms. And two mechanisms depend on the precipitate size with respect to a critical size. When $\gamma’$ phase is small in size and keeps a good coherent relationship with matrix, $\gamma’$ phase is sheared by dislocations and the contribution of $\gamma’$ can be expressed as [35]:

$$\tau_p = \chi \varepsilon \left( \frac{2f}{b} \right)^{\frac{1}{2}}$$

where $\tau_p$ is the shear stress, $\chi = 2.6$ is the constant, and $\varepsilon$ is a function of the mismatch between the $\gamma’$ phase and the matrix, $\varepsilon = |\delta| [1 + 2G(1 - 2\nu)/G_\gamma] \approx 1.5|\delta|$. Through the interplanar spacing between matrix phase and $\gamma’$ phase, $\delta$ can be calculated to be approximately 0.005. $G$ is the elastic modulus of matrix, $r$ is the radius of $\gamma’$ phase, $f$ is the volume fraction of the second phase, $b$ is the Burgers vector. When the size of $\gamma’$ phase reaches a certain degree. And $\gamma’$ phase is not easily sheared by dislocations. At this time, Orowan looping mechanism plays a leading role and the stress required for a dislocation to bow between precipitates can be expressed as equation (3) [36]:

$$\tau_p = \frac{G\delta^{\frac{1}{2}} \ln \frac{r}{b}}{2r(1 - \nu)}$$

where $\nu = 0.3$ is the Poisson’s ratio of the A286 alloy matrix, $\tau_p$ is defined as the $\gamma’$ phase radius when the dislocation shearing strengthening is equal to the Orowan strengthening. $\gamma’$ phase has the maximum

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Figure 13. Microstructure and dislocation configuration of stress rupture at 750 °C/250 MPa.
strengthening effect when the radius of $\gamma'$ phase is $r_c$. Combining equations (2) and (3), the (4) related to $r_c$ can be obtained as

$$\frac{(2r_c)^3}{\ln \left( \frac{b}{\xi} \right)} = 0.1864 \frac{b^2}{\chi (1 - v)\varepsilon^3}$$  \hspace{1cm} (4)

The value of $r_c$ calculated by MATLAB software is 14.383 nm, and the size of $\gamma'$ phases sheared by dislocations was counted by Image-Pro Plus software (figure 16(a)). It is observed from figure 16(b) that the calculated $r_c$ value is very close to the experimental $r_c$ value.

The above TEM observation results indicated that Orowan looping plays an important role in the stress rupture of A286 alloy. The smaller $\gamma'$ phases can be sheared by dislocations and make less contribution to the strength. When the size of $\gamma'$ phase is larger than $r_c$, the interaction of $\gamma'$ phase dislocation is transformed into Orowan looping mechanism. The density of Orowan loops increases with decreasing stress and increasing temperature, thereby significantly increasing the stress rupture life. Additionally, the existence of microtwins can effectively block the movement of dislocation. Particularly at a high temperature, the dislocations will slip and undergo dynamic recovery, and microtwins can also provide a location for dislocation nucleation, thereby improving the strength and plasticity of the alloy [37, 38].

4. Conclusions

The stress rupture properties of the A286 alloy at different temperature ranges and stress levels have been systematically studied, and the deformation mechanism and fracture behavior were analyzed. Based on the results, the following conclusions were derived.
Under the given temperature and stress condition, with the decreasing stress and increasing temperature, the stress rupture life and elongation of the A286 alloy clearly increased. The stress rupture life of the A286 alloy was predicted using the Larson-Miller parameter equation, and linear fitting obtained the following formula for the stress:

$$\sigma_r = 2872.8 - 107.36 \times 10^{-3} \times \text{LMP}.$$ 

Figure 15. Microstructure and dislocation configuration of stress rupture under 600 °C/700 MPa.

Figure 16. (a) Typical TEM image of γ' phase sheared by dislocations; (b) Comparison between experimental $r_e$ value and calculated $r_e$ value.

(1) Under the given temperature and stress condition, with the decreasing stress and increasing temperature, the stress rupture life and elongation of the A286 alloy clearly increased. The stress rupture life of the A286 alloy was predicted using the Larson-Miller parameter equation, and linear fitting obtained the following formula for the stress: $\sigma_r = 2872.8 - 107.36 \times 10^{-3} \times \text{LMP}$.
(2) In the stress rupture tests at 700 °C–750 °C/150–400 MPa, the precipitation of the closely arranged cellular \( \eta \) phase and the coarse MC at the grain boundary acted as the crack source. For the stress rupture test at 600 °C–700 °C/400–700 MPa, the coarse MC at the grain boundary gave rise to stress concentration that led to crack initiation and propagation during deformation. With decreasing temperature and increasing stress, the fracture mode of the A286 alloy changes from the ductile fracture mode of microvoids to the brittle fracture mode of intergranular.

(3) Orowan loops, microtwins and dislocation pairs shearing are the most important stress rupture mechanisms of A286. Orowan loops can effectively block the movement of dislocations, and their density decreased with increasing stress and decreasing temperature. The critical transition radius of \( \gamma' \) phase during the transition from dislocation shearing mechanism to Orowan mechanism is 14.383 nm. And the existence of microtwins inhibits the movement of the slip band and further increases the stress rupture life.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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