Influence of thermal ageing on creep rupture mechanism and creep life of P92 ferritic steel

Hui Kang1,2, Junjie Shen1,2,4, Shanjun Zhang1,2, Hongguang Han1,2, Fuyong Hu1,2, Biao He3, Qingquan Zhao3 and Gongye Xiao4

1 Tianjin Key Laboratory for Advanced Mechatronic System Design and Intelligent Control, School of Mechanical Engineering, Tianjin University of Technology, Tianjin, 300384, People’s Republic of China
2 National Demonstration Center for Experimental Mechanical and Electrical Engineering Education, Tianjin University of Technology, Tianjin, 300384, People’s Republic of China
3 Tianjin Pipe Corporation, Tianjin, People’s Republic of China
4 Author to whom any correspondence should be addressed.

E-mail: sjj1982428@sina.com

Keywords: thermal ageing, P92 heat-resistant steel, creep, creep rupture life

Abstract

In order to analyze the effect of thermal ageing on creep rupture mechanism and establish a method for predicting the creep rupture life of P92 heat-resistant steel under thermal ageing, creep tests were performed on P92 steel specimens, aged at 650 °C for different times under different applied stresses. Optical microscopy and scanning electron microscopy were utilized to observe the microstructure after thermal ageing and creep rupture. Voids and cracks were distributed in the grains of P92 steel before thermal ageing, whereas the voids were clearly distributed around M23C6 precipitates and Laves phase along sub-grain boundaries after thermal ageing. The creep damage tolerance factor of the unaged and short-time aged samples ranged from 2.5 to 5, and the creep rupture was caused by dislocation movement. After high-temperature thermal ageing for 3000 h, the damage tolerance factor increased to > 5 and creep rupture was caused by precipitate coarsening. A theoretical method was established to predict the creep rupture life of heat-resistant P92 steel under thermal ageing, providing consistent results with the American Society of Mechanical Engineers (ASME) long-term test and trend.

1. Introduction

Heat-resistant steels, with 9%–12% chromium, are widely used in thick-wall components of the main steam pipes in ultra-supercritical power plants due to their excellent high-temperature resistance, oxidation resistance and creep resistance under high-temperature conditions. The high chromium ferritic steel possesses unstable martensitic structure, which is obtained by rapid cooling. Hence, the microstructure evolution due to heat instability influences long-term service performance and mechanical properties [1–4]. During long-term service, the dislocation density is decreased and martensitic laths are coarsened under the influence of thermal energy. However, thermal ageing has a negligible influence on MX change. On the other hand, the Laves phase precipitates during thermal ageing and the relative proportion of Laves phase initially increases with ageing time, followed by a gradual decrease after 1500 h [3]. The average diameter growth rate of Laves phase is found to be significantly faster than that of M23C6. In addition, the spherical Laves phase precipitates are more stable than the rod precipitates. The M23C6 phase gradually coarsens during thermal ageing and the chemical stability has a significant influence on M23C6 coarsening.

In recent years, several research groups [5, 6] have analyzed the influence of microstructural evolution on mechanical properties, i.e., tensile behavior, creep strength and creep-rupture properties, and identified key microstructural features which influence the mechanical properties. It has been reported that the microstructural changes during ageing exert a slight effect on room-temperature tensile properties and
hardness, whereas the impact properties decrease significantly after ageing for 2000 h \[5\]. Khayatzadeh et al \[6\] investigated the influence of thermal ageing (650 °C, 1000 h) on creep behavior of P92 martensitic steel and demonstrated that thermal ageing does not influence the room-temperature tensile properties, i.e., Young’s modulus and yield strength. Moreover, thermal ageing does not influence the creep stress relaxation and primary creep. However, thermal ageing alters the creep strength, minimum creep strain and creep life. One should note that the low dislocation density, unstable Laves phase and M23C6 carbide particles significantly decrease the creep strength \[5\]. Saini et al \[7\] studied the effect of thermal ageing on Laves phase evolution and mechanical properties of P92 steel, and demonstrated that the evolution of Laves phase degrades the mechanical strength. We have previously reported the influence of M23C6 and Laves phase evolution on creep strength of P92 steel under low-stress conditions \[6\]. At \(\sigma < 35\) MPa, the Laves phase affects the low-stress creep behavior, whereas the M23C6 carbide determines the low-stress creep behavior at \(\sigma > 35\) MPa. Furthermore, thermal ageing reduces the creep life over a relatively narrow stress range (85–140 MPa). Thermal ageing initially leads to crack incubation and distributes micro-cracks around the crack tip. The precipitates are strongly associated with crack initiation of T92 steel at high temperatures \[8\]. The MX precipitates provide excellent resistance to dislocation cutting during creep, whereas M23C6 and Laves phase precipitates are prone to cutting by dislocation glide, which provides a dislocation slipping path. Crack initiation is observed in the cut of M23C6 and Laves phase.

![Figure 1. Creep strain versus time curves of P92 heat-resistant steel samples, thermally-aged for different times, under applied stress of (a) 100 MPa; (b) 120 MPa; (c) 140 MPa; and (d) 160 MPa at 650 °C.](image)

| Table 1. The chemical composition (mass fraction, %) of P92 steel. |
|------------------|---|---|---|---|---|---|---|---|---|---|
| Element | C  | Si | Mn | S  | P  | Cr | Mo | W  | V  | Nb | Ni |
| Content   | 0.012 | 0.370 | 0.550 | 0.003 | 0.003 | 9.430 | 0.510 | 1.820 | 0.220 | 0.081 | 0.30 |
phase precipitates [8]. It has been reported that the cavities produced during high-temperature long-term service nucleate at coarse Laves-phase particles on grain boundaries, triggering a fracture of T92 steel [9].

Hence, the evolution of different precipitates, such as Laves phase, strongly affects creep-rupture properties. As the Laves phase is gradually formed during the creep process, the influence of Laves phase precipitates on creep rupture mechanism may vary with ageing time. Therefore, the current study aims to investigate the influence of thermal ageing on creep rupture mechanism and creep life of P92 ferritic steel.

2. Materials and methods

The chemical composition of P92 steel is shown in table 1. The test samples were cut along the axial direction of steel tubes and processed into standard creep samples with a length of 80 mm and a cross-section of 2 × 15 mm. The pipe was homogenized at 1050 °C for 1 h and tempered at 780 °C for 2 h, followed by air cooling. The P92 steel samples were aged for 0, 3000, 5000, and 7200 h at 650 °C in a box furnace (CHY-M1700). The thermal ageing temperature was maintained within a deviation range of ±1 °C. The tensile creep tests were carried out by using an electronic creep endurance testing machine according to the HB5151-1996 standard, i.e., high-temperature tensile creep testing method for metals, at 650 °C under 100 MPa, 120 MPa, 140 MPa and 160 MPa. The elongation was recorded by computer software.

Before tensile creep testing, various samples (5 mm × 5 mm × 5 mm) were prepared by using different thermal ageing conditions and treated with a corrosive mixture of picric acid (1 g), hydrochloric acid (5 ml) and alcohol solution (100 ml) for metallographic analysis. The metallographic changes were observed by using an Olympus BX51 optical microscope. The composition was analyzed by a ZEISS GeminiSEM500 scanning electron microscope (SEM) and the structure was determined using an x-ray diffractometer (XRD, SmartLab.)
9 KW). The creep fracture surface was sampled by a Q-2 metallographic sample cutting machine and observed by SEM.

3. Results and discussion

3.1. Creep properties

Figure 1 shows the creep strain versus time curves of P92 steels, thermally aged for different times, under applied stress of 100 MPa (figure 1(a)), 120 MPa (figure 1(b)), 140 MPa (figure 1(c)) and 160 MPa (figure 1(d)). The creep curves can be divided into three stages: the initial creep stage, where the creep strain gradually decreases with time; the secondary creep stage, where the creep strain remains unchanged; and the accelerated creep stage, where the creep strain rapidly increases with time until rupture. Under different thermal ageing times and stress conditions, the creep behavior of samples before thermal ageing was significantly different from the thermally aged samples. The creep rupture time of the thermally-aged samples was significantly shorter than that of the unaged samples. Moreover, the creep strain of thermally-aged samples was larger than that of unaged samples, which indicates that the thermal ageing influences the creep plastic deformation. Figure 2 shows the creep strain rate versus time curves in the double logarithmic scale. It can be seen that the minimum creep strain rate gradually increased with the increase in thermal ageing time, which indicates that thermal ageing leads to a
significant decrease in creep strength due to the change in dislocation substructure and instability of precipitated particles during ageing [5].

Figure 3(a) shows the dependence of minimum creep rate on stress at 650 °C. The minimum creep rate increased with the increase in thermal ageing time and stress. The relationship between minimum creep rate and
stress follows Norton’s power law:

\[ \dot{\varepsilon}_{\text{min}} = A \sigma^n \]  

(1)

Figure 3(b) shows the dependence of creep rupture life on stress at 650 °C. The relationship between creep rupture life \((t_r)\) and stress \((\sigma)\) under the same thermal ageing conditions conforms to the power-law equation:

\[ \sigma = A' t_r^{n'} \]  

(2)

3.2. Rupture mechanism

The Monkman-Grant relationship describes the correlation between \(\dot{\varepsilon}_{\text{min}}\) and \(t_r\):

\[ \dot{\varepsilon}_{\text{min}} t_r = C_{\text{MG}} \]  

(3)

where \(\dot{\varepsilon}_{\text{min}}\) refers to the minimum creep rate, \(t_r\) represents the creep rupture life, and \(C_{\text{MG}}\) denotes the Monkman-Grant (M-G) constant at constant temperature and stress. Dobes \textit{et al} [10] revised the M-G relationship and added the influence of rupture strain \((\varepsilon_r)\), which can be given as:

\[ \dot{\varepsilon}_{\text{min}} t_r / \varepsilon_r = C_{\text{MMG}} \]  

(4)

The modified M-G relationship describes the correlation between the minimum creep rate and rupture life. The creep deformation increases the minimum creep rate and leads to the final rupture. Phaniraj \textit{et al} [11] defined the Monkman-Grant toughness as:

\[ \varepsilon = \dot{\varepsilon}_{\text{min}} t_r = C_{\text{MGD}} \]  

(5)

whereas Kachanov \textit{et al} [12] and Rabotnov \textit{et al} [13] proposed the mechanism of continuous creep rupture. The continuous creep rupture mechanism considers creep damage as an intrinsic variable and defines the ratio of creep rupture to \(C_{\text{MGD}}\) as the damage tolerance factor:

\[ \lambda = \varepsilon / \dot{\varepsilon}_{\text{min}} t_r \]  

(6)

The constant \(\lambda\) can be related to the creep and ultimate rupture mechanism during the third creep stage. According to the literature [14], \(\lambda\) represents the ability of a material to resist deformation before it ruptures. Wilshire \textit{et al} [15] found that, during the third stage, the creep and ultimate rupture in the range of \(\lambda \approx 1–2.5, 2.5–5\) and \(>5\) are caused by the voids, dislocation movement and precipitates coarsening, respectively.
At $\lambda = 2.5 - 5$, the creep and ultimate rupture of the unaged and short-time thermally-aged samples during the third stage are caused by the dislocation movement, as shown in figure 4. However, the creep and ultimate rupture of the thermally-aged samples ($>3000$ h) are caused by precipitate coarsening at $\lambda > 5$. Hence, the Laves phase is precipitated after ageing for $>3000$ and the creep rupture in long-time thermally-aged samples is affected by the precipitation of M$_{23}$C$_6$ and Laves phases. It can be concluded that lower stress and longer ageing time result in higher $\lambda$ values. Hence, a correlation exists between the final creep rupture and precipitated phase.

### 3.3. Creep life prediction

Isotherm extrapolation was used to extrapolate the long-time creep endurance strength of lower stress with short-time experimental data of higher stress under a certain temperature. Figure 3(b) shows the linear relationship between stress log($\sigma$) and creep rupture time log($t_r$), which can be given as:

$$\ln \sigma = a + b \times \ln t_r$$  \hspace{1cm} (7)

This extrapolation method [16, 17] can be used to calculate the creep endurance strength of P92 heat-resistant steel during long-term creep under different thermal ageing conditions at 650 °C (figure 5).

Figure 5 shows that the creep endurance strength of P92 heat-resistant steel at 50000 h was 60 MPa after thermal ageing at 650 °C for 3000 h, which conforms to the American Society of Mechanical Engineers (ASME) standard [18], as shown in figure 7. After thermal ageing at 650 °C for 5000 h, the creep endurance strength at 100000 h was about 50 MPa, which is close to the predicted value by ASME. Moreover, the creep endurance strength at 100000 h of the unaged sample was 66.7 MPa, which is much higher than the value determined by the ASME standard.

In order to understand the effect of thermal ageing on long-term creep rupture life, the Origin software was used to fit and extrapolate the experimental data, resulting in two curved surfaces (figure 6). The yellow dots represent the experimental data points, the red dots represent isothermal extrapolation data points, and the

---

Figure 8. Optical microstructure (OM) of P92 steel after thermal ageing at 650 °C for different times: (a) 0 h, (b) 3000 h, (c) 5000 h and (d) 7200 h.
black triangles represent ASME data points. Upper surface $a_1$ in figures i and ii is the fitted surface with experimental data points and isothermal extrapolation data points. Lower surface b in figure i is fitted by experimental data points and ASME data points.

The results of upper surface fitting were compared (ii – iv) in figure 6. Table 2 and figure 6 show that the method (ii) provides optimal fitting results for the top surface. The fitting equation can be given as:

$$t_r = c\sigma^2 + d\sigma t + e\sigma + f\sigma + gt + z$$

where $\sigma$ represents the creep endurance strength, $t$ refers to the thermal ageing time, $t_r$ denotes the rupture time, and $c, d, e, f, g,$ and $z$ are constants (table 2). Figure 6 shows the obvious differences between the isothermal extrapolation points and ASME data points of the unaged condition for the long-term creep life. However, both surfaces tend to coincide with the decrease in stress and increase in thermal ageing time. The difference between both surfaces becomes minimal after thermal ageing for 3000 h.

The extrapolated points of stress versus creep rupture life under thermal ageing are plotted along the ASME data [18], as shown in figure 7. Black triangles are ASME data points and the blue dots are data points which are extrapolated points considering thermal aging.

It can be observed that the stress versus creep rupture life prediction curves without thermal ageing, derived by using L-M and M-H parametric methods [19, 20], significantly deviate from the data obtained by the long-term creep rupture test when the creep rupture time is greater than 10000 h. On the other hand, the stress versus creep rupture life data with thermal ageing, derived by using the isothermal extrapolation method, is consistent with the ASME data.

L-M and M-H methods are the analytical expression of time-temperature parameter (TTP) method [21, 22] (figure 7). Both methods are functions of temperature, stress and creep fracture time, and their relations are as follows:
L-M method \[ \text{[19]} \]:

\[
P_{LM}(\sigma) = T(C + \log t_r)
\]  

(9)

where \(\sigma\) is stress, \(T\) is temperature, \(t_r\) is rupture time, \(C\) is the material constant independent of temperature \((C = 38)\) \[\text{[23]}\], and \(P_{LM}(\sigma)\) represents a function of \(\sigma\).

M-H method \[\text{[20]}\]:

\[
P_{MH}(\sigma) = \frac{\log t_r - \log t_a}{T - T_a}
\]  

(10)

where \(\sigma\) is stress, \(T\) is temperature, \(t_r\) is rupture time, \(T_a\) and \(t_a\) are constants \((T_a = 190, t_a = 31)\) \[\text{[24]}\], and \(P_{MH}(\sigma)\) represents a function of \(\sigma\).

3.4. Microstructural analysis

3.4.1. Influence of thermal ageing on microstructure evolution

Figure 8 presents the microstructure of P92 steel under different thermal ageing times at 650 °C, showing the typical tempered martensite microstructure, including strip/block martensite and several precipitates, which are uniformly distributed in the matrix. The lath thickness and grain size increased with the increase in thermal
Figure 11. XRD pattern of P92 steel after thermal ageing for 7200 h.

Figure 12. The crack rupture morphology of P92 heat-resistant steel at 650 °C under 140 MPa: (a) before thermal ageing and (b) after thermal-ageing for 7200 h.

Figure 13. (a) The crack morphology of P92 heat-resistant steel after thermal-ageing for 7200 h, and SEM-EDS analysis of (b) phase A and (c) phase B.
ageing time. Also, the martensite bundles, blocks, laths, and other structural units became slightly more discernible with the increase in thermal ageing time.

Figures 9(a)–(d) show SEM images of the P92 heat-resistant steel after thermal ageing at 650 °C for 0–7200 h. It can be observed that the precipitates are mainly located at grain and sub-grain boundaries of the original austenite.

3.4.2. Analysis of precipitated phases

Figures 10(a) and (b) show the backscattered electron (BSE) morphologies of the precipitated phases in P92 after thermal ageing for 3000 h and 7200 h. The precipitated phase is obviously coarsened. Figures 10(b) and (c) show that two types of precipitates exist with various sizes, as depicted by bright and dark shades. The energy dispersive spectrometer (EDS) analysis demonstrates that the bright precipitates (phase A) are rich in W and Fe, whereas the dark precipitates (phase B) are rich in Fe and Cr (figures 10(d) and (e)).

Moreover, x-ray diffraction (XRD) analysis was carried out to identify the precipitates (figure 11). Three types of precipitates were observed, i.e., Laves phase, M23C6, and MX precipitates. Therefore, the brightest and large-size precipitates can be identified as the Fe2W-type Laves phase, the dark and medium-size precipitates are Cr23C6-type M23C6 carbide, and the dark and small-size precipitates are MX carbides.

The creep strength of ferritic heat-resistant steels, with 9%–12% chromium, is caused by precipitation strengthening of MX, M23C6, and Laves phases [25, 26]. During long-term service, the precipitate evolution leads to decreased creep strength. Under high stresses, the decrease in creep strength is related to the coarsening of M23C6 phase, whereas, under low stresses, the decrease in creep strength is due to the precipitation and growth of the Laves phase [27].

3.4.3. Creep rupture morphology

Figure 12 shows the SEM images of P92 heat-resistant steel, which was creep ruptured at 650 °C under 140 MPa. In the case of unaged sample (figure 12(a)), the voids and cracks are distributed in the grains and do not exhibit coalescence. However, the voids in thermally-aged sample (7200 h) are clearly distributed around the precipitates along sub-grain boundaries (figure 12(b)). Figure 13 shows the crack morphology in P92 heat-resistant steel thermally-aged for 7200 h. The long-term thermal ageing resulted in voids and cracks around the precipitates, i.e., Laves and M23C6 phases, and along the sub-grain boundaries.

The rupture surface morphology of thermally-aged creep specimen (5000 h) is shown in figure 14. The thermally-aged P92 steel rupture surface exhibited small ductile dimples and large transgranular cleavage facets (figure 14(a)). Moreover, a few tear ridges were observed (figure 14(b)). The Laves phase and M23C6 precipitates at grain boundaries acted as the crack nucleation sites (figure 12(b)), making it susceptible to inter-granular cracking [28]. Therefore, the thermally-aged P92 steel (5000 h) exhibited brittle intergranular fracture due to the coarsening of Laves phase and M23C6 precipitates.
4. Conclusions

In summary, the influence of thermal ageing on creep rupture mechanism and creep life of P92 ferritic steel was systematically investigated. Moreover, a novel method to predict the creep life of P92 steel under thermal ageing was established. The following conclusions can be drawn from the current results:

(1) The damage tolerance factor ($\lambda$) ranged from 2.5 to 5 in the case of unaged and short-time aged (<3000 h) samples, indicating that the creep rupture was caused by dislocation movement. However, after thermal ageing for >3000 h, $\lambda$ increased to >5 and the creep rupture was caused by precipitate coarsening. In short, lower stress and longer thermal ageing time resulted in higher $\lambda$ values.

(2) In as-prepared samples, the voids and cracks were distributed in the grains, whereas the voids were distributed around the $M_{23}C_6$ and Laves phase precipitates along the sub-grain boundaries in thermally-aged samples.

(3) An ASME method was established to predict the creep rupture life of thermally-aged P92 heat-resistant steel. The results revealed that the creep rupture life, obtained by the proposed method, is in accordance with the long-range test values and ASME trends.

Acknowledgments

This project was supported by the National Natural Science Foundation of China (Grant No.51605330), Natural Science Foundation of Tianjin (No.18JCYBJC88700).

ORCID iDs

Hui Kang @ https://orcid.org/0000-0002-9960-7878
Junjie Shen @ https://orcid.org/0000-0002-1415-476X
Shanjun Zhang @ https://orcid.org/0000-0003-0393-4565

References

[1] Yan W, Wang W, Shan Y Y and Yang K 2013 Front. Mater. Sci. 71–27
[2] Lopez Barrilajo I, Kuhn B and Wessel E 2018 Micron (Oxford, England) 108 11–8
[3] Matsunage T et al 2019 Mater. Sci. Eng. A 760 267–76
[4] Han H G, Shen J and Xie J X 2019 Sci. Rep. 9 9567
[5] Sklenicka V, Kucharova K, Svobodova M, Krala P, Kvasnikovaa M and Dvorak J 2018 Mater. Charact. 136 388–97
[6] Khayatzadeh S, Tanner D W J, Truman C E, Flewitt P E J and Smith D J 2017 Mater. Sci. Eng. A 708 544–55
[7] Saini N, Mulik R S and Mahapatr M M 2018 Mater. Sci. Eng. A 716 179–88
[8] Wang S B, Chang X F and Key J 2017 Mater. Charact. 127 1–11
[9] Nie M et al 2014 J. Alloy. Compd. 588 348–56
[10] Dobes F and Milicka K 1976 Met. Sci. 10 382–4
[11] Phaniraj C, Choudhary B K, Bhanu Sankara Rao K and Baldev R 2003 Scripta. Mater. 48 1313
[12] Kachanov L M 1961 J. Appl. Math. Mech. 25 234–7
[13] Backhaus G 1971 J. Appl. Math. Mech. 51 (Amsterdam: North-Holland Publishing Company) 575–6
[14] Zhao Q, Peng X K and Wang R 2010 journal of Iron and Steel Research 22 56–8
[15] Wilshire B and Burt H 2008 Int. J. Pres. Ves. Pip. 88 47–9
[16] Chen H X, Yang K and Shan Y Y 2012 Guangdong Electric power 25 5–8
[17] Peng Z F, Dang Y Y and Peng F F 2010 Acta. Metal. Sin. 46 435–43
[18] European Creep Collaborative Committee 2005 ECCC Data Sheets
[19] Larson F R and Miller J 1952 ASME Trans. 74 765–75
[20] Manson S S and Haferd A M 1953 NACA TN 2890 1–49 A linear time-temperature relation for extrapolation of creep and stress rupture data (http://dl.wanfangdata.com.cn/instId9pkeEnw53byJu%25252532FCAhBnUK585nE%25252532FVssl%25252532FtReIPho%25252532F3D)
[21] Abdallah Z, Gray V, Whitaker M and Perkins K 2014 Mater. 7 3371–98
[22] Kim W G, Ye X S, Lee G G, Kim Y W and Kim S J 2010 J. Pres. Ves. Tech. 87 289–95
[23] Wang X W, Guo J M, Guo X F and Jiang Y 2014 Journal of Nanjing Technology University 36 32–8
[24] Jiang F 2015 Analyses of Several Prediction Techniques Of Creep Rupture Life for P92 Steel (Dalian, China: Dalian University of Technology)
[25] Guo X F, Gong J M and Jiang Y 2015 Mater. Mech. Eng. 39 41–3
[26] Zhang H J, Zhou R C, Yang L Y and Yu Z S 2009 Journal of Electrical Engineering of China 29 174–7
[27] Zhang J S 2010 High Temperature Deformation and Rupture of Materials (Beijing: Science Press Publishing)
[28] Thomas Paul S, Saroja S and Vijayalakshmi M 2008 J. Nucl. Mater. 378 273–81