Study on creep behavior and microstructure of a Fe–Cr–Al–Nb–W ferritic heat-resistant steel based on C, N and Al element control

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Abstract

In this work, the effects of C, N, and Al on the microstructures and creep properties of Fe–Cr–Al–Nb–W ferritic alloys were investigated through scanning electron microscopy, x-ray diffractometry, as well as uniaxial creep testing and hardness testing. The results demonstrated that the matrix of the Fe–Cr–Al–Nb–W heat-resistant steel was ferrite, while the precipitation phases were Laves phases, M23C6 carbides and MX nitride phases. M23C6 and MX precipitated at grain interior, Laves phases precipitated at grain interior, grain boundary and around MX phases. C, N and Al affected microstructure and creep of heat-resistant steel. As the Al content increased or as N content decreased along with the C increase, the average diameter of the Laves phases, along with M23C6 and MX phase grain interior decrease. Moreover, the unit density increased and the phase spacing decreased, which led to the minimum creep rate decrease and creep rupture time increase. Compared to M23C6, Laves and MX phases mainly affected the alloy creep strength. The decrease of Laves phase continuity coefficient (ratio of Laves phase particle spacing and size) on the grain boundary was conducive to the plasticity improvement of heat-resistant steel.

1. Introduction

Carbon dioxide emissions reduction constitutes an important method to inhibit the global warming increase. The efficiency improvement of coal-fired thermal power generation is an important technology for carbon dioxide emission reduction. To utilize this important technology, thermal power plants must be required to operate at higher steam pressures and temperatures [1–5]. One of the key points restricting the development of thermal power units is the development of high-temperature materials for high-temperature superheaters and reheaters. At present, the candidate materials for the high-temperature part of ultra-supercritical (pressure exceeding 25 MPa, temperature exceeding 580 °C) generator sets are mainly nickel-based alloys, austenitic steels and ferritic heat-resistant steels [6]. Nickel-based alloys (Inconel 740 H and Haynes 282) are mainly used as superheater/reheater tubes for advanced ultra-supercritical boilers at 700 °C. The corresponding high purchase costs and operation costs limit large scale utilisations and applications in thermal power plants [7]. Austenitic heat-resistant steels have lower thermal conductivity compared to ferritic heat-resistant steels above 650 °C, as well as high thermal expansion coefficient and low stress corrosion resistance. During the actual service, once high temperature difference and thermal stress exist between inner and outer walls of pipelines, easy cracking and certain security risks might occur [8, 9]. Ferritic heat-resistant steels (P/T91, P/T92, P/T93) have become the first chosen material for supercritical (supercritical) power plants, due to outstanding creep resistance, good corrosion resistance and high temperature oxidation resistance. Moreover, the cost is significantly lower compared to austenitic heat-resistant steels and nickel base alloys [10–14]. However, the upper limit of the corresponding service temperature is 650 °C, which limits any application in advanced ultra-supercritical units.
Therefore, the effective ferritic heat-resistant steel modification to meet the application of advanced ultr-supercritical units at 700 °C is one of the future research directions for heat-resistant steels.

The high-chromium (9–12Cr) ferritic heat-resistant steel would sustain a significant decrease in creep strength, due to ‘structural degradation’ during long-range service. The room temperature structure of high Cr ferritic heat resistant steel is martensite + a low amount of δ-ferrite [2]. High density dislocations and fine precipitates in martensitic laths are mainly utilized to ensure the stability of microstructure. It has been reported [14–16] that the precipitation phases of high-Cr ferritic heat-resistant steel are mainly composed of M\textsubscript{23}C\textsubscript{6}, located at the boundaries or subgrain boundaries, MX grain interiors and Laves phases (type Fe\textsubscript{2}W), generated during aging. In the long-term service [15, 16], the dislocation density decreased, the size of the martensite lath increased, M\textsubscript{2}N phases disappeared, Z-phase formed, Mo and W decreased in amount, while M\textsubscript{23}C\textsubscript{6} phases became coarser. In recent years, researchers [5] have attempted to qualitatively analyze the influence of microstructure degradation on creep strength. The evolution of the precipitates had high influence on creep properties. H G Han and J J Shen investigated the evolution of precipitates of P92 high-chromium heat-resistant steel and corresponding effects on creep properties under low and medium stress [17]. The results demonstrated that 1) no Z-phase formation was observed; 2) the microstructures of M\textsubscript{23}C\textsubscript{6} and Laves phases affected the creep properties; also, the microstructure of the M\textsubscript{23}C\textsubscript{6} under high and medium stresses affected the creep properties; in addition, under low stresses, the morphologies of the Laves phases had high effect on creep performance.

The high-temperature strengthening of ferritic heat-resistant steel might be further improved through microstructure and structural characteristics control of the precipitated Laves phases [18]. A Fe–Cr–Al–Nb–W ferritic heat-resistant steel was prepared through eutectic reaction utilization [19, 20], while the influence of alloying elements on microstructure of Laves phase was studied. The precipitated Laves phases were coarse and strength was low. In addition, the purpose of this work was to refine the Laves phases through Nb and W content reduction, to improve the oxidation resistance, while simultaneously different amounts of Al were added. Furthermore, the steel was strengthened through the addition of various amounts of C and N. The content effects of C, N and Al on microstructure and creep properties were studied.

### 2. Materials and method

The experimental alloy was prepared through vacuum arc melting with non-consumable electrode. Melting in a vacuum melting furnace along with forging resulted in production of three cylindrical steel ingots of 120 mm in large head diameter, of 90 mm in small head diameter and of 300 mm in height. In order to ensure the alloy composition uniformity, each alloy ingot was repeatedly smelted 5 times and subsequently sucked into a mold for ingot production. The compositions are presented in table 1. The smelted samples were used for microstructure observations and creep testing. Cube samples of 5 mm × 5 mm × 5 mm were utilized for microstructure observation and a rectangular specimen for creep testing (as presented in figure 1). These were processed with a DK77100 EDM machine without heat treatment. The cube samples were roughed, finely ground and polished with an etching solution (FeCl\textsubscript{3} 5 g + HCl 100 ml + Ethanol 100 ml) for corrosion testing. Scanning electron microscopy (ZEISS Gemini500) and x-ray diffractometry (SmartLab 9KW) were conducted to observe the tissue characteristics and to conduct phase analysis. Vickers hardness was measured with a micro hardness tester (MC010). A uniaxial creep testing machine (RI-50) was utilized to obtain the creep properties at 650 °C and 80 MPa.

According to the XRD and Mapping results, precipitates of different colors were labeled as different shapes in the SEM image. The Image-Pro Plus software was used to repeatedly extract the precipitated particles from the SEM image until the result remained constant, while parameters, such as precipitate size and unit density, were quantitatively measured.

| Steels | Cr | Al | W | Nb | C | N | Fe |
|--------|----|----|---|----|---|---|----|
| S1     | 9.0| 4  | 2 | 0.5| 0.012| 0.04| Bal |
| S2     | 9.0| 4  | 2 | 0.5| 0.02 | 0.03| Bal |
| S3     | 9.0| 4  | 2 | 0.5| 0.008| 0.05| Bal |
| S4     | 9.0| 2  | 2 | 0.5| 0.012| 0.04| Bal |
| S5     | 9.0| 1  | 2 | 0.5| 0.012| 0.04| Bal |

### Table 1. Chemical composition of experimental steels, wt-%.
3. Results and discussion

3.1. Phase identification

The x-ray diffraction pattern of sample S2 is presented in figure 2. Through the position, intensity and number analysis of diffraction peaks, it could be observed that the constituent phases were ferrite, Laves phases, as well as M$_{23}$C$_6$ and MX phases. Figure 3 presents a SEM image of sample S2. The precipitates in the heat-resistant steel differed in sizes and shapes. Rod-shaped precipitates, large spherical particles and small spherical particles existed. In order to identify the phases of precipitates, an elemental surface scan was performed upon the area presented in figure 3 (figure 5), while an energy spectrum analysis of the different shapes and sizes of the precipitates within the grain and at grain boundary was performed (figures 4(a)–(d)). Energy spectrum results in figure 4(a) present that the main elements of the small spherical precipitate 1 were Al and N. Combined with XRD results (figure 2) and element mapping results analysis (figure 5), it is calibrated to be nitride MX. Figure 4(b) presents that the major elements of the large spherical precipitate 2 were Fe, Cr, and C. Combined with XRD results (figure 2) and element mapping results (figure 5) analysis, it is calibrated to be M$_{23}$C$_6$ carbide. The results of energy spectrum in figure 4(c) indicated that the main elements of rod-shaped precipitate 3 (within the grain) were Fe and Nb. Combined with XRD results (figure 2) and element mapping results (figure 5) analysis, it is calibrated to be Laves phase (within the grain). The results of energy spectrum in figure 4(d) indicated that the main elements of rod-shaped precipitate 4 were Fe and Nb. It was characterized as Laves phase (at grain boundary) through stoichiometric composition analysis.

3.2. Influence of C, N and Al on microstructure characteristics

Figure 6 presents a secondary electron image of samples S1–S5. The matrix was ferrite. The carbides M$_{23}$C$_6$ and nitride MX both precipitated within the grain. The Laves phase precipitated at the ferrite grain boundary and within the grain, while the precipitation occurred around the MX phase.

When the contents of Al were the same (S1–S3): In sample S3 with the highest content of N and the lowest content of C, the Laves phase, as well as MX and M$_{23}$C$_6$ phases had the lowest density; as the N decreased and the
C increased (samples S1 and S2), the density of Laves phase increased, the volume increased and the shape was uniform (sample S2). The volumes of M23C6 and MX did not significantly change, but the density increased.

When the contents of C and N were the same (S1, S4 and S5): As the Al content decreased, the Laves phase tended to gradually become coarser and the density decreased (the density was lowest and the volume was highest at S5), which signified that Al was beneficial to the Laves phase precipitation, as also reported by F Liu [21]. As the content of Al decreased, the density of MX increased and the volume decreased, while the density of M23C6 decreased and the volume increased. The Al content (1%–4%) increase was conducive to the second phase particles refinement.

The precipitates at the grain boundary in the study were only Laves phases, without any M23C6 existence through the analysis of energy spectrum results (figure 4) and element mapping images (figure 5). The Fe–Cr–Al–Nb–W alloys were melted and cooled down to room temperature. In this case, M23C6 was formed by eutectic reaction: \( \alpha \rightarrow \alpha + M_{23}C_6 \) (figure 7) [22], when the C content was extremely lower (<0.02 wt-% in this work). Therefore, M23C6 precipitated within the grain.

The arithmetical average and Bootstrapping statistical methods were utilized to obtain the average diameters and particle space \( \lambda \) of Laves, M23C6, and MX phases. In order to avoid a non-uniform microstructure within the steel, the arithmetical average statistical method was utilized. Five pictures of each group of materials were taken for data statistics, while the average values of diameters and particle spaces of Laves, M23C6, and MX phases were calculated.

Based on Bootstrapping in statistics, according to the results of XRD and Mapping, Image-Pro Plus was utilized to determine the boundaries of different precipitates and different colors were used to calibrate different precipitates, extracting the precipitates for data statistics. Based on Bootstrapping, the latter operations were carried out multiple times, to achieve the most accurate range. Based on Bootstrapping, Image-Pro Plus measurement software was utilized to repeatedly extract differently colored particles from SEM images until the measurement results remained constant, while quantitative parameters, such as precipitation number per unit area \( N \) and average diameter \( \bar{d} \) of precipitates (within the grain), were measured. Through consideration of sample S1 as an example, the statistical process is presented in figure 8. Five pictures of each group of materials were utilized for data statistics. The standard deviation of each group of material data was calculated. Subsequently to the standard deviation comparisons, the average value of each group of material data was used as the final result. The results are presented in figure 9.

### 3.3. Influence of C, N and Al on hardness

Figure 10 presents the hardness values of heat-resistant steels for samples S1–S5. When the content of C and N were the same, the hardness values of heat-resistant steels presented slight differences. When the content of C and N were the same, as the content of Al increased (S5 \( \rightarrow \) S4 \( \rightarrow \) S1), the hardness of heat-resistant steels firstly decreased and subsequently increased. When the Al content was 2 wt-%, the hardness of heat-resistant steel was the lowest, which coincided with the results [23].
3.4. Influence of C, N and Al on creep behavior

Figures 11(a) and 12(a) present the strain versus time creep curves of heat-resistant steel at 650 °C and 80 MPa. Figures 11(b) and 12(b) present the strain rate versus time creep curves in double log scale. The creep curve consisted of three stages: initial creep stage with reduced creep rate, steady creep stage with constant creep rate and accelerated creep stage with increased creep rate. Considering the influence of microstructure

Figure 4. (a) EDS of Precipitate 1. (b) EDS of Precipitate 2. (c) EDS of Precipitate 3. (d) EDS of Precipitate 4.
Figure 5. (a) Sample S2 EDS layered image. (b) Sample S2 Element Mapping image.

Figure 6. SEM Image of S1–S5.

Figure 7. Fe–Cr–C phase diagram.
characteristics on creep properties of heat-resistant steel, the unit density $N$ along with average diameters $d$ of Laves, $M_{23}C_6$, and MX phases were calculated. Also, the second phase particle spacing $\lambda$ was calculated according to equation (1) [24]. The results are presented in figure 13.

$$\lambda = 1.25N^{\frac{1}{2}} - \bar{d}$$  \hspace{1cm} (1)

Three mechanisms existed for the second phase strengthening: dislocations bypass of particles through the Orowan mechanism, dislocations climbing over the precipitates (local and global climbing) and dislocation cutting mechanisms. The Orowan stress required for dislocations to bypass particles is [24]:

$$\tau = \frac{Gb}{\lambda}$$  \hspace{1cm} (2)

where, $G$ is the shear modulus (80 GPa), $b$ is the Burgers vector (0.25 nm), and $\lambda$ is the second-phase particle spacing. Figure 14 presents the Orowan stress of Laves phase, MX, and $M_{23}C_6$ for samples S1–S5. The Orowan stress generated by Laves phase exceeded the Orowan stress generated by $M_{23}C_6$ and MX phases. In this work, the external stress of 80 MPa exceeded the Orowan stress value, generated by the precipitates. Therefore the creep was controlled by the Orowan mechanism.
Figure 10. Hardness value of different samples with different element content.

Figure 11. (a) Time-strain creep curves of heat-resistant steel S1, S4, and S5 at 650 °C and 80 MPa. (b) Time-strain rate creep curve in double log scale of heat-resistant steel S1, S4, and S5 at 650 °C and 80 MPa.

Figure 12. (a) Time-strain creep curves of heat-resistant steel S1–S3 at 650 °C and 80 MPa. (b) Time-strain rate creep curve in double log scale of heat-resistant steel S1–S3 at 650 °C and 80 MPa.
Figure 15 presents the dependence of creep properties (creep rupture time and minimum creep rate) of S1, S4 and S5 on the microstructure characteristics of the precipitates ($\lambda$), caused by different Al contents. The change trend of the minimum creep rate and $\lambda$ value was the same, but the change trend of fracture time and $\lambda$ value was opposite. As the Al content increased, the growth rate of Laves, $M_{23}C_6$ and MX phases within ferrite crystal decreased, while the average particle size and unit density of Laves, $M_{23}C_6$ and MX phases decreased. According to equation (1), the second phase particle spacing decreased. Consequently, the Orowan stress increased, which provided an additional obstacle for dislocation movement \[25\], resulting in a decrease in the minimum creep rate (figure 11(b)). Consequently, the creep strength increased.

Figure 16 presents the dependence of S1–S3 creep properties on the microstructure characteristics of the precipitates caused by different C and N contents. The trend of minimum creep rate and $\lambda$ value was the same, but the trend of fracture time and $\lambda$ value was opposite. As the C content increased and the N content decreased, the growth rate of the Laves, $M_{23}C_6$ and MX phases increased significantly, while the average diameter increased. According to equation (1), the second phase particle spacing decreased, while the Orowan stress increased. This provided an additional obstacle for dislocation movement \[25\], resulting in a decrease of the minimum creep rate (figure 12(b)) and creep strength increase.

Compared to $M_{23}C_6$, Laves and MX phases mainly affected the alloy creep strength. Figure 17 presents the relationship among the $\lambda$ values of Laves, $M_{23}C_6$ and MX phase and the minimum creep rate at S1–S5. The
change trend of λ values of Laves and MX phases was similar to the minimum creep rate at S1-S5, but the change trend of λ values of M$_{23}$C$_6$ was the opposite for S3 to S4.

From figures 11(a) and 12(a), it could be observed that the plasticity of the heat-resistant steels differed. Creep plasticity is determined by the formation and growth of micropores (microcracks). The higher the
formation time was, the lower the propagation rate was. Also, the material plasticity was improved [27]. Recent researches [27, 28] demonstrated that when the alloys contain non-metallic inclusions and the second phase or other brittle phases (collectively referred to as heterogeneous phases), the micrometallic inclusions and the second phase or other brittle particles in the alloy became the core of micropores formation (microcracks). In this study, the external stress exceeded the Orowan stress. Dislocations could bypass the particles to reach the grain boundaries through the Orowan mechanism. The hard and brittle phase Laves phases on the grain boundaries inhibited the movement of dislocations, which induced dislocation pileup and cause stress concentration. Therefore, micropores (microcracks) formed, while growing along the grain boundary until fracture. The size and continuous morphology of the precipitates on the grain boundary had different effects on the high temperature endurance strength and toughness of the alloy [29, 30]. Small and discontinuous grain boundary precipitates prevented the grain boundary sliding and increased the creep resistance and ductility, while coarse continuous grain boundary precipitates reduced the ductility and plasticity of the alloy. The ratio of the Laves phase spacing \( \lambda \) to the size \( d \) was used to define the distribution of the Laves phase along the grain boundary, while the ratio was defined as the Laves phase at the grain boundary continuity coefficient:

\[
F_C = \frac{d}{\lambda}
\]

The results are presented in table 2.

Figure 18 presents that the elongation of the heat-resistant steel was inversely proportional to the Laves phase continuity coefficient, which indicated that the Laves phase on the grain boundary was the core of micropores (microcracks). As the Laves phase continuity coefficient decreased, the fewer the formed micropores (microcracks) were. Also, the time, during which, micropores (microcracks) grew and interconnected until fracture was longer. This induced the better plasticity of the heat-resistant steel.

The ferritic heat-resistant steel developed for this study had large crystal grains and irregular Laves precipitates. These reasons led to insufficient strength of heat-resistant steel. The next research step will be the heat treatment conditions adjustment: heat treatment at 600 °C for 10 h to promote nucleation of Laves phase, heat treatment at 700 °C for 10 h to finely and uniformly precipitate Laves phase within the crystal [31], as well as cooling rate increase to refine the grains [32, 33].

![Graph showing the relationship between Laves phase continuity coefficient and elongation of heat-resistant steel.](image)

**Table 2.** Grain boundary Laves phase continuity coefficient \( F_C \), grain boundary Laves phase size \( d \) and spacing \( \lambda \).

| Steels | S1  | S2  | S3  | S4  | S5  |
|--------|-----|-----|-----|-----|-----|
| \( d (\mu m) \) | 1.13 | 1.41 | 1.24 | 1.23 | 1.11 |
| \( \lambda (\mu m) \) | 5.35 | 3.80 | 10.60 | 8.45 | 8.48 |
| \( F_C \) | 0.2116 | 0.3711 | 0.1770 | 0.1456 | 0.1309 |

Figure 18. Relationship between continuity coefficient and elongation of heat-resistant steel.
4. Conclusions

In order to further optimize the high-temperature creep properties of Fe–Cr–Al–Nb–W ferritic heat-resistant steel, C, N and Al were added. The microstructure of new Fe–Cr–Al–Nb–W heat-resistant steel with different C, N and Al contents was observed, while creep testing was carried out, to analyze the relationship between element content, microstructure and creep properties. The following conclusions are drawn:

(1) The matrix of Fe–Cr–Al–Nb–W steel with C, N and Al addition was ferrite, while three types of precipitation phases existed: Laves phases, M23C6 and MX phases. M23C6 and MX precipitated at grain interior, Laves phases precipitated at grain interior, grain boundary and around MX phases.

(2) The ferritic steel hardness was not affected by contents of C and N, but it was affected by Al content. As the Al content increased, the hardness of heat-resistant steel increased first and subsequently decreased. When the Al content was 2%, the hardness as the lowest.

(3) C, N and Al affected microstructure and creep of heat-resistant steel. As the Al content increased or as N content decrease and C element increased, the average diameter of the Laves, M23C6 and MX phases at grain interior decreased, the unit density increased and the phase spacing decreased. This led to the minimum creep rate decrease and creep rupture time increase.

(4) Compared to M23C6, Laves and MX phases mainly affected the alloy creep strength.

(5) The continuous coefficient $F_C = d/\lambda$ decrease of the Laves phase at the grain boundary was beneficial to the plasticity improvement of the heat-resistant steel.

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