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Microstructure and creep strength evolution in G115 steel during creep at 650 °C

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Abstract

Microstructure and creep strength evolution in the ruptured 2.6 wt% W G115 specimens after creep tests were investigated under stresses of 120–200 MPa at 650 °C. The growth of the lath during the creep tests was accompanied with the coarsening of second phase particles and the reduction of dislocation density. The tempered martensitic lath structure (TMLS) transformed into polygonal subgrain structure when the pinning force due to the precipitates was lower than about 0.68 MPa. The Laves phase particles were irregular during the short-term creep and grew to be equiaxed shape after long-term creep.

1. Introduction

In order to raise the service temperature of martensitic steel to 650 °C, to improve the stream efficiency of coal-fired plants and reduce coal consumption, Liu’s team has developed 9Cr-3W-3Co G115 steels in recent years [1–3]. This steel is typical martensitic steel with a great amount of dislocation and precipitates [3–7]. The precipitates in this steel are M 23 C 6 with mean size of 90–300 nm, MX (20–50 nm), and Laves phase (LP) particles (100–400 nm), respectively. M 23 C 6 and LP particles are mainly distributed at high-angle boundaries (HABs), while the MX particles are located homogeneously in the matrix [8–10]. M 23 C 6 and MX particles precipitate in the tempered state, while the LP particles precipitate during the following aging [11, 12]. Because of these particles pinning dislocation and preventing lath from migrating, martensitic lath structure is very stable. However, with the increase of service time at high temperature, these particles will gradually coarsen, and their pinning ability to the dislocation will weaken, leading to the growth of lath and the decrease of dislocation density [13–15]. Thus the strength of the steel decreased.

The structure evolution of these G115 steels at high temperature has been studied by other researchers [16, 17]. The coarsening of M 23 C 6 and LP particles are responsible for the degrading of high temperature properties of the G115 steels. The composition of this 2.6 wt% W G115-type steel is shown in table 1. Cu has a low solid solubility in martensitic steel and tends to exist in the form of a Cu-rich phase. The addition of Cu acts to precipitate strengthening and improve the resistance to high temperature steam corrosion. The concentration of Cu in this G115 steel is 0.94 wt%. As the amount of Cu-rich phase is very small, it is not the focus of this paper.

According to the researches by Fang L, Boron was enriched in the M 23 C 6 particles during aging and creep, which reduced the coarsening rate of M 23 C 6 particles and improved the stability of the martensitic microstructure [6]. So the concentration of the Boron in this steel is relatively high with the value of 150 ppm as shown in table 1. The main composition of LP particles is Fe 2 W, the precipitation of which will consume W from the matrix. As reported by the researcher Liu [7], the coarsening rate of LP particles increased with the increase of W concentration in G115 steels during aging. Therefore, the W concentration in the experimental steel was adjusted to 2.6 wt%. The aim of this article is to elucidate the microstructure evolution and find the reasons that
affect this evolution during the creep test in G115 steels. Creep tests were conducted at 650 °C under 120∼200 MPa and the microstructure and creep strength evolution have been investigated.

2. Experimental procedure

The steel was casted and hot forged into a rod at Bao Steel Company. The specimens for the experiments were taken from the rod. They were heat-treated before tests, normalizing at 1100 °C for 1 h, cooled by air, and subsequently tempering at 780 °C for 3 h, cooled by air [7, 16]. The specimens were machined in the standard size: 25 mm in a gauge length and Φ5 mm in cross section. All of the specimens were tested at 650 °C with a constant load until rupture. The creep tests were conducted under the stresses of 120∼200 MPa in steps of 20 MPa. The schematic diagram of fractured specimens was analyzed as shown in figure 1. The focus of this paper is on the grip portions and the neck portions.

For the purpose of observing the comprehensive microstructure of the G115 steel, another tempered specimen was taken and observed after aging at 650 °C for 100 h to ensure that parts of LP particles has precipitated. The microstructure of this specimen would be discussed in section 3.1. Microstructural characterization of laths and precipitates was observed in the grip and neck portions of ruptured specimens by field emission scanning electron microscopy (FESEM) and on thin foils using TECNAIG220 field emission transmission electron microscope (FETEM). The lath and sub-grain widths were statistically counted in the TEM images covering total areas over 200 um². More than 150 laths or sub-grains were measured and the average values of them were recorded.

Dislocation density in this paper was obtained by XRD measurement. This method was proven to be effective by the researches [18, 19]. This method was described in detail in the paper by Liu [7]. The number density of precipitates distributed at HABs was measured as the number of precipitates for each unit boundary length. Using the Thermo-Calc software, the volume fractions of the precipitates could be obtained.

For measurement of precipitates sizes, 134 to 338 particles were measured for each sample. The mean diameter and the size distribution of the M₂₃C₆ and LP particles were measured by Image-Pro Plus 6.0 (IPP 6.0) software in 20 TEM images, magnification for 20,000 times, corresponding to over 100 particles for each precipitates. The M₂₃C₆, MX and LP particles were distinguished by the selected area electron diffraction (SAED) pattern by TEM.

3. Results

3.1. Microstructure of G115 steel

The microstructure in TEM images of the specimens aged at 650 °C for 100 h was shown in figure 2. There were three kinds of precipitates observed within the TMLS. They were MX, M₂₃C₆ and LP particles. With the elongation of aging, MX, M₂₃C₆ and LP particles showed the tendency of coarsening. The mean sizes of these precipitates were shown in table 2. The size of the smallest M₂₃C₆ and LP particles was 70∼90 nm, while the
maximum size of MX particles didn’t not exceed 35 nm. It was easy for MX particles to be distinguished with the other two particles. The microstructure and precipitates were shown in figure 2. The precipitates were MX, M23C6 and LP particles, respectively. The M23C6 and MX particles were mainly equiaxed shape while Laves phase particles were mainly irregular, which was in accordance with the research by Yan [3].

### 3.2. The creep rate versus time curves of the specimens

Figure 3 displayed the creep rate versus time curves obtained during tests under the stresses from 120~200 MPa. The curves were divided into two parts: the transient creep region and the acceleration creep region. The transient creep region represented the creep rate decreasing slowly; and the acceleration creep region represented the creep rate increasing quickly after reaching a minimum creep stage. The minimal creep rate increased from approximately $10^{-9} \text{ s}^{-1}$ to $10^{-6} \text{ s}^{-1}$ when the applied stresses rose from 120~200 MPa. With the increase of stress, the creep rate of the specimens tended to increase, while the time to rupture tended to decrease. The creep rupture times were 15187 h (120 MPa), 8792 h (140 MPa), 4082 h (160 MPa), 659 h (180 MPa) and 101 h (200 MPa), respectively.

### 3.3. Microstructure observations in the grip portions

Figure 4 showed the lath structure in the grip portions of the specimens as a function of aging. After aging for 101 h and 4082 h, the lath width increased in the grip portions compared with the initial state. The lath width
increased from 0.30 ± 0.01 μm in the initial state to 0.41 ± 0.01 μm after short-term aging for 4082 h and almost no polygonal sub-grains growth was found (table 2, figure 4(a)). It should be stressed that the martensitic lath was bundle-shaped, and the sub-grain structure was polygonal in shape. However, about 20~30% of martensitic laths have transformed to sub-grains with the average size of about 0.50 ± 0.05 μm after long-term aging for 8792 h to 15187 h (table 2, figure 4(c)). The lath widths increased from original 0.30 ± 0.01 μm to
0.49 ± 0.05 μm and 0.52 ± 0.05 μm after aging for 8792 h and 15187 h, respectively. The dislocation density decreased from 2.7 × 10^{14} \text{ m}^{-2} to 1.5 × 10^{14} \text{ m}^{-2} after aging for 15187 h.

Figure 5 showed the size distribution of M23C6 and LP particles in the grip portions aged for different time at 650 °C. The height of the histogram indicated the proportion of the precipitates in the range of sizes with the steps of 20 nm (figure 5(a)) and 30 nm (figure 5(b)) respectively. M23C6 particles have existed since the tempered state and they distributed mainly at boundaries such as prior austenite, packets and block boundaries (figure 4). About 80% of M23C6 particles distributed at the high-angle boundaries (HABs) and the volume fraction of M23C6 particles was 1.7% from Thermo-Calc software. The initial mean size of M23C6 particles was 98 nm and they would coarsen when exposed at high temperature of 650 °C. With the elongation of aging, the proportion of small particles with size of 70∼130 nm decreased, and the proportion of large particles with size of 150∼210 nm increased (figure 5(a)). The final mean size of M23C6 particles was 235 ± 2 nm after aging for 15187 h in the grip portions. High temperature accelerated the diffusion of carbon atoms and other atoms, making the M23C6 particles gradually coarsen.

The distribution of LP particles in the grip portions after aging for different time was shown in figure 5(b). Above 80% of LP particles precipitated at the HABs, which was similar to the distribution of M23C6 particles. It should be stressed that the LP particles were mainly irregular after short-term aging. With the elongation of aging, the proportion of large particles increased gradually, while the proportion of small particles gradually decreased (figure 5(b)). Finally, the Laves phase particles grew to 263 ± 3 nm when exposed at 650 °C for 15187 h and many of them become equiaxed.

The mean sizes of MX particles were 20 ± 1 nm before the thermal aging time of 659 h and they grew to 25 ± 1 nm after aging for 15187 h. This indicated that MX particles would coarsen during long-term aging. They were uniformly distributed in the martensitic matrix and prevent the migration of dislocation.

3.4. Fractography

Figure 6 showed the morphology of the rupture surface of specimens, which was strongly depended on the applied stress. The dimples of the fracture were mostly even, large and deep at 200 MPa (figure 6(a)), while the dimples under 120 MPa were mostly inhomogeneous, small and shallow (figure 6(b)). The microporous aggregation and fracture process was divided into three stages: cavity nucleation, growth and merging. The size of dimples was closely related to the plasticity of materials. As shown in figure 7, with the stresses decreased from 200 MPa to 120 MPa, the reduction of area of specimens decreased from 78% to 29% and the elongation dropped from 22% to 7.4%.

The plastic deformation was severe under high stress, which would consume more energy and produce larger dimples. It should be stressed that coarse secondary phase particles often acted as nucleation sites for dimples (figures 6(a), (b)). These particles mainly consist of two types: M23C6 and LP particles. The coarsened secondary phase particles often served as nucleation sites for dimples.

3.5. Microstructural evolution in the neck portions

The microstructure evolution in the neck portions showed significant difference between short-term and long-term tests. Some laths structure transformed into sub-grain structure in the neck portions after long-term creep.
The proportion of laths transformed to sub-grains increased under low-stresses of 140∼120 MPa, reaching to about 40∼50% (figure 8).

Compared with the grip portions, the growth rate of lath was bigger and the laths transformed to sub-grains easier at the neck portions. At high-stresses of 200∼160 MPa, the lath width has increased by 1∼7% in comparison with the aging specimens. The proportion of sub-grains increased at the expense of the laths and the growth of sub-grains/laths was often accompanied by the annihilation of internal dislocations. The dislocation density in the neck portions decreased more rapidly as shown in table 2. The dislocation density decreased over 2 times relative to the tempering state when specimens ruptured at the stress of 120 MPa. Under the stress of 120 MPa, the rupture time was 15187 h. This long-term rupture time led to fully developed sub-grains with the size of 0.71 ± 0.05 μm and the laths with the width of 0.59 ± 0.05 μm.

Figure 8 showed the microstructure of steel in the neck portions under 200 MPa, 160 MPa and 120 MPa at 650 °C. From the pictures of (figures 8(a), (c), (e)), some results can be concluded. Figure 8(a) showed elongated microstructure because large deformation occurred in the neck portion due to high reduction of area. On the other hand, figure 8(e) showed equiaxed microstructure since deformation was small in the neck portion due to low ductility. Figures 8(f) showed that the secondary phase particles in HABs were larger than those located at other sites. The reason for this phenomenon was that atoms at HABs tended to rapid diffusion along these boundaries.

Figure 9 showed the size distribution of M23C6 and LP particles in the neck portions. The height of the histogram indicated the proportion of the precipitates in the range of sizes with the step of 20 nm (figure 9(a)) and 30 nm (figure 9(b)). In the neck portions, the majority of M23C6 and LP particles were located at HABs,
which was similar to the distribution of these precipitates in the grip portions. However, as they were induced by the stresses in the neck portions, both of these two precipitates coarsen faster than those in the grip portions. When specimens ruptured at high stresses of 200–160 MPa, the M$_{23}$C$_{6}$ particles with 80 nm still existed. However, when the samples ruptured under the low stresses of 140–120 MPa, the smallest size of M$_{23}$C$_{6}$ particles was 100 nm. With the increase of rupture time, the proportion of small particles with size of 80–140 nm decreased, and the proportion of large particles with size of 180–300 nm increased. At 120 MPa, the mean size of M$_{23}$C$_{6}$ particles has increased 22% in comparison with the same particles in grip portions, which could be the direct evidence for stress-induced M$_{23}$C$_{6}$ particles coarsening.

The coarsening rule of LP particles in the neck portions was similar to M$_{23}$C$_{6}$ particles. With the extension of rupture time, the proportion of large particles with size of 240–320 nm increased at the expense of small

![Figure 8. Microstructure of specimens in the neck portions at 650 °C and stresses, (a) (b) 200 MPa, (c) (d) 160 MPa, (e) (f) 120 MPa, (a) (c) (e) SEM, (b) (d) (f) TEM.](image-url)
particles with size of 100–160 nm (figure 9(b)). At 120 MPa, the mean size of LP particles had increased 17% in comparison with the same particles in grip portions. Under the stresses of 200–160 MPa, LP particles were mainly irregular (figure 8(b)). While under the stress of 140–120 MPa, LP particles were mainly equiaxed shape (figure 8(f)). Equalization of interfacial energy between particles (and within particles as well) was the driving force for coarsening. The Laves phase particles could be seemed as consisting of one or several grains. The coarsening of the particles was actually the growth of the internal crystal grains. When the grain grew, the grain boundary always moved toward the center of curvature and was continuously flattened. Therefore, the process of grain growth was the process of ‘big swallowing small’ and flattening the concave surface. Therefore, after a long-term aging, the LP particles became equiaxed from the initial irregular shape.

What’s more, the sizes of MX particles in the neck portions were the same with those in the grip portions. So the stress had no effect on the coarsening of MX particles. Figures 10(a), (b) showed the number density of second particles including M23C6 and LP particles. With the elongation of rupture time, the number density of two kinds of particles decreased and the decrease scale in the neck portions was larger than that in the grip portions, which stemmed from the theory that stress induced the growth of the secondary particles. Some LP particles distributed nearby the M23C6 particles as shown in figure 2(a). As explained by the researchers [18, 19], these LP particles would swallow the nearby M23C6 particles during their growth. The faster the coarsening rate of the particles was, the faster the number density of the particles would decrease.

Figure 10(c) showed the number density of MX particles in grip and neck portions. With the elongation of aging, the number density gradually decreased and there was almost no difference between the grip portions and neck portions.

4. Discussion

In order to reveal the reason why the laths transformed into sub-grains, it was very important to calculate the pinning force generated by the precipitates. The pinning force $P_z$ due to MX particles could be obtained as [20]:

$$P_z = \frac{3\gamma F_v}{d}$$

$d$ was size of particles and $F_v$ was the volume fraction. Through the Thermo-Calc software, the volume fraction of MX particles was 0.23%. $\gamma$ was the boundary surface energy per unit area, which was calculated from the Read and Shockley equation:

$$\gamma = Gb(A - \ln \theta) / 4\pi(1 - \nu)$$

$G$ and $b$ were the shear modulus (64 GPa at 650 °C) and Burgers vector (0.25 nm), respectively. The term $A = 0.45$ [17], $\theta = 1$ degree, $\nu$ was the Poisson ratio ($\nu = 0.25$).
The pinning force from M\textsubscript{23}C\textsubscript{6} and LP particles distributed at the boundaries could be calculated as:

\[ P_B = \frac{E_B D}{d^2} \]  

D was the lath/sub-grains width. \( E_B \) the volume fraction of boundary particles, which was calculated by the boundary particle density \( \beta \) in figures 9(a) and (b). \( E_{M23C6} \) and \( E_{Laves} \) were calculated by Thermo-Calc, 1.7% and 1.8%, respectively, during long-term aging. Assume that all M\textsubscript{23}C\textsubscript{6} and LP particles were located at boundaries.

\[ E_{M23C6} = E_{BM23C6} \]  
\[ E_{Laves} = E_{BLaves} \]

According to the research by Fedoseeva and Dudova [21, 22], there was an equation among the density ratio of M\textsubscript{23}C\textsubscript{6} and LP particles on boundaries, their sizes \( d_{M23C6} \) and \( d_{Laves} \) and their corresponding volume fractions.

\[ \frac{E_{Laves}}{E_{M23C6}} = \frac{d_{Laves}}{d_{M23C6}} \]  

\( E_{B} \) of the M\textsubscript{23}C\textsubscript{6} and LP particles distributed at the boundaries under different stresses could be obtained from the formula below.

\[ \frac{E_{B}}{E_{B0}} = \frac{D_0}{D_i} \left( \frac{\beta_i}{\beta_0} \right) \left( \frac{d_i^2}{d_0^2} \right) \]  

\( E_{B0}, D_0, \beta_0, d_0 \) were the parameters for the M\textsubscript{23}C\textsubscript{6} or LP particles in the initial state, and \( E_{Bis}, D_i, \beta_i, d_i \) were the parameters of specimens at different stresses. Because the Thermo-Calc software could only calculate the volume fraction of precipitates after long time aging, the volume fraction of precipitates under different stresses should be calculated by above formulas. As 80% M\textsubscript{23}C\textsubscript{6} and Laves phase particles were located at HABs, the \( P_{BLaves} \) and \( P_{BM23C6} \) could be calculated by:

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Figure 10. Particle densities of precipitates during aging and creep test (a) M\textsubscript{23}C\textsubscript{6} and LP particles in grip portion and (b) M\textsubscript{23}C\textsubscript{6} and LP particles in neck portion (c) MX in grip and neck portion.
The pinning force from all precipitates could be calculated as follows:

\[ D = 0.8D_{\text{HAB}} + 0.2D_{\text{lath}} \]  
\[ D_{\text{HAB}} = 2.5D_{\text{lath}} \]  
\[ P_{\text{Blaves}} = 0.9P_{\text{Blaves}} + 0.1P_{\text{ZLaves}} \]  
\[ P_{\text{BM_{23}C_{6}}} = 0.8P_{\text{BM_{23}C_{6}}} + 0.2P_{\text{ZM_{2}C_{6}}} \]

The pinning force from all precipitates could be calculated as follows:

\[ P_{1} = P_{Z} + P_{\text{BM_{23}C_{6}}} + P_{\text{Blaves}} \]

Figure 11 showed the evolution of the pinning force (from the precipitates) at 650 °C and 120~200 MPa. The pinning force in the grip portion gradually decreased with the elongation of the aging, from 1.41 MPa at 101 h to 0.55 MPa at 15187 h. When the aging time arrived 8792 h, the pinning force reduced to 0.66 MPa, and some bundled martensitic laths transformed into irregular polygonal sub-grains. With the aging time increase, the lath and sub-grain structure coarsened, and the proportion of sub-grain increased. When the specimens ruptured at 120 MPa, the aging time reached 15187 h, the proportion of the sub-grain structure increased to nearly 30%, and their average size reached 0.58 ± 0.05 μm.

A similar phenomenon was observed in the neck portion. At 200 MPa, the pinning force derived from the precipitates was 1.39 MPa, and no sub-grain structure was observed. With the stresses in the neck portion reducing from 200 MPa to 120 MPa, the pinning force generated by the precipitates gradually decreased. In the neck portion of the ruptured specimen at 140 MPa, it could be observed that part of the laths had transformed into sub-grains, and the pinning force from the precipitates was 0.60 MPa. With the stress applied to the specimen decreasing, the sub-grain structure at the neck portion increased gradually. The proportion of the sub-grain structure increased to 40~50% in the neck portion ruptured at 120 MPa, and their average size was 0.71 ± 0.05 μm.

M_{23}C_{6} and LP particles in G115 steel mainly existed at HABs such as the boundaries of packet, block and lath bundles. In the short-term aging, the precipitates were finer, which could effectively hinder the dislocation recovery and the lath boundary migration. As the aging time increased, the precipitates gradually coarsened, and their number density decreased. The pinning force generated by the precipitates reduced, the pinning effect on the HABs weakened, and the martensitic lath bundle could not be effectively fixed. According to the results of this experiment combined with the calculation of the pinning force, the martensitic lath structure in the steel would transform into the polygonal sub-grain structure when the pinning force reduced to lower than about 0.68 MPa during the creep process.

5. Conclusions

The creep strength and microstructures evolution of 2.6 wt% W G115 steel crept at different stresses were investigated. Some main results could be obtained:
1. The microstructure was mainly composed of martensitic lath and three kinds of precipitates: M$_{23}$C$_6$, MX and LP particles. M$_{23}$C$_6$ and MX particles were mainly equiaxed shape, while LP particles were irregular during the short-term aging or creep test. After long-term aging or creep test, the LP particles would transform into equiaxed shape.

2. The precipitates and lath would coarsen and the dislocation density would reduce after the creep test. The number density of precipitates would decrease after the creep test. The mean sizes of M$_{23}$C$_6$, LP particles and lath in the neck portions were larger than those in the grip portions. The dislocation density in the neck portions was smaller than that in the grip portions.

3. The pinning force from precipitates had a major impact in the stabilization of the TMLS. The transformation of lath into sub-grain structure occurred when the pinning force was lower than about 0.68 MPa in this 2.6 wt% W G115-type steel.

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