Research Article

Microstructural Evolution during Thermal Exposure of G115 Steel and Its Influence on Mechanical Properties

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Microstructural evolution and mechanical property degradation of the newly developed G115 steel during thermal exposure are highly important for the safe power plant operation. In this paper, the microstructural stability and strength as well as impact toughness were investigated after aging at 650 °C and 700 °C up to 5000 h. It was found that when aged at 650 °C in the G115 steel, the size of $\text{M}_{23}\text{C}_6$ carbides and the width of martensitic laths had little change even increasing aging time up to 5000 h. Both the room-temperature and high-temperature strengths remained stable after aging for 5000 h. In comparison, after aging at 700 °C, high-density Laves phases precipitated accompanied by coarsening with increasing aging time. The majority of martensitic lath structure was replaced by sub-grains and the strength decreased linearly. Most importantly, the impact toughness of G115 steel aged at both 650 °C and 700 °C decreased sharply and embrittlement occurred rapidly in the initial aging period. The evidence provided by fracture morphologies proved that Laves phase was the culprit of decreasing the impact toughness.

1. Introduction

The 9-12Cr martensite heat-resistant steel is deemed as the most common structural material in ultra-super-critical (USC) power plants for its advantages such as high thermal conductivity, low thermal expansivity, and excellent anti-fatigue performance [1]. Increase of the steam temperature and pressure is essential to realize the efficient utilization of fossil energy sustainable development. Service temperature of commercial 9-12Cr heat-resistant steel restricts the increase of operating parameter of USC power plants. Under this background, G115 steel was developed in China for the USC power plants aiming to improve the service temperature to 630 °C and above [2, 3].

Higher service temperature requires higher micro-structural stability and higher creep rupture strength. G115 steel was made for this purpose. It is one of such 9-12Cr martensitic heat-resistant steels, in particular one member of the 9Cr3W3Co steel family. As compared to the 9Cr3W3Co steel which was firstly developed in Japan, the contents of B and N have been optimized [4]. B is a key alloying element in G115 steel. It is believed that a proper amount of B contributes to the creep rupture strength [5]. B prefers to segregate to grain boundaries after normalizing and then enters into $\text{M}_{23}\text{C}_6$ carbides and forms B-bearing carbides [6–12]. Consequently, the coarsening rate of $\text{M}_{23}\text{C}_6$ carbides is suppressed, leading to improvement of microstructural stability and creep rupture strength. Besides, another important alloying element Cu was added into G115 steel to further enhance precipitation strengthening [13]. Moreover, some microalloying elements, e.g., V and Nb, were also included, which enable the formation of stable nano-sized precipitates for improving the creep strength. Most importantly, completely tempered martensite can be obtained by adding 3% W and 3% Co which can also strengthen the matrix by acting as solute elements [14]. Therefore, G115 steel is expected to be used in USC power plants above 630 °C.

Nowadays, extensive studies have been conducted on G115 steel, such as the effect of key chemical composition on microstructure and mechanical properties [4, 15, 16], creep behaviour [3, 5, 17–19], microstructural evolution and mechanical property degradation after long-term aging [20, 21], critical element segregation behaviour during...
thermal exposure [22–24], and so on. However, as a new developed steel, lots of experimental work must be done before practical engineering application. The experimental data are still far from enough. Therefore, the present study aims to investigate the microstructural evolution during the aging of G115 steel and its influence on mechanical properties. Emphasis is placed on the microstructural stability and strength as well as impact toughness after different aging temperatures. In this study, the direct reason of the sudden decline of impact toughness during aging was observed. Furthermore, the present work will also provide more support data for the practical engineering application of G115 steel.

2. Materials and Methods

The G115 steel used in this study was produced by Shanghai Changqiang Industrial Technology Co., Ltd., China, according to T/CISA 003-2017 standard. The main chemical compositions of the steel were analysed by the Q8 photoelectric direct reading spectrometer (Bruker). Particularly, C and S were analysed by the CS600 carbon sulfur analyser (LECO) and B was analysed by the Agilent-5100 inductively coupled plasma emission spectrometer (Agilent). The results are listed in Table 1. It can be seen that all the elements are within the standard range. The G115 steel was melted in a 100 kg vacuum induction melting furnace. The steel ingot was austenitized at 1150°C for 4h and then forged into several plates with size of 400 mm × 200 mm × 40 mm. The initial forging temperature is about 1100°C, and finished temperature is about 950°C. Before the plate delivery, heat treatment consisting of normalizing at 1100°C for 195 min and tempering at 780°C for 390 min was carried out. The schematic diagram of heat treatment process is shown in Figure 1.

Several blocks cut from the steel plate were subjected to thermal exposure at 650°C and 700°C for different times of 0, 500, 1000, 3000, and 5000 h, respectively. After that, samples with the dimension of 10 mm × 10 mm × 10 mm were cut out and mechanically ground to 2000# with sand papers, polished, and then etched in Vilella’s reagent (1% trinitrophenol, 5% alcohol hydrochloric acid, and 94% alcohol). Microstructural evolution of experimental steel was observed using an optical microscope (OM), scanning electron microscope (SEM), and transmission electron microscope (TEM). The SEM of FEI Quanta 600 equipped with an energy-dispersive analysis system (EDS) was used to study the Laves phase evolution during aging at different temperatures. For the TEM observation, 400μm thick discs cut from the metallographic samples were first mechanically thinned to about 50μm thick foils and then electropolished at −25°C by a 15 V twin-jet electropolisher in a solution of 10% perchloric acid and 90% glacial acetic acid.

Tensile and impact specimens were all cut from the aging blocks. They were machined into tensile test specimens of 5 mm in gage diameter and 25 mm in gage length and impact specimens of 10 mm × 10 mm × 55 mm containing a 2 mm long 45° V-notch with a root radius of 0.25 mm. The tensile tests were conducted at 630°C, 650°C, 675°C, and room temperature according to GB/T 228.1 specification. To ensure the reliability of the tensile results, three specimens were tested to obtain the average value. The errors in the yield strength and tensile strength measurement were about ±3%. Charpy impact tests were carried out at room temperature according to GB/T 229 standard. The average of three results was recorded as the impact value for the specimens. The fracture morphology of the Charpy impact specimens was observed by the SEM.

3. Results

3.1. Microstructure Characterization of G115 Steel under As-Received Condition. The microstructure of as-received G115 steel is shown in Figure 2. The normalizing and tempering heat treatment made the steel show a typical martensite matrix (Figure 2(a)) and precipitates distributed along grain boundaries (Figure 2(b)) and lath boundaries (Figure 2(c)). The width of martensitic lath varied in the range of 100–400 nm, and its average width was about 250 nm (Figure 2(c)). The precipitates decorated the prior austenite grain boundaries, and lath boundaries were M23C6 carbides. They were confirmed by EDS (Figure 2(b)) and selected area electron diffraction (Figure 2(c)). It is worth noting that no Laves phase was found in the as-received steel.

3.2. Microstructural Evolution during Aging. The OM microstructure of the evolution of G115 steel aged at 650°C and 700°C for different times is shown in Figure 3. After aging at 650°C, the tempered martensite matrix showed nearly no change. Even aging up to 5000 h, the microstructure still remained the initial martensitic structure, and the prior austenite grain boundaries and packets were still clear. When the aging temperature went up to 700°C, the martensitic lath did not change obviously within 1000 h, and a clear martensitic lath structure could still be observed. After aging up to 3000 h, the microstructural stability of G115 steel was greatly damaged, and the martensitic lath boundaries were no longer visible clearly. At this stage, a large amount of martensite was broken into sub-grains, and dislocation cells were formed. With increasing aging time to 5000 h, the OM image was basically the same as that of 3000 h. It is worth noting that the prior austenite grain boundaries without martensitic lath still existed. Moreover, coarsened precipitates could be observed in the vicinity of grain boundaries. It is reasonable to speculate from the composition of G115 steel and the long-term microstructural evolution characteristics of 9–12Cr martensitic steel that they were coarsened M23C6 carbides and Laves phases.

Figure 4 traces the tiny microstructural evolution of G115 steel after aging at 650°C and 700°C for different times. It can be seen in Figures 4(a)–4(c) that the tempered martensitic lath structure was still clear when aging at 650°C, even for 5000 h. Rod-like M23C6 carbides, not excluding Laves phase, could be observed in the vicinity of both prior austenite grain boundaries and lath boundaries. Compared with as-received condition in Figure 2(c), martensitic lath got slightly wider with increasing aging time. The width of
Table 1: Chemical compositions of the G115 steel.

| Elements | C    | Si   | Mn   | P     | S    | Cr   | W    | Co   | V    | Cu   | Nb  | N   | B    | Fe   |
|----------|------|------|------|-------|------|------|------|------|------|------|-----|-----|------|------|
| In this study | 0.079 | 0.31 | 0.47 | 0.006 | 0.002 | 8.90 | 2.79 | 2.98 | 0.19 | 0.87 | 0.063 | 0.01 | 0.0135 | Bal. |
| Standard  | 0.06–0.10 | ≤0.55 | 0.27–0.73 | ≤0.02 | ≤0.01 | 8.4–9.6 | 2.33–3.17 | 2.8–3.25 | 0.13–0.27 | 0.4–1.2 | 0.03–0.1 | 0.005–0.015 | 0.01–0.022 | Bal. |
Martensitic lath increased from 0.25 \( \mu \text{m} \) to 0.5 \( \mu \text{m} \). At this temperature, the motion of dislocations was significantly improved. With aging time, dislocations moved and rearranged to form dislocation cells, and some small-scale changes, such as dislocation annihilation, occurred. Finally, the dislocation density decreased. It is worth noting that when aging at 650 °C, M\(_{23}\)C\(_6\) carbides both on grain boundaries and lath boundaries did not coarsen obviously, and needle-like Laves phase can be detected on grain boundaries (Figure 4(c)). So, the matrix showed good microstructural stability. This could be somehow attributed to the pinning effect of the M\(_{23}\)C\(_6\) carbides along the grain boundaries. However, when aging at 700 °C, the width of martensitic lath reached about 0.5 \( \mu \text{m} \) only aging for 1000 h. The lath width was almost the same as that aged at 650 °C for 5000 h. Moreover, dislocation networks formed by dislocation rearrangement could be observed in some regions. After aging up to 3000 h, the regular martensitic lath was destroyed and the majority of them were replaced by sub-grains. With aging time up to 5000 h, the number of sub-grains increased further and the size of precipitates (M\(_{23}\)C\(_6\) and Laves phase) coarsened significantly. On high-temperature thermal exposure, the widening of martensitic lath and the formation of sub-grain were mainly result from Ostwald ripening of precipitates [1]. The M\(_{23}\)C\(_6\) carbides play an important role in the microstructural stability. As mentioned above, B is a critical alloying element in G115 steel. It can suppress the coarsening of M\(_{23}\)C\(_6\) carbides. However, as a light weighing element, B is very difficult to detect. It needs to be explored by using high-end microscopy.
such as TEM coupled with energy electron loss spectroscopy (EELS). Only small-sized $\text{M}_2\text{C}_6$ carbides have a more effective effect on hindering grain boundary and sub-grain boundary as well as dislocation migration, so that the steel can maintain a stable microstructure during long-term aging. For aging at 700°C, $\text{M}_2\text{C}_6$ carbides grew rapidly (Figure 4), resulting in a sharp decrease in pinning effect. When the pinning force of $\text{M}_2\text{C}_6$ carbides was lower than the driving force of grain boundary migration, the martensitic lath boundary would be relaxed. At this time, the migration of lath boundary was no longer hindered by $\text{M}_2\text{C}_6$ carbides, and the lath boundary would be widened or merged to be sub-grains. Therefore, it can be summarized that microstructural instability could take place in a short time at 700°C for G115 steel.

$\text{M}_2\text{C}_6$ carbide, MX carbonitride, and Laves phase are three important precipitates in 9-12Cr martensitic heat-resistant steel during long-term aging [25]. The size of MX

Figure 3: OM images of the evolution of G115 steel aged at 650°C (a–d) and 700°C (e–h) for (a, e) 500 h, (b, f) 1000 h, (c, g) 3000 h, and (d, h) 5000 h.
phase was usually less than 50 nm [26], which was easy to
distinguish from the other two. M$_{23}$C$_6$ carbide and Laves
phase have little difference in morphology on secondary
electron (SE) image, so they need to be distinguished in
back-scattered electron (BSE) mode. For BSE mode in SEM,
the contrast of the precipitates was directly proportional
to the atomic number [27]. In the present steel, one of the main
compositions of Laves phase is W, while Cr is the main
element of M$_{23}$C$_6$ carbide. As W is heavier than Cr in atom
weight, Laves phase is brighter than M$_{23}$C$_6$ in the BSE
images. This characteristic is helpful in identifying the Laves
phase particles. Besides, Laves phase is an AB$_2$ type inter-
metallic compound, which is a hexagonal close-packed
structure composed of two atoms with different sizes [1]. In
general, they are usually presented as large black rectangular
particles with one side closely attached to lath boundaries,
grain boundaries, or M$_{23}$C$_6$ carbides. According to Figure 5,
the chemical composition of Laves phase in G115 steel
consisted of Cr, Fe, V, Nb, and W, and it was basically stable
during aging.

Figure 6 shows the evolution behaviour of Laves phase in
G115 steel during aging at 650°C and 700°C. No Laves phase
was found in the experimental steel at as-received condition,
indicating Laves phase nucleated should be guaranteed a
certain incubation period. The incubation period usually
depended on the composition of matrix and aging tem-
perature. The research results of Saini et al. [28] showed that
Laves phase was presented in P92 steel after the aging of
720 h at 650°C. Due to the high content of W and Co in G115
steel, Laves phase has been observed after aging at 650°C for
500 h (Figures 5 and 6(a)). It can be inferred that the incu-
bation time of Laves phase in G115 steel at 650°C may be
earlier than 500 h. Yan et al. [21] reported that Laves phase
appeared in 9Cr3W3Co martensitic steel at 650°C only for
300 h. However, there are some differences in the evolution
behaviour of Laves phase at different aging temperatures. In
the present study, after aging for 500 h at 650°C, only a small
amount of fine Laves phase precipitated along the grain
boundaries and lath boundaries, and its growth rate was
slow. Till aging for 3000 h, their size was basically not
changed. When the aging time was extended to 5000 h, the
number density of Laves phase increased significantly, and
M$_{23}$C$_6$ and Laves phase overlapped together. It is reasonable
to believe that with the aging time, Laves phase first pre-
cipitated mainly along the prior austenite grain boundaries
and lath boundaries. When the aging time is long enough
(such as 5000 h), they precipitated not only along the grain
boundaries but also attached to M$_{23}$C$_6$, resulting in a sig-
nificant increase in number density (Figure 6(d)). Never-
theless, the sizes of M$_{23}$C$_6$ and Laves phase of G115 steel

![Figure 4: Typical TEM micrographs of G115 steel after aging at 650°C (a–c) and 700°C (d–f) for different times: (a, d) 1000 h; (b, e) 3000 h; (c, f) 5000 h.](image-url)
Figure 5: BSE image of G115 steel after aging at 650°C for 500 h and its EDS result.

Figure 6: Typical SEM images of G115 steel after aging at 650°C (a–d) and 700°C (e–h) for different times: (a, e) 500 h; (b, f) 1000 h; (c, g) 3000 h; (d, h) 5000 h.
were still small after aging at 650°C for 5000 h, and there is no obvious coarsening tendency. With increasing aging temperature from 650°C to 700°C, Laves phase precipitated in large quantities after aging just for 500 h. In contrast, the number density and size of Laves phase were enough to exceed the level of aging at 650°C for 5000 h, and the former decreased and the latter increased with aging time. Till aging for 5000 h, the size increased to about 1.5 μm. It can be seen that the precipitation and coarsening of Laves phase were promoted at a temperature of 700°C.

3.3. Mechanical Property Degradation. In general, the microstructure of 9-12Cr martensitic heat-resistant steel would undergo the following changes during long-term high-temperature exposure [25]: (1) microstructural recovery, (2) dislocation density decrease, (3) accelerated migration rate of grain boundary, (4) martensitic lath widening, (5) disappearance of prior austenite grain boundary, (6) emergence of sub-grains, and (7) coarsening of precipitates. All of them would lead to the degradation of mechanical properties of steel.

Figure 7 shows the variations of room-temperature strength and impact toughness of G115 steel at different time cutoff points within 5000 h of aging at 650°C and 700°C. For aging at 650°C, the strengths, including yield strength and tensile strength, decreased slightly. The former decreased from 650 MPa to 602 MPa, while the latter decreased from 783 MPa to 757 MPa. Both of them decreased within 50 MPa. Furthermore, the strength remained stable after aging for 500 h. This result was somehow attributed to the pinning effect of grain boundaries and sub-grain boundaries of finer M7C3 carbides, which made the matrix maintain good microstructural stability. In addition, the precipitation strengthening effect of fine-sized M7C3 carbides and Laves phase in the steel made up for the decrease of strength caused by the decrease of solid solution and dislocation strengthening. Therefore, the strength decreased slightly and remained stable after aging at 650°C. For aging at 700°C, the strength decreased linearly. During the first 1000 h aging, as mentioned above, martensitic laths widened and high-density Laves phases were observed. Fortunately, the martensite lath still retained complete structure. So, the strength decreased slowly. However, with further increasing aging time, some martensitic laths have been replaced by subgrains, and the size of the precipitates continued to grow. Thus, the strength decreased drastically. Finally, the yield strength decreased from 650 MPa to 427 MPa and from 783 MPa to 621 MPa for tensile strength after 5000 h.

Compared with tensile properties, the effect of aging on impact toughness was more remarkable. On aging both at 650°C and 700°C, the variations of impact toughness were mainly divided into two stages: (1) during the first 500 h aging, the impact toughness decreased sharply; (2) after aging for 500 h, the impact toughness remained stable. The impact toughness of G115 steel drastically decreased from 52 J to 18 J and 17 J at the aging temperature of 650°C and 700°C, respectively. In order to figure out the reason why the impact toughness decreased so sharply at the initial period of aging, the fracture morphologies were observed in Figures 8 and 9. It can be seen that the fracture surfaces aged at 650°C and 700°C for 500 h consisted of cleavage platform and river pattern, showing obvious brittle cleavage characteristics. Moreover, large Laves phases were observed in the vicinity of the initial crack source. This may be an important reason for the decrease of impact toughness.

The variations of high-temperature strength of G115 steel aged at 650°C and 700°C are compared in Figure 10. These results can further verify the microstructural stability of G115 steel after aging at different temperatures. It can be also seen that the effect of aging at 700°C on the high-temperature strength was more significant than that at 650°C. Compared with the tensile strength, the yield strength decreased greatly at the initial stage of aging (within 500 h). Moreover, the high-temperature strength decreased with increasing temperature in the range of 630–675°C. The main difference was that the strength decreased rapidly after aging at 700°C, while it decreased slowly and remained basically stable after aging at 600°C. The evolution trend of high-temperature strength further verified that the microstructural stability of G115 steel aged at 650°C was obviously better than that aged at 700°C.

4. Discussion

During the thermal exposure, in contrast to tensile properties, the impact toughness exhibited considerable variations (Figure 7). Microstructural evolution under thermal exposure should be the main reason. It is well known that the main methods by which G115 steel can be strengthened are solution strengthening (mainly W and Co), dislocation strengthening, and precipitation strengthening. Under the condition of high-temperature and long-term exposure, on the one hand, the motion of dislocations would be increased, making the opposite dislocations annihilate each other and disappear. Finally, the dislocation density decreased and its contribution to strength was reduced. On the other hand, high temperature and long time made it possible for atomic diffusion and grain boundary migration. The ripening of M23C6 carbides decreased its ability to pin grain boundaries, making the martensite lath widen and further forming subgrains. At the same time, the precipitation strengthening was also weakened. In addition, the precipitation and growth of Laves phase would continuously consume the solid solution atom W. Accordingly, the solution strengthening got small in steel, and these processes occurring fast or slow mainly depended on the aging temperature. When the temperature was enough, the aging time would be shortened sharply, just as G115 steel aged at 700°C. When the temperature was low, such as 650°C, these processes occurred slowly or did not occur for a long time, showing the long-term microstructural stability. In the present study, the results showed that G115 steel still exhibited good microstructural stability aging at 650°C even up to 5000 h, which was also verified by the strength variations.

Unfortunately, after aging at 650°C and 700°C for 500 h, the impact toughness of G115 steel decreased greatly and embrittlement occurred rapidly in a short time. There is no
doubt that this result was closely related to alloy design in G115 steel. First of all, G115 steel contained much more solid solution strengthening elements, such as C, Cr, W, Co, and Cu. They can cause strong lattice distortion, making it easy to induce the initiation and propagation of microcracks. Secondly, the high-density precipitates in the aged G115 steel destroyed the continuity of the matrix and damaged the toughness. Last but not least, Laves phase was the main factor that deteriorated the toughness of 9-12Cr martensitic heat-resistant steel [28–33]. In the present study, the toughness of the experimental steel decreased rapidly at the same time after aging at temperatures of 650°C and 700°C.
When aging at 650 °C for 500 h, although the number density of Laves phases was low, they precipitated and grew along the grain boundaries and lath boundaries (Figures 4 and 6). It is noted that M23C6 carbides did not coarsen at this time (Figure 6). The precipitation and growth time of Laves phase was consistent with the decrease time of toughness. Thus, it is reasonable to suspect that Laves phase was the culprit of decreasing the impact toughness. More importantly, the
evidence of this conjecture was found from the results of impact fracture (Figures 8 and 9). When aging at 650℃ exceeded 500 h, M$_{23}$C$_{6}$ carbides still maintained the nano-scale size. Till aging at 700℃, the size of M$_{23}$C$_{6}$ carbides was obviously coarsened. However, the toughness of the steel hardly changed at this period (Figure 7). Therefore, the effect of coarsened M$_{23}$C$_{6}$ carbides on the impact toughness of G115 steel can be excluded. In addition, the physical properties of Laves phase also determined its adverse effect on toughness. Compared with M23C6, laves phase is hard and brittle and not easy to deform [34]. Moreover, the results calculated by Hald and Korcakova [35] showed that the interface energy between Laves phase and matrix was about 0.8 J/m$^2$, which means that it has a non-coherent relationship with the matrix. Laves phase precipitated along the interface after aging has poor adhesion and is not easy to deform. Therefore, under the action of impact stress, microcracks were easy to nucleate and expanded there and eventually led to brittle fracture. Therefore, Laves phase was the main reason for the early embrittlement of G115 steel aged at 650℃ and 700℃.

5. Conclusions

In the present work, the microstructural evolution and mechanical property degradation in G115 steel during long time exposure at 650℃ and 700℃ were investigated. Based on the experimental results, the following conclusions were obtained:

(1) There is little change in the aspects of the size of M$_{23}$C$_{6}$ carbides and the width of martensitic lath in G115 steel aged at 650℃ even increasing aging time up to 5000 h.

(2) The time interval for Laves phase to start coarsening and martensitic lath destruction was cut down with increasing temperature from 650℃ to 700℃.

(3) Both the room-temperature and high-temperature strengths remained stable after aging at 650℃ for 5000 h, while they decreased linearly aged at 700℃.

(4) The impact toughness of G115 steel aged at both 650℃ and 700℃ decreased sharply and embrittlement occurred rapidly in the initial aging period.

(5) Large-sized Laves phases were detected in the vicinity of the initial crack source, which proved that Laves phase was the culprit of decreasing the impact toughness.

(6) Laves phase particles are harmful to the toughness during long time service; however, they are very important for the creep properties in G115 steel. Therefore, how to improve the impact toughness of G115 steel without decreasing creep properties is to be solved in the future work.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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