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Research on the Thermostability of G115 Martensite Heat-Resisting Steel Aged at 650°C and 700°C

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Abstract. In this study, we conducted a preliminary examination of the microstructure and properties at room and high temperatures of G115 martensite heat-resisting steel following exposure at temperatures of 650° C and 700° C for a duration of 8000 hours. Our findings indicate that after aging at 650° C, the martensitic microstructure remained intact, with no significant change in the width of the martensitic laths. Additionally, the precipitation of dense and small-sized Laves phases was observed. Notably, the strength of G115 steel did not exhibit any substantial deterioration, except for a decrease in impact toughness. These results provide evidence of the favorable microstructural stability of G115 steel at 650° C. However, upon aging at 700° C, we observed the coarsening of high-density and large-size Laves phases, accompanied by the transformation of the majority of martensitic laths into sub-grains. Consequently, both the strength and impact toughness of the material experienced a sharp decline.

Keywords. Carbon dioxide (CO₂) emissions, microstructure and properties, thermostability, G115 martensite heat-resisting steel

1. Introduction

Carbon dioxide (CO₂) emissions and climate change have become a focal point of global concern, prompting significant changes in the power generation industry. The ultrasupercritical (USC) unit generator technology has emerged as the most notable change in fossil power plants, as it aims to enhance power generation efficiency and reduce carbon dioxide emissions. However, the adoption of stricter steam operating parameters, such as higher temperatures and/or pressures, imposes more stringent requirements on materials.

The 9%-12% Cr martensite heat-resisting steel has been extensively used due to its desirable properties of good creep resistance, good conductivity, and low thermal expansion [1]. However, as the service temperature increases to above 600°C, steels like A335-P91, A335-P92, and A335-P122 can no longer meet the elevated requirements. To address this need, a novel G115 steel was developed with the goal of improving service temperature to 630°C and above [2, 3].

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As higher temperatures demand greater microstructural stability, which has a direct impact on creep behavior, G115 steel was developed as one of the 9%-12% Cr martensite heat-resisting steels to address this challenge. Based on the chemical composition of commercial P91/P92 steel, G115 steel takes advantage of the theories of "multi-element alloving strengthening" and "selective strengthening" in composition design. In comparison to P92 steel. W substitutes Mo in G115 steel, and 3 wt.% Co and an appropriate amount of B are added. Both typical solid solution elements W and Mo have excellent strengthening effect, but the effect of W is more pronounced than that of Mo [4]. Furthermore, an appropriate amount of W can inhibit the coarsening of $M_{23}C_6$ and enhance creep strength [5], while B improves creep rupture strength of G115 steel significantly [6]. During normalizing, B tends to precipitate to grain boundaries, enter $M_{23}C_6$ carbides, and form B-bearing coexist carbides [7]. As a result, the coarsening rate of M23C6 carbides is restrained, leading to excellent stability of microstructure and creep properties. Additionally, another element of Cu can enhance precipitation strengthening greatly [8]. Micro-alloying elements, such as V and Nb, were added to improve creep strength through forming stable nano-sized precipitates. Notably, G115 steel produces unique martensite after heat treatment, making it suitable for use in USC boilers above 630°C.

This study investigates the stability of microstructure of G115 steel aged at 650°C and 700°C after 8000 hours of thermal exposure, as well as the corresponding strength and impact toughness. The relationship between microstructure and mechanical properties at room and high temperatures is also studied.

2. Materials and Methods

The experimental steel specimens were obtained from a G115 steel tube with dimensions of 254 mm in outer diameter and 204 mm in inner diameter. Table 1 presents the chemical composition of the studied steel in weight percentage. Prior to thermal exposure, the specimens underwent a heat treatment process consisting of normalization at 1100°C for 1 hour, and then tempered at 780°C for 3 hours.

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C	Si	Mn	P	S	Cr	W	Co	V	Cu	Nb	Ν	В	Fe
0.089	0.27	0.47	0.006	0.004	8.70	2.99	2.98	0.19	0.88	0.068	0.006	0.015	Bal.

Table 1. Chemical compositions of the G115 steel (wt.%).

To investigate the microstructural changes, the experimental steel specimens were subjected to aging at temperatures of 650°C and 700°C for a duration of 8000 hours. Subsequently, microstructure samples were cut, mechanical grinded with sandpapers, polished, and etched in Vilella reagent (comprised of 1% trinitrophenol, 5% alcohol hydrochloric acid, and 94% alcohol). The microstructure samples were then analysed by means of optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

For TEM analysis, discs with a thickness of 0.5 mm were initially cut from the microstructure samples. These discs were subsequently thinned to approximately 50 μ m and electro-polished at -25°C using a twin-jet electro-polisher in a solution containing 10% perchloric acid and 90% glacial acetic acid.

Mechanical samples were also prepared from the aged steel tube. The gage diameter and the gage length of the tensile test specimens are 5 mm and 25 mm, respectively, while the impact specimens measured 10 mm \times 10 mm \times 55 mm. These specimens were examined at room temperature according to the specifications outlined in GB/T 228.1 and GB/T 229, respectively. To ensure the accuracy of the test results, each test consists of three specimens, and take the average values of the final results.

3. Results and Discussion

3.1. Microstructure Characterization

Figure 1 depicts the initial microstructure of G115 steel, revealing a solitary martensite microstructure subsequent to the processes of normalizing and tempering (Figure 1a). Figures 1b and 1c exhibit the presence of precipitates distributed along crystal boundaries and lath boundaries, which we can reasonably infer to be $M_{23}C_6$ precipitates, given the composition elements of the steel. Furthermore, the martensitic lath width fluctuated within the range of 100 nm to 400 nm, and the average width is approximately 250 nm (Figure 1c).



Figure 1. The initial microstructure of G115 steel: (a) OM; (b) SEM; (c) TEM.

Figure 2 showcases the OM, SEM, and TEM images of the studied steel following an 8000-hour aging period at temperatures of 650°C and 700°C. Upon aging at 650°C, the microstructure retained its martensitic nature, and the PAGB(prior austenite grain boundaries) and packets remain discernible (Figure 2a). However, an increase in the aging temperature to 700°C led to a severe impairment of the microstructure stability of G115 steel, with the lath boundaries of martensite structure invisible clearly (Figure 2b). At the temperature of 700°C, most martensite laths underwent sub-grain transformation, accompanied by the observation of large-sized precipitates along grain boundaries.

Notably, the Laves phase, a significant precipitate in 9%-12%Cr martensite heatresisting steel during prolonged aging, was evident in Figures 2c and 2d after aging at temperature of 650°C and 700°C for 8000 hours. No Laves phase was detected in the initial state of G115 steel, implying the requirement of a certain incubation period for nucleation. The duration of this incubation period is typically dictated by the material's chemical composition and aging temperature. The large amount of W and Co elements in the studied steel has been confirmed to result in the formation of dense Laves phases under back-scattered electron (BSE) mode. By comparison, the number density of Laves phases aged at 650°C exceeded that of those aged at 700°C, although the size of Laves phases aged at 700°C was larger. This indicates a significant increase in the coarsening rate of the precipitates at 700°C.



Figure 2. The microstructure of G115 steel after thermal exposure at (a) and (c) 650°C; (b) and (d) 700°C for 8000 hours: (a) and (b) OM mode, (c) and (d) BSE mode.

Figure 3 traces the subtle microstructural changes in G115 steel after an 8000-hour aging period at 650°C and 700°C. Figure 3a demonstrates that the tempered martensitic lath structure remains discernible after aging at 650°C. Compared to the initial state depicted in Figure 1c, the martensitic laths exhibit a slight increase in width even after 8000 hours of aging, expanding from 0.25 μ m to 1.0 μ m. Notably, the presence of Laves phase, measuring approximately 500 nm in size, is detected when aging at 650°C. These large black phases manifest as rectangular particles adhering to martensitic lath boundaries closely (Figure 3a). Conversely, aging at 700°C leads to the destruction of the regular martensitic lath structure, with most laths transforming into sub-grains (Figure 3b). Consequently, microstructural stability is significantly compromised, and the size of Laves phase increases to approximately 1.5 μ m.



Figure 3. TEM micrographs of the studied steel aged at (a) 650°C and (b) 700°C for 8000 hours.

3.2. Mechanical Properties

Table 2 showcases the mechanical properties of G115 steel following thermal aging at temperatures of 650°C and 700°C for a duration of 8000 hours. The observed mechanical properties, such as yield strength, tensile strength, and impact toughness, exhibited a decrease compared to the initial properties. Notably, the impact toughness displayed

significant variations. This can be attributed to the development characteristics of the microstructure. The chemical composition of the studied steel indicates that the primary strengthening mechanisms involve solid solution strengthening, dislocation strengthening, and precipitation strengthening [9-11]. Serving at high temperatures and prolonged conditions, dislocation movement increases, leading to their annihilation and disappearance. Consequently, the decrease in dislocation density reduces their contribution to strength. Additionally, elevated temperatures and extended durations lead to atomic moving and grain boundary migrating faster. The coarsening of precipitates diminishes their ability to impede grain boundaries, resulting in the widened martensite laths and the appearing of sub-grains. For this reason, precipitation strengthening is weakened. Furthermore, the formation and coarsening of Laves phase constantly consumes the solid solution element W, thereby reducing solution strengthening. The rate at which these processes occur, either rapidly or slowly, primarily depends on the aging temperature when the duration is fixed. Increasing the temperature significantly shortens the aging time, as observed in G115 steel aged at 700°C. Conversely, at lower temperatures such as 650°C, evolution process above occur gradually or may not occur for an extended period, indicating long-term stability of the microstructure. The current study demonstrates that G115 martensite heat-resisting steel exhibits favorable microstructural stability even aged at 650°C for a duration of 8000 hours.

Aging temperature	Yield strength (MPa)	Tensile strength (MPa	n) Impact toughness (J)
Initial	649	806	78
650°C	564	731	18
700°C	431	612	14

Table 2. Mechanical properties of G115 steel after aging at 650°C and 700°C for 8000 h.

4. Conclusions

In this paper, we studied the microstructural stability and mechanical properties of G115 steel following exposure at temperatures of 650°C and 700°C for a duration of 8000 hours. Our findings reveal that G115 steel exhibits favorable microstructural stability at 650°C, whereas its stability is compromised at 700°C. Remarkably, even after prolonged exposure at 650°C, the strength of G115 steel remained largely unaffected, with the exception of a decrease in impact toughness. This can be attributed to the robust microstructural stability exhibited by the material. However, at 700°C, we observed the coarsening of high-density and large-size Laves phases, leading to the replacement of most martensitic lath structure by sub-grains. Consequently, both the strength and impact toughness of the material experienced a significant decline.

Acknowledgments

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