Microstructure analysis of P91 heat resistant steel in service for 3 years

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Abstract. P91 heat resistant steel is an important material for supercritical units. In this paper, a P91 heat resistant steel pipe which has been used for about 3 years is analyzed by means of hot tensile test, metallography and scanning electron microscope. The results show that the microstructure is martensite and a large number of Cr-rich precipitates are distributed at the grain boundary. There are pores and cracks in the fracture of high temperature tensile specimen.

1. Introduction

With the improvement of steam temperature and pressure parameters of ultra supercritical units, higher and more stringent requirements are put forward for service materials of core structure of units, especially for high temperature oxidation resistance, corrosion resistance and thermal strength of materials. Therefore, the key to the development of ultra supercritical units is to develop the materials and manufacturing technology of core components. For the material selection of ultra supercritical units, the most basic requirement is to ensure that all components of the unit can work efficiently and reliably under the highest steam and pressure parameters. With the continuous improvement of steam temperature and pressure parameters, the materials of core components of ultra supercritical units must also be selected according to the requirements, so as to meet the requirements of strength design.

P91 steel is a new type of martensitic heat resistant steel, which is developed on the basis of 9Cr-1Mo heat resistant steel by controlling the content of C element in the steel and adding some strengthening elements such as V, Nb, N, etc [1, 2]. In recent years, with the development of ultra supercritical units in the direction of large capacity and high parameters, P91 heat resistant steel has become the main material for the core components of ultra supercritical units with its good oxidation resistance, high endurance strength and excellent process performance. In recent years, many scholars have studied the properties of heat resistant steel. Jin et al. [3] studied the microstructure evolution of P92 heat resistant steel under different service time. The results show that the high temperature strength of P92 steel is mainly affected by lath and dislocation hardening at the initial stage of service. With the extension of service time, the strengthening mechanism changes to lath and precipitation hardening. Hu et al. [4] studied the superheater pipes of martensite heat resistant steel with different service time of boiler and put forward the quantitative relationship. The results show that the decrease of austenite grain boundary hardness is greater than that of matrix hardness. Liu et al. [5] studied the microstructure evolution of austenitic heat resistant cast steel during aging and long-term service. The
results show that the secondary carbides precipitated in austenite increase obviously after long-term service. Guo et al. [6] studied the microstructure of a heat resistant steel pipe of cracking furnace and its effect on the properties of heat resistant steel. The results show that the primary carbides Cr7C3 and NbC have transformed into chromium rich Cr23C6 and niobium rich carbides G phase.

The performance degradation of heat resistant steel will affect the safety of power plant structural components in long-term use. In this paper, the metallographic analysis of P91 heat resistant steel pipe matrix which has been in service for 3 years and the specimens after high temperature tensile test are carried out.

2. Material and Experimental
The P91 heat resistant steel pipe was cut from the steam extraction valve installed in the thermal power plant, and it has been in service for about 3 years, as shown in Fig. 1. The diameter of the pipeline is 244.5mm, the wall thickness is 45mm, the working pressure is 17.7MPa, the working temperature is 537°C, and the heat treatment state is normalized. According to the actual operation and design temperature of P91 heat resistant steel pipe, the high temperature tensile test temperature is 537°C and 565°C.

![Figure 1. P91 heat resistant steel pipe.](image)

The results of chemical analysis are shown in Table 1. It can be seen that the element content of service pipe meets the requirements of ASTM A335 <Standard Specification for Seamless Ferritic Alloy-Steel Pipe for High-Temperature Service> standard.

| Element | C     | Mn   | Si   | Ni   | Cr  | Mo  | P     |
|---------|-------|------|------|------|-----|-----|-------|
| Sample  | 0.09  | 0.51 | 0.34 | 0.18 | 8.87| 1.02| 0.012 |
| ASTM A335 Grade91 | 0.08-0.12 | 0.30-0.60 | 0.20-0.50 | ≤0.40 | 8.00-9.50 | 0.85-1.05 | ≤0.02 |

| Element | Ti   | V    | Nb   | Zr   | Al  | S   |
|---------|------|------|------|------|-----|-----|
| Sample  | <0.01| 0.22 | 0.083| <0.01| <0.01| <0.005|
| ASTM A335 Grade91 | ≤0.01 | 0.18-0.25 | - | ≤0.01 | ≤0.02 | ≤0.01 |

3. Results and Discussions

3.1. Microstructure analysis of the serviced raw materials
Metallographic analysis was carried out from the P91 heat resistant steel pipe matrix, and the distribution of matrix structure is shown in Figure 2. It can be seen that the matrix structure tends to be banded. At high magnification, the microstructure is martensite, retained austenite and precipitated phase.
The SEM morphology of P91 heat-resistant steel is shown in Fig. 3. The results show that the microstructure is martensite and there are Cr containing precipitated strengthening phases. Mo, Cr, Mn, Fe and other alloying elements can promote the formation of Cr-rich M23C6 precipitates. The coarse Cr-rich M23C6 precipitates are mainly distributed in grain boundaries, original austenite grain boundaries and lath boundaries.

3.2. Microstructure analysis after 537 ℃ tensile test
Metallographic analysis was carried out on the fracture section of 537 ℃ tensile specimen of P91 heat resistant steel pipe. The microstructure distribution of tensile fracture section is shown in Fig. 4(a), the fracture surface is undulating, and there are pores in the section. The pore morphology at high magnification is shown in Fig. 4(b).
3.3. Microstructure analysis after 565 ℃ tensile test

Metallographic analysis was carried out on the fracture section of 565 ℃ tensile specimen of P91 heat resistant steel pipe. The microstructure distribution of tensile fracture section is shown in Fig. 5(a). It can be seen that the fracture surface is rough, and there are pores in the section. At high magnification, the pore morphology is shown in Fig. 5(b).

![Figure 5. Metallographic of tensile at 565 ℃.](image)

4. Conclusion

The microstructure of P91 heat resistant steel which has been in service for 3 years is mainly martensite with Cr-rich precipitates. There are holes and cracks in high temperature tensile fracture.

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