Research Article

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Study on microstructure and mechanical properties of P92 steel after high-temperature long-term aging at 650°C

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Abstract: A series of long-term high-temperature aging tests of ASME A335-P92 steel from 1,000 to 29,000 h at 650°C were carried out. The microstructure evolution of as-received and aging specimens at different stages was investigated using optical microscope observations, scanning electron microscope examinations, and TEM investigations. The static mechanical strength properties (yield strength/ultimate tensile strength) at room and 600°C test temperatures and the plastic performance (elongation/reduction in area) were also analyzed. The experimental results show that the Laves phase can be precipitated rapidly in the early aging period. After a certain aging period, the continuous precipitation of $M_{23}C_6$ and the relatively high coarsening rate of Laves resulted in a rapid decrease of room and high-temperature mechanical properties in the early aging period. However afterwards for the long aging time, a slow decline in tendency of mechanical properties was presented.

Keywords: P92 steel, long-term aging, microstructure, mechanical properties

1 Introduction

In order to meet the comprehensive performance requirements of high efficiency and low emission ultra-supercritical unit (USC) boiler steels, Cr (9–12 wt%) ferritic heat-resistant steels are widely used. P92 steel is designed (ASME grade 92) based on P91 steel (ASME grade 91), which reduces the molybdenum (Mo) content of P91 steel to 0.3–0.6% and adds 1.5–2.0% W [1]. Compared with other commonly used heat-resistant steels of the 9% Cr series, P92 steel has similar corrosion resistance and oxidation resistance, but its high-temperature strength and creep performance have greatly improved [2]. Under the same service conditions, P92 steel can significantly reduce the size of the components, and it has been widely used at 600°C or even at 630°C USC in China.

The microstructure has a great influence on the performance of 9–12 wt% Cr steel [3]. Studies on the microstructure evolution of ferrite/martensitic steel show that the precipitated phases after tempering are mainly $M_{23}C_6$ ($M = Cr$)-type carbides and MX-type carbo-nitrides ($M = V, Nb$ and $X = C, N$) [4,5], which are the main strengthening phases of P92 steel. In addition, in the aging process of P92 steel, Laves phase and Z phase are formed [6]. The quantity and size of these precipitates will have an important effect on the applied performance of the steel. Based on the long-term aging test of P92 steel, the microstructure evolution at different aging stages and variation of mechanical properties of P92 steel at room and high temperatures were studied in this work.

2 Experimental details

The sample studied in this paper is an as-received ASME P92 steel pipe of an unused reheat pipeline in a certain USC. After normalizing heat treatment at 1,065°C for 1.5 h

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Table 1: Chemical composition of an as-received P92 steel sample (wt%)

Element	C	Mn	P	S	Si	Cr	Mo
Mass fraction	0.126	0.366	0.0192	0.0091	0.277	9.35	0.360
Wt%							
Element	V	W	Ni	Al	Nb	B	Fe
Mass fraction	0.165	1.85	0.349	<0.001	0.0778	0.0038	85.3
Wt%							

and high-temperature tempering at 775°C for 3 h, the size of the pipe is ID 616 mm $\times$ 47 mm. The chemical composition of the material conforms to the ASTM A335 Standard Specification, as shown in Table 1. The test samples with a cross-section bigger than 25 mm $\times$ 25 mm were cut off from the pipe along the axial direction. The schematic diagram is shown in Figure 1. The stress-free high-temperature aging test was carried out at 650°C in a TXCS9-II muffle furnace with the furnace temperature control within $\pm 2^\circ\text{C}$. The aging time interval of A1–A9 series from 1,000 to 29,000 h is shown in Table 2.

Circular standard samples with a diameter of 5 mm and a standard distance of 50 mm from the original materials and each aging group were prepared (as per the ASTM-E8/E8M). Mechanical static tensile performance tests were carried out with a MTS C45.305 electronic universal testing machine at room temperature and elevated temperature (600°C) at which the P92 steel was widely selected and applied for the main steam and high reheat pipelines in ultra-supercritical power units at present. The results are the average value of three samples in each test group. The test rate is based on the strain rate ISO 6892-1:2016 A224. During the testing, yield strength (YS/0.2% proof stress) was

measured under the strain mode at the strain rate of 0.00025 s^{-1} in the first period. In the second period, these specimens controlled by the strain rate of 0.0067 s^{-1} were pulled up to the fracture to determine the ultimate tensile strength (UTS). The metallographic structure was observed by ZEISS DMI 5000 optical microscope (OM). The microstructure, precipitation phase, and distribution of elements were observed by FEI Quanta 450 field emission scanning electron microscope (SEM) and TECNAI F30 field emission transmission electron microscope (TEM). The sample for TEM observation is prepared by the following method: first, the samples were cut into thin slices with the wire cutting method and then polished with sandpaper to make it smooth on both sides, and the thickness was polished to about 100 microns. Second, a small wafer with a diameter of 3 mm was processed. Then, the small wafer was electrolytically polished using an electrolytic double spray polisher. The electrolyte was 5% perchloric acid–alcohol solution, the double spray voltage was 50 V, and the temperature was about -20°C . Finally, the electrolyte on the surface of the sample was washed off with alcohol and dried naturally on filter paper.

3 Results and discussion

3.1 Microstructure

The original structure of P92 steel (sample A0) under standard heat treatment (normalizing + tempering) is a typical lamellar tempered martensite structure. A large number of carbides and MX-type precipitates disperse along the prior austenitic grain boundaries (PAGB), subgrain boundaries, and lath boundaries, as well as inside the laths, as shown in Figure 2(a).

Figure 1: Schematic diagram of the test sampling.

Table 2: Aging time of P92 steel samples at 650°C

Samples	A0	A1	A2	A3	A4	A5	A6	A7	A8	A9
Aging time (h)	0 (as-received)	1,000	2,000	3,000	4,000	5,000	6,000	7,000	20,000	29,000

Most of the precipitated carbide phases are $M_{23}C_6$ carbides with centered cubic lattice structure (Cr-rich, M is partly replaced by Fe), which is mainly formed during the tempering process [7]. Their shapes are granular and rod-shaped, as shown in Figure 2(b–d). There are a large number of granular precipitates which are mainly distributed along the PAGB and martensitic lath boundaries, while the number of rod-shaped precipitates is small and they are mainly distributed along the subgrain boundaries and inside the martensitic lath [8,9].

The MX-type precipitates are Nb-rich Nb(C,N) compound and V-rich V(C,N)/VN carbonitride compound or nitride compound distributed in the martensitic lath matrix and subgrain crystal with a small size. The shape of Nb-rich NbX is granular, and a part of NbX are retained because they are not dissolved during normalizing. The other part of them is formed during tempering, and finer than the former. The V-rich VX precipitation is mainly formed in the tempering process with a plate-like shape [7–10].

As shown in Figure 3, the martensitic laths of the sample A4 after aging for 4,000 h are coarser than those of sample A0 (Figure 3(a)). Compared with the original material (Figure 2(b)), the number of precipitates in the SEM image (Figure 3(b)) is significantly reduced, but the size of the precipitates is larger with a tendency of gathering and growing up. The precipitates are still dominated by $M_{23}C_6$ carbides (Figure 3(c and d)) and MX carbides. The SEM-EDS analysis results of the grain boundary precipitates (Figure 3(e)) show that a part of grain boundary precipitates have a relatively high W content, which indicates that the Laves phase with a high W content has been precipitated at the grain boundary. Laves phase is a $Fe_2(W,Mo)$ -type intermetallic compound with a hexagonal structure. In the aging process of P92 steel, Laves phases, which are attached to the $M_{23}C_6$ carbide, nucleate and grow up at the grain boundaries [11,12]. Figure 3(c) shows that the Laves phase has grown and has a tendency of coarsening,

Figure 2: (a) OM image, (b) SEM image, (c) TEM bright-field image, and (d) TEM dark-field image and SAED of $M_{23}C_6$.

Figure 3: (a) OM image, (b) SEM image, (c) TEM bright-field image, (d) TEM dark-field image and SAED of $M_{23}C_6$, (e) SEM-EDS result of P92 steel after 650°C–4,000 h aging, (f) SEM-EDS mapping image of W in P92 steel after 650°C–4,000 h aging, and (g) SEM-EDS result of P92 steel after 650°C–1,000 h aging.

indicating that the Laves phase has been precipitated before the aging time reaches 4,000 h. In fact, the EDS

analysis (Figure 3(e and f)) shows that the W content of precipitates at the grain boundary is higher than that of

the matrix when the aging time reaches 1,000 h, indicating that the Laves phase had been precipitated, as shown in Figure 3(g). Studies of Saini et al. [12] show that after aging at 650°C for 720 h, the content of Mo and W of the grain boundaries increased while the Fe content decreased, indicating that the Laves phase had been nucleated. Guo et al. [13] also reported that the Laves phase rapidly precipitated after aging at 650°C for 200 h. The related studies and the present research of this paper have shown that the Laves phase had been precipitated in the early aging stage of 1,000 h or even earlier under the service condition of 650°C. However, the study of Sun et al. showed that the Laves phase had been rapidly precipitated within 1,000 h under 650°C aging condition, and then the quantity of precipitation decreased and the coarsening occurred [14].

The metallographic structure (Figure 4(a)) of the sample A7 aged for 7,000 h shows that the martensitic lath coarsened obviously. A SEM image (Figure 3(b)) combined with a TEM image (Figure 4(c and d)) and EDS results (Figure 4(e)) show that both the $M_{23}C_6$ carbide and the Laves phase were further coarsened and grown as compared with 4,000 h aging, and that the growth rate of the Laves phase was significantly faster. The EDS mapping image (Figure 4(f)) also shows further aggregation of the tungsten (W) element in the Laves phase precipitated at the boundaries, compared with that of aging to 4,000 h (Figure 3(f)). The size of the MX carbonitride is relatively stable with no obvious change. Czyrska-Filemonowicz et al. [11] found that there is no obvious coarsening phenomenon of the MX phase in P92 steel after 33,000 h creep at 600°C. Research studies of Panait et al. also show that the MX phase still did not have a significant size coarsening phenomenon after aging or creep for 1,00,000 h [15].

When the aging test lasted up to 20,000 h, as shown in Figure 5, the martensitic lath is obviously wider than that of 7,000 h aging, and the recovery phenomenon of the lath structure is observed. The martensitic lath interface in most areas is still visible, but the lath interface in the local area is disappearing (Figure 5(a)), which is a manifestation of the lath fragmentation and subgrain growth. The content and size of the second phase particles have increased significantly, especially at the lath boundaries. The $M_{23}C_6$ carbides keep growing up when the new precipitates were observed in subgrain boundaries. The size of Laves has reached the micron level, and element W showed an extremely obvious agglomeration in the precipitate.

After 29,000 h aging, the recovery degree of the martensitic lath structure is more obvious than that at

20,000 h, and the width of the lath is wider, and the morphology of the precipitates inside and along the grain boundaries was observed clearly in the metallographic structure (Figure 6(a)). The SEM image (Figure 6(b)) shows that the amount of precipitates reduced, but the particle size increased. The Laves phase was characterized by aggregate coarsening with a size up to 1.2 microns, surrounded by small $M_{23}C_6$ carbides (Figure 6(c–f)) [11]. In P92 steel, $M_{23}C_6$ carbide continuously precipitates out and grows with the extension of aging time; however, the Laves phase has a higher coarsening rate than $M_{23}C_6$. Comparatively speaking, the $M_{23}C_6$ carbide is more stable than the Laves phase [13]. Saini et al. reported that after 3,000 h aging, the Laves phase was larger than $M_{23}C_6$ and became the largest phase in P92 steel [12].

3.2 Strength and plasticity

P92 steel cylindrical tensile specimens ($d_0 = 5$ mm/ISO 6892 standard specimen, according to ASTM e8-04) of each aging stage from A1 to A9 were prepared for room temperature and 600°C elevated temperature static tensile tests. Each group has three specimens.

The influence of long-term aging at the temperature of 650°C on mechanical properties of P92 steel is shown in Figure 7. The determined results are the average value of three samples. As shown in Figure 7(a), the yield strength and tensile strength of as-received P92 steel are 514 and 683 MPa at room temperature, respectively. During the aging time at 650°C, the yield strength at room temperature decreased from 500 MPa at 1,000 h to 479 MPa at 7,000 h, and the tensile strength decreased from 685 to 662 MPa at 7,000 h. The yield strength and tensile strength showed almost no change at all after aging for 20,000 h and 29,000 h, and the yield strength was 483 MPa and the tensile strengths were 665 and 663 MPa, respectively. Compared with the as-received original material, the yield strength decreased by 6% and the tensile strength decreased by 3%. The experimental results in the research [16] also showed that the yield strength only decreased by about 10% and the tensile strength decreased by about 6% for the aged P92 steel after the exposure time of 30,000 h. The variation tendency of strength in the aging process is consistent with the present research results in this paper (Figure 7(a)). Generally speaking, the yield strength and tensile strength of the steel decrease with increasing the aging time. However, the strength appeared to show an obvious decreasing trend in the early stage of aging;

Figure 4: (a) OM image, (b) SEM image, (c) TEM bright-field image, (d) TEM dark-field image and SAED of $M_{23}C_6$, (e) SEM-EDS result of P92 steel after 650°C–7,000 h aging, and (f) SEM-EDS mapping image of W in P92 steel after 650°C–7,000 h aging.

especially, the yield strength decreased rapidly to a stable value within 1,000 h aging time, while the tensile strength decreased slowly throughout the whole aging stage. Guo *et al.* studied the room temperature tensile properties of P92 steel after 650°C aging for 12,000 h and the results showed that the strength decreased rapidly within 1,000 h and then the decrease slowed down [17].

The static tensile test results examined at 600°C after aging at 650°C are shown in Figure 7(b). The high-temperature yield strength decreased from 258 MPa at 1,000 h to 250 MPa at 7,000 h, while the tensile strength decreased from 327 to 314 MPa. The yield strength and tensile strength were almost unchanged after aging from

20,000 to 29,000 h, and the yield strengths were 254 and 250 MPa, and the tensile strengths were 313 and 308 MPa, respectively. Similarly to the static tensile properties at room temperature, it can be seen from Figure 7 that the high-temperature strength also shows a rapidly decreasing trend within 1,000 h to a stable level.

In this study, a significant reduction was not found in strength properties, such as the yield strength and tensile strength, both at room temperature and 600°C elevated temperature after long-term aging at 650°C and 29,000 h soaking.

Room and elevated temperature tensile test results show that the strength decreases with the increase of

Figure 5: (a) OM image, (b) SEM image, (c) TEM bright-field image, (d) TEM dark-field image and SAED of $M_{23}C_6$, (e) SEM-EDS result of P92 steel after 650°C–20,000 h aging, and (f) SEM-EDS mapping image of W in P92 steel after 650°C–20,000 h aging.

aging time. However, the decrease of both yield strength and tensile strength is not large. A similar course of changes in mechanical properties of aged high-chromium steels was observed inter alia in the works [18,19]. Khayatzadeh et al. [20] carried out the 650°C tensile test after aging at 650°C for 1,000 h and the results showed that the yield strengths were the same, and the tensile strength decreased by about 4%. A stress-free thermal aging study with a maximum aging time of 5,000 h at 650°C was conducted on P92 steel in ref. [21]. The results showed that the yield strength and tensile strength decreased slightly within 2,000 h, then became stable. Saini et al. [12] also observed a small decrease in

strength in the study of thermal aging at 650°C for 3,000 h. The variation of mechanical properties of P92 steel during the long-term simultaneous temperature and time acting is closely related to the complex microstructure evolution and microprecipitates in each different aging stage. In the early aging period, especially within 1,000 h, the rapid decrease in strength is mainly attributed to the precipitation and rapid growth of the Laves phase (Figure 3(g)), which consumes both W and Mo elements' supersaturated solute in the matrix, and thus weakens the solid solution strengthening effects. Although the strength will be improved by the precipitation strengthening effect caused by Laves

Figure 6: (a) OM image, (b) SEM image, (c) TEM bright-field image, (d) TEM dark-field image and SAED of $M_{23}C_6$, (e) SEM-EDS result of P92 steel after 650°C–29,000 h aging, and (f) SEM-EDS mapping image of W in P92 steel after 650°C–29,000 h aging.

phase in the initial short stage of aging at 650°C up to 500 [17] or 200 h [21], the high coarsening rate of Laves phase cannot make up the loss of solid solution strengthening and therefore leads to a decrease in mechanical properties.

Wang *et al.* [22] show that the precipitation of Laves phases has been completed in the first 2,000 h thermal aging time at 650°C when the partition coefficients of the Mo and W elements supersaturated in the matrix reduce from 86% before aging to about 50%, and after the 2,000 h time, the concentration of both the elements keeps a constant value or does not reduce anymore in the matrix. Obviously, the latter result is simultaneously

attributed to the much lower coarsening rate of $M_{23}C_6[(Cr,Fe,Mo,W)_{23}C_6]$ carbides than that of Laves phases $[(Fe,Cr)_2(Mo,W)]$, which can be observed in Figures 3(c), 5(b, c), and 6(b, c)). $M_{23}C_6$ carbides are relatively stable, as compared to Laves phases during aging exposure [17,22]. Gustafson and Hättstrand [23] and Hättstrand and Andrén [24] revealed very low coarsening rates of $M_{23}C_6$ precipitates and almost unchanged coarsening rates of MX, based on both experimentally measured precipitate sizes that existed in P92 steel at 650°C thermally aged from 1,000 h time up to 26,000 h, and correspondingly in this aging time stage, the content of W in the matrix almost keep a

Figure 7: (a) Variations of static tensile properties examined at room temperature during the long-term aging exposure of 650°C. (b) Variations of static tensile properties examined at 600°C temperature during the long-term aging exposure of 650°C.

constant value. Actually, the addition of W can suppress the coarsening of $M_{23}C_6$ carbides [25]. All the above factors indicate that the solid solution hardening enhanced by W and Mo elements in the P92 steel matrix as one of the main strengthening mechanisms supports the fact that the strengths drop extremely slowly or almost remain unchanged after aging beyond 2,000 h in the present work (see Figure 7).

On the other hand, Li [26] and Bao et al. [19] have revealed that the martensitic laths in 9–12% Cr martensitic steels are dominant microstructures contributing to strengthening. $M_{23}C_6$ carbides mostly precipitated on the martensitic lath/subgrain boundaries stabilize the subgrain and lath microstructure by pinning and prohibiting the migration of subgrain boundaries. The fine MX phases precipitated inside subgrains with an extremely high thermal stability against coarsening [27] constitute an effective obstacle for inhibiting the dislocation movement. Thus, it is highly important to increase the stability of $M_{23}C_6$ carbides to retain the lath substructure.

In the present study, an obvious coarsening of the Laves phase starts at approximately 4,000 h of aging and its size becomes larger at the aging times of 20,000 and 29,000 h (Figures 3(c), 5(c), and 6(c)). However, according to the research by Panait et al. [15], the Laves phase should not strongly affect the resistance to creep deformation but could affect the resistance to creep fracture. This is another possible contribution to maintain the stability in tensile strength properties during the aging exposure.

In the present study, the microstructure for aging time up to 29,000 h at 650°C was characterized by the

still preserved lath martensitic microstructure, although a local or partial progressing decomposition of the lath martensitic microstructure was observed in tempered martensite areas (Figure 6(a)). This similar feature appeared in the work [16]. Furthermore, Dobrzański et al. in the research [28] revealed that even when the aging time reached up to 70,000 h at 650°C, a significant disintegration of the lath martensite microstructure and considerable degraded zones of martensite could be found. Generally, the constitutions and characteristics of microstructure evolved in different aging stages of up to 29,000 h in the present work contribute to a significant flat tendency in the tensile strength, especially after the aging time of 2,000 h, which reveals the process of extremely slow recovery and polygonization of the matrix in the P92 steel [29] (Figure 7).

Figure 8 shows the variation tendency of room temperature plasticity of the as-received samples. The plasticity of the as-received material at room temperature $A = 28\%$, $Z = 71\%$ decreased to $A = 22\%$, $Z = 61\%$ when the aging time reached 2,000 h, then recovered to $A = 26\%$, $Z = 61\%$ when aging for 5,000 h, then basically remained stable $A = 25\%$, $Z = 63\%$ to after 29,000 h aging. The plasticity of the as-received material under the high-temperature test at 600°C $A = 37\%$, $Z = 83\%$ decreased to $A = 23\%$, $Z = 82\%$ after aging for 1,000 h and then fluctuated in a narrow range to $A = 30\%$, $Z = 76\%$ after aging for 29,000 h. The elongation decreased rapidly within 1,000 h in the early aging period, while the reduction of area showed a slow decrease during the whole aging tests. In general, the plasticity of the as-received material at room temperature and high

Figure 8: The elongation and reduction in area during different aging periods.

temperature decreased rapidly in the early aging period (room temperature 2,000 h, high temperature 1,000 h), and then the plasticity recovered a bit and decreased gradually and slowly. The rapid decrease in plasticity in the early aging stage is related to the precipitation of the Laves phase, its growth and coarsening between slats [12]. The plasticity recovery is due to the matrix softening caused by the recovery of the lath martensite structure [17]. The slow decrease of plasticity in the later aging stage is mainly attributed to the saturation of Laves phase precipitation and the decrease of coarsening rate [14].

4 Conclusion

Based on the 29,000 h long-term aging test of P92 steel at 650°C, the microstructure evolution at different aging stages was characterized by OM, SEM, and TEM. The static mechanical properties of the as-received material and specimens of each aging stage at room temperature and high temperature 600°C were also investigated. The conclusions are as follows:

- (1) In the early aging stage, the structure of martensitic lath is stable. However, the width of the laths begins to increase after a certain period of aging time, especially after aging for 20,000 h, the recovery phenomena of the martensitic lath structure are relatively obvious.
- (2) With the extension of aging time, $M_{23}C_6$ continues to precipitate and grow in PAGB and subgrain boundaries. The Laves phase nucleates and grows up

around the $M_{23}C_6$ phase. The Laves phase precipitates rapidly in the early stage of aging within 1,000 h. It can be observed that the growth and coarsening rate of Laves phase are much higher than those of the $M_{23}C_6$ phase. However, the MX seems to be stable with a little change in size throughout the whole aging period.

- (3) During the whole aging stage, the static room temperature strength, high-temperature strength (yield strength YS, tensile strength UTS), and the plastic index (elongation A, reduction in area Z) show a decrease in tendency; however, the decrease in range is small. The strength and plasticity decrease rapidly in the initial aging stage within 1,000 h, but maintain a slow decrease in trend until aging 29,000 h. The precipitation reinforcement effect caused by the simultaneous precipitation of secondary particles and the recovery softening effect of the martensitic lath structure during the long-term aging process are the main mechanisms for the slow degradation of the mechanical properties. Besides, the precipitation and coarsening of Laves phase play an important role in affecting the stability of the martensitic lath structure.

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