Evolution of Microstructures and Mechanical Properties of Zr-Containing Y-CLAM During Thermal Aging

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Abstract

The thermal stability and mechanical properties of China low activation martensitic steel with Zr and Y were investigated via thermal aging at 550 °C for 8000 h. The Laves phase content monotonically increased with thermal aging, and the volume fraction of the Laves phases stabilized in the alloy after 3000 h of thermal aging. The observed degradation in mechanical properties was because of the coarsening of $M_{23}C_6$ carbides and matrix grains during the earlier stages of thermal aging. The precipitation of Laves phases and V_3Zr_3C particles increased the strength and hardness of the alloy. Grain coarsening was the primary reason for the decrease in impact properties, and the ductile-to-brittle transition temperature increased from −71 to −48 °C after 8000 h of thermal aging.

Keywords Thermal stability · Zr-containing RAFM · Precipitation · Microstructures · Mechanical properties

1 Introduction

Reduced-activation ferritic/martensitic (RAFM) steels have been considered as the primary candidate structural materials for advanced fusion reactors because of their excellent microstructure stability, high-temperature mechanical properties, and irradiation resistance [1]. In the beginning, RAFM steels were modified from heat-resistant 9Cr–1Mo–Nb steels for thermal power plants by the substitution of Mo and Nb, high activation alloying elements, with W and Ta, low activation alloying elements, respectively [2]. Several countries have developed their own RAFM steels: for instance [3], Eurofer 97 (Europe), F82H (Japan), 9Cr–2WVTa (USA), CLF-1 (China), and CLAM (China).

To ensure the low activity of alloys, only a limited number of elements can be added to the RAFM steel to improve its mechanical properties. Currently, the main research is focused on Y, Ti, and Zr element. The authors have systematically studied the effects of Y on inclusions, structure, and properties of CLAM steel and developed a Y-containing CLAM steel (Y-CLAM) [4]. It is indicated that Y acts as deoxidizers, desulfurizers, and modifiers. The CLAM steel modified by 0.01 wt.%–0.05 wt.% Y can have better properties than other RAFM steels. In addition, yttria or yttrium oxide is commonly introduced to oxide dispersion strengthened (ODS) steels, which are widely studied worldwide [5].

Darling et al. [6] have reported that the ODS alloys with Zr still have the ability to resist grain growth at 1373 °C. Xu et al. [7] have reported that an addition of Zr leads to the finer grain size of the 9Cr–Zr–ODS alloy, which was only 0.8 µm. Zr can also be used to refine Y-containing oxide particles in 9Cr–ODS alloys prepared via mechanical alloying, which are other candidate structural materials for fusion reactors. Currently, Zr is usually used for grain refinement and strengthening in RAFM steel alloys [8, 9]. Since 2011, Korea has developed a Zr-containing advanced reduced activation alloy (ARAA) for fusion reactors [8]. ARAA alloy exhibits a significantly longer creep–rupture time than Eurofer 97 steel, suggesting a positive influence of Zr addition. Chun et al. [10] reported that the improvement in impact and creep resistance by the addition of small amounts of Zr is attributed to the solution softening the

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effect of Zr, which is associated with a reduced temperature-dependence of the yield strength. The authors have investigated the effects of Zr on the inclusions and mechanical properties of the Y-CLAM steel [11–13]. The compound addition of Zr and Y can effectively improve the purity of the steel, refine the grain size, and improve the mechanical properties of steel. As a structural material for nuclear reactors, it is necessary to assess the thermal stability of Y-CLAM steel with Zr to ensure the structural integrity at prolonged exposure temperatures. The microstructure evolution of Eurofer 97, F82H, and CLAM after thermal aging had been described as precipitation of Laves phase, coarsening of grains and carbides, causing degradation in material strength and toughness [14]. However, few studies were conducted on the aging effects of Y-CLAM steel with Zr at 550 °C. The aim of this study was to investigate the evolution of the microstructures and mechanical properties of Zr-containing Y-CLAM steel that has been aged at 550 °C (the upper limit operating temperature) for up to 8000 h.

2 Materials and Experiments

2.1 Materials

The alloy used in this study was prepared by the vacuum induction melting method, and its chemical composition is given in Table 1. The ingot was first hot-forged into a 35 mm × 50 mm plate and then hot-rolled into a 12-mm-thick plate at temperatures between 950 and 1100 °C. The plate was normalized at 1050 °C for 0.5 h and then tempered at 750 °C for 1.5 h. All the samples were air-cooled. The tempered specimens were aged at 550 °C for 1000, 1500, 3000, 5000, and 8000 h in an air atmosphere to study their microstructural stability and mechanical properties.

2.2 Measurement of Particle Size and Number

The most common second phases in RAFM steel are $M_{23}C_6$ ($M = \text{Cr, Fe}$), MX , Laves phases (Fe, Cr, W), and the Z phase (Cr, V, N) [14]. Backscattered electron (BSE) imaging was conducted to clearly identify the particles in the samples because different types of particles show different Z-contrasts in the matrix [15]. Hald [16] reported that the Z phase (Cr, V, N) can only be found in 9Cr steel after 30,000–40,000 h of thermal aging at 650 °C. The fine MX particles (< 40 nm) cannot be detected in scanning electron microscope (SEM) images at low magnification because of the resolution limit of the SEM. Unlike the $M_{23}C_6$ precipitates, the W-rich Laves phase precipitates

are visible as bright precipitates in a dark matrix, such that the Laves phases can be clearly distinguished in the BSE images at 2500× magnification. The particles were counted using 15–20 BSE images from each sample. The mean spatial particle diameter ($\bar{d}_V$) can be expressed as [17]

$$\bar{d}_V = \pi \cdot n / 2 \sum_{i=1}^n (1/d_{Ai}),$$

where n is the total number of particles in the observed area and d_{Ai} is the equivalent diameter, which is estimated as the diameter of a circle with the same area of a sectioned inclusion phase.

The number of particles per unit volume (N_V) was evaluated from the following relationship [17]:

$$N_V = N_A / \bar{d}_V.$$

The volume fraction of the particle sections (f_V) can be expressed as [17]

$$f_V = \sum_{i=1}^n (\pi \cdot d_{Ai}^2 / 4) / A_{\text{obs}},$$

where A_{obs} is the total observed area.

2.3 Experiment

Optical microscopy (OM), SEM, transmission electron microscopy (TEM), impact test, and tensile test samples were spark machined from the aging plates along the rolling direction. Villella's reagent (1 g muriatic acid, 5 mL picric acid, and 100 mL alcohol) was used to etch the OM and SEM samples. The TEM samples were twin-jet polished using an electrolyte solution (95% acetic acid and 5% perchloric acid) at 25 °C. Bar samples (25 mm length, 5.0 mm diameter) were used for the tensile tests at room temperature. Dog-bone-shaped flat specimens (6.0 mm × 2.0 mm × 30 mm) were used for the tensile tests at 600 °C. The impact tests were conducted using full-size V-notch Charpy specimens in the range of −140 °C to 25 °C, with the Charpy impact transition curves determined by fitting the Boltzmann function to the impact test data, to obtain the ductile–brittle transition temperature (DBTT). The hardness of the samples was measured with an FV-800 hardness tester.

Table 1 Chemical composition of the steel (wt.%)

C	Si	Mn	Cr	W	V	Ta	Y	S	P	N	O	Zr
0.11	0.05	0.45	9.2	1.50	0.22	0.15	0.043	0.005	0.0078	0.0157	0.0070	0.013

3 Results and Discussion

3.1 Microstructure

OM micrographs of the thermally aged alloys are shown in Fig. 1, with typical martensitic structures observed in the samples. There was an obvious trend in grain growth, as shown in Fig. 2, where the average grain size increased during the thermal aging process and began to stabilize at the longest aging times. The grain size of the alloys increased from 11.2 to 14.2 μm after 8000 h of thermal aging, which was $\sim 26.8\%$ higher than that of the as-received sample. This grain coarsening is a spontaneous process, where the surface energy is the driving force for the migration of grain boundaries [18]. Therefore, grain coarsening is an obvious characteristic of microstructure evolution during the thermal aging process, which affects the mechanical properties of the alloy [14].

Details of the alloy microstructures are shown in Fig. 3. Typical martensitic laths were observed in the thermally aged alloy. The microstructures underwent minimal changes, with the exception of lath coarsening during the thermal aging process. The width of the as-received lath martensite sample was 0.29 μm (Fig. 3a), which increased by 45% after 1500 h of aging (0.42 μm) (Fig. 3b). The martensite lath migrated and merged during the thermal aging process, with the width of the lath doubling to 0.58 μm after 8000 h of thermal aging. Furthermore,

Fig. 2 Grain size evolution

subgrains were observed in the sample that was thermally aged for 8000 h.

The misorientation angle distribution in the samples is shown in Fig. 4. The thermal aging time had a significant effect on angle distribution, particularly at the low-angle grain boundary (LAB: 4° – 10°) and the high-angle grain boundary (HAB: 50° – 60°). The same result was also reported by Wang et al. [19] and Li et al. [20]. The LAB proportion gradually increased, whereas the HAB proportion decreased during the thermal aging process [21]. The

Fig. 1 Microstructure images of the as-received and aged samples: **a** as-received, **b** 1000 h, **c** 1500 h, **d** 3000 h, **e** 5000 h, and **f** 8000 h

Fig. 3 TEM images of the as-received and aged samples: **a** as-received, **b** 1500 h, **c** 5000 h, and **d** 8000 h

Fig. 4 Misorientation angle distributions in the samples

increase in the LAB proportion indicates that subgrains gradually formed during the thermal aging process, which is consistent with the results in Fig. 3. The reason for the

decrease in HABs can be grain coarsening, which reduced the HABs per unit volume.

3.2 Physical Properties of the Laves Phase Precipitates

A BSE image of the alloy before and after thermal aging is shown in Fig. 5, where the bright particles are Laves phase precipitates and the gray particles are $M_{23}C_6$ carbide particles. There were hardly any Laves phases observed in the as-received sample because the tempering temperature (750 °C) was higher than the precipitation temperature of the Laves phases (650 °C [4, 11–13]), as shown in Fig. 5a. Some fine Laves phases can be found in the sample that was thermally aged for 1000 h (Fig. 5b). The number and size of the Laves phases in the sample that was thermally aged for 1500 h significantly increased (Fig. 5c), indicating that the Laves phases in the thermally aged samples simultaneously grew and precipitated. The Laves phases primarily precipitated at the grain and martensitic lath boundaries.

The physical properties of the Laves phase precipitates, which were calculated from the BSE images, are shown in Fig. 6. Rapid Laves phase precipitation occurred within

Fig. 5 BSE images of the as-received and aged steels: **a** as-received, **b** 1000 h, **c** 1500 h, **d** 3000 h, **e** 5000 h, and **f** 8000 h

3000 h, with observed increases in both the density and size of the precipitates, resulting in a rapid increase in volume fraction. The volume fraction of the Laves phase precipitates stabilized after 3000 h of thermal aging and remained approximately constant with further thermal aging. The density of the Laves phase precipitates began to significantly decrease after 3000 h of thermal aging, but the size of the Laves phase precipitates continued to increase. This observation is consistent with the Ostwald ripening mechanism, which states that the larger particles grow by drawing materials from the smaller particles [22].

The relationship between the volume fraction of the Laves phase precipitates and thermal aging time can be described by the Johnson–Mehl–Avrami equation [23]:

$$f_V(t) = 1 - \exp(-(t/t_0)^n),$$

where t is the time (s), t_0 is the time constant, and n is a time exponent that is dependent on the type of phase transition, particularly the nucleation and growth mechanisms. The precipitation of the second phase at the grain boundary is defined by $n = 1.5$ when the nucleation rate is constant, and $n = 0.5$ when the nucleation rate rapidly decreases to zero for the diffusion-controlled phase transition. The nucleation mechanism of the Laves phase precipitates was investigated using n , which was calculated from the Johnson–Mehl–Avrami equation, where $n = 0.37$ based on the observed physical properties of the Laves phase precipitates (Fig. 7). The Laves phase precipitates primarily precipitated at the grain and martensitic lath boundaries, as shown in Fig. 5, which indicates that $n = 0.5$ – 1.5 [23]. The observed n value (0.37), which is close to 0.5, indicates that

the nucleation rate of the Laves phase precipitates rapidly decreased. The saturation phenomenon of the nucleation position can easily occur because the grain boundary area was limited. However, further nucleation was not possible because of the absence of a suitable nucleation location, which would result in a rapid decay in the nucleation rate. Furthermore, the segregation of W at the grain boundaries would accelerate Laves phase precipitation [24].

3.3 TEM–EDS Results

Some precipitates were $M_{23}C_6$ particles (50–120 nm length, 30–80 nm width), which were observed along the grain boundaries in the bright-field image, EDS image (Fig. 8c), and diffraction pattern; these precipitates have frequently been found in other RAfM steels [1–4]. The $M_{23}C_6$ particles were notably coarsened (70–200 nm length, 50–110 nm width) after 8000 h of thermal aging at 550 °C (Fig. 8b). Furthermore, W can be found in the $M_{23}C_6$ particles in the thermally aged alloy (Fig. 8d), which would reduce solid-solution strengthening via Wang et al. [14] evaluated the coarsening behavior of $M_{23}C_6$ in CLAM steel during the thermal aging process and obtained $M_{23}C_6$ particle diameters of 100, 130, 180, and 175 nm before and after 2000, 4000, and 10,000 h of thermal aging at 550 °C [25]. The volume fraction of the $M_{23}C_6$ carbides in 9Cr steel was 0.6–1.5% at 550 °C. The precipitate contribution to the yield strength can be expressed by Orowan strengthening [24]:

$$\sigma_{\text{Orowan}} = 3.32 G b \sqrt{f_v} / \bar{d}_v,$$

where σ_{Orowan} is the Orowan yield stress (MPa), G is the shear modulus (76 GPa), and b is the Burgers vector

Fig. 6 Physical properties of the Laves phase precipitates in the alloy: **a** mean spatial diameter, **b** number of particles per unit volume, and **c** volume fraction

Fig. 7 Evaluation of the time exponent (n) based on the physical properties of the Laves phase precipitates

(0.2487 mm) [3]. The σ_{Orowan} values of the $M_{23}C_6$ carbides with thermal aging time in steel, which are based on Wang's results and the aforementioned Orowan strengthening model, are given in Fig. 9, where the yield stress of the $M_{23}C_6$ carbides gradually decreases during the thermal aging process.

The Laves phase precipitates formed and coarsened during the thermal aging process. They had an elongated shape after 1500 h of thermal aging, as shown in Fig. 10a. The bright-field and EDS images, and the diffraction pattern indicated that the Laves precipitates (252 nm length, 51 nm width) were generally much larger than the $M_{23}C_6$ particles and then underwent rapid grain coarsening with continued thermal aging, with large Laves phase precipitates (365 nm length, 188 nm width) observed after 8000 h of thermal aging, as shown in Fig. 10b. The Orowan stress of the Laves phase precipitates with thermal aging time is shown in Fig. 9, where a rapid decrease occurred between 1000 and

Fig. 8 TEM micrographs (a, b) and EDS results (c and d) of the $M_{23}C_6$ carbides in the as-received alloy (a, c) and sample that was thermally aged for 8000 h (b, d)

Fig. 9 Orowan yield stresses of the $M_{23}C_6$ carbides (black) and the Laves phase precipitates (red) with thermal aging time

5000 h, stabilizing at ~ 15 MPa with further thermal aging. There was good correspondence between the Orowan stress changes and the precipitation and coarsening of the Laves phase precipitates, with the Orowan stresses being much lower than those of the $M_{23}C_6$ carbides.

Figure 11 shows that some small particles precipitated in the alloys. Fine precipitates were present in the as-received alloy, as shown in Fig. 11a, with the EDS results indicating that they primarily comprised Ta, Fe, and Cr (Fig. 11e). The bright-field and EDS images, and the diffraction pattern identified the precipitates as TaC particles that were 10–25 nm in size. A small degree of growth was observed in these TaC precipitates after 3000 h of thermal aging (Fig. 11b). New particles (NPs) formed, which were ~ 30 nm in size, after 5000 h of thermal aging (Fig. 11c), with the EDS results and diffraction pattern (Fig. 11f) identifying these NPs as V_3Zr_3C (Cubic, Fd-3 mS, $a=b=c=1.212$ nm, $\alpha=\beta=\gamma=90^\circ$). The MX particles coarsened after 8000 h of thermal aging, with grain sizes in the 15–40 nm range (Fig. 11d). Furthermore, Ta-rich MX precipitates and V_3Zr_3C particles were frequently observed along the dislocation planes, which indicated that the dislocation movement would be effectively impeded by these particles.

3.4 Evolution of Mechanical Properties

Hardness is an important property that is indicative of the degree of material aging, because a decrease in hardness results in a decrease in the creep properties of heat-resistant

Fig. 10 TEM micrographs (a, b) and EDS results (c, d) of the samples that were thermally aged for 1500 h (a, b) and 8000 h (c, d)

steel [26, 27]. The effect of thermal aging time on alloy hardness is demonstrated in Fig. 12, where the hardness first decreased with thermal aging, stabilized at 3000 h, peaked at 5000 h, and then rapidly decreased with further thermal aging. The mean hardness of the as-received sample was 231 HV, which decreased to 216 HV after 8000 h of thermal aging. The hardness peak at 5000 h was 233 HV, which was due to the new V- and Zr-containing precipitates. The dislocation mobility can be effectively hindered by the MX carbides and the V- and Zr-containing precipitates, as shown in Fig. 4, which was responsible for the increased hardness. The grain coarsening of carbides resulted in the decreased hardness after 8000 h of thermal aging.

The effect of thermal aging on alloy strength is shown in Fig. 13. The strength first decreased and then increased, with an inflection point and peak strength at 1500 and 5000 h, respectively. The yield strength (σ_y) of materials during the thermal aging process can be expressed as [28]

$$\sigma_y = \sigma_p + \sigma_s + \sigma_M,$$

where σ_p is the contribution of precipitation strengthening, σ_s is the contribution of solution strengthening, and σ_M is the contribution of the matrix (e.g., microstructure, grain

size). The rapid decrease in $M_{23}C_6$ Orowan stress (Fig. 9), coupled with the grain coarsening (Fig. 2), during the early stages of thermal aging led to the decrease in yield strength (574 MPa $\rightarrow$ 546 MPa). The subsequent increase in yield strength (546 MPa $\rightarrow$ 583 MPa) was due to the precipitation of the Laves phases and V_3Zr_3C particles. Both the size and volume fraction of the precipitates in the alloy stabilized after 5000 h of thermal aging, with the yield strength slightly decreasing (583 MPa $\rightarrow$ 543 MPa) because of additional grain coarsening after further thermal aging. The elongation fluctuated between 20% and 24% during the thermal aging process.

The impact energy of the samples is shown in Fig. 14, with the DBTT obtained by fitting the Boltzmann function to the Charpy impact data [4, 11–13]. The grain size and second phase of the precipitates were the primary factors that effectively influenced the observed impact properties [4]. The effect of grain size on alloy toughness is primarily manifested by hindering microcrack growth along the grain boundaries in the materials. Grain refinement can effectively increase the intergranular area per unit volume and then increase the resistance to microcracking. However, not all types of grain boundaries can effectively

Fig. 11 TEM images (a–d) and EDS results (e, f) of the MX carbides in the as-received and thermally aged samples: as-received (a, e), 3000 h (b), 5000 h (c, f), and 8000 h (d)

hinder microcrack growth. It has been reported that the effect of HABs, such as austenite grain boundaries, on crack growth was much greater than that of LABs (e.g., subgrain boundaries) [29]. Although some LABs formed during the thermal aging process, they cannot effectively hinder crack growth and improve the impact properties of the alloy. The phase boundary between the second phase and the matrix not only effectively prevented crack propagation but also easily initiated microcracks. The low-temperature brittleness of the alloy was due to the reduced fracture strength of the alloy relative to its yield strength [30]. Cracks will form and rapidly expand in the material once the stress is greater than the fracture strength,

particularly at the grain or phase boundary, such that brittle fracture will occur without plastic deformation. The critical fracture stress (σ_{cf}) can be expressed as follows [30]:

$$\sigma_{cf} = \sqrt{\pi E / (1 - \nu^2)} \cdot \sqrt{\gamma / D},$$

where E is the Young's modulus, ν is the Poisson ratio, γ is the interfacial energy at the particle–matrix interface (0.4–1.1 J/m² [23]) or matrix–matrix interface (1–2.1 J/m² [23]), and D is the grain or second phase size. E and ν were constant for the thermally aged alloys. The critical fracture stress equation can be rewritten as

Fig. 12 Effect of thermal aging on alloy hardness

Fig. 13 Effect of thermal aging on alloy strength

Fig. 14 Impact curves of the as-received and thermally aged samples

$$\sigma_{cf} = A \cdot \sqrt{\gamma/D},$$

where

$$A = \sqrt{\pi E / (1 - \nu^2)},$$

which is constant for the thermally aged alloys. The size ranges of the second phase and grains in the as-received and thermally aged alloys were 10–355 nm and 11.2–14.2 μm , respectively. The critical fracture stress caused by the grain size was 0.27–0.43 A, and the critical fracture stress caused by the second phase was 1.06–10.49 A. Therefore, the change in fracture stress of steel as a function of thermal aging was primarily controlled by the grain size. The toughness of the steel alloy decreased with increasing grain size in the thermally aged alloy, and the DBTT increased from -71 to -48 $^{\circ}\text{C}$ during the thermal aging process.

4 Conclusions

The thermal stability and mechanical properties of Zr-containing RAFM steel during thermal aging at 550 $^{\circ}\text{C}$ for up to 8000 h were studied. The main results are summarized below:

1. The lath martensite morphology of Zr-containing RAFM steel is maintained after thermal aging at 550 $^{\circ}\text{C}$ for 8000 h. The average grain size of the steel gradually increased with aging and tended to be stable.
2. The $M_{23}C_6$ particles underwent significant grain coarsening after 8000 h of thermal aging with lengths of 70–200 nm and widths of 50–110 nm observed. There was a slight increase in the size of the TaC precipitates with thermal aging. Some NPs (V_3Zr_3C), ~ 30 nm in size, were observed in the sample after 5000 h of thermal aging, which effectively impeded dislocation movement to improve the mechanical property of the steel.
3. The Laves phase precipitates formed along the grain and martensite lath boundaries after 1000 h of thermal aging, and grew rapidly. The Laves phase precipitates increased monotonically with thermal aging, and the volume fraction of the Laves phase precipitates in the alloy stabilized at $\sim 0.17\%$ after 3000 h of thermal aging. Both the strength and the hardness of the alloy decreased with thermal aging time, although a hardness peak did appear at 5000 h due to the precipitation of Laves phase particles.
4. The yield strength of the steel decreased from 574 to 543 MPa, the tensile strength decreased from 685 to 680 MPa, and the DBTT increased from -71 to -48 $^{\circ}\text{C}$ after thermal aging for 8000 h because of the coarsening of grains and the second phases.

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