Relationship Between Microstructure and Corrosion Behavior of Cr12Ni3Co12Mo4W Ultra-High-Strength Martensitic Stainless Steel

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Abstract The effect of tempering temperature on the microstructure and corrosion behavior of Cr12Ni3Co12Mo4W ultra-high-strength martensitic stainless steel was investigated using transmission electron microscopy, atomic force microscopy, X-ray diffraction, and electrochemical tests. The microstructures of the ultra-high-strength martensitic stainless steel consisted of some retained austenite and lath/plant martensite with the carbides distributed within the matrix and at the grain boundaries. Tempering of the steel for 4 h at various temperatures resulted in various carbide grain sizes and different amounts of the retained austenite. The results showed that larger carbide grains led to diminished corrosion resistance, whereas larger amounts of the retained austenite resulted in improved corrosion resistance. The steels exhibited good corrosion resistance in 0.017 mol/L NaCl solution and exhibited pitting corrosion in 0.17 mol/L NaCl solution. The martensite and prior austenite crystal boundaries dissolved in solution with pH 1.

KEY WORDS: Heat treatment; Martensite; Retained austenite; Carbide; Corrosion behavior

1 Introduction

Applications such as tissue engineering, aircraft devices, and ocean equipment require steels with a combination of high specific strength and good durability. The use of multi-component martensitic stainless steels in high stress and corrosive environments requires a better understanding of the passive film formation, the breakdown behavior during exposure to aggressive ions, and the consequent effects on pitting or failure. Previous studies have demonstrated that the corrosion resistance

of the martensitic stainless steel is generally better in the hardened condition than in the annealed or soft condition [1–5]. Martensitic stainless steels containing (11–13) % Cr exhibit good electrochemical properties. Sun et al. studied the electrochemical behaviors of Cr9Ni5MoCo14 and Cr12Ni4Mo2Co14 martensitic stainless steels, and their results indicated that the steels' passive films dramatically affected their corrosion resistances [6, 7].

Because the properties of the martensitic stainless steels can be greatly changed by heat treatment, therefore the heat treatment parameters must be carefully controlled to ensure that the steels possess appropriate structures achieving the desired properties. For example, the amount and the grain size of the carbide precipitates can affect the steel's hardness [8–12]. Martensitic stainless steel consists of the martensite, undissolved carbides, and retained austenite [13] and exhibits greater strength than austenite, which makes the steel achieve ultra-high strength in the martensitic steels. However, the poor corrosion resistance of the martensite is problematic [14].

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Heat treatment parameters play a critical role in producing suitable microstructures and can affect the mechanical properties and corrosion behavior of the steels [15–19].

The presence of the retained austenite, making the austenite fail to transform into martensite during the cooling process, may positively affect the wear resistances, fatigue properties, and ductility of the steels. However, the retained austenite deteriorates the pitting corrosion resistances of the steels because it results in the formation of Cr-depleted zones [15]. Li *et al.* [20] deduced that the coarsened grains were the main contributor to good intergranular corrosion resistance. Choi *et al.* [21] concluded that the better corrosion resistance of the martensitic stainless steels with 13% Cr is related to the dissolution of the carbides, whereas the precipitation of Cr-rich $M_{23}C_6$ and M_7C_3 carbides reduces the resistance of passive films, enhancing pitting corrosion. Bilmes *et al.* reported that the presence and amount of the retained austenite as a microstructural component resulting from the applied heat treatments can improve the pitting corrosion resistances of both weld and parent metal 13CrNiMo stainless steels [22]. Most of the previous researches have focused on the microstructures and corrosion behaviors of the austenite stainless steel [23–26] and the martensitic stainless steel. However, the literature contains few studies on the effect of the microstructure on the corrosion behavior of ultra-high-strength martensitic stainless steel (UHSMSS).

Further work based on our previous studies is underway on the subject of passivity and pitting corrosion of Cr12Ni3Co12Mo4W in support of our principal objective of elucidating the correlations among the heat treatment process and the steel's microstructure, mechanical properties, and corrosion behavior. We are seeking new insights to enhance the corrosion resistance of this steel, which combines the advantages of the martensite and stainless steel.

In the present study, we investigated the effects of heat treatment on the microstructure and corrosion behavior of a novel UHSMSS by using transmission electron microscopy (TEM), atomic force microscopy, X-ray diffraction (XRD), and electrochemical analyses. The samples with various carbide grain sizes and different amounts of the retained austenite were tempered at different temperature for 4 h.

2 Experimental

2.1 Experimental Material

Cr12Ni3Co12Mo4W UHSMSS with the chemical composition listed in Table 1 was used in this study. The UHSMSS was heated by austenitizing at 1080 °C for 30 min, oil quenching to room temperature, followed by treatment for 1 h at −73 °C to transform the retained austenite, and finally tempering at 400, 520, and 600 °C for 4 h. A schematic of the heat treatment process is shown in Fig. 1.

The samples were ground, polished, and then etched with a solution (HF: HCl: H₂O = 4:4:92) to reveal the microstructure under a stereologic microscope (KEYENCE VHX-2000C). The microstructure of UHSMSS consists of some retained austenite and lath/plant martensite with the carbides distributed within the matrix and at the grain boundaries.

2.2 Transmission Electron Microscopy and Atomic Force Microscopy

A JEM-2100F high-resolution transmission electron microscope operated at 200 kV was used to observe the microstructures of as-prepared NPC specimens. The specimens were thinned by a Gatan 691 precision ion polishing system using Ar ions at 5 kV.

The surface height and potential morphology of the etched surface of the Cr12Ni3Co12Mo4W UHSMSS tempered at 600 °C were observed using an atomic force microscope (Bruker MultiMode 8.0) in tapping mode.

2.3 X-ray Diffraction

Volume fractions of the retained austenite in three kinds of the samples were analyzed by Rietveld analysis of XRD data. The XRD patterns were collected at room temperature with a Philips PW 1710 diffractometer equipped with a diffracted-beam graphite monochromator. The data were collected using CuK_α radiation at angles of $10^\circ \leq 2\theta \leq 100^\circ$ in steps of 0.02° . Rietveld analysis was performed using the Fullprof program. The amounts of the retained austenite in three kinds of UHSMSSs, as determined by this analysis, are listed in Table 2.

Table 1 Chemical composition of the Cr12Ni3Co12Mo4W UHSMSS (wt%)

C	Cr	Co	Mo	Ni	W	V	Ti	Si	Mn
0.089	12.41	12.3	4.43	2.59	0.98	0.24	<0.005	0.053	<0.10

Fig. 1 Schematic of heat treatment process on the Cr12Ni3Co12-Mo4W UHSMSS

2.4 Electrochemical Tests

All of the samples were embedded in epoxy resin, leaving a working area of 1 cm²; the samples were subsequently ground to 1000 grit and cleaned with ethanol and deionized water before the tests. The test solutions were 0.1 mol/L Na₂SO₄ + 0.017 mol/L NaCl (pH 3) and 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3). All of the solutions were prepared from analytical-grade 98% H₂SO₄ and deionized water, and all of the tests were carried out at room temperature. The electrochemical measurements were taken in a conventional three-electrode cell using an M370 advanced electrochemical system. The UHSMSS samples were used as the working electrodes, a platinum sheet was used as the counter electrode, and all of the potentials were measured against a saturated calomel electrode (SCE) connected to the cell via a Luggin probe.

Prior to the electrochemical measurements, the working electrodes were initially reduced potentiostatically at $-1.2 V_{SCE}$ for 3 min to remove air-formed oxides. Electrochemical impedance spectroscopy (EIS) was performed using an applied potential amplitude of 10 mV at the open-circuit potential in the frequency range from 100 kHz to 10 mHz. The polarization curves were measured potentiodynamically from $-0.3 V$ (vs. corrosion potential E_{corr}) to $1.8 V_{SCE}$ at a scanning rate of 0.5 mV/s.

The surface morphologies of the UHSMSS were observed under a stereologic microscope (KEYENCE VHX-2000C) after the samples had been immersed for 10 min and subjected to a potentiodynamic polarization test in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 1) solution.

3 Results and Discussion

3.1 Microstructure of UHSMSS

The microstructures of the samples subjected to various heat treatments are shown in Fig. 2. The microstructure of UHSMSS consists of some retained austenite and lath/plant martensite with the carbides distributed within the matrix and at the grain boundaries. The lath martensites are arranged in parallel along the austenite grain boundaries. The amount of retained austenite (the blank areas in the figures) in the microstructure of the steel tempered at 400 °C is larger than those in the microstructures of the steel tempered at other temperatures.

The carbide precipitates at the boundaries of the martensite plates and within the martensite matrix were further inspected by TEM, as shown in Fig. 3. The microstructures of the steels subjected to various heat treatments are clearly different. The grain sizes of the carbides are listed in Table 2. No obvious carbide precipitates are evident in the UHSMSS sample tempered at 400 °C for 4 h (Fig. 3a). The carbide grain size is approximately 200 nm in the sample tempered at 520 °C (Fig. 3b) and decreases dramatically to 100 nm in the sample tempered at 600 °C (Fig. 3c). These carbides were indexed as $M_{23}C_6$ based on their corresponding electron diffraction patterns (Fig. 3d), similar to the carbides in 1Cr12NiMo martensitic stainless steel also reported by Fan [9].

The results in Fig. 3 also reveal that the martensite lath sizes differ in the microstructures of the steels subjected to different heat treatments. When the tempering temperature increases from 400 to 520 °C, the martensite laths appear to be more refined. However, when the aging temperature increases from 520 to 600 °C, the martensite laths appear to be coarsened. We assume that, for the experimental steel, the best temperature for refining martensite lath is

Table 2 Microstructure of Cr12Ni3Co12Mo4W UHSMSS subjected to various heat treatments

	400 °C	520 °C	600 °C
Microstructure	Martensite + retained austenite	Martensite + retained austenite + carbide	Martensite + retained austenite + carbide
Retained austenite	4.74%	0.4%	1.48%
Carbide grain size	0	200 nm	100 nm

Fig. 2 Microstructures of the Cr12Ni3Co12Mo4W UHSMSS samples subjected to various heat treatments of **a** 400 °C, **b** 520 °C, **c** 600 °C

520 °C. Refined martensite lath is generally considered to enhance the strength of martensitic stainless steels.

The TEM bright-field image (Fig. 3a) clearly shows that the retained austenite formed within the martensite matrix after tempering at 400 °C for 4 h and formed along the martensite lath boundaries in the specimen tempered at 600 °C (Fig. 3c). (Retained austenite formed within the martensite matrix and at the boundaries at both 400 and 600 °C; the images show local areas.) Retained austenite is hardly observable in the microstructure of UHSMSS tempered at 520 °C for 4 h. The amounts of the retained austenite in the microstructures of the UHSMSS samples subjected to different heat treatments, as measured by XRD, are listed in Table 2. The amount of the retained austenite is 4.74% in the UHSMSS sample tempered at 400 °C, which is twice the amount in the sample tempered at 600 °C. The smallest amount (0.4%) is evident for the sample tempered at 520 °C.

The height and potential morphology of the etched surface of the Cr12Ni3Co12Mo4W UHSMSS tempered at 600 °C are shown in Fig. 4. As evident in Fig. 4a, the surface height difference is approximately 200 nm, consistent with the size observed in Fig. 3. In addition, the 3D potential morphology shows that the surface potential of the UHSMSS is uniform. According to the results of the metallographic observations and TEM analysis, the martensite is the predominant phase in Fig. 4.

3.2 Effect of Heat Treatment on Microstructure

Martensitic stainless steels are commonly used in their quenched and tempered conditions. The quenching heat treatment involves annealing to obtain austenite and dissolve the carbides, followed by cooling to transform the austenite into martensite and induce carbide precipitation. The heat treatment parameters play a critical role in determining the carbide grain size and the amount of the retained austenite. Garcia *et al.* [27] reported that the

amount of the carbides in the quenching microstructures of 0.45–13Cr steel can be substantially modified through control of the heating and cooling conditions used in the heat treatment. Although they studied the influences of the heating parameters and the cooling rate on the carbide amounts, they did not investigate the influence of the tempered temperature.

The UHSMSS was tempered to transform the austenite into the martensite. The sample tempered at 400 °C contained more retained austenite than that tempered at 520 °C. We concluded that the amount of the retained austenite in the tempered microstructure of the UHSMSS decreases with increasing tempering temperature, resulting in more complete transformation. However, the amount of the retained austenite in the steel sample tempered at 520 °C was smaller than that in the sample tempered at 600 °C. We therefore concluded that the amount of the retained austenite in the tempered microstructure of the UHSMSS decreased as the tempering temperature increased from 400 to 520 °C and increased as the tempering temperature increased from 520 to 600 °C. Because the mechanical properties of the steels are known to be improved with decreasing amount of the retained austenite, a tempering temperature of 520 °C is the optimal condition for achieving excellent mechanical properties.

The carbides were dissolved during the annealing process, but were precipitated during the tempering process. For the test steel, the carbide grain size was largest in the sample tempered at 520 °C and was second largest in the sample tempered at 600 °C; by contrast, the carbides were hardly observed in the microstructure of the UHSMSS sample tempered at 400 °C. Because the carbides are known to cause high hardness of steels, the tempering temperature of 520 °C is optimal for achieving excellent mechanical properties; tempering at 600 °C is the second-best tempering condition, whereas tempering at 400 °C results in the lowest hardness. The optimal parameters for achieving a specific property vary for different steels, and

Fig. 3 TEM micrographs of the Cr12Ni3Co12Mo4W UHSMSS subjected to various tempered temperatures **a** 400 °C, **b** 520 °C, **c** 600 °C, **d** corresponding electron diffraction pattern from the carbide in **b**

the optimal parameters for achieving excellent mechanical properties are not always the best conditions for achieving good electrochemical properties.

4 Effect of Carbides on Corrosion Behavior

Figure 5 shows the potentiodynamic polarization curves of three kinds of steels measured in 0.1 mol/L Na₂SO₄ + 0.017 mol/L NaCl (pH 3) solution. As shown in

Fig. 4, the three kinds of steels exhibit good electrochemical characteristics in the mild environment. The polarization curves consist of four primary parts: (1) cathodic section (a–b), (2) anodic dissolution sections (b–c), (3) passivity sections (c–d), and (4) trans-passive sections (d–e). A similar phenomenon was also observed in the ultra-high-strength steel AerMet100 in H₂SO₄ + Na₂SO₄ solution [28].

The corrosion potential of the UHSMSS in 0.1 mol/L Na₂SO₄ + 0.017 mol/L NaCl (pH 3) solution is

Fig. 4 3D AFM height (a) and potential (b) morphologies of the etched surface of the Cr12Ni3Co12Mo4W UHSMSS tempered at 600 °C

Fig. 5 Potentiodynamic polarization curves for the UHSMSS in 0.1 mol/L Na₂SO₄ + 0.017 mol/L NaCl (pH 3) solution

Fig. 6 Potentiodynamic polarization curves for the UHSMSS in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution

approximately $-0.4 \text{ V}_{\text{SCE}}$, which is lower than that of some other ultra-high-strength steels such as AerMet100 steel [28]. The potential in the passivity region ranges from $-0.25 \text{ V}_{\text{SCE}}$ to $0.85 \text{ V}_{\text{SCE}}$. The low concentration of Cl^- did not cause pitting corrosion on the UHSMSS. The three kinds of UHSMSS subjected to different heat treatments exhibit excellent corrosion resistances in 0.1 mol/L Na₂SO₄ + 0.017 mol/L NaCl (pH 3) solution.

Figure 6 shows the potentiodynamic polarization curves for three steels measured in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution. The corrosion potentials for the steels are similar: approximately $-0.4 \text{ V}_{\text{SCE}}$. However, the pitting potentials differ substantially. The pitting potential of the steel tempered at 400 °C is the highest, at $0.85 \text{ V}_{\text{SCE}}$ (point d₃); thus, this steel exhibits the best pitting corrosion resistance. As shown in Fig. 3a, the microstructure of the steel tempered at 400 °C is mainly

lath/plant martensitic with a few carbide precipitates in the martensitic matrix. We assume that this microstructure of the steel contributed to its good corrosion resistance.

The pitting potential of the steel tempered at 520 °C is the lowest, at 0 V_{SCE} (point d₁). This sample exhibits the worst pitting corrosion resistance among the investigated samples. As shown in Fig. 3b, the microstructure of the steel tempered at 520 °C is mainly lath/plant martensitic, with some large carbide precipitates in the martensitic matrix. Choi *et al.* [21] reported that the better corrosion resistance of the martensitic stainless steels with 13% Cr is related to the dissolution of the carbides; the precipitations of Cr-rich $M_{23}\text{C}_6$ and $M_7\text{C}_3$ carbides reduce the resistances of passive films, thereby enhancing pitting corrosion. Based on the selected-area electron diffraction pattern, we indexed these large carbides as $M_{23}\text{C}_6$. We concluded that the carbide precipitation induces pitting corrosion. The

microstructure was uneven when the carbide precipitates were present; these steels were easily corroded because the potential of the carbide is lower than that of the martensitic matrix. Thus, the carbide concentration decreases, which leads to pitting.

The pitting potential of the steel tempered at 600 °C is 0.6 V_{SCE} (point d₂), which is in the intermediate between the potentials of the steels tempered at 400 and 520 °C. As shown in Fig. 3c, the microstructure of the steel tempered at 600 °C is mainly lath/plant martensitic, with a few small carbide precipitates in the martensitic matrix.

Figure 7 shows the carbide grain size plotted against the pitting potential E_p for steel samples subjected to different heat treatment conditions. The pitting potential, which is the breakdown potential of passivation, decreases from 0.85 V_{SCE} to 0 V_{SCE} as the carbide grain size increases from 0 to 200 nm, indicating that the bigger carbide grains result in greater susceptibility to pitting corrosion. We assume that the undissolved and precipitated carbides deteriorated the corrosion resistance of the specimen in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution [15].

We try to investigate the correlation between the carbide grain size and the pitting potential E_p for a given set of conditions. However, such a correlation is difficult to be discerned because E_p is affected by a combination of the testing parameters (e.g., exposure time, temperature, and scan range), the carbide grain size, and the texture of the material. Small variations in these parameters can result in large changes in E_p , especially variations in the potentials of the experimental samples or in the solution temperature. In fact, efforts have been made to elucidate a relationship between the grain size and the pitting potential, but a specific relationship was difficult to be observed [20].

Fig. 7 Curves of pitting potential versus carbide grain size and amount of the retained austenite

Fig. 8 EIS results of the UHSMSS in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution

Figure 8 shows the EIS results for the steel specimens in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution. Similar impedance features are observable among the three samples; the electrochemical equivalent circuits are also shown in Fig. 8 (insert), where R_s is the solution resistance, CPE_f is the double-charge-layer capacitance, R_f is the charge-transfer resistance, CPE_{dl} is the capacitance of the corrosion product formed on the electrode surface, and R_t is the resistance of the corrosion product. The fitted parameters are given in Table 3. In the case of the steel tempered at 520 °C, the carbide grain size is the largest, R_f is small, and CPE_f is large; thus, this sample exhibits the worst corrosion resistance among the investigated samples. By contrast, in the case of the steel tempered at 400 °C, R_f is the largest and CPE_f is the smallest; thus, this sample exhibits the best corrosion resistance among the investigated samples.

4.1 Effect of Retained Austenite on Corrosion Behavior

Figure 7 also shows the amount of the retained austenite plotted against the pitting potential E_p for the steels heat treated under different conditions. E_p increases with increasing amount of the retained austenite. The shift of E_p in the more noble direction with increasing retained austenite content indicates the enhanced pitting resistance [22, 29]. We assume that the retained austenite promotes the corrosion resistance of the steel in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 3) solution.

It is generally accepted that it affects strongly the electrochemical performances of the martensitic stainless steels. Retained austenite deteriorates the pitting corrosion resistance because of the formation of Cr-depleted zones [30]. Although retained austenite has been demonstrated to deteriorate the tensile strengths and ductilities of the steels,

Table 3 Values of fitting parameters observed from the Nyquist diagrams

	R_s (Ω cm ²)	CPE_f (μ F cm ⁻²)	R_f (Ω cm ²)	CPE_{dl} (μ F cm ⁻²)	R_t (Ω cm ²)
400 °C	12.72	0.0001562	7417	0.002058	8295
520 °C	10.66	0.0001866	2987	0.008394	4158
600 °C	8.487	0.0001476	6705	0.002451	5626

Fig. 9 Surface morphologies of the UHSMSS tempered at 400 °C (a, c, d) and 600 °C (b) after immersion for 10 min (a, b) and potentiodynamic polarization test (c, d) in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 1) solution

it nonetheless exists in our martensitic stainless steel. As previously illustrated, the steel tempered at 400 °C for 4 h, which contains the largest amount of the retained austenite among the three investigated steel samples, exhibits the best resistance to pitting corrosion. Isolating the carbide grain size effect from the retained austenite effect is difficult. Therefore, we concluded that both the grain refinement and the increase in the amount of the retained austenite contribute to the improvement of the pitting corrosion resistance of UHSMSS. However, compared with the effect of the increase in the amount of retained

austenite, the carbide grain refinement is the main contributor.

Figure 9 shows the surface morphologies of the UHSMSS tempered at 400 and 600 °C after immersion for 10 min and potentiodynamic polarization tests in 0.1 mol/L Na₂SO₄ + 0.17 mol/L NaCl (pH 1) solution. As evident from Fig. 9a, b, when the UHSMSS was immersed in the strongly acidic solution, the surface state was clearly altered, initially by dissolution of the martensite and prior to austenite crystal boundaries. The UHSMSS exhibits pits with depths on the order of tens of microns in the solution with pH 1.

5 Conclusions

1. The microstructure of UHSMSS consists of retained austenite and lath/plant martensite with the carbides distributed within the matrix and at the boundaries.
2. The carbide grain size and the amount of the retained austenite are affected by the tempering temperature. The amount of the retained austenite in the microstructure of the UHSMSS decreases as the tempering temperature increases from 400 to 520 °C and increases as the tempering temperature increases from 520 to 600 °C. The carbide grain size increases as the tempering temperature increases from 400 to 520 °C and decreases as the tempering temperature increases from 520 to 600 °C.
3. The carbides can induce pitting corrosion because larger carbide grains result in a lower pitting potential, thereby diminishing the steel's corrosion resistance. By contrast, the retained austenite improves the corrosion resistance of UHSMSS.

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