Effects of Si and Sn Contents and Heat-Treatment Temperature on the Corrosion Behavior of AISI 439 Ferrite Stainless Steel

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Abstract: This study was conducted to evaluate the corrosion resistance and optimize the heat-treatment process of AISI 439 ferrite stainless steel silicon and tin alloys with reduced chromium. The microstructure of the specimens and deposition under each condition were analyzed. The production of oxide films was compared based on the thickness of the film and the change in the contents of each element. In addition, electrochemical analyses of each heat-treatment condition was used to quantitatively compare corrosion resistance and passive film stability based on the relative chromium, silicon, and tin contents. It was found that the addition of silicon and tin compensated for the decrease in corrosion resistance induced by the chromium reduction. The addition of the two elements inhibited iron (Fe) oxide production in the surface oxide film, thereby improving the corrosion resistance of the material and improving the stability of the passive film. Moreover, the SiO_2 and SnO_2 layers inhibited the production of Fe oxide and contributed to the stability of the film along with Cr_2O_3 , the main component of the passive film. However, when the heat treatment temperature increased above a specific temperature, the oxide inhibitory effect of the two elements was relatively offset. Nevertheless, further research to optimize the content of the three elements will help develop materials with superior mechanical properties and corrosion resistance.

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Keywords: ferrite stainless steel, corrosion resistance, oxide layers, passive film, pitting corrosion

1. Introduction

Stainless steel is a type of steel with excellent corrosion resistance compared to ordinary steel materials, due to a passive film. The passive film is an ultra-thin film formed during the initial stainless steel oxidation process. This film plays an important role in the corrosion resistance of stainless steel because it prevents the exposure of the base metal to the external environment, thereby preventing oxidation of the surface. A wide variety of stainless steel species are available, depending on the intended purpose and field of use. These materials are applied extensively, in petrochemical industries, power plants, plumbing, facilities, home appliances, and household goods [1,2]. Compared to austenite stainless steel, ferrite stainless steel contains only a small amount of expensive nickel (Ni), making it relatively inexpensive. In

addition, this steel species has a good surface gloss, formability, and oxidation resistance. Hence, it can be used for various applications, including heat-resistant appliances, enclosures, home appliances, and electronic components. Moreover, ferrite stainless steel is economical and has excellent corrosion resistance, in line with the demand for inexpensive and efficient steel materials. Accordingly, many studies have been conducted on ferrite stainless steel, which has been recommended as an alternative to steel materials in various fields [3].

The corrosion resistance of most stainless steel is generally superior to other steel species. However, stainless steel is highly susceptible to pitting corrosion due to destruction of the passive film [4]. Pitting corrosion results when anodic dissolution occurs in a limited area of the material surface covered with the passive film. In particular, pitting corrosion can occur when the external environment destroys the passive film locally, and the melting rate of the film is faster than the recovery rate. Pitting corrosion arises with specific reactive ionic atmospheres and electrochemical potentials,

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including chlorine ions (Cl^-), and ultimately shortens the life span of the stainless steel [5]. Although several studies have been conducted on the reduction of the lifespan of stainless steel by pitting corrosion, not many have reported on the corrosion mechanisms and their causes. Therefore, further research is necessary to improve the corrosion resistance of stainless steel and its resistance to pitting corrosion, which is a globally important problem.

Stainless steel undergoes several heat-treatment processes during its manufacture. The secondary annealing process, following roll pressing, is performed several times in a high-temperature atmosphere to effectively remove hydrogen embrittlement inside the material [6]. However, this process results in rapid iron oxidation on the surface. Although the oxide produced on the surface is typically eliminated through an acid pickling process, it is complicated and challenging to eliminate the iron oxidation, and is thereby inefficient for production. Furthermore, the generation of precipitates, including chromium (Cr) carbide and titanium (Ti) nitride, can induce galvanic effects in the material, which is fatal to the corrosion resistance and the lifespan of the material [1,7]. Accordingly, there are ongoing efforts to study and develop steel species to minimize the generation of iron (Fe) oxide on the Fe surface, and simultaneously improve the corrosion resistance of the materials.

AISI 439 ferrite stainless steel typically contains 17-19 wt.% Cr. Chromium is one of the main constituent elements of stainless steel, essential for improving corrosion resistance. During the initial oxidation process of stainless steel, the Cr_2O_3 passive film plays a key role in preventing corrosion. Several studies have been conducted on how Cr content affects various properties of stainless steel. Generally, the corrosion resistance increases as the Cr content of stainless steel increases. Specifically, studies have reported that every 1 wt.% increase in Cr content increases the value of the pitting potential, a major corrosion resistance indicator of stainless steel, by approximately 100 mV [8,9]. However, excessive Cr content degrades the mechanical properties of the stainless steel, such as formability, which limits the use of stainless steel in certain fields. With these considerations, the goal of the present study is to reduce Cr content to preserve various mechanical properties while supplementing corrosion resistance by adding other corrosion resistance-enhancing

elements. In this study, the decrease in corrosion resistance resulting from the Cr reduction was offset by adding silicon (Si) and tin (Sn). Si acts as a reducing agent and ferrite stabilizer in the manufacturing process. Si is also reported to inhibit the production of oxides on the iron surface [10,11]. Moreover, SiO_2 layers produced on iron surfaces enhance the stability of the passive film by inhibiting the iron oxidation of the base metal with a passive film, thereby improving the corrosion resistance of the material. In addition, an appropriate amount of Si improves the formability of stainless steel, thus improving its mechanical properties.

Adding small amounts of Sn to stainless steel can also improve its corrosion resistance and passive film stability. Similar to Si, Sn reacts with oxygen on the surface to produce SnO_2 layers, which has been reported to improve the stability of the passive film [5,12]. Moreover, the electromotive force (EMF) potential of Sn is -0.136 V, higher than iron (-0.440 V). Elements with higher EMF values will exhibit higher electrochemical stability. However, when the passive film of the stainless steel is destroyed, the galvanic effect of the potential difference between the Sn and Fe in the base metal accelerates the corrosion of the base metal [4,5,13]. Therefore, studies have reported that the addition of Sn in the range of 0.05 to 0.1 wt.% is optimal for effectively improving the corrosion resistance of stainless steel [12]. Based on the above studies, the present work evaluated corrosion resistance and optimized the heat-treatment process for Si and Sn alloys that replaced the Cr composition of AISI 439 ferrite stainless steel.

2. Experimental Methods

The stainless steel samples were acquired from POSCO Co., Ltd. (South Korea). The samples were produced by roll pressing a thin sheet with a 1 mm thickness. The composition of AISI 439 ferrite stainless steel samples are illustrated in Table 1, with (#A) containing 17 wt.% Cr and stainless alloys

Table 1. Components of ferritic stainless steel #A and #B

Specimen	Cr	Si	Sn	Ti	Mn	Fe
#A	17.6	0.15	-	0.35	0.1	bal
#B	15.2	0.98	0.057	0.35	0.1	bal

(#B) containing 15 wt.% Cr, Si, and Sn. Each sample was prepared with dimensions of 20 mm × 20 mm × 1 mm. In addition, to analyze the surface conditions before and after the heat treatment, grinding and polishing were performed from #600 grit to #2000 grit using a silicon carbide (SiC) paper, and micro-grinding was performed using a 1 μm diamond suspension.

Subsequently, heat treatment was conducted for a short time after the roll pressing process to remove the hydrogen brittleness generated during the production of stainless steel during the manufacturing process at POSCO. Each heat treatment was performed for one minute in a high-temperature atmosphere at 900, 950, and 1000 °C, followed by air-cooling. The obtained specimens were oxidized and etched in Viella's Reagent (ethanol + 10 mL/L HCl + 10 g/L picric acid) to analyze surface morphology and microstructure using an optical microscope (OM, Nikon, ECLIPSE LV150N, Japan). Additionally, a field-emission scanning electron microscope (FE-SEM, Hitachi S4800, Japan) and energy-dispersive X-ray spectrometer (EDS, ELECT PLUS, AMETEK, USA) were employed to obtain high-magnification images and component changes for each condition. Depth profiles (NEXA Thermophosphorous) were analyzed by X-ray photoelectron spectrophotometer (XPS) to quantitatively assess the component changes based on the depth direction of the specimen cross section. The analysis was conducted for 2500 s at a sputtering energy of 3 kV, sputter rate of 0.49 nm/s, and sputter size of 2 mm × 2 mm. An electrochemical analysis of each specimen was performed using a potentiostat (VersaSTAT 4, AMETEK, USA) to conduct a potentiodynamic polarization test, and an electrochemical impedance spectrometer (EIS, VersaSTAT 4, AMETEK, USA). A saturated calomel electrode (SCE) was used as the reference electrode. The relative electrode (CE) used a platinum mesh of 2 cm × 2 cm. Additionally, the gap between the working electrode (WE) and CE was set to 12 cm. The test was conducted at a temperature of 25 °C under the conditions of NaCl and H₂SO₄ mixed solution (0.5 M NaCl + 0.5 M H₂SO₄) according to ASTM G 5-14. The potentiodynamic polarization test was conducted at a scan rate of 0.167 mV/s under conditions of −0.2 V (vs OC) to 1.0 V (vs SCE) to quantitatively evaluate the corrosion resistance and electrochemical properties of the specimen.

EIS was conducted to measure the solution resistance (R_s) and polarization resistance (R_p), depending on the conditions of the specimen, and proceeded in the range of 1 Hz to 100 kHz.

3. Results and Discussions

3.1 Microstructure (Optical microscope)

Based on the heat-treatment conditions, the surface microstructures of specimens #A and #B were analyzed using an optical microscope, as shown in Figure 1. Similar precipitates, including Cr carbide and Ti nitride, were formed under heat-treatment conditions in both #A and #B. These deposits were randomly created inside the grain boundaries and grains. However, no significant changes were observed in the deposits produced at varying heat-treatment temperatures, due to the short heat-treatment time. Conversely, it was confirmed that the grain size of each specimen slightly increased with increasing heat-treatment temperature. In general, the grain growth occurred during the annealing process, due to an increase in the heat-treatment temperature of the stainless steel, thereby increasing the grain size. In

Fig. 1. Optical microscope image of surface microstructure according to heat treatment conditions.

Fig. 2. SEM images before and after corrosion of surfaces by heat treatment conditions of #A and #B specimen.

addition, studies have reported that the corrosion resistance of stainless steel is inversely proportional to the grain size. Thus, the contact area between the base metal and the corrosion environment increased with the grain size, allowing easy exposure to the corrosion environment [14,15].

3.2 Surface analysis (SEM and EDS)

The surface morphology of specimens under each condition was analyzed using SEM and EDS to compare and analyze the surface changes before and after corrosion, as shown in Figure 2. No significant changes were observed before and after corrosion from the SEM results. However, the formation of oxide films was common under all conditions, allowing the identification of non-uniform surfaces from the images. The results of the before- and after-corrosion specimen surface composition ratio measured by the EDS spectrum of the surface are shown in Table 2.

An increase in O content from 1 wt.% to approximately 30 wt.% was common for all specimens after corrosion. This is a common phenomenon, indicating that corrosion creates an oxide film on the surface. In addition, the contents of Cr and Fe were relatively reduced, indicating the production of Fe

Table 2. Results of EDS analysis of components before and after corrosion of surfaces by heat treatment conditions of #A and #B specimen

	wt.%	O	Cr	Fe	Si	Sn	Ti
#A Bal	Before	0.9	32.9	56.1	1.3	-	1.9
	After	34.1	12.8	38.3	2.5	-	13.3
#A 900 °C	Before	0.7	34.8	54.9	1.3	-	1.9
	After	32.9	12.7	38.6	2.7	-	13.0
#A 950 °C	Before	1.0	33.5	55.7	1.4	-	1.8
	After	34.4	15.1	38.9	3	-	8.6
#A 1000 °C	Before	0.9	33.0	55.9	1.3	-	2
	After	33.2	13.6	38.7	3.2	-	11.4
#B Bal	Before	1.2	31.3	59.5	2.8	1.1	1.7
	After	30.4	13.0	38.8	3.9	3.2	10.7
#B 900 °C	Before	1.0	29.3	57.4	2.6	1.0	1.5
	After	29.6	11.1	40.3	4.0	3.7	11.3
#B 950 °C	Before	0.7	29.4	57.9	2.8	1.4	1.5
	After	28.0	11.0	41.6	4.3	4.1	11.0
#B 1000 °C	Before	0.7	30.6	56.7	2.4	1.5	1.8
	After	29.3	11.8	40.4	3.9	3.4	11.3

oxide and Cr oxide in the oxide films.

Furthermore, in the case of Si present in specimens #A and #B, the Si content on the surface increased after corrosion, indicating that the oxide film consisted of Si oxide. Studies have reported that adding Si to stainless steel resulted in the formation of SiO₂ layers, which increased the stability of the passive film. These layers inhibited the oxidation of Fe by contributing to the stability of the passive film [16,17]. The amount of Si in specimen #B was approximately twice that in specimen #A, which was attributed to the difference in Si content. For specimen #B containing Sn, the Sn content increased by approximately 2 wt.% after corrosion. This indicated the formation of SnO₂ layers on the surface oxide film. The following chemical reaction can account for the formation of Sn oxide layers on the surface: [2,12,18]

It has been reported that the Si and Sn oxide films formed on stainless steel surfaces improved the stability of passive films by inhibiting the Fe oxidation of the base metal. Thus,

Fig. 3. Scanning electron microscope (SEM) analysis results for cross sections of #A and #B specimens under heat-treatment conditions.

the decreasing Fe oxidation ratio on the surface lead to the formation of Si and Sn oxide films, thereby contributing to enhancement of the passive film stability.

3.3 Cross-sectional analysis (Depth profiles)

SEM images of the cross-section of each specimen based on the heat treatment conditions are shown in Figure 3. No significant differences in the Cr, Si, and Sn contents were observed on the surfaces of the base metal. Thus, to quantitatively clarify the differences based on the conditions, depth profiles using XPS analysis were obtained, as shown in Figure 4. These profiles determined the changes in the content of each element based on the depth direction of the specimen. Etch level indicated the length, measured in the direction of depth from the surface.

In addition, the XPS analysis showed that the film thickness of the material was approximately 1 μm [19]. The oxide film thickness for each condition was relatively comparable through the intersection of Fe and O. For specimen

Fig. 4. Results of Depth profiles for each cross section by heat treatment conditions for #A and #B specimens.

Fig. 5. Results of potentiodynamic polarization test by heat treatment conditions of #A and #B specimens.

#A, the increasing temperature increased the Fe content and decreased the O content (etch levels 200 nm, 500 nm, and 600 nm). This suggests that the amount of Fe oxide produced on the surface increased as the heat-treatment temperature increased.

In contrast, the amount of oxide was constant at etch level 350 nm until 950 °C and subsequently increased to 750 nm from 1000 °C. Thus, the production of Fe oxide was inhibited up to 950 °C, and the effect of Si and Sn was relatively halved at 1000 °C. The (c, d) graph shows the change in the contents of Cr and Ti as the temperature increases. The contents of Cr and Ti on the surface decreased with the direction of the base metal, implying that the oxidation ratio of Cr and Ti on the surface increased with temperature.

The (e, f) graph shows the change in content based on the depth direction of Si and Sn [20]. For specimen #B with a high Si content, the ratio of oxidized Si on the surface was high. Moreover, Sn exhibited behavior similar to Si. This suggests that Si and Sn reacted with O to form oxide layers

on the surface of specimen #B, reducing the production rate of Fe oxide. Thus, the addition of Si and Sn contributed to an improvement in the passive film stability of the stainless steel.

3.4 Corrosion test

Graphs and data were obtained from the potentiodynamic polarization test results of each specimen for the various heat-treatment conditions, as shown in Figure 5 and Table 3. The overall corrosion resistance data of specimen #B, including Si and Sn, were relatively superior to specimen #A. For the corrosion potential (E_{corr}) indicating the corrosion resistance of the material, #A and #B were measured in the range of -580 to -570 mV and -540 to -530 mV, respectively. The corrosion potential of #B was approximately 40 mV higher than that of #A, implying an improved resistance to corrosion on the surfaces of the stainless steel, due to the effect of Si and Sn [2,21]. In addition, the E_{corr} of both specimens decreased when the temperature increased. This is considered to be a slight decrease in corrosion resistance, attributed to an increase in the material grain size.

The Nyquist plot of the EIS analysis allows comparison of the corrosion resistances of the #A and #B specimens, as shown in Figure 6 and Table 4. The R_s was measured at 4.0 ohms for all specimens. However, the R_p for #A and #B were measured in the range 280-325 ohms and 505-510 ohms, respectively, indicating that #B has better corrosion resistance than #A in the same corrosion environment [13,21]. Furthermore, the polarization resistance of specimen #B showed a relatively smaller decrease with increasing temperature than specimen #A, implying that specimen #B has higher stability due to the temperature changes. Thus, the addition of Si and Sn contributed to the improvement in the corrosion resistance of the materials [6].

Table 3. Results of potentiodynamic polarization test by heat treatment conditions of #A and #B specimens

Condition		Corrosion resistance		Passive Range	
		E_{corr} (mV)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	I_d ($\mu\text{A}/\text{cm}^2$)	E_{pit} (mV)
#A	900 °C	-569 $\pm$ 5	160 $\pm$ 20	45.5 $\pm$ 5	391 $\pm$ 5
	950 °C	-572 $\pm$ 5	280 $\pm$ 20	53.5 $\pm$ 5	388 $\pm$ 5
	1000 °C	-577 $\pm$ 5	276 $\pm$ 20	48.3 $\pm$ 5	395 $\pm$ 5
#B	900 °C	-535 $\pm$ 5	34 $\pm$ 5	15.8 $\pm$ 10	295 $\pm$ 5
	950 °C	-520 $\pm$ 5	67 $\pm$ 5	10.7 $\pm$ 10	293 $\pm$ 5
	1000 °C	-541 $\pm$ 5	82 $\pm$ 5	7.9 $\pm$ 10	290 $\pm$ 5

Fig. 6. Nyquist plot curves of EIS analysis results by heat treatment conditions for #A and #B specimens.

Table 4. R_s and R_p data of EIS analysis results by heat treatment conditions for #A and #B specimens.

Condition	R_s (Ohms)	R_p (Ohms)
#A 900 °C	4.0	325.9
#A 950 °C	4.0	302.4
#A 1000 °C	4.0	287.4
#B 900 °C	4.0	508.7
#B 950 °C	4.0	507.5
#B 1000 °C	4.0	506.1

The corrosion current density (I_{corr}) was less than $100 \mu A/cm^2$ for specimen #B compared to specimen #A, indicating that the corrosion rate in the same environment was reduced. In addition, the I_{corr} increased with temperature. Furthermore, the increase in I_{corr} with temperature was similar to the decrease in E_{corr} . Thus, the decrease in corrosion resistance increased the corrosion rate [22].

The measured passive film maintenance current values (I_d) of specimen #B were between 7 and $20 \mu A/cm^2$, lower than that of specimen #A (45 – $53 \mu A/cm^2$). This implies that the passive film of specimen #B was produced in an electrochemically stable region, due to the improvement in passive film stability of the SiO_2 and SnO_2 layers.

The pitting potential (E_{pit}) of specimen #B was measured to be approximately 100 mV, lower than that of specimen #A. This indicates that the destruction of passive film in #B was faster, leading to the onset of pitting corrosion. Such behavior could have two causes. First, the Cr content of specimen #B was approximately 2 wt.% lower than that of #A. However, the amount of added Si and Sn was only about 1 wt.%.

Studies have reported that the value of E_{pit} decreased by approximately 100 mV each time the Cr content of the stainless steel decreased by 1 wt.% [8,23]. However, the decrease in the Cr content of the stainless steel led to a decrease in the Cr_2O_3 content, which is the main component of the passive film. This reduces the Fe oxidation inhibiting effect on the surface; therefore, an appropriate content of Cr is essential for corrosion resistance [24,25].

A significant difference in E_{pit} of approximately 100 mV was observed for specimens #A and #B. This suggests that the Si and Sn oxide layers added to #B were equivalent to the corrosion resistance effect of approximately 1 wt.% Cr. Therefore, the corrosion resistance of specimen #B might decrease because Cr was reduced less. The addition of Si and Sn could also supplement the corrosion resistance. Consequently, we were able to reduce the Cr content and confirm improvements in the corrosion resistance of the stainless steel by Si and Sn. Second, Sn has a higher potential than Fe. Thus, the acceleration in pitting corrosion could be attributed to the galvanic effect based on the difference in electromotive force (EMF). Other studies have confirmed that when the passive film was destroyed and the pitting corrosion began, the galvanic effect between the Sn layers and the base metal accelerated the pitting corrosion of the base metal.

4. Conclusions

This study analyzed changes in the corrosion resistance of AISI 439 ferrite stainless steel with heat-treatment temperature and the amount of added Cr, Si, and Sn contents, which

changed the components of the surfaces and cross sections. The following conclusions were drawn from this study:

1. It was confirmed that adding Si and Sn to AISI 439 ferritic stainless steel increased its corrosion resistance.

2. As the heat-treatment temperature of stainless steel increased, the grain size increased slightly due to grain growth. This change is attributed to the reduced corrosion resistance of the materials. In addition, precipitates such as Cr carbide and Ti nitride were produced during the heat treatment, which commonly affect corrosion resistance.

3. As the heat-treatment temperature increased from 900 to 1000 °C, the production of Fe oxide on the surface increased. This is the leading cause of reduced corrosion resistance. Simultaneously, the production of Si and Sn oxides on the surface increased. This inhibited production of the Fe oxide on the surface, thereby improving the passive film stability.

4. Although the suppression of Fe oxide up to 950 °C was confirmed, the oxide generation increased at temperatures above 1000 °C. This suggests that the heat treatment temperature of 950 °C is most effective in the manufacturing process.

5. The Si and Sn added specimens showed improvement in corrosion potential and corrosion current density. It was also confirmed that a passive film was produced in electrochemically stable areas by reducing the passive maintenance current.

6. The pitting potentials of the specimens containing Si and Sn were relatively low. This is believed to have affected the reduction in the passive film properties due to a 2 wt.% Cr reduction. Thus, further studies on the appropriate contents of Cr, Si, and Sn could simultaneously improve mechanical properties, formability, and corrosion resistance.

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