Effect of Alloying Tin on the Corrosion Characteristics of Austenitic Stainless Steel in Sulfuric Acid and Sodium Chloride Solutions

Min Sun¹ · Ming Luo² · Chao Lu² · Tian-Wei Liu³ · Yan-Ping Wu³ · Lai-Zhu Jiang² · Jin Li¹

Received: 30 January 2015 / Revised: 14 May 2015 / Published online: 15 August 2015
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Abstract The effect of tin on general and pitting corrosion behaviors of the austenitic stainless steel in sulfuric acid and sodium chloride solutions was investigated by potentiostatic critical pitting temperature, cyclic potentiodynamic polarization, electrochemical impedance spectroscopy, and scanning electron microscopy. The results showed that there is an optimal tin addition which is around (0.062–0.1) wt%, and the general corrosion resistance of B316LX with 0.08 wt% tin addition in boiling H₂SO₄ increased remarkably with a corrosion rate of an order of magnitude lower than that of 316L. Hydrolyzation of tin ions induces more metastable pit occurrence on the material surface. However, the pitting resistance of B316LX increases because tin oxides improve the density and uniformity of the passive film, and hydroxide and oxide of tin inhibit the process of pit growing. The effect of tin on pitting corrosion process is illustrated schematically.

KEY WORDS: Stainless steel; Corrosion resistance; Passivation; Pitting corrosion

1 Introduction

Austenitic stainless steels are the most common materials used in construction industries because of the good combination of mechanical, fabrication, and corrosion resistance properties [1–4]. High alloying contents, such as chromium, nickel and molybdenum, are the reason of good corrosion resistance. However, nickel and chromium are rather pricey, and the price change of the raw materials greatly influences the cost of stainless steels these years.

Therefore, their replacement using small amounts of other alloying elements, such as nitrogen and manganese, is commonplace [5]. As a trace element, tin has been added into the austenitic stainless steels. The use of tin addition can be justified as follows: (a) Tin increases general corrosion resistance and helps to improve the corrosion resistance [6]. (b) It improves the formability and surface finish with fewer ridging pattern. (c) It conserves alloy resources and is stable in cost by minimizing the effect of fluctuations of raw material prices.

Some researchers have studied the beneficial effect of tin addition on corrosion resistance of austenitic stainless steels in acid mediums. Nam et al. [7] have reported that the corrosion rate of low-alloy steel decreased with increasing tin content, because tin favored partial regeneration of the passive layer, which was rich in Cr, Fe, and Sn elements. The deposition of these elements and compounds drastically inhibited the steel dissolution in H₂SO₄ solutions [7–9]. In addition, it has been reported that combination of tin with other elements, such as Cu and Sb, may improve the corrosion resistance. There is reference about the positive synergy of Cu and Sn on corrosion

Available online at <http://link.springer.com/journal/40195>

✉ Jin Li
corrosion@fudan.edu.cn

¹ Department of Materials Science, Fudan University, Shanghai 200433, China

² Research and Development Center, Baosteel Co., Ltd., Shanghai 201900, China

³ Science and Technology on Surface Physics and Chemistry Laboratory, P. O. Box 718-35, Mianyang 621907, China

resistance of the austenitic stainless steel by the formation of a stable film on the steel surface, and the reduction in the corrosion rate produced by tin addition was smaller than the synergic effect of Cu and Sn in both AISI 304 and AISI 316 stainless steels in a 30% H₂SO₄ solution [6]. Similarly, Nam *et al.* [7] have showed that the positive effect of the interaction of tin with Cu and Sb can improve the formation of a continuous, adherent rust layer.

However, according to Pardo, there was a limit of tin content (<0.1%) in AISI 304 and 316 to increase the corrosion resistance. The addition of large amount of tin would inhibit pits nucleation but favor its growth, because its hydrolysis generated more acidification than Cr and Fe and then the occurrence of more acidification resulted in an advance of the pit [10].

In summary, the studies on the alloying effect of a small amount of tin on the corrosion resistance of the austenitic stainless steels are important. However, the effect of tin addition on corrosion resistance of the austenitic stainless steels in a sulfuric-chloride-containing environment is seldom studied, and the mechanism of the tin addition improving the corrosion resistance is not clear. This work aims to study the corrosion behavior and the mechanism of tin addition into the austenitic stainless steel in acid chloride-containing solutions. The corrosion resistance of the steels was determined by critical pitting temperature (CPT), cyclic potentiodynamic polarization (CPP), electrochemical impedance spectroscopy (EIS), surface analysis techniques of XPS, and scanning electron microscopy (SEM).

2 Experimental

2.1 Electrochemical Tests

The test materials were the austenitic stainless steels, 316L, B316LX, and 317L, obtained from Baosteel Research Institute. The different content of Tin was added into the based 316L. Chemical compositions of these materials are given in Table 1.

The materials for gravimetric test were cut from cold-rolled plates of the austenitic stainless steels leaving a working area of 30 mm × 20 mm, with a thickness of 0.5 mm. All samples were ground to 1000 grit and cleaned by ethanol and deionized water prior to tests. According to GB/T 4334.6-2000, gravimetric test was carried out in 5 wt% H₂SO₄ solution under the boiling state for 6 h, and condense tubes were used to sustain the H₂SO₄ concentration. Before the experiment, the sample areas were measured and weighted to a precision of 0.01 mg. Upon completion of the experiment, the sample was extracted, cleaned with 30 wt% HNO₃ solution and then alcohol, dried at 70 °C for 30 min in a furnace, and then weighed again at room temperature. The subsequent loss of mass per unit of surface area was then calculated for the each material. Three samples were used to guarantee the reliability of the results.

The electrochemical measurements were carried out with a PARSTAT 2273 electrochemical system. The working electrodes for electrochemical measurement were embedded in epoxy resin leaving a working area of 10 mm × 10 mm. All samples were ground to 1000 grit and cleaned by ethanol and deionized water prior to tests, and all the tests were carried out at room temperature. The electrochemical measurements were performed in a conventional three-electrode cell. The austenitic stainless steel were used as the working electrode, a platinum sheet as the counter electrode, and all potentials were measured against a saturated calomel electrode (SCE) connected to the cell via a Luggin probe.

The CPT measurements were carried out in 1.0 mol/L NaCl deaerated solutions. Before CPT measurements, cathodic polarization at −900 mV versus SCE for 120 s was used to deoxidize the surface of working electrode. Then open-circuit potential was carried out for about 30 min to stabilize the surface of the specimen. In order to determine the CPT, anodic potential of 700 mV versus SCE was applied and the solution temperature was increased at a rate of 1 °C/min. The current density was recorded simultaneously with increasing the temperature throughout the test. The experiment was stopped after the

Table 1 Chemical composition of tin addition austenitic stainless steel (wt%)

Materials	C	Si	Mn	S	P	Cr	Ni	Cu	Mo	N	Sn
Sample 1	0.024	0.52	1.41	0.003	0.007	16.12	10.05	0.21	2.03	0.025	0
Sample 2	0.021	0.54	1.42	0.004	0.007	16.15	10.06	0.23	2.01	0.031	0.062
Sample 3	0.018	0.51	1.35	0.006	0.007	15.95	10.07	0.20	2.03	0.033	0.1
Sample 4	0.02	0.47	1.38	0.004	0.006	15.90	9.92	0.25	2.03	0.027	0.229
316L	0.0149	0.49	1.41	0.002	0.022	16.16	10.4	0.09	2.05	0.030	–
B316LX	0.0188	0.50	1.43	0.002	0.024	16.14	10.27	0.15	2.01	0.027	0.08
317L	0.0171	0.43	1.52	0.001	0.023	18.07	13.41	0.11	3.06	0.0415	–

current density increased to $100 \mu\text{A}/\text{cm}^2$ and then kept for 1 min. the CPT was defined as the temperature where the current density equaled to $100 \mu\text{A}/\text{cm}^2$. During the test, pure nitrogen gas was bubbled to get rid of oxygen gas. The measurements were carried out three times to guarantee the repeatability.

The CPP curves and EIS plots were carried out in 3.5% NaCl deaerated solutions. The CPP measurement was measured potentiodynamically from -250 mV (vs. open-circuit potential E_{oc}) to the positive potential with a scanning rate of 1.667 mV/s . When the current density reached a value approximately $1 \text{ mA}/\text{cm}^2$, the potential was scanned in reversed direction with the same scanning rate. EIS plots were measured at the open-circuit potential, and frequency was ranged from 100 kHz to 0.01 Hz with an applied ac perturbation of 10 mV . The impedance data were analyzed by a commercial ZSimpWin software package.

2.2 XPS Measurement

To investigate the composition of the oxide films on the sample surface in sulfuric acid and sodium chloride solutions, the tested samples were polarized at 0.4 V versus SCE for 2 h in 3 wt% $\text{H}_2\text{SO}_4 + 0.5 \text{ wt\% NaCl}$ solutions in order to form a thin oxide film. Then the chemical composition of the oxide films obtained by XPS were acquired using a PHI Quantera SXM equipped using AlK_α source ($h\nu = 1486.6 \text{ eV}$) operated at 200 W . For XPS characterization, $\text{Cr}2\text{p}$, $\text{Fe}2\text{p}$, $\text{Sn}3\text{d}$, $\text{Cu}2\text{p}$, $\text{O}1 \text{ s}$ and $\text{C}1 \text{ s}$ core-level spectra have been recorded. The minimum X-ray beam spot from the microprobe was less than $9.0 \mu\text{m}$ in both the x and y directions. Due to the atmospheric contamination on the surface of the samples, a 1-nm-thick passive film was sputtered to increase the intensity of the signal. The sputtering gun used an Ar-ion source with energy of 2.0 keV and an emission current 20 mA . The sputtering rate was determined to be 14 nm/min . The measurement depth for XPS analysis was approximately $5\text{--}7 \text{ nm}$. Peak decomposition was carried out with XPS Peak 4.1 peak-fitting software using Gaussian–Lorentzian peak shapes and a Shirley background [11].

3 Results and Discussion

3.1 Influence of Tin Addition on General Corrosion Resistance

Figure 1 shows the corrosion rates of the austenitic stainless steels with different tin contents in 5 wt% boiling sulfuric acid solutions. The corrosion rate is $33.76 \text{ g}/(\text{m}^2 \text{ h})$ for the steel without tin addition, and it decreases

Fig. 1 Corrosion rates of tin addition austenitic stainless steel with different tin contents in 5 wt% boiling sulfuric acid solution

Fig. 2 Corrosion rates of 316L, B316LX and 317L in 1, 5, 10, 20 wt% boiling sulfuric acid solutions

dramatically to 3.192 when $0.062 \text{ wt\%}$ tin is added into the austenitic stainless steel. With the further increase in tin content, the corrosion rate continues to decrease. It is worth noting that a little trace tin addition into the austenitic stainless steel can improve the general corrosion resistance remarkably; however, the further tin addition causes a limited increase in the general corrosion resistance. Then it can be deduced that there is an optimal tin addition is around $(0.062\text{--}0.1) \text{ wt\%}$ to increase the general corrosion resistance and maintain the material cost.

316L, B316LX (with $0.08 \text{ wt\%}$ tin), and 317L stainless steels are used to investigate the effect of tin on general corrosion behavior. Figure 2 shows the corrosion rates of 316L, B316LX, and 317L stainless steels in 1, 5, 10, and 20 wt% boiling sulfuric acidic solutions. In 1% boiling sulfuric acidic solutions, the corrosion rates are 0.032 , 0.022 , and $0.014 \text{ g}/(\text{m}^2 \text{ h})$ for 316L, B316LX and 317L, respectively, which are low and close to each other. In 5% boiling sulfuric acidic solutions, the corrosion rate of 316L is $52.97 \text{ g}/(\text{m}^2 \text{ h})$, and it dramatically decreases to $8.582 \text{ g}/(\text{m}^2 \text{ h})$ when 0.08% tin was added into B316LX, which is close to the corrosion rate of 317L with a value of $2.995 \text{ g}/$

Fig. 3 SEM morphologies of corrosion products deposited on 316L **a**, B316LX **b**, 317L **c** in 5 wt% boiling sulfuric acid solution after 6 h

($\text{m}^2 \text{ h}$). With increasing the sulfuric acid concentration, the corrosion rates of these austenitic stainless steels increase dramatically. In 20% H_2SO_4 solutions, corrosion rate of 316L increases to more than $400 \text{ g}/(\text{m}^2 \text{ h})$, and the rates are 142.99, $118.89 \text{ g}/(\text{m}^2 \text{ h})$ for B316LX and 317L, respectively. Addition of tin reduces the corrosion rate of B316LX in sulfuric acidic solution, which means that the effect of tin increases the corrosion resistance of B316LX immersed in H_2SO_4 boiling solutions significantly. However, the corrosion resistance for 317L is still better than that of B316LX because of the more amounts of chromium and molybdenum.

Figure 3 shows the morphologies of the corrosion products deposited on the 316L, B316LX, and 317L in 5 wt% H_2SO_4 boiling solutions after 6 h. It can be seen that severe uniform corrosion occurs all over the specimen surface for 316L, whereas there are local corrosion on the surfaces of B316LX and 317L because the corrosion only occurs at local and small areas with some holes. Meanwhile, the corrosion of B316LX is more severe than 317L, and it has more corrosion holes on the surface. According to Refs. [7, 12], the addition of tin created a protective SnO_2 layer on the steel surfaces to inhibit steel dissolution. The presence of SnO_2 on the surface of the tin addition steels decreased the corrosion rate due to a decrease in the anodic reaction kinetics.

3.2 Influence of Tin Addition on Pitting Corrosion

The simulated specimens were tested for three times under the same conditions. Figure 4 shows typical curves of the current density versus temperature for three austenitic stainless steels during the CPT measurement. These results show that the current density presents a lower value than $5 \mu\text{A}/\text{cm}^2$ during the initial heating, suggesting that the specimen is protected well by the passive film formed on the surface. With the solution temperature increasing, the current density fluctuations are found below the CPT, which could be associated with a passive film breakdown in the form of metastable pit. As the solution temperature

Fig. 4 Typical curves of current density versus temperature for 316L, B316LX and 317L at 700 mV versus SCE during CPT test in 1 mol/L NaCl solution

was increased further up to CPT, the current density rises markedly due to the occurrence of the stable pit [13]. The CPT value for 316L is 21.7°C , similarly hereinafter. However, the mean CPT values are 30.7 and 42.5°C for B316LX and 317L, respectively, which can be concluded that the later two steels possess a better pitting corrosion resistance in chloride solutions. However, it is worth noting that before the stable pits form on the sample, there are more current fluctuations for B316LX marked with dashed lines. One current fluctuation corresponds a metastable pit occurring, which cannot grow into stable pit because of the passive film reformed on the surface of the pit preventing the further dissolution. Compared with 316L, B316LX has a higher resistance to occur stable pits meaning a better pit corrosion resistance; however, metastable pits are easy to form which is probable to be explained by the dissolution and hydrolyzation of tin ion in the next section [14].

Figure 5 shows the EIS plots of 316L, B316LX, and 317L measured in 3.5 wt% NaCl solutions at 30°C . The measure impedance of 316L is the smallest, and the impedance of B316LX is bigger than that of 316L. Meanwhile, 317L shows the biggest impedance among them.

Fig. 5 EIS plots and equivalent circuit diagram of 316L, B316LX, 317L in 3.5 wt% NaCl solution at 30 °C

The equivalent electrical circuit shown in Fig. 5 is used to fit the impedance data, where R_s represents the solution resistance, R_f is the resistance due to the ionic paths through the passive layer, Q_f is the capacitive behavior of the passive film formed, Q_{dl} presents the capacitive behavior of the electric double layer, and R_{ct} is the charge transfer resistance. In view of the dispersion effect caused by the roughness of the electrode interface, the constant phase angle element Q is used to replace the pure capacitance element C to reflect the capacitance behavior [15]. The values of the impedance parameters for the three austenitic stainless steels are shown in Table 2. Values of R_f and R_{ct} show the same trend: 316L < B316LX < 317L. According to these results, the protective property of passive film for 317L is the best, and B316LX has a better property than that of 316L. The better protective property of passive film is always due to its thickness and compactness with fewer defects [11, 16, 17].

3.3 Influence of Tin Addition on Pitting Heal Behavior

The breakdown potential of pitting corrosion (E_b) is the potential above which pits nucleate and grow, the higher E_b , the better resistant of pitting corrosion. The protect potential of pitting corrosion (E_p) is the potential below which pits never occurs. If E_p is high, the metal is said to be very resistant to pitting corrosion [18]. $\Delta E(E_b - E_p)$ represents the corrosion resistance of the materials, and the

smaller the ΔE value, the better the anti-corrosion property [19]. When the direction of the potential scan is reversed, the ΔE value can be used to evaluate the self-healing property of the produced pits owing prior polarization [20], accordingly detecting resistance to pitting corrosion. Figure 6 shows the CPP curves of 316L, B316LX, and 317L SSs in 3.5 wt% NaCl solution. Compared with 316L SS, the E_b of the B316LX SS is 0.412 V versus SCE, which is higher than 316L SS, indicating more effective inhibition of localized corrosion. The E_p of the 316L is 0.074 V versus SCE; meanwhile, it is 0.179 V versus SCE for B316LX with a preferable resistance to pitting corrosion. The ΔE of B316LX is 0.233 V versus SCE, significantly less than that of 316L (0.320 V vs. SCE), indicating that passive film of B316LX shows a better repair property to protect the substrate from corrosion susceptibility.

To further substantiate the discussions above, XPS measurements are used to directly characterize the composition of passive film formed on the specimens after polarization at potentials 0.4 V versus SCE for 2 h in 3 wt% H_2SO_4 + 0.5 wt% NaCl solutions. Figure 7 displays the detailed spectra of Fe 2p_{3/2}, Cr 2p_{3/2}, Cu 2p_{3/2} and Sn 3d in the passive films. After background subtraction according to Shirley [11], the XPS results are separated into contributions of the different oxidation states by a fit procedure which was described in previous papers. The evaluation of possible species in passive films could be

Fig. 6 CPP curves of 316L, B316LX, and 317L in 3.5 wt% NaCl solution at 30 °C

Table 2 Values of impedance parameters determined for 316L, B316LX and 317L in 3.5 wt% NaCl solution at 30 °C

	R_f (Ω cm ²)	Y_f Ω^{-1} s ⁿ cm ²	n	R_f (Ω cm ²)	Y_{dl} Ω^{-1} s ⁿ cm ²	n	R_{ct} (Ω cm ²)
316L	8.04	0.00051	0.86	1.19×10^4	0.00015	0.84	817.5
B316LX	8.44	0.00035	0.82	1.96×10^4	0.00019	0.84	2162
317L	7.85	0.00021	0.81	3.05×10^4	0.00024	0.80	8147

Fig. 7 XPS spectra of $\text{Fe}3d_{3/2}$, $\text{Cr}3d_{3/2}$, $\text{Cu}3d_{3/2}$, $\text{Sn}3d_{3/2}$ for B316LX SS after polarization at 0.4 V versus SCE for 2 h in 3 wt% H_2SO_4 +0.5 wt%NaCl solutions

performed on the basis of well-characterized standards from Handbook of X-ray photoelectron spectroscopy [21].

It can be seen that there exist several obvious peaks for Fe $2p_{3/2}$, which are related Fe_3O_4 (708.2 eV), FeO (709.4 eV), and Fe_2O_3 (710.6 eV). The intensities of the FeO and Fe_3O_4 states are higher than those of the Fe_2O_3 . The main peaks of Cr $2p_{3/2}$ spectra are fitted with four peaks, which are attributed to metal state Cr (574.1 eV) and the Cr(III). The predominant state of chromium is Cr(III) in Cr_2O_3 (576.4 eV), with minor contribution of Cr(III) in $\text{Cr}(\text{OH})_3$ (577.3 eV) and CrO_3 (579.8 eV). It can be noted that Cr_2O_3 is the primary iron-oxidized species in the passive film. These oxidized species are the primary constituents of the passive film for the austenitic stainless steels in sulfuric acid solutions. Moreover, it is well known that the bilayer passive film formed on the stainless steel in acid solutions consists of a spinal inner layer rich in chromium and outer layer rich in iron.

The binding energy of Sn $3d_{3/2}$ and Sn $3d_{5/2}$ was approximately 494.60 and 486.18 eV, respectively. The presence of SnO_2 on the surface of the tin addition steels decreased the corrosion rate due to a decrease in the anodic

reaction kinetics [7]. The XPS spectra of Cu 2p were shown in Fig. 7. The Cu signal presented two superimposed peaks at 933.6 eV corresponding to Cu (II). The oxides of Cu and Sn improved the formation of the continuous rust layer. This may be preferred to maintain the uniformity of the rust layer, and then the corrosion resistance was improved by cuprous oxide as a result of an interaction of Sn with Cu [2].

3.4 Mechanism of the Influence of Tin Addition on Corrosion Resistance

When the austenitic stainless steels are exposed to sulfuric acid solutions and chloride media, the passive films can form on the surface, which consist of oxide of iron, chromium, molybdenum, and nickel, showing a good resistance in the sulfuric or neutral environments [8, 22]. However, the chloride ions can adsorbed on the specimens and destroy the protective passive film and induce pitting corrosion [23].

Figure 8 summarizes the mechanism whereby tin increases corrosion resistance in the austenitic stainless

Fig. 8 Schematic diagram of effect of tin on pitting corrosion of austenitic stainless steel in chloride-containing solutions

steel B316LX in sulfuric acid chloride media. In the acid or neutral solutions containing chloride ions, the sample surfaces are easy to form absorbed thin electrolyte layers containing chloride ions, which can break the oxidation film and lead to pitting corrosion (Fig. 8a). In acid and neutral media, two cathodic reactions are possibly the hydrogen reduction in acid solutions or the oxygen reduction in neutral media, as Eqs. (1) and (2) [14]:

The anodic reactions are mainly the iron dissolution, and other alloys dissolution, such as chromium, nickel, as [14]:

Simultaneously, based on the Pourbaix diagram of the Sn/H₂O–Cl system [24] and the surface characteristics of the tin addition steels in the acid chloride solution, the formation of tin oxide in this environment was proposed to proceed as:

The oxidation of tin produces initially Sn(OH)₂ which is unstable and may undergo both progressive dehydration to 2SnO·H₂O, 5SnO·2H₂O, 3SnO·H₂O and/or SnO, and oxidation to SnO₂·nH₂O, where n are 0, 1, and 2. SnO₂ is the predominant passivating species in the Sn/H₂O system, under all conditions, as shown in Fig. 8b. SnO₂ can be formed as the dehydration reaction which takes place as:

However, the hydroxides are unstable with respect to the corresponding oxides as:

In the chloride solutions, these anodic reactions can take place also as:

Unlike most metals, tin is passivated in moderately strong chloride solutions, due to the formation of SnOHCl·H₂O.

The breaking down of the passive film consists of two process. On the first stage, the chloride ions are absorbed on the specimen surface and induce the occurrence of metastable pits; on the second stage, the metastable pits are converted into stable pits and the stable pits continue to grow in the depth. During the pit nucleation process, Sn⁴⁺ in the pit holes can generate more acidification and cause an advance of the pits because of Sn⁴⁺ hydrolysis. This is the reason more metastable pits occur on the tin addition stainless steel in the CPT test. On the second stage, the hydroxide and oxide of tin, including Sn(OH)₄, SnO₂, and SnOHCl·H₂O, can promote the heal of passive film in pits to prevent corrosion continuing growing. According to results of the CPT and CPP curves, the effect of increasing corrosion resistance for tin addition material in the second process is more pronounced.

4 Conclusions

1. The corrosion rates of the austenitic stainless steels decrease dramatically with a little trace of tin addition. The optimal content of tin is about (0.068–0.1) wt% for improving the corrosion resistance and maintaining material cost. The corrosion resistance of B316LX with the addition of tin in boiling H₂SO₄ increases

remarkably with a corrosion rate one order of magnitude lower than that of 316L.

2. Sn^{4+} hydrolysis generates more acidification and causes more metastable pits occurring on the tin addition stainless steel during the pit nucleation process.
3. The corrosion resistance of B316LX is increased because hydroxide and oxide of tin oxides changed the density and uniform of the passive film to protect the substrate from corrosion susceptibility.

Acknowledgments The authors acknowledge the supports of the China Postdoctoral Science Foundation (2013M541463), the National Natural Science Foundation of China (No. 51,131,008), the National Key Technology R&D Program (No. 2012BAE04B00), and the Doctoral Fund of Ministry of Education of China (No. 20120071110013).

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