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Electrochemical and Stress Corrosion Cracking Behavior of Alpha-Al Bronze in Sulfide-Polluted Salt Water: Effect of Environmentally-Friendly Additives

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ABSTRACT

The stress corrosion cracking (SCC) behavior of α-Al bronze alloy (Cu$_7$Al) was investigated in 3.5% NaCl solution in the absence and in the presence of different concentrations of Na$_2$S under open circuit potentials using the constant slow strain rate technique. Also, the addition of different concentrations of cyseine (cys), and alanine (ala) to the test solution, as corrosion inhibitors, was studied. Increasing the sulfide ions concentration in polluted salt water resulted in a reduction in the maximum stress (σ_{max}) and an increase in the susceptibility of α-Al bronze towards SCC. The addition of ala and cys to the test electrolyte increased the time to failure by changing the mode of failure from brittle transgranular cracking to ductile failure. Electrochemical tests were performed to assist the interpretation of the SCC data. Electrochemical impedance spectroscopy (EIS) was used to investigate the mechanism of corrosion inhibition. The results support film rupture and anodic dissolution at slip steps as the operating mechanism of the SCC process. Therefore, cys and ala can be considered as potential environmentally-friendly corrosion inhibitors for the SCC of α-Al bronze in 3.5% NaCl solution containing sulfide ions.

KEYWORDS

Bronze; Sulfide; Corrosion; SCC; Inhibitor; Amino Acids

1. Introduction

Copper-base alloys have a long history of service in marine environments. Aluminum bronzes contain copper as the major component and aluminum as a minor component. They have good strength [1-6] and superior resistance to cavitation erosion and impingement attack [7]. Al-bronzes have been used in various sea water applications [1,8-10], the manufacturing of acid handling equipment, and dental restoration [2]. Small quantities of Fe, Ni, or Ag may be added to Al-bronzes to improve their corrosion resistance and mechanical properties [1-4]. The corrosion resistance was attributed to the formation of a thin film of Al$_2$O$_3$, which heals rapidly when dam-

aged [11-13]. The corrosion resistance of Al-bronze increases with increasing the aluminum content up to about 9.6%, which represents the limit of the ∞-phase [14]. Beyond that limit, the two-phase alloys undergo severe corrosion and loss of ductility, which had been attributed to selective loss of aluminum [10,15-17] *i.e.* dealumini-fication. In this regard, the electrochemical and stress corrosion cracking (SCC) behavior of Al-bronzes has been reported in marine and desalination environments [18-20] and in 3.4% NaCl solutions [13].

Various pollutants, such as nitrite ions [18], are often introduced in the work environments, which might have a delirious effect on the workability of such alloys [19]. More seriously, the presence of sulfide ions in seawater, from seaweed, sulfide reduced bacteria, or industrial

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waste discharge, is known to promote aqueous corrosion of copper and its alloys with greater damage than that caused by Cl⁻ ions [19,20]. However, it seems that the study of the effect of such pollutant on the stability of Al-bronzes is very rare [18].

Therefore, the aim of the present work is to study the propensity of ∞-Al bronze to stress corrosion cracking in sulfide-polluted salt water. A complete set of electrochemical characterization techniques have also been used to understand the electrochemical behavior of the alloy in such environments. Moreover, cysteine (cys) and alanine (ala) have been investigated as positional environmentally-friendly additives to suppress the observed damage.

2. Experimental Procedure

Tests were performed on commercial ∞-Al bronze alloy supplied by Non-ferrous industries company, Helwan, Egypt, with the composition: 7% Al, 0.04% iron (Fe), 0.01% nickel (Ni), 0.04% silicon (Si), 0.006% magnesium (Mg), and the balance Cu. The mechanical properties are: Ultimate tensile stress (UTS) of 150 kg/mm^2, yield stress (YS) of 48 kg/mm^2, Hardness is 141 (VHN) and elongation is 6%. A constant slow strain rate machine was used at a constant strain rate of 5.53×10^{-6} s^{-1}. The test cell was in the form of a glass cylinder (120 mm in height and 80 mm in diameter. It was closed at the top and bottom by a rubber stopper through which the ends of the specimens protruded. The specimens were machined to a gauge length of 25 mm and a diameter of 6 mm.

The samples were polished with 320, 600, and 800 grit silicon carbide (SiC) paper, degreased with acetone, and coated with silicon rubber so that only the gauge length was exposed to the test solution under open circuit potential. The experiments were carried out at 24°C ± 1°C in air, and in aerated 3.5% NaCl with different concentrations of Na$_2$S (2, 16, 100 ppm). Potentiostatic polarization measurements were performed using a PS6 Meinsperger Potentiostat/Galvanostat (Germany). A three electrode electrochemical cell made of pyrex glass, with a saturated calomel electrode (SCE) as a reference electrode, a platinum foil as a counter electrode, and α-Al bronze specimen as the working electrode, was used. Electrochemical impedance spectroscopy (EIS) was carried out with a potentiostat/galvanostat (Gamry PCI 300/ 4) and a personal computer with EIS 300 software for calculations at E$_{opc}$ after 30 min. of immersion in solution. After the determination of steady—state current at a given potential, sine wave (10mV) peak to peak, at frequencies between 5×10^{3} Hz and 0.02 Hz were superimposed on the rest potential. The impedance diagrams are given in the Nyquist and Bode representation. Cracking specimens were removed from the solution after fail-

ure, cut 1cm beyond the crack tip and subjected to scanning electron microscopy (SEM) using JEOL, JXA-840 A Electron Probe Microanalyzer, Japan.

3. Results and Discussion

3.1. Stress Corrosion Cracking (SCC) Behavior of α-Al Bronze

3.1.1. Effect of Sulfide Ions

Stress-strain curves were measured in 3.5% NaCl in the presence and absence of different concentrations of sodium sulfide under open circuit conditions. Measurements were also performed in air for comparison. The results are shown in **Figure 1(a)** and summarized in **Table 1** in terms of the maximum stress (σ), time to failure (T_f), stress ratio (τ), time to failure ratio (r), susceptibility to SCC (S) and mode of failure for samples tested under the various conditions.

The curves show the same conventional shape usually seen for other alloys and are characterized by an increase in strain with increasing stress until the yield stress point (σ_{max}) is reached, followed by a slight gradual increase in the form of plateau until it reaches a maximum, after which the strain begins to decline to reach the point of failure. The susceptibility to stress corrosion cracking was measured by the ratios of both the time to failure (τ) and the maximum stress (r), where the maximum stress ratio is defined as [21]:

$$r = \frac{\sigma_{max}\left(sol\right)}{\sigma_{max}\left(air\right)} \qquad (1)$$

and the ratio of time to failure as:

$$\tau = \frac{t_f\left(sol\right)}{t_f\left(air\right)} \qquad (2)$$

The values of r and τ and the metallographic appearance of the fracture surfaces are useful criteria for assessing susceptibility to SCC. In a previous report [21] both r and τ were combined in a quantitative phenomenological expression for the susceptibility (S) to SCC, given by:

$$S = \left[\left(1-r\right)\left(1-\tau\right)\right]^{1/2} \qquad (3)$$

Figure 1(a) shows the stress-strain curves of ∞-Al bronze in 3.5% NaCl with different concentrations of sulfide ions (2.16 and 100 ppm) at a strain rate of 5.53×10^{-6} s^{-1} under open-circuit potential (OCP). The increase of sulfide ion to 100 ppm led to a decrease in the maximum stress. Note the similarity in behavior for the samples tested in air and in sulfide-free NaCl electrolytes. However, upon the addition of sulfide ions, the propensity of α-Al-bronze to SCC was found to depend on the

(a)

(b)

(c)
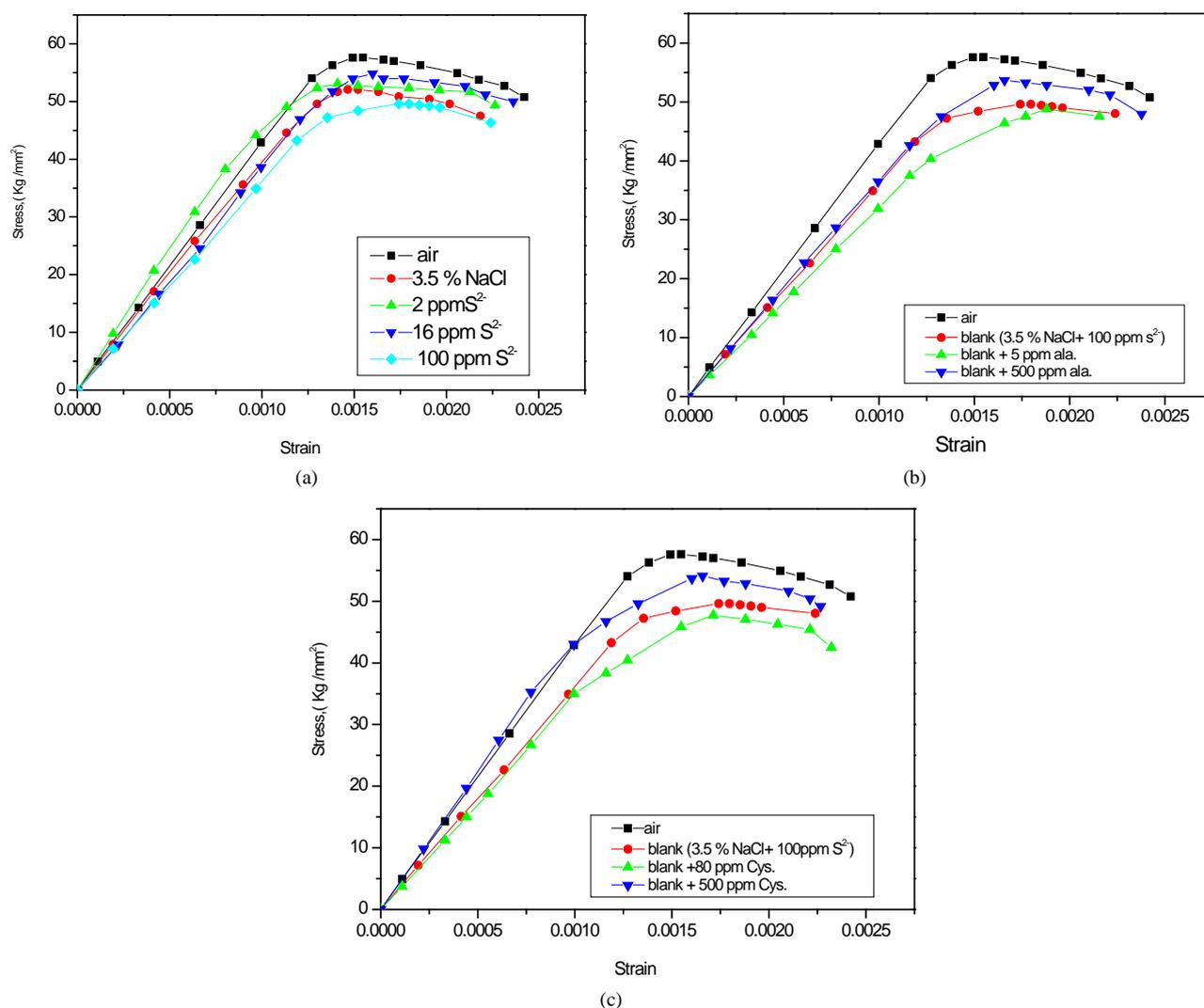
Figure 1. Stress-strain curves of Al-bronze at strain rate 5.53×10^{-6} s^{-1} in air and in 3.5% NaCl containing (a) different concentrations of sulfide ions; (b) 100 ppm·S^{2-} with and without ala; and (c) 100 ppm·S^{2-} with and without cys.

Table 1. Stress corrosion cracking (SCC) parameters of α-Al bronze alloy in 3.5% NaCl in the presence of different concentrations of sulfide ion with the different concentration of alanine and cysteine at strain rate of 5.53×10^{-6} s^{-1}.

Medium	σ	T_f	r	τ	S	Failure mode
air	57.61	437	-	-	-	ductile
3.5% NaCl	52.08	395	0.90	0.90	0.10	ductile
2 ppm·S^{2-}	53.13	405	0.92	0.93	0.08	ductile
16 ppm·S^{2-}	54.83	427	0.95	0.98	0.03	ductile
100 ppm·S^{2-}	49.60	405	0.86	0.93	0.09	brittle
100 ppm·S^{2-} +5 ppm ala	48.79	390	0.84	0.89	0.13	brittle
100 ppm·S^{2-} + 500 ppm ala	53.69	430	0.93	0.98	0.04	ductile
100 ppm·S^{2-} + 80 ppm cys	47.71	400	0.82	0.91	0.13	brittle
100 ppm·S^{2-} +500 ppm cys	54.10	410	0.94	0.94	0.06	ductile

sulfide ion concentration, indicating the promoting effect of sulfide ions on the SCC of α-Al bronze. The first step is believed to be an initial adsorption process of the sul-

fide species:

$$HS^-_{(aq)} + Cu = HS^- : Cu \qquad (4)$$
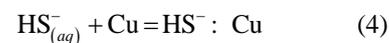
where $HS^- : Cu$ refers to the adsorbed sulfide species on the bare copper surface. This adsorbed species catalyzes the anodic dissolution reaction:

$$HS^- : Cu = CuS + H^+ + 2e^- \qquad (5)$$

The resulting sulfide scale (CuS and Cu_2S), as well as dissolved sulfide [22,23] catalyzes the partial anodic Equation (5) and cathodic Equation (6) reactions:

$$\frac{1}{2}O_2 + H_2O + 2e^- = 2OH^- \qquad (6)$$
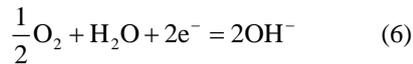
The overall corrosion reaction is the sum of the two partial reactions Equations (5) and (6), which can be represented as [24]:

$$HS^- : Cu + \frac{1}{2}O_2 = CuS + OH^- \qquad (7)$$
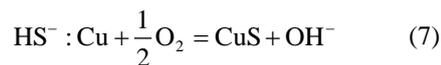
Consequently, the rates of reactions 5 - 7 are affected by the sulfide ions that are adsorbed on the alloy surface.

3.1.2. Effect of Alanine and Cysteine

The inhibitive effect of two amino acids, alanine (ala) and cysteine (cys), as environmentally-friendly corrosion inhibitors was investigated for the SCC of α-Al bronze under the most severe conditions identified above (3.5% NaCl + 100 ppm·S^{2-} at strain rate of 5.53×10^{-6} s^{-1}). The results are shown in **Figures 2**, **3** and **Table 2**. **Figures 1(b)** and **(c)** shows the stress-strain curves obtained for Al-bronze alloy samples tested in air as well as in electrolytes containing 3.5% NaCl in the presence of alanine and cysteine inhibitors. It is noted that the addition of alanine (500 ppm) increases the ultimate stress and decreases the susceptibility (S) of the alloy to SCC from 0.09 to 0.04 as listed in **Table 1**. Similarly, the addition of cysteine increases the ultimate stress and decreases the susceptibility of α-Al bronze to SCC from 0.09 to 0.06. This indicates that the addition of ala or cys can be used to improve the resistance of the alloy to SCC in sulfide-polluted salt water.

3.1.3. Metallographic Characterization

Figure 2 displays the obtained SEM micrographs of the fractured surfaces obtained in air, in 3.5% NaCl and in 3.5% NaCl with different concentrations of sulfide ions (2, 16, 100 ppm). It can be seen that the mode of failure (**Figures 2(a)-(d)**) is mainly ductile. However, in the presence of 100 ppm sulfide, the fracture mode was found to be brittle transgranular (**Figure 2(e)**). Upon the addition of 500 ppm ala or cys to an electrolyte containing 3.5% NaCl + 100 ppm·S^{2-}, the mode of failure changes from brittle transgranular to ductile failure (**Figures 2(f)** and **(g)**), which is the same mode of failure obtained upon testing the material in air, confirming that amino acids (ala and cys) can act as potential environ-

mentally friendly corrosion inhibitors for the SCC of α-Al bronze alloy in sulfide-polluted salt water. However, it is important to note that the absence of cracking is not the only factor determining the immunity of the material to SCC (24). The inhibitive effect of ala and cys can be attributed to the adsorption of inhibitor on the alloy surface, which inhibits the anodic dissolution of the alloy by blocking the active sites on the alloy surface and subsequently the SCC.

3.2. Electrochemical Measurements

3.2.1. Potentiostatic Polarization

Figure 3(a) shows the polarization curves of α-Al bronze alloy in 3.5% NaCl in the presence of different concentrations of sulfide ion (2, 16, 100 ppm). Note that the presence of sulfide ions shifts the potential towards more negative potential region and the shift was found to be dependent on the sulfide ion concentration. The sulfide ion results in an increase in the measured anodic current, which can be attributed to the anodic oxidation of the alloy. The current increases abruptly with potential at values near the OCP. At cathodic potentials to E_{corr}, the current is usually a measure of the oxygen reduction reaction in aerated sea water environments.

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \qquad (8)$$
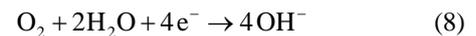
However, in the presence of sulfide ions, unstable black cuprous sulfide is formed:

$$2Cu + S^{2-} \rightarrow Cu_2S + 2e^- \qquad (9)$$

According to Tromans *et al*. [25], Cu is involved in this reaction:

$$2Cu + H_2O \rightarrow Cu_2O + 2H^+ + 2e^- \qquad (10)$$

$$Cu + Cl^- \rightarrow CuCl + e^- \qquad (11)$$
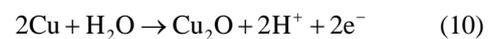
Ashour *et al*. (12) documented the presence of SCC of a single—phase α-Al bronze in 3.4% NaCl solution and explained the results as a dealloying process occurred through a film of copper chloride (CuCl). The electrochemical behavior of the α-Al bronze in 3.4% NaCl reveals the formatiom of mostly solid corrosion products at anodic potentials to the free corrosion potential. These are defined as Cu_2O and CuCl on the basis of thermodynamics calculation and X-ray diffraction measured on the treated α-Al bronze alloy surface (10). This insight is in line with results reported by Neodo *et al*. [26] confirming that the formation of cuprite (Cu_2O) during the copper oxidation in a chloride solution, at pH 6.2, is thermodynamically favoured over CuCl.

Ateya *et al*. (10, 13) as well as Schussler and Exner [27] identified Al_2O_3 as an inner adherent layer, acting as a barrier for ionic transport across the corrosion product
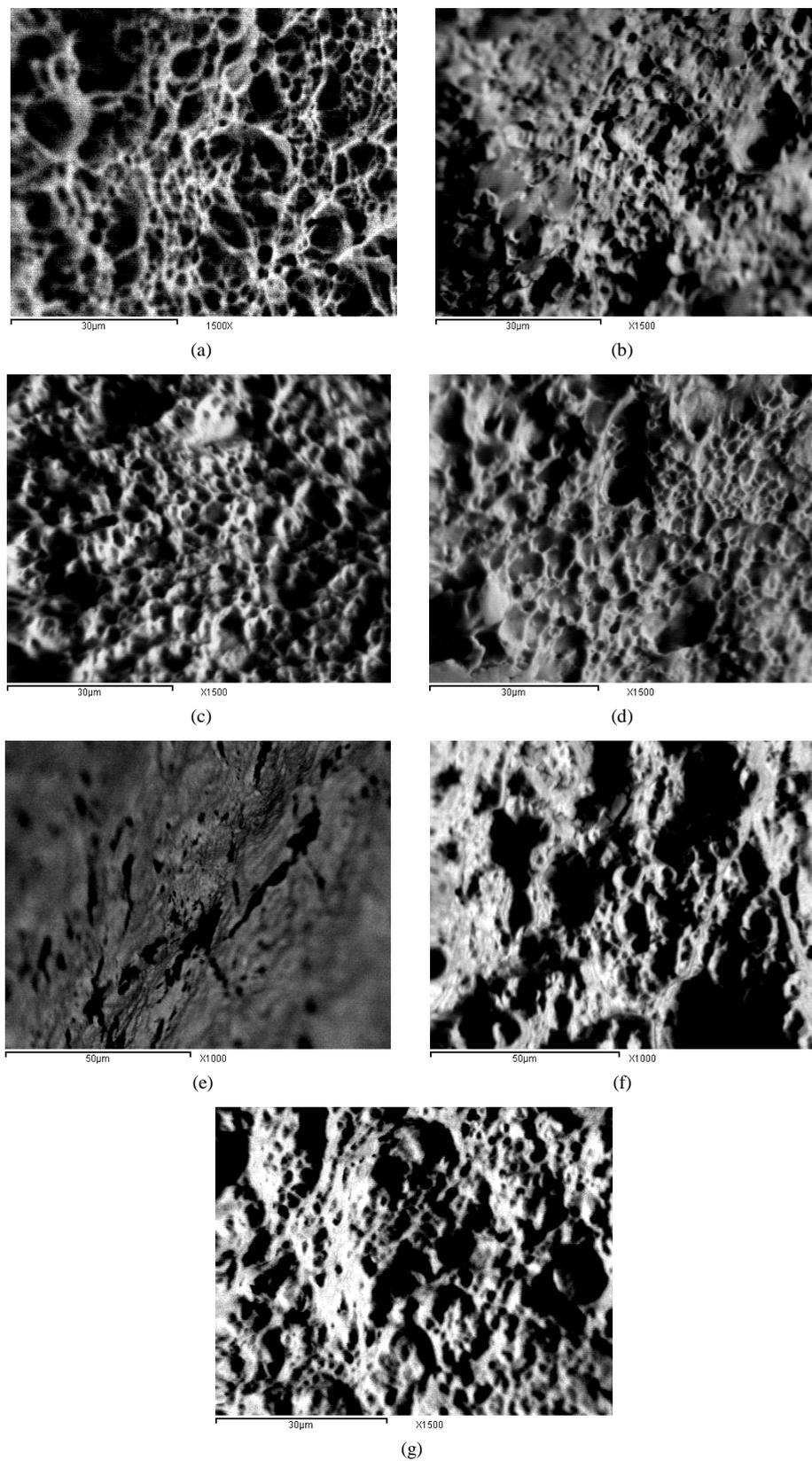
Figure 2. SEM fractographs of α-Al bronze in (a) air, (b) 3.5% NaCl, (c) 3.5% NaCl + 2 ppm·S^{2-}, (d) 3.5% NaCl + 16 ppm·S^{2-} and (c) 3.5% NaCl + 100 ppm·S^{2-}, (f) 3.5% NaCl + 100 ppm·S^{2-} + 500 ppm ala, and (g) 3.5% NaCl + 100 ppm·S^{2-} + 500 ppm cys.

(a)

(b)
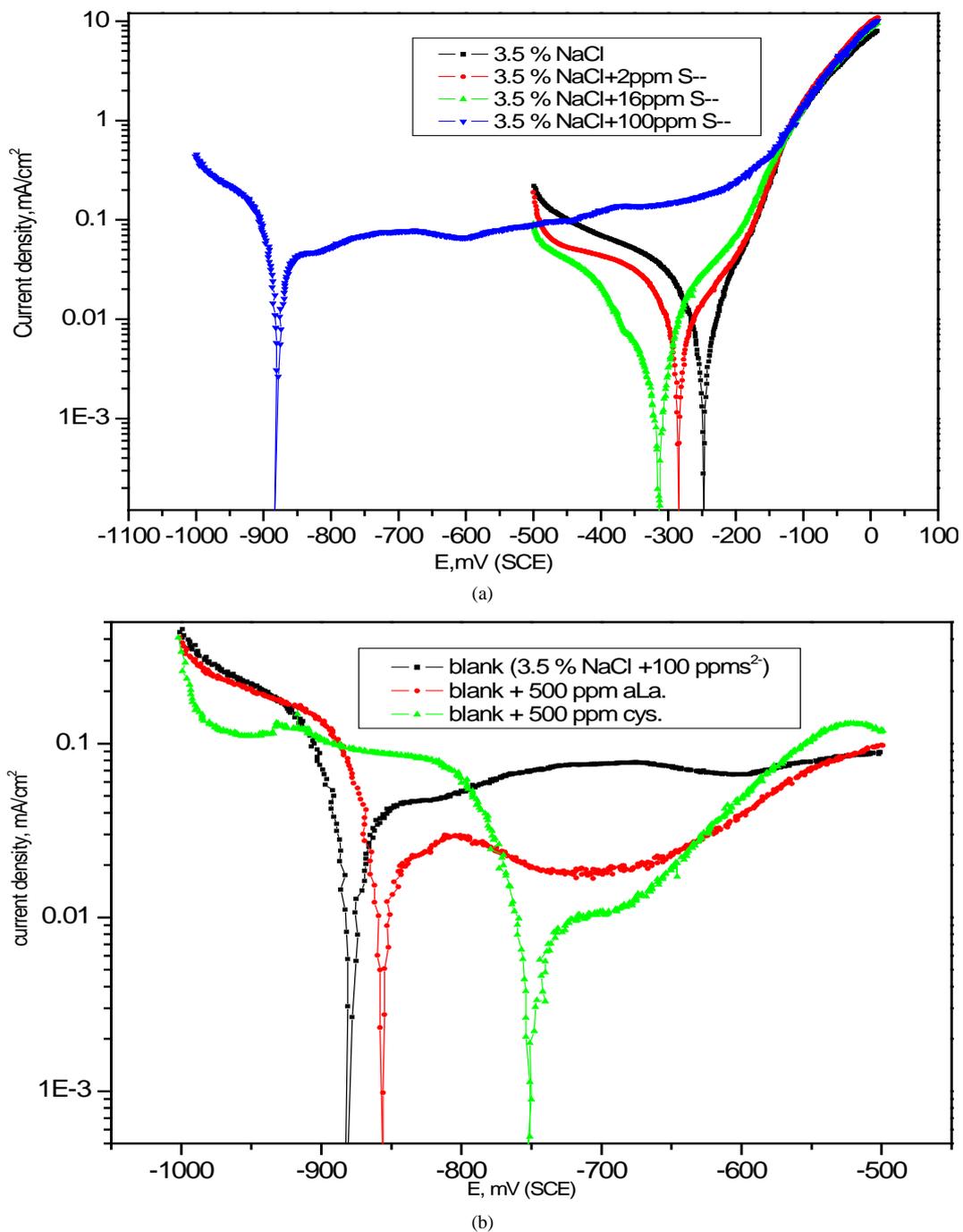
Figure 3. Polarization curves of Al-bronze in (a) 3.5% NaCl with different concentrations of sulfide ions, and (b) 3.5% NaCl + 100 ppm·S^{2-} (blank), blank + 500 ppm ala, blank + 500 ppm cys.

Table 2. The effect of the optimum doses of inhibitors (alanine and cysteine) on the free corrosion potential (E$_{corr}$), corrosion current density (I$_{corr}$), Tafel slopes (β_a, β_c), percentage inhibition efficiency (%IE),corrosion rate (CR) of α-Al bronze alloy in (3.5% NaCl + 100 ppm·S^{2-}).

Conc., ppm	I$_{corr}$. mAcm^{-2}	E$_{corr}$. −mV	β_c −mVdec.$^{-1}$	β_a mVdec.$^{-1}$	IE%	Corrosion rate (CR) mm/year
blank (3.5% NaCl + 100 pm·S^{2-})	0.00751	885	20.3154	30.6427	-	0.08712
blank + 500 ppm ala	0.00492	857	17.1083	14.6820	34.48	0.05093
blank + 500 ppm cys	0.00183	750	18.2768	18.6279	75.63	0.02126

and Cu_2O as an outer layer using X-ray diffraction and auger electron spectroscopy (AES) techniques on Al-bronze surfaces in sodium chloride solutions.

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \qquad (12)$$
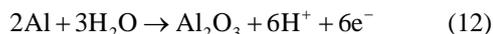
The presence of aluminum oxide (Al_2O_3) often has been postulated to explain the improved corrosion resistance of Cu-Al alloys as compared to pure copper. The ability of this oxide to heal rapidly in the work environment is the important criterion for application. **Figure 3(b)** shows the polarization curves of the tested alloy in 3.5% NaCl + 100 ppm·S^{2-} (blank) with 500 ppm of alanine (ala) and blank + 500 ppm of cysteine (cys). In the presence of inhibitor, the cathodic and anodic curves were shifted toward more-positive potentials, compared to that in the absence of inhibitor. This indicates that the presence of cys and ala species enhances the passivation and retardation of the alloy dissolution due to their adsorption on the alloy surface. The electrochemical corrosion and SCC measurements are in good agreement.

3.2.2. Electrochemical Impedance Spectroscopy (EIS)

Electrochemical impedance spectroscopy (EIS) is a powerful, non-destructive electrochemical technique for characterizing electrochemical reactions at the metal/electrolyte interface and the formation of corrosion products [28]. Impedance measurements provide information on both the resistive and capacitive behavior of the interface, evaluation on the performance of studied compounds as possible corrosion inhibitor, and investigation of the corrosion inhibition processes [28].

EIS measurements have been carried out for Al-bronze alloy in the absence and presence of different inhibitor concentrations. **Figures 4** and **5** illustrate the influence of both ala and cys on the impedance response of Al-bronze alloy in 100 ppm·S^{-2} at 25°C. The semicircular appearance of Nyquist plot shows that the charge transfer process takes place during dissolution [29-31]. The simplest fitting is represented by Randles equivalent circuit (**Figure 4(c)**), which is a parallel combination of the charge-transfer resistance (R_{ct}) and a constant phase element (C_{dl}) both in series with the solution resistance (R_s). **Table 3** presents the values of R_{ct} and C_{dl} obtained for Al-bronze alloy in the absence and presence of optimum concentration of ala and cys at 25°C. In all cases, R_{ct} values increase, while those of C_{dl} decrease in the presence of ala and cys, possibly due to the adsorption of the inhibitor on the electrode surface [32].

(a)

(b)

(c)
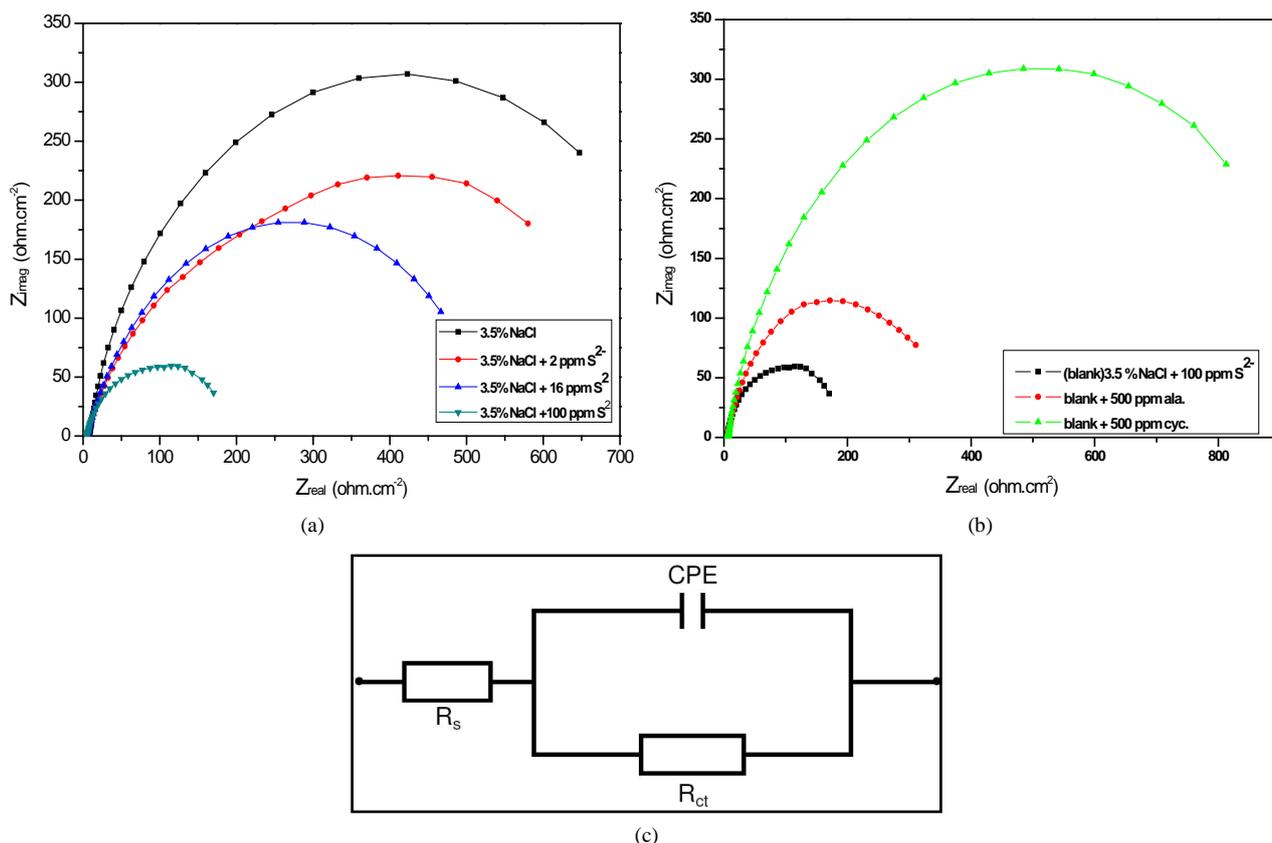
Figure 4. The Nyquist plots for Al-Bronze alloy in 3.5% NaCl solutions containing (a) different concentrations of sulfide ions, (b) 100 ppm·S^{2-} + 500 ppm of ala or cys, and (c) The equivalent circuit model used to fit the impedance data.

(a)

(b)
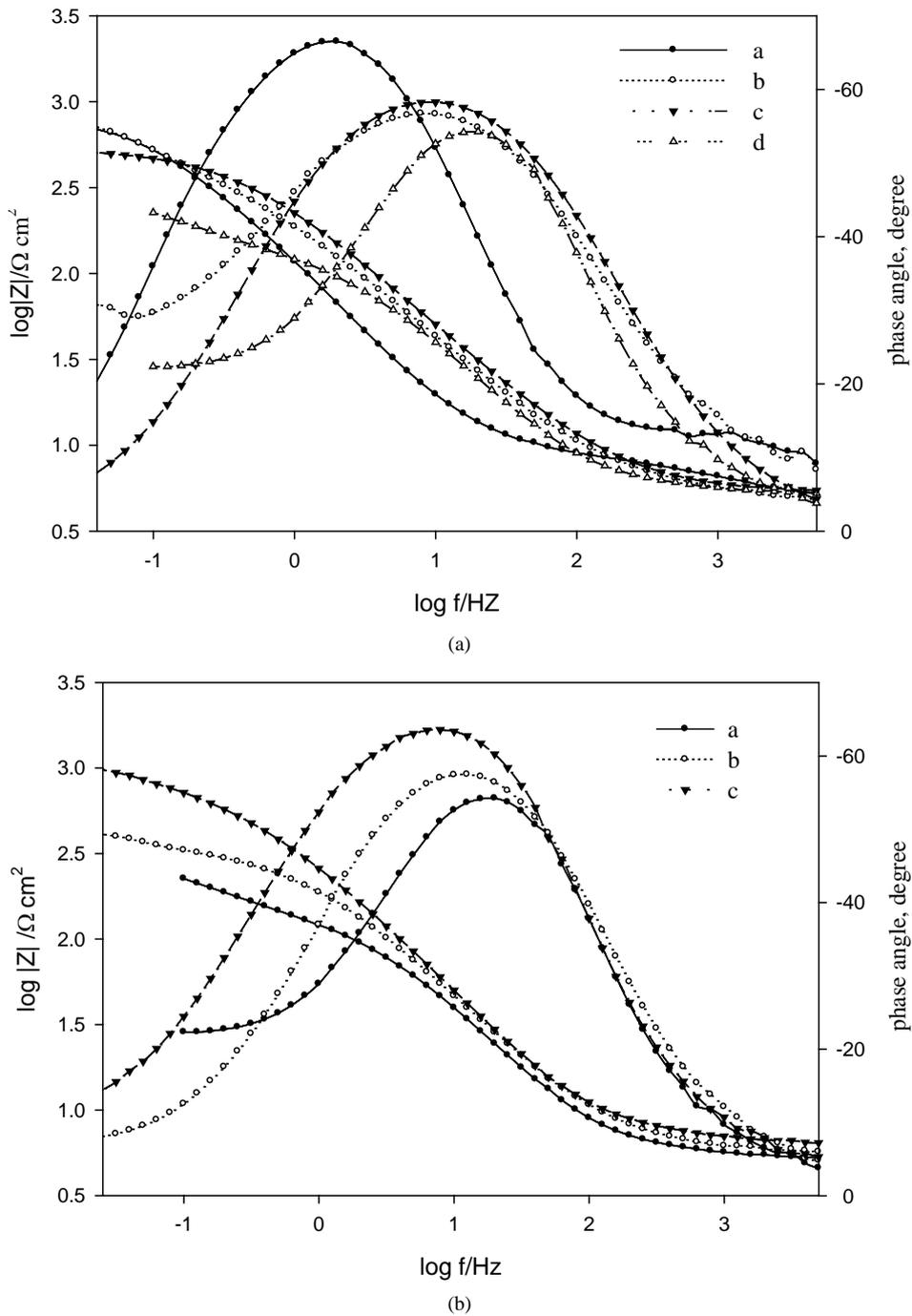
Figure 5. The Bode plots for Al-bronze alloy in (a) 3.5% NaCl in the absence (curve a) and in presence of 2 ppm·S^{2-} (curve b), 16 ppm·S^{2-} (curve) and 100 ppm·S^{2-}, and (b) 3.5% NaCl + 100 S^{2-} (curve a) and in the presence of 500 ppm ana (curve b), 500 ppm cyst (curve c)

Table 3. Electrochemical kinetic parameters obtained by EIS technique for the corrosion of Al-bronze alloy in 3.5%NaCl + 100 ppm·S^{2-} (blank) and in the presence of 500 ppm alanine, 500 ppm cysteine.

Concentration	R_s Ohm·cm^2	R_{ct} Ohm·cm^2	C_{dl} μF/cm^2	n	IE%
Blank(3.5% NaCl + 100 ppm·S^{2-})	5.576	204.3	402.0	0.91	-
blank + 500 ppm ala	5.576	365.8	381.2	0.94	43.0
blank + 500 ppm cys	6.386	962.9	317.4	0.93	78.7

The obtained R_{ct} values were used to calculate the inhibition efficiency ($\eta\%$) for the different inhibitors as:

$$\eta(\%) = \left[(R_{ct} - R'_{ct})/R_{ct}\right] \times 100 \qquad (13)$$

where R_{ct} and R'_{ct} are the charge transfer resistances for the inhibited and uninhibited solutions, respectively. The obtained $\eta\%$ values are listed in **Table 3**. Cys was found to act as a better corrosion inhibitor than ala, achieving an inhibition efficiency close to 80% under the most aggressive conditions used. Cysteine (denoted as R-SH) has more inhibition efficiency than alanine, cys contains -SH group which has strong affinity for copper. Cys can be adsorbed as bidentate ligands, in which surface coordination is taking place through both amino group and the -SH moiety. The corrosion inhibition of cys can be attributed to the adsorption of the -SH group on Cu surface blocking more corrosion sites [33,34].

4. Conclusions

1) α-Al bronze was found to be susceptible to SCC in sulfide-polluted 3.5% NaCl solutions.

2) Increasing the sulfide ions concentration leads to a decrease in the maximum stress (σ_{max}) and changes the mode of the fracture surface from ductile to brittle transgranular.

3) The addition of alanine and cysteine to the test electrolytes reveals the positive effect of amino acids to improve the resistance of the alloy toward SCC in sulfide-polluted salt water, with changing the mode of fracture from brittle transgranular cracking to ductile failure.

4) EIS showed that the presence of ala and cys in the test solution decreases C_{dl} and increases R_{ct}.

5) The electrochemical and SCC measurements are in good agreement.

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