

## Research Article

## Effect of Cr and Mo on the Corrosion Behavior of Some Low Alloy Steels in Acidic Media

Hesham T.M. Abdel-Fatah<sup>1,\*</sup>, Aliaa A. M. Hassan<sup>2</sup>, Maison M. Shetify<sup>2</sup>,  
Hala E.E. El-Sehiety<sup>1</sup>, Hesham S. Abdel-Samad<sup>3,4</sup>, Khalid M. Zohdy<sup>5</sup>

<sup>1</sup> Central Chemical Laboratories, Egyptian Electricity Holding Company, Sabtia, Cairo, EGYPT.

<sup>2</sup> Faculty of Science and Arts in Al-Ardha, Jazan University, Al-Ardha, Jazan, KSA.

<sup>3</sup> Chemistry Department, Faculty of Science, Ain Shams University, Abbassia, Cairo, EGYPT.

<sup>4</sup> Basic Sciences Department, Community College, Taibah University, Madina, KSA.

<sup>5</sup> Higher Technological Institute, Tenth of Ramadan City, EGYPT.

**Abstract**

Low alloy steels are widely employed for boiler tubing and steam piping in thermal power plants. This is mainly due to its excellent oxidation and corrosion resistance as well as superior strength at high temperature and pressure. Scale formation in internal surface of boiler tubes is one of the major problems in most of high-pressure boilers of thermal power stations. Two major problems result from deposit fouling on the interior tube surfaces: (1) Loss of heat transfer, and (2) Under-deposit corrosion. Therefore, it is necessary to clean the heat transfer surfaces to remove scale formed during normal operation, in order to minimize corrosion and maintain the desired heat transfer

capabilities of industrial equipment. Mineral acids are typically employed for this purpose. The present study addresses the corrosion behavior of two low alloy steels (ASTM A213 grade T22 and T92) in hydrochloric and sulfuric acid solutions at different temperatures and stirring velocities using gravimetric test, electrochemical impedance spectroscopy (EIS) method and the new technique electrochemical frequency modulation (EFM).

**\*Correspondence**

Hesham Tawfik M. Abdel-Fatah  
E-mail: hesham\_tm@yahoo.com  
Tel/Fax: +20 2 22755341  
Mobile: +20 12 22678654

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**1. Introduction**

In a power plant, during operation processes progressive build-up of scale occurs inside the boiler tubes. Therefore, maintaining clean boiler tubes is vital importance to reliable, efficient power plant operation. Two major problems result from deposit fouling on the interior tube surfaces: (1) Loss of heat transfer, and (2) Under-deposit corrosion. Therefore, in order to minimize corrosion and maintain the desired heat transfer capabilities of industrial equipment, it is necessary to clean the heat transfer surfaces to remove scale formed during normal operation. One way of removing scale is to chemically clean the boiler tubes. Chemical cleaning is a multiple stage process that seeks to remove all the existing scale from the boiler internals, leaving a clean, passivated waterside system. One step in the process involves the use of acid to dissolve the scales. This acid stage is potentially damaging to the boiler and therefore, it requires careful monitoring to prevent serious metal loss. Hydrochloric, sulfuric, sulfamic and citric acids are commonly used for such purpose [1,2].

There are two basic methods for acid cleaning process, circulation method and static method (sometimes called fill and soak methods). Most of the investigations on the corrosion characteristics of different metal materials in acid media have been made in stagnant conditions. Only few studies in the literatures discuss the effect of solution movement (non-stagnant condition) on corrosion rate [3-5].

Many different low alloy steels have been used worldwide for boiler tubing and steam piping in thermal power plants [6,7].

Therefore, the present work attempts to provide a further understanding of the effect of Cr and Mo on the corrosion resistance of two low alloy steels (ASTM A213 grade T22 and T92) in 0.5M hydrochloric and sulfuric acid solutions at different temperatures and stirring velocities using weight loss test, electrochemical impedance spectroscopy (EIS) method and the recent technique electrochemical frequency modulation (EFM).

## 2. Experimental

Low alloy steels (ASTM A213 grade T22 and T92) strips were used. Table 1 presents the chemical composition (wt. %) of the used alloys.

**Table 1** Chemical composition of the two steels (Weight Percent)

Alloy	Si	Mn	Cr	Fe	Ni	Mo
ASTM A213 grade T22	0.37	0.51	2.28	95.78	-	1.06
ASTM A213 grade T92	0.54	0.46	9.07	89.13	0.34	0.46

Weight loss measurements were carried out with the dried rectangular strips of size (1.5 cm x 1 cm x 0.2 cm) at 25, 45 and 65 °C for 5 days by immersion the low alloy steel coupons into acid solutions. After the elapsed time, the specimens were taken out, washed with ultrapure water, dried, and weighed accurately.

The electrochemical experiments were carried out using a three-electrode glass cell assembly of 150 cm<sup>3</sup> volume capacity. The cell consists of a low alloy steel electrode embedded in epoxy resins with an exposed area of (1cm<sup>2</sup>) as a working electrode, a saturated calomel electrode as a reference electrode, and a platinum foil (1cm<sup>2</sup>) as a counter electrode. The working electrodes were abraded mechanically with fine grade emery papers, degreased with acetone in an ultrasonic bath, washed with ultrapure water and finally dried before use.

All solutions were freshly prepared from analytical grade chemical reagents using washed with ultrapure water. The experiments were conducted in stagnant aerated solutions at different temperatures 25, 45 and 65 °C. Some experiments were carried out at different stirring velocities 600 and 1200 RPM using adjustable digital magnetic stirring hotplate DAIHAN, Model MSH 20.

Electrochemical impedance spectroscopy (EIS) measurements were carried out using AC signals of 10 mV amplitude and sweeping the frequency from 10 kHz to 300 mHz. The electrode was kept immersed for 30 min in acid solutions before starting measurement to attain steady state.

The Electrochemical Frequency Modulation (EFM) is an electrochemical technique in which two sinusoidal potential signals are summed and applied to a corrosion sample through a potentiostat. The resultant current is measured and the time-domain data are converted to the frequency domain. This frequency

domain is used to measure the signal at the applied fundamental frequencies, at harmonics of the fundamental frequencies, and at intermodulation frequencies. The intermodulation spectra contain current responses assigned for harmonically and intermodulation current peaks. By the appropriate mathematical manipulation, the large peaks are used to directly determine the values of corrosion current density ( $I_{corr}$ ), corrosion rate, Tafel constants ( $\beta_c$  and  $\beta_a$ ) and the causality factors (CF2 & CF3). This is in accordance with the theory of EFM technique, which was reported previously [8]. EFM carried out using two frequencies 2 and 5 Hz and the base frequency was 1 Hz. EFM has many features such as [8]:

- 1) It is considered to be a non-destructive technique.
- 2) It is a fast test (requires only few minutes).
- 3) The EFM technique has internal checking parameters called causality factors, by which the experimental data can be verified. These two factors CF2 & CF3 should have the values 2.0 and 3.0 if all of the conditions of EFM theory have been met.

All Electrochemical experiments were carried out using Gamry PCI300/4 Potentiostat/Galvanostat/Zra analyzer, EIS300 Electrochemical Impedance Spectroscopy software, EFM140 Electrochemical Frequency Modulation software and Echem Analyst 5.21 for results plotting, graphing, data fitting and calculating.

## 3. Results and Discussion

### 3.1 Effect of solution temperature on the corrosion rate:

#### 3.1.1 Weight loss studies:

Measurements of weight changes were performed on rectangular coupons of total exposed area of (4 cm<sup>2</sup>). The coupons were immersed in aerated static 0.5M hydrochloric and sulfuric acid solutions for different time intervals, up to 5 days.

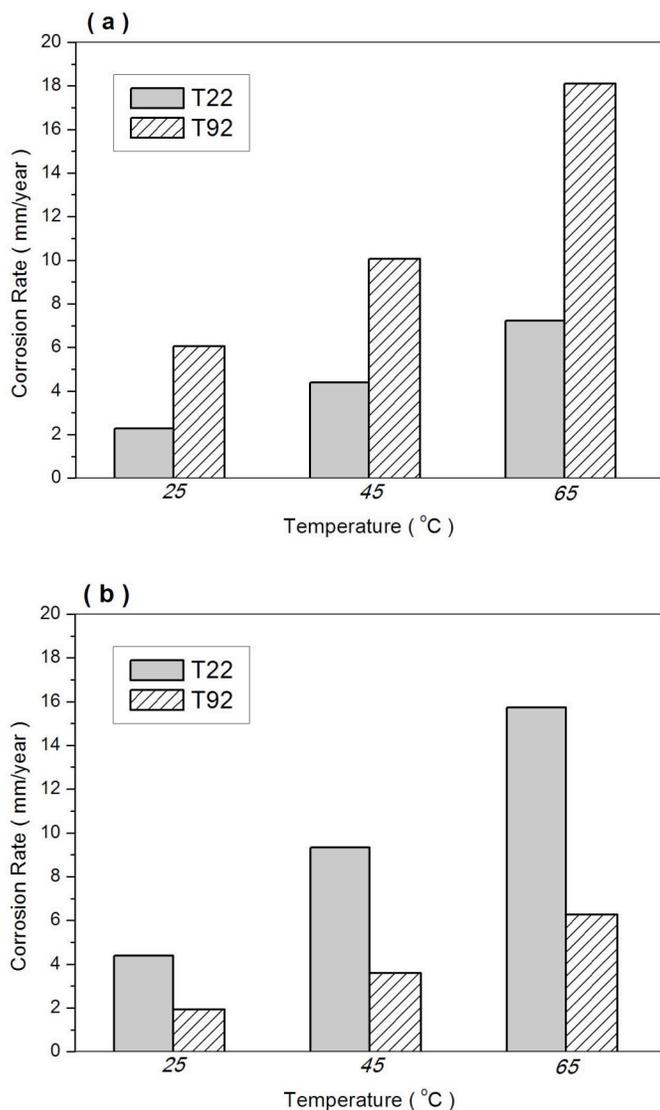
The corrosion rate in units of millimetres per year (mm/year) was calculated from the weight loss, time of exposure and original exposed surface area of the material by the following formula [9,10]:

$$\text{Corrosion rate (mm/year)} = 3.16 \times \left( \frac{W}{DA t} \right) \quad (1)$$

where  $W$  is the weight loss in milligrams,  $D$  is the density in g/cm<sup>3</sup> ( $D = 7.88$ ),  $A$  is the area in square inches ( $A = 0.62$ ) and  $t$  is the time of exposure in hours ( $t = 120$ ).

The corrosion rate values obtained from weight loss method for the two steels were demonstrated in Figure

1, which represent the comparison between the corrosion rates of T22 and T92 steels at different temperatures in both acids.



**Figure 1** Corrosion rates of T22 and T92 steels after 120 hours of immersion in 0.5 M a) HCl and b) H<sub>2</sub>SO<sub>4</sub> at different temperatures

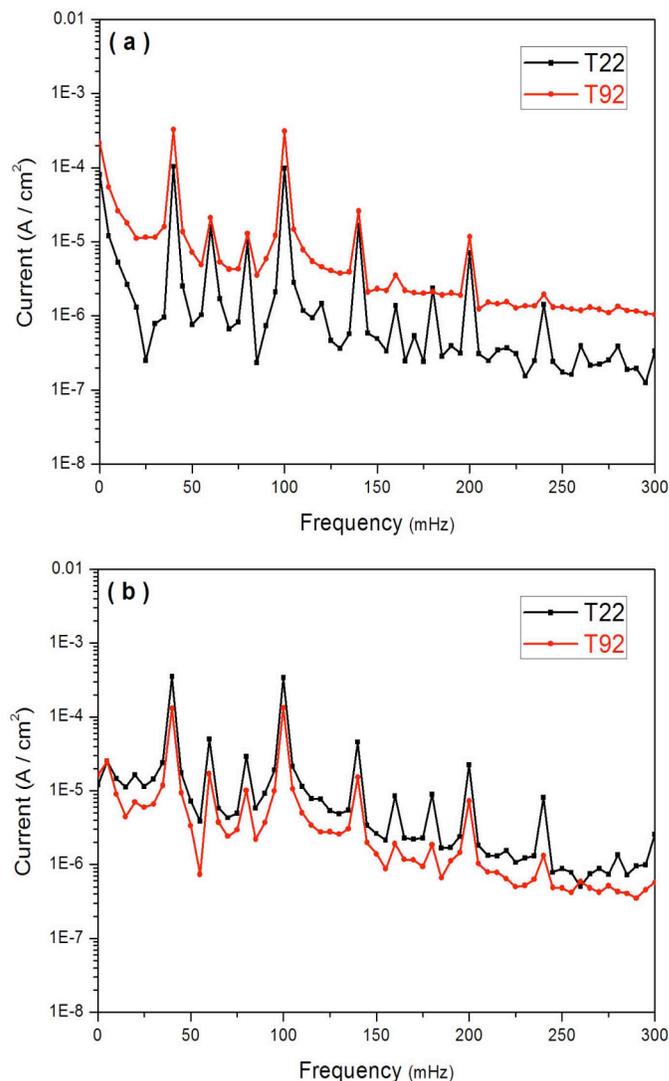
Inspections of the obtained data presented in Figure 1 reveal the following:

- 1) The weight loss and therefore the rate of corrosion enhance of each alloy with increasing the solution temperature.
- 2) The corrosion rate of the two alloys in 0.5 M hydrochloric acid obeys the order: T92 > T22. While, the corrosion rate was found to follow the sequence: T22 > T92 in sulfuric acid.
- 3) The corrosion rate of T22 steel in sulfuric acid is higher than that in hydrochloric acid. In contrast, the

corrosion rate of T92 steel in sulfuric acid is lower than that in hydrochloric acid.

### 3.1.2 Electrochemical frequency modulation studies:

Figure 2 - as an example - shows intermodulation spectra obtained from the EFM measurements for both alloys (T22 and T92) in 0.5 M hydrochloric and sulfuric acid solutions at 25 °C.



**Figure 2** Intermodulation Spectra of T22 and T92 steels in 0.5 M a) HCl and b) H<sub>2</sub>SO<sub>4</sub> at 25 °C

Obtained parameters from EFM technique,  $I_{corr}$ ,  $\beta_a$ ,  $\beta_c$ , corrosion rate and both causality factors (CF2 and CF3) were given in Table 2.

Inspections of the obtained data listed in Table 2 indicate the following:

- 1) Corrosion current density ( $I_{corr}$ ), and hence the corrosion rate of each steel increases with increasing temperature.

2) High corrosion rate is observed for T92 steel followed by T22 steel in 0.5 M hydrochloric acid. While, this  
 3) The values of corrosion rate for T22 steel in hydrochloric acid are less than that in sulfuric acid.  
 4) The corrosion rate of T92 alloy in hydrochloric acid is higher than that in sulfuric acid.

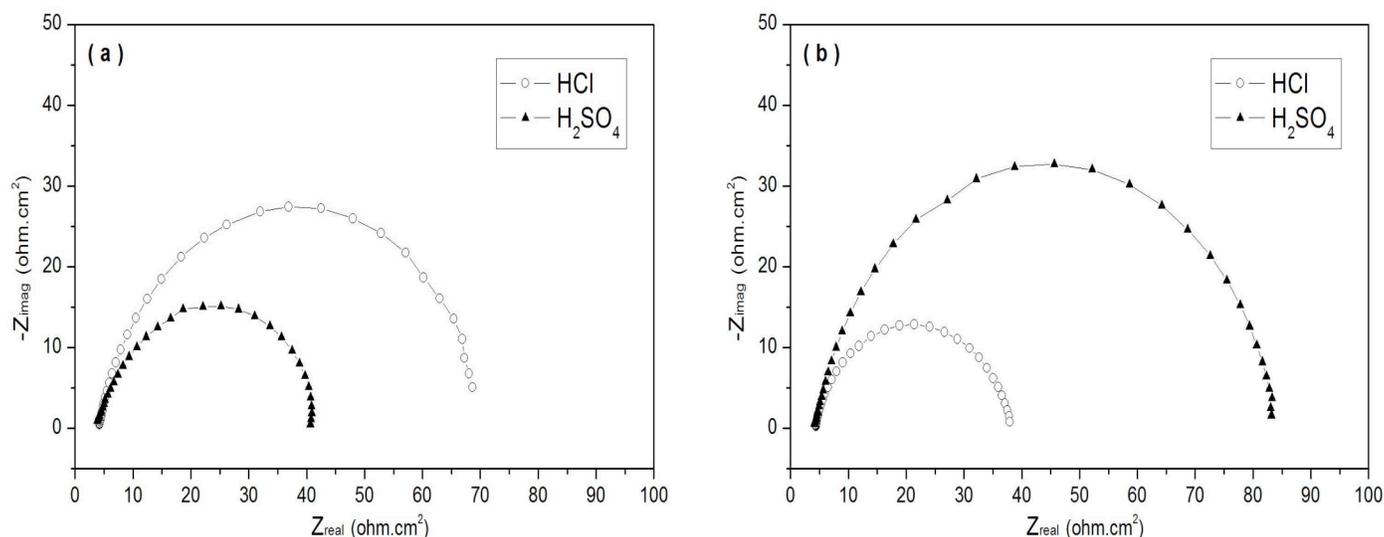
sequence changed in case of 0.5 M sulfuric acid to obey the order: T22 > T92.

5) The values of causality factors CF2 and CF3 obtained under different experimental conditions are approximately equal the theoretical values 2 and 3 respectively, indicating that the measured data obtained from EFM technique are reliable [8].

**Table 2** Corrosion rates of the two alloys in 0.5 M hydrochloric and sulfuric acid solutions at different temperatures

Acid	Alloy	Temp. (°C)	$\beta_a$ (mV.dec <sup>-1</sup> )	$\beta_c$ (mV.dec <sup>-1</sup> )	CF(2)	CF(3)	$I_{corr}$ ( $\mu$ A.cm <sup>-2</sup> )	CR (mm/y)
HCl	T22	25	89.1	171.7	1.912	2.984	243.4	2.82
	T92		87.6	168.4	1.688	3.245	493.5	5.71
	T22	45	86.3	161.7	1.896	2.754	400.2	4.63
	T92		89.6	156.7	2.117	2.974	891.2	10.31
	T22	65	87.8	158.2	1.982	2.876	715.6	8.28
	T92		88.5	161.2	2.088	3.129	1458	16.87
H <sub>2</sub> SO <sub>4</sub>	T22	25	86.7	147.6	1.818	3.256	357.2	4.13
	T92		88.5	150.2	1.964	3.176	187.2	2.17
	T22	45	89.3	149.3	2.198	2.721	770.3	8.91
	T92		83.6	144.8	2.225	2.942	288.6	3.34
	T22	65	84.1	147.7	1.755	3.356	1289	14.91
	T92		85.7	142.7	2.113	2.897	647.7	7.49

### 3.1.3 Electrochemical impedance studies:



**Figure 3** Nyquist plots for the two alloys a) T22 and b) T92 in both acids at 45 °C

The above Figure 3 - as an example - shows a typical set of Nyquist plots for T22 and T92 in both acids at 45 °C.

The impedance spectra of the two alloys T22 and T92 were traced at open circuit potentials in aerated stagnant 0.5 M hydrochloric and sulfuric acid solutions at different temperatures were carried out and presented as Nyquist plots.

The determined values of charge-transfer resistance ( $R_{ct}$ ) and double layer capacitance ( $C_{dl}$ ) were listed in Table 3.

The corrosion current density ( $I_{corr}$ ) can be calculated using the charge-transfer resistance ( $R_{ct}$ ) based on the following Stern-Geary equation [11,12];

$$I_{corr} = \frac{1}{2.303} \times \left( \frac{\beta_a \beta_c}{\beta_a + \beta_c} \right) \times \frac{1}{R_p} \quad (2)$$

where  $I_{corr}$  is the corrosion current density (mA/cm<sup>2</sup>),  $\beta_a$  is the anodic Tafel slope (mV/decade),  $\beta_c$  is the cathodic Tafel slope (mV/decade), and  $R_p$  is the polarization resistance (ohm.cm<sup>2</sup>). Both Tafel slopes ( $\beta_a$  and  $\beta_c$ ) were collected from EFM method.

The corrosion current density ( $I_{corr}$ ) can be converted to corrosion rates in units of millimetres per year (mm/year) based on the following Faraday's Law [11,12];

$$\text{Corrosion rate (mm/year)} = 3.15 \times 10^5 (M / nFD) \times I_{corr} \quad (3)$$

where  $M$  is atomic mass of metal in g/mole ( $M = 55.85$ ),  $n$  is number of electrons involved of the corrosion reaction in mole ( $n = 2$ ),  $F$  is Faraday's constant in coulombs/mole ( $F = 96500$ ), and  $D$  is density of metal in g/cm<sup>3</sup> ( $D = 7.88$ ).

The calculated corrosion rate values obtained from the charge-transfer resistance ( $R_{ct}$ ) were also listed in Table 3.

The results obtained from EIS technique are compatible with the above results, where the following points could be observed through the achieved results extracted from EIS method:

1) By increasing the solution temperature, charge-transfer resistance ( $R_{ct}$ ) values will decrease, and therefore values of double layer capacitance ( $C_{dl}$ ) will increase.

2) The corrosion resistance for the two alloys in 0.5 M hydrochloric acid obeys the sequence: T22 > T92. While, it obeys the order: T92 > T22 in 0.5 M sulfuric acid.

3) The corrosion rate of T22 alloy in sulfuric acid is higher than that in hydrochloric acid and the opposite happens in the case of T92 alloy.

**Table 3** Corrosion rates of the two alloys in 0.5 M hydrochloric and sulfuric acid solutions at different temperatures

Acid	Alloy	Temp. (°C)	$C_{dl}$ ( $\mu\text{F.cm}^{-2}$ )	$R_{ct}$ (ohm.cm <sup>2</sup> )	CR (mm/y)
HCl	T22	25	136.4	115.8	2.55
	T92		300.4	53.81	5.39
	T22	45	277.1	58.63	4.83
	T92		818.7	29.34	9.77
	T22	65	582.6	35.90	7.91
	T92		1349	15.92	18.06
H <sub>2</sub> SO <sub>4</sub>	T22	25	264.7	60.87	4.51
	T92		111.8	123.5	2.27
	T22	45	621.2	33.79	8.32
	T92		172.8	72.49	3.68
	T22	65	1018	19.46	13.85
	T92		538.7	37.11	7.26

### 3.2 Effect of solution stirring on the corrosion rate:

Corrosion rate of the two steels T22 and T92 in aerated stirring 0.5 M hydrochloric and sulfuric acid solutions at stirring speed 600 and 1200 RPM and at 25 °C were determined using EFM and EIS techniques. The produced results from both techniques were listed in Table 4.

Revisions of these results infer the following:

1) Corrosion current density ( $I_{corr}$ ) increases while charge-transfer resistance ( $R_{ct}$ ) decreases with increasing stirring speed.

2) The corrosion rates under static conditions are lower than that under stirring conditions.

3) At a given stirring speed, the rate of corrosion for the two alloys in 0.5 M hydrochloric acid obeys the sequence: T92 > T22. While the corrosion rate in 0.5 M sulfuric acid obeys the order: T22 > T92.

4) Corrosion rate values produced from EFM measurements are comparable with those obtained from EIS method.

**Table 4** Effect of stirring speed on the corrosion rates of the two alloys in 0.5 M hydrochloric and sulfuric acid solutions using the obtained data from EFM and EIS techniques

Acid	Alloy	RPM	$I_{corr}$ ( $\mu\text{A}\cdot\text{cm}^{-2}$ )	CR (mm/y)	$R_{ct}$ ( $\text{ohm}\cdot\text{cm}^2$ )	CR (mm/y)
HCl	T22	600	368.7	4.27	62.86	4.38
		1200	682.6	7.90	38.57	7.16
	T92	600	722.6	8.36	32.92	8.42
		1200	1501	17.36	17.43	16.12
H <sub>2</sub> SO <sub>4</sub>	T22	600	694.4	8.03	36.67	7.68
		1200	348.6	4.03	76.49	3.71
	T92	600	1394	16.13	17.25	15.64
		1200	804.7	9.31	33.95	8.35

### 3.3 Effect of alloying elements on the corrosion rate

A particular aspect of steel development receiving much attention recently is the role of passive films to limit the corrosion [13]. Chromium (Cr) and Molybdenum (Mo) additions have an important role in this regard. Chromium is commonly added to steel to increase corrosion resistance and harden ability as well as to improve high-temperature strength [14].

It is known that Molybdenum may prevent corrosion in three ways. First, it may improve the resistance of the passive film to break down. Second, it may enhance repassivation rates. Third, it may retard the dissolution process and allow more time for repassivation to repair the film break down sites. Molybdenum was found to retard corrosion by forming Mo salt film on the surface of the alloy. This film is difficult to break down. If this film is broken, the surface of the alloy will be enriched with Mo, which then further retards the dissolution of the alloy [15-18].

Additionally, many authors have suggested other mechanisms for the beneficial influence of Mo [19-21]. It is proposed that the main effect of Mo is to decrease the rate of dissolution in active zones by formation and retention of molybdenum oxyhydroxide or molybdates at these sites. Other mechanisms include; dissolving Mo from the substrate, into the solution, and then oxidized to molybdate, enrichment of Cr in the oxide layer by the selective dissolution of Mo and thickening of the passive film and stabilisation of the Cr oxides by the presence of Mo<sup>6+</sup> forming an amorphous oxide film with a glassy structure. In the present investigation, two types of low alloy steels T22 and T92 have been subjected to general corrosion studies in 0.5 M hydrochloric and sulfuric acid solutions.

The corrosion resistance of the two steels in sulfuric acid was found to obey the following order: **T92 (9.0Cr-0.5Mo) > T22 (2.25Cr-1.0Mo)**

This trend indicates that the corrosion rate of these samples decreases with increasing the chromium content in the alloy. The presence of higher content of Cr in T92 alloy has attributed to the Cr enrichment in the passive layer and a thickening of the passive film and hence increases its corrosion resistance [18]. It is clear that the corrosion resistance of T92 alloy is higher than that of T22 alloy even though the T22 steel contains higher Mo content (1.0%) than the T92 steel (0.5 %).

While, in hydrochloric acid solutions, the opposite takes place, the corrosion resistance of the two steels was found to obey the following order: **T22 (2.25Cr-1.0Mo) > T92 (9.0Cr-0.5Mo)**. Although, T92 steel contains 9.0% chromium, the corrosion rate is more than T22 steel, which contains only 2.25% chromium. This result suggests that chromium alone is not helpful in minimizing corrosion in hydrochloric acid. The above trend can be explained to a chemical reaction between the chlorine and chromium. The resultant product is chromium chloride (CrCl<sub>2</sub>), which is water soluble and thus dissolves out of the steel into the solution. The chemical reaction between the chromium and chlorine tends to occur in where the chromium in the steel is at its highest. Such suggestion was documented by Chu et al. [22] who discovered that, although the presence of chromium in the alloys promotes the formation of Cr<sub>2</sub>O<sub>3</sub>, high levels of chromium have an adverse effect on the chlorinated resistance. Thus, the increase of chromium content of T92 steel and the small percentages of molybdenum can cause an increase of the chemical reaction between the chlorine and chromium. This conclusion can also explain the increase of corrosion rate

for T92 steel in hydrochloric acid than that in sulfuric acid.

Furthermore, the high resistance to corrosion of T22 steel than that of T92 steel in HCl may be also due to an increase in the Mo content in the T22 alloy, which increases the stability of the passive film and, hence, the ability of the steel to resist the corrosion. This finding reveals that both Cr and Mo act cooperatively [23]. Thus, the increase of molybdenum content of T22 steel ( $\text{Mo} \approx 1\%$ ) than that in T92 alloy ( $\text{Mo} \approx 0.5\%$ ), may be the cause of reducing the chemical reaction between the chlorine and chromium in T22 steel.

In this regard, several works concluded that Mo promotes the repassivation process by forming insoluble molybdenum chloride complexes [24,25] or  $\text{MoO}_4^{2-}$  [26] at the active sites. Furthermore, Sugimoto and Sawada [16] considered that the adsorption of  $\text{MoO}_4^{2-}$  in pits could inhibit the early stages of pit growth. Additionally, other researchers have proposed transient enrichment of  $\text{Mo}^{6+}$  in the active region, which blocks the active surface sites by formation of Mo oxyhydroxide or Cr-Fe molybdate [27,28].

It is worth interesting to observe that the corrosion resistance of T22 steel in hydrochloric acid is more than that in sulfuric acid. This means that the beneficial effect of Mo content in T22 alloy observed in HCl medium does not appear in  $\text{H}_2\text{SO}_4$  solution. This effect is due to the dissolution of Mo in  $\text{H}_2\text{SO}_4$  to generate  $\text{Mo}^{3+}$  and/or  $\text{Mo}^{5+}$  soluble species. Both  $\text{Mo}^{3+}$  and  $\text{Mo}^{5+}$  do not exert inhibition effect on corrosion process [14,29].

From the above results and interpretations, the following important points can be drawn:

- 1) The acid cleaning process for boiler parts made of T22 and T92 alloys will be quite satisfactory, when it is applied at stagnant conditions (fill and soak method) and at ambient temperature.
- 2) In the case that boiler parts are made of T22 alloy, the acid cleaning process can be carried out using hydrochloric acid.
- 3) For the boiler parts made of T92 alloy should not be used hydrochloric acid during an acid cleaning process due to its high corrosivity, but can be used the sulfuric acid.

## Conclusion

The corrosion behavior of two low alloy steels T22 and T92 in hydrochloric and sulfuric acid solutions has been investigated. The following conclusions can be made:

- 1) Corrosion rate of both steels increases by increasing the solution temperature and stirring movement.
- 2) Corrosion rate of T92 steel (9.0Cr-0.5Mo) is

significantly lower than its value of T22 steel (2.25Cr-1.0Mo) in sulfuric acid solutions. This behavior can be explained to an increase in the Cr content in the T92 alloy which promotes the formation of passive layer ( $\text{Cr}_2\text{O}_3$ ) and a thickening of this film and hence increases its corrosion resistance. Therefore, it is preferable to carry out acid cleaning process for the boiler parts manufactured from T92 steel with sulfuric acid and not with hydrochloric acid.

3) The rate of corrosion for the two alloys in hydrochloric acid was found to follow the sequence:  $\text{T92} > \text{T22}$ . This trend is due to combine the chloride with the chromium in the passive layer of T92 and form soluble chromium chloride. While high molybdenum content in T22 alloy minimizes this reaction. Additionally, Mo promotes the repassivation process by forming insoluble molybdenum chloride complexes. Accordingly, it is recommended to use hydrochloric acid and not sulfuric acid during the acid cleaning process for the boiler parts made of T22 alloy.

4) There is a great harmony between the results achieved from the three techniques. This asserts that EFM technique can be relied upon to determine the corrosion measurements under the studied conditions.

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