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Corrosion of Heat-Treated 304SS in the Presence of Molybdate Ions in Hydrochloric Acid

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The electrochemical behaviors of quenched (non-sensitized) and furnace-cooled (sensitized) 304SS stainless steels have been thoroughly investigated in 0.1M HCl as a function of molybdate ions concentration at 22 °C and 50 °C. Standard corrosion techniques that have been used include potentiostatic polarization, polarization resistance *vs.* time (R_p *vs.* t), and cyclic polarization measurements. The non-sensitized steel showed better resistance to pitting corrosion than the sensitized steel when tested in 0.1M HCl at 22 °C and 50 °C. Furthermore, both steels showed better resistance at 22 °C in comparison to 50 °C. While the addition of molybdate ions at sufficient concentrations (0.075M) enhanced significantly both the resistance to general corrosion and to pitting corrosion in both steels, the non-sensitized steel showed better resistance especially when tested at 22 °C. Testing both steels at low concentrations of molybdate ions (0.001M) at 22 °C and 50 °C resulted in increasing the corrosion current densities, i_{corr}, combined by increasing the repassivation (protection) potential, E_{pp}. It is suggested that molybdate ions increase resistance to general and pitting corrosion by lowering the current densities in the active and passive regions by means of chemical reactions, enhancing repassivation in the process and making it difficult for stable pits to grow.

Keywords: sensitized and non-sensitized stainless steel; HCl; molybdate ions; repassivation.

1. INTRODUCTION

The hexavalent chromium is an effective commercial corrosion inhibitor widely used for surface conversion and as a component in the primer paints. Unfortunately its toxicity poses serious health and environmental hazards. While practical environment-friendly replacements for chromates have been developed, none are as effective as chromates for inhibition of metal corrosion. The existing chromate based inhibitor for metal is believed to be effective for reducing the number of accessible cathodic sites [1]. Moreover, it is well-known that in an intermediate temperature range of 425 ^{o}C to 815 ^{o}C chromium carbides are insoluble and precipitate at grain boundaries creating chromium-depleted zones in the process [2]. The chromium depleted zones are much less corrosion resistance that the surrounding matrix.

Alloying Mo to steels reduces the steel's susceptibility to localized (pitting) corrosion in marine and chemical environments [3]. Environment-friendly, also known as a green inhibitor, molybdate ions (MoO_4^{2-}) have been of interest as a corrosion inhibitor due to their very low toxicity which makes them a candidate to replace chromate (toxic) as a corrosion inhibitor [4]. It has been reported that the effect of alloying Mo to the steel or adding MoO_4^{2-} ions have the same effect in terms of increasing repassivation rate and enhancing resistance to localized corrosion [5-9]. It was proposed [8] that in acidic-chloride solutions, molybdenum may dissolve as molybdate and consequently reacts with Fe cations forming insoluble molybdate precipitates and as a result hindering the transpassive reaction leading to lower current densities. Others argue that the formation of Mo-rich stable oxide film (MoO_3) in the outer region of the passive film is responsible for the enhanced corrosion resistance of stainless steel tested in acidic solutions [10].

Interestingly, a minimum concentration of 5 wt% of Mo was needed in order to achieve positive results in Fe-Mo alloys and concentrations below 5 Wt% were reported to have detrimental effects on repassivation kinetics [5]. Furthermore, Nishimura *et al.* [11] showed that pitting initiation of 304SS in HCl solutions was completely inhibited by the addition relatively high concentration of molybdate ions.

Ilevbare and Burstein [12] suggested that the addition of molybdate ions affect both passivity-lower passive current densities were observed- and pit nucleation by deactivating the sites at which pit formation occur and by reducing the size of the pits, resulting in making these pits more difficult to develop into stable pits.

Isaacs and Huang [13] argued that the major improvement in the corrosion resistance of Mo-containing stainless steels were *not* related to Mo preventing pit initiation, but related to reduction of active dissolution rates of salt-free surfaces by Mo leading in the process to repassivation and termination of localized corrosion.

Interestingly**,** S. A. M. Refaey reported that while molybdate ions acted as inhibitors for vanadium steels tested in 0.1M HCl, their inhibition efficiency (IE) decreased with increasing molybdate ions concentrations [14].

Virtanen et al. [9] concluded that the beneficial effect of alloyed Mo and MoO_4^{2-} was inhibiting metastable pitting corrosion as current transients observed in the passive region at potentials below the pitting potentials decreased with added molybdate.

In conclusion, molybdate ions improve both the resistance to general and localized corrosion (pitting) when added in sufficient concentrations and may have detrimental effect at low concentrations which needs to be further examined. Furthermore, there is no widely accepted mechanism on how Mo or MoO_4^{2-} enhances the corrosion resistance of steels. Needless to mention, the effect of heat treatment, an important metallurgical factor, has not been given the proper attention. The main objectives of this study are to further investigate the effect of MoO_4^{2-} on both the general and localized corrosion of 304SS in HCl solutions as a function of temperature and molybdate ions concentrations taking heat-treatment into consideration by using two different heat-treatments of the same steel.

2. EXPERIMENTAL

The stainless steel-304 (304SS: Fe/Cr18/Ni10) used in all experiments was supplied by Goodfellow, Cambridge, the UK. The chemical composition of the steel is given in Table 1. Both the HCl and Na_2MoO_4 were provided by Panreac, Spain.

Data were collected automatically with the aid of a potentiostat/galvanstat (Gamry G750). All data analysis and extrapolations were performed using specialized Gamry corrosion software (Gamry Echem Analyst).

Prior to each experiment the sample was wet-ground using P 280 to P 1200 grit SiC papers, cleaned with distilled water, acetone and air-dried. An average of three independent experiments was conducted for a given set of conditions in order to verify the results.

Table 1. Composition in wt.% of 304SS used in experiments from EDX analysis.

Steel (304SS)	Cr	Ni	Mn	Si	Fe
wt%	19.23	9.77	1.73	0.46	balance

2.1 Preparation of quenched (non-sensitized) samples

The 304SS (Fe/Cr18/Ni10) samples were vacuum-sealed in quartz tube, heat-treated at 1050 ^{O}C for 30 minutes, followed by quenching in saline water in order to prevent sensitization

2.2 Preparation of furnace-cooled (sensitized) samples

The 304SS samples were vacuum-sealed in a quartz tube, heat-treated at 1050 ^{o}C for 30 minutes, allowed furnace cooling to 750 ^{o}C then samples were kept at 750 ^{o}C for approximately four hours in order to induce sensitization. Finally, the samples were allowed furnace cooling to room temperature.

2.3 Polarization measurements

The samples were electrically connected to copper wires using a silver-based conductive epoxy, allowed to cure at room temperature, and finally mounted in epoxy. The area exposed to the testing solution is 0.3165 cm^2 in all tests. Then the samples were placed in a 3-electrode cell with Pt used as a counter electrode and Ag/AgCl (*KCl*, *sat*.) as a reference electrode. All experiments were conducted in deaerated solutions by pumping purified nitrogen for 30 minutes prior to starting the polarization and continuing throughout the experiments. Magnetic stirrer with a constant rate was maintained in all experiments. All experiments were conducted at 22^oC and 50 ^oC; ±1 ^oC.

The corrosion potential, E_c, was monitored for 30 minutes prior to starting the polarization experiment. The cyclic polarization measurements were conducted using a scanning rate of 1.0 *mV/s*. The cyclic polarization tests were initiated at -500 mV, scanned to +600 mV, and then reversed to -500 mV. The polarization resistance *vs*. time (R_p *vs. time)* measurements were conducted using a scanning rate of 0.1 *mV/s* with experiments conducted within ± 20 *mV vs*. the corrosion potential, E_c. Ten measurements were collected per experiment with 10 minutes time interval between measurements. The potentiostatic experiments were conducted at -250 mV and 400 mV, in the active and pitting regions, respectively.

3. RESULTS AND DISCUSSION

3.1 Measurements at 22 ^oC

3.1.1 Cyclic Polarization Measurements

Figures 1a shows the cyclic polarization plots of 304SS, both non-sensitized and sensitized, in 0.1M HCl in the absence of molybdate ions at 22 ^oC. Figures 1b and 1c show the cyclic polarizations in the presence of different concentrations of molybdates ions. Examination of Figure 1a shows that the quenched (non-sensitized) steel shows slightly better corrosion resistance that the furnace-cooled (sensitized) as apparent from the lower critical current density; 30 μA/cm^2 *vs*. 700 μA/cm^2 and lower passive current densities. On the other hand, the two steels show similar hysteresis in terms of size and shape, with the reverse scans for both coincide with each other. The non-sensitized steel, however, shows a slightly higher repassivation (protection) potential, E_{pp}, (the potential where the forward and reverse scans intersect each other in the cyclic polarization curve) which indicates slightly better pitting resistance.

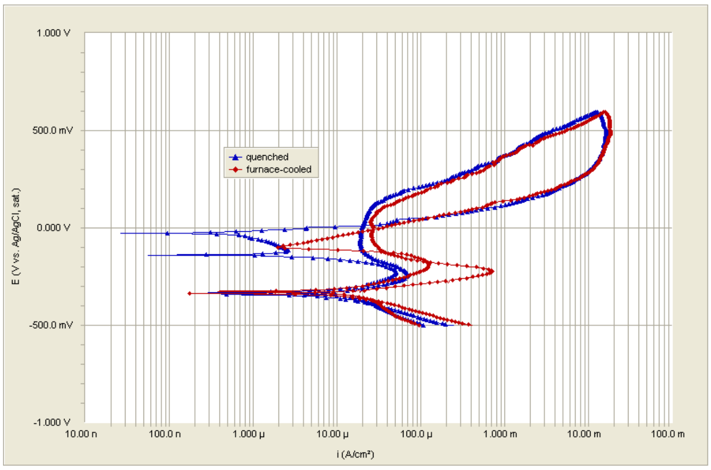

Figure 1a. Cyclic polarization curve of 304SS in 0.1M HCl at 22 ^oC.

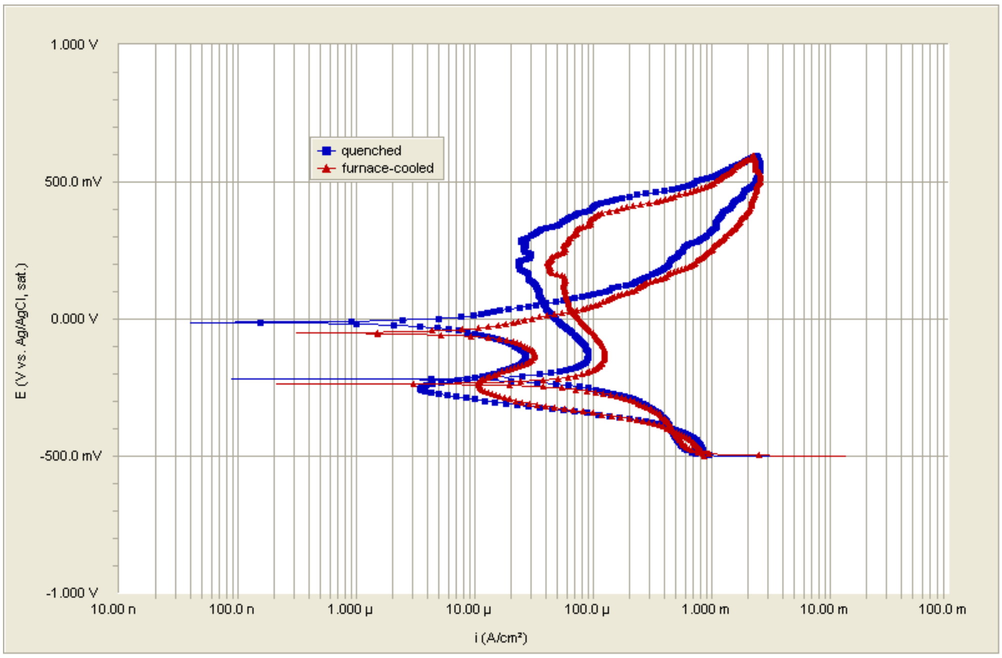

Figure 1b. Cyclic polarization curve of 304SS in 0.1M HCl+0.001M Na_2MoO_4 at 22 ^oC.

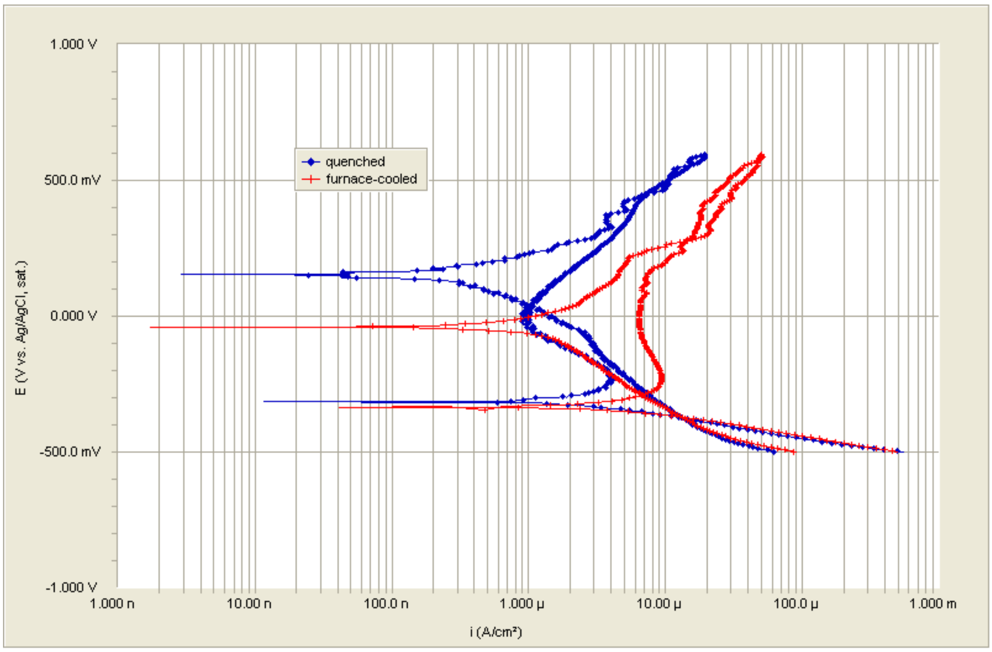

Figure 1c. Cyclic polarization curve of 304SS in 0.1M HCl+0.075M Na_2MoO_4 at 22 ^oC.

The electrochemical corrosion parameters including corrosion potential (E_c), corrosion current density (i_{corr}), and protection potential (E_{pp}) were extrapolated from the cyclic polarization curves of quenched (non-sensitized) and furnace-cooled (sensitized) steel, in the absence and presence of various concentrations of inhibitor (*molybdate ions*) in 0.1 M of hydrochloric acid at 22 °C, and summarized in Tables 2a and 2b, respectively.

Table 2. Data extrapolated from cyclic polarization curves of non-sensitized and sensitized 304SS in 0.1M HCl as a function of molybdate ions concentration at 22 °C. A) Quenched (non-sensitized) tested at 22 °C; and B) Furnace-cooled (sensitized) tested at 22 °C.

A-

Solution	E_c (mV *vs.*Ag/AgCl, sat.)	i_{corr} (A/cm^2)	E_{pp} (mV)
0.1M HCl	-343	1.59×10^{-5}	11.88
0.1M HCl + 0.001M Na$_2$MoO4	-219	1.69×10^{-4}	56.83
0.1M HCl + 0.075M Na$_2$MoO4	-315	6.87×10^{-6}	NA

B-

Solution	E_c (mV *vs.* Ag/AgCl, sat)	i_{corr} (A/cm^2)	E_{pp} (mV)
0.1M HCl	-340	1.49×10^{-5}	-9.14
0.1M HCl + 0.001M Na_2MoO_4	-235	8.34×10^{-4}	26.80
0.1M HCl + 0.075M Na_2MoO_4	-335	3.14×10^{-6}	275.6

The addition of relatively low concentration of molybdate ions (0.001M) resulted in shifting the E_c in the noble direction with a little effect on the passive current densities for both steels. Furthermore, the addition of 0.001M molybdate ions resulted in a smaller hysteresis loop for both steels combined with reduction of the maximum current density at 600 mV from about 20 mA/cm^2 in the absence of molybdate ions to about 2 mA/cm^2 in the presence of 0.001 M molybdate ions. Furthermore, while the pitting resistance of both steels increased with the addition of molybdate ions, the non-sensitized showed better pitting corrosion resistance that the sensitized steel as apparent from lower passive current densities, lower current densities in the reverse scan, and higher repassivation potential. The molybdate ions are most effective in enhancing both general corrosion resistance and pitting resisitance when 0.075 M is used as clear from further reduction in the critical current densities, the passive current densities, the great reduction in the hysteresis loop for the sensitized steel and almost the disappearance of the loop in the non-sensitized steel as shown in Figure 1c. Such reduction of the hysteresis loop combined with noticeable increase in the repassivation potentials are a clear indication of the ability of molybdate ions to reduce and even eliminate pitting corrosion when added in sufficient concentrations.

Figure 1c clearly shows molybdate ions are more effective in reducing pitting corrosion in the non-sensitized steel than the sensitized steel.

Further inspection of Table 2 shows that adding a small amount of molybdate ions (0.001M) may in fact have detrimental effect on the corrosion behavior of 304SS in 0.1M HCl, as i_{corr} slightly increases in the presence of small amount of molybdate ions. Only when added at relatively sufficient concentrations, molybdate ions result in decreasing i_{corr}. Such increase in i_{corr} in the presence of low concentrations of molybdate ions has been reported by some investigators. Xianghong Li and co-workers reported that molybdate ions acted as a corrosion accelerator for Al tested in HCl [15]. M. R.

Ali and co-workers reported that the addition of molybdate accelerated the corrosion of mild steel in acidic chloride media [16].

3.1.2 R_p vs. time measurements

Figure 2a shows the R_p *vs. t* for both steels in 0.1M HCl in the absence of molybdate ions. The non-sensitized has higher average R_p values than the sensitized one indicating its relatively better corrosion resistance. Figures 2b and 2c show the R_p *vs. t* for non-sensitized 304SS and sensitized as a function of molybdate ions concentrations at 22 ^{o}C. Inspection of the Figures shows that the addition of molybdate ions results in increasing the R_p values for both steels as a function of molybdate ions concentration.

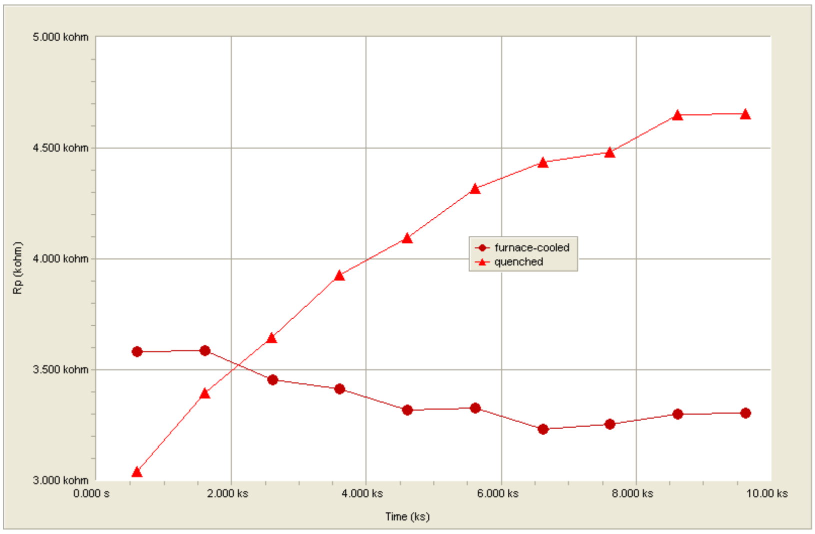

Figure 2a. R_p *vs.* t of 304SS in 0.1M HCl at 22 ^{o}C.

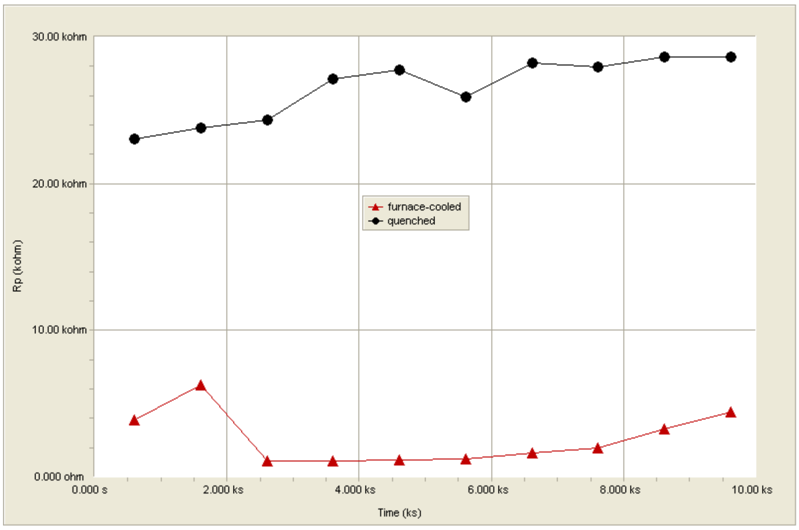

Figure 2b. *Rp vs. t* of 304SS in 0.1M HCl+0.001M Na_2MoO_4 at 22 ^{o}C.

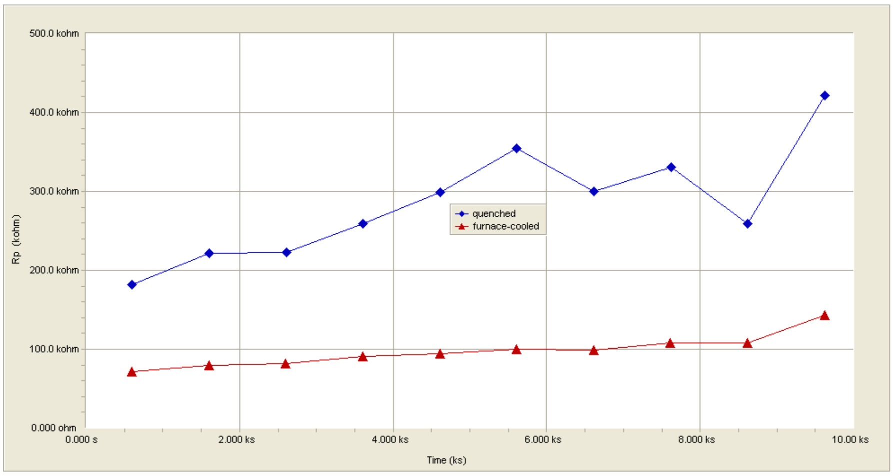

Figure 2c. *Rp vs. t* of 304SS in 0.1M HCl+0.075M Na_2MoO_4 at 22 ^oC.

The polarization resistance (R_p) of both steels tested in the absence and presence of various concentrations of inhibitor (*molybdate ions*) in 0.1 M of hydrochloric acid at 22 °C were summarized in Table 3. As shown in Table 3, the R_p values for the non-sensitized steel are, on average, higher than the sensitized ones for a given concentration of inhibitor.

Table 3. Polarization resistance data for 304SS tested in 0.1M HCl as a function of molybdate ions concentrations tested at 22 ^oC.

Solution	R_p (kΩ-cm^2, quenched, from R_p *vs. t*)	R_p (kΩ-cm^2, furnace-cooled, from R_p *vs. t*)	R_p (kΩ-cm^2, quenched, from cyclic polarization)	R_p (kΩ-cm^2, furnace-cooled, from cyclic polarization)
0.1M HCl	1.29	1.07	1.699	1.048
0.1M HCl + 0.001M Na_2MoO_4	8.40	0.827	0.458	0.332
0.1M HCl + 0.075M Na_2MoO_4	90.2	30.8	7.994	4.561

It is well know that the corrosion current density (i_{corr}) depends on the polarization resistance (R_p) by the following equation: $i_{corr} = B/R_p$, where B is a constant given by B = βa βc/ [2.3(βa + βc)], where βa and βc are the anodic and cathodic Tafel slopes extrapolated from the polarization curves; respectively. Thus, it is obvious that the higher the R_p values, the lower the expected the $i_{corr.}$

Table 3 shows that in absence of molybdate ions the R_p values extrapolated from both techniques (cyclic polarization and R_p *vs. t*) are in agreement with each other for both steels. In addition, both the non-sensitized and the sensitized steels have comparable R_p values and as a result are expected to have comparable i_{corr} as shown in Table 2. Interestingly, in the presence of molybdate ions, the R_p values extrapolated from the cyclic polarization curves are lower than the ones obtained from the R_p. *vs. t* measurements; this is especially true for the non-sensitized steel tested in the presence of 0.075*M* molybdate ions. These lower R_p values extrapolated from the cyclic polarizations curves in the presence of moybdate ions can be attributed to film formation when the steel is cathodically polarized well below the E_c, and the consequence dissolution of the film near E_c, where the R_p values are evaluated, resulting in lower R_p values in comparison to R_p *vs. time* measurements.

3.2 Measurements at 50 ^{o}C

To study the effect of temperature on corrosion inhibition of both steels tested the in absence and presence of inhibitor, the cyclic polarization and R_p vs. *time* measurements were reported at *50 ^{o}C***.**

3.2.1 Cyclic Polarization Measurements

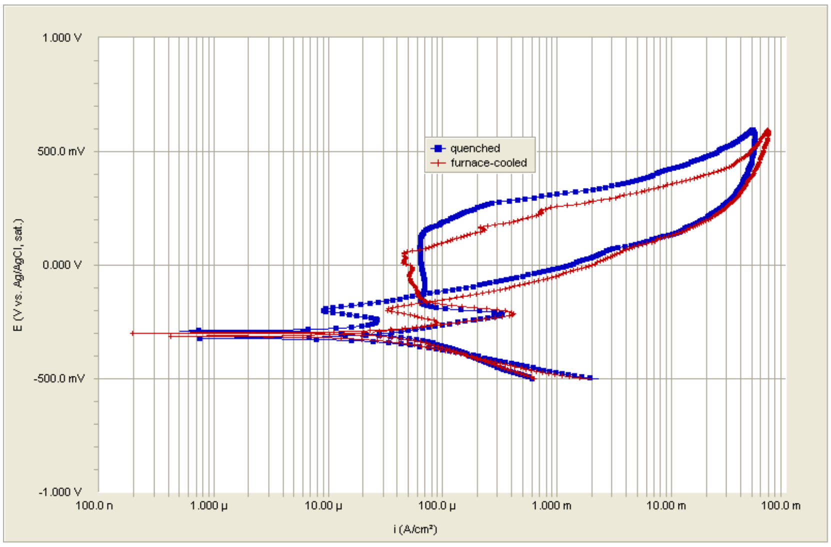

Figure 3a. Cyclic polarization curve of 304SS in 0.1M HCl at 50 ^{o}C.

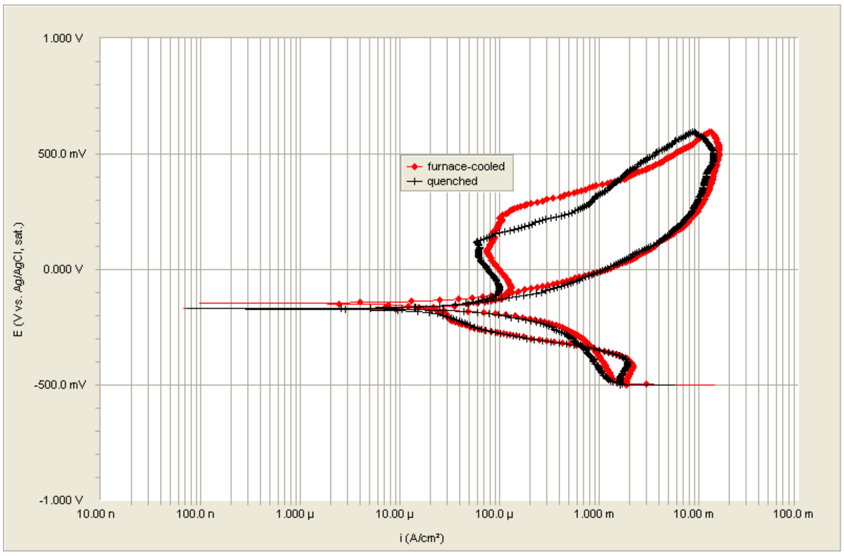

Figure 3b. Figure 1b: Cyclic polarization curve of 304SS in 0.1M HCl+0.001M Na_2MoO_4 at 50 ^{o}C

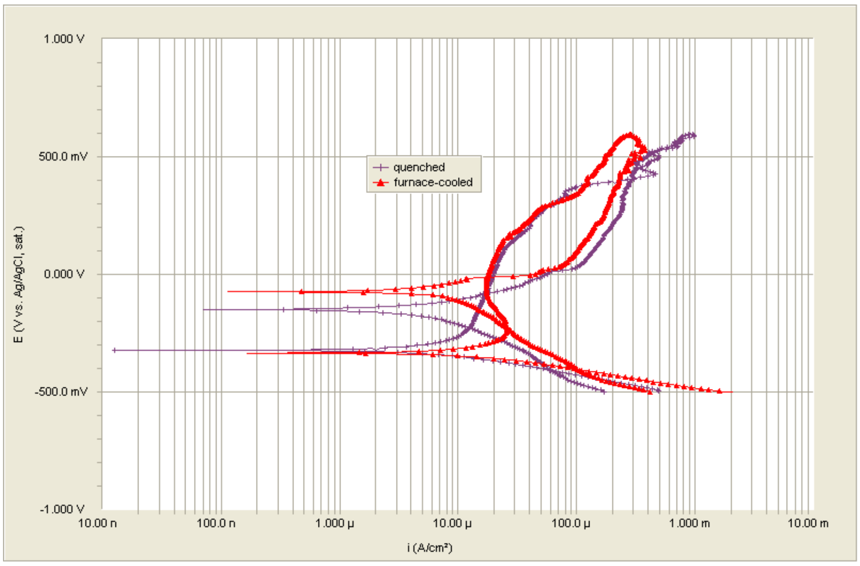

Figure 3c. Cyclic polarization curve of 304SS in 0.1M HCl+0.075M Na_2MoO_4 at 50 ^oC

Figures 3a shows the cyclic polarization plots of 304SS, both non-sensitized and sensitized, in 0.1M HCl in the absence of molybdate ions at 50 ^oC. Figures 3b and 3c show the cyclic polarizations in the presence of different concentrations of molybdates ions.

By comparing Figures 1a and 3a; the main effects of testing at 50 ^oC in comparison to 22 ^oC temperature on both steels in the absence of molybdate ions are the increase in the passive current densities, the increase in the size of the hysteresis loops, and the decrease in the repassivation potential which indicates that 304SS in more prone to pitting at 50 ^oC. While the addition of molybdate ions enhanced the pitting corrosion resistance of both steels tested at 50 ^oC, molybdate ions become less effective at 50 ^oC (Figure 3b and 3c) in comparison to 22 ^oC (Figure 1b and 1c), where larger hysteresis loops appear in the sensitized steel and the non-sensitized steel at 50 °C in comparison to that at 22 ^oC. The later is combined with lower $E_{pp.}$, indicating both steels are more prone to pitting corrosion at 50 ^oC. The above is consistent with Alentejano and Aoki findings who reported an increase of pitting potential with increasing molybdate ions concentrations and a decrease in pitting potential with increasing temperature for 304SS tested in chloride media [17].

Corrosion parameters extrapolated from the cyclic polarizations curves of both steels tested at 50 ^oC are given in Table 4.

Table 4. Results extrapolated from cyclic polarization curves for quenched and furnace-cooled 304SS in 0.1M HCl as a function of molybdate ions concentration at 50 ^oC. A) Quenched tested at 50 ^oC, and B) Furnace-cooled tested at 50 ^oC.

A)

Solution	E_c (mV *vs.* Ag/AgCl, sat)	i_{corr} (A/cm^2)	E_{pp} (mV)
0.1M HCl	-320	1.49×10^{-5}	-103.1
0.1M HCl + 0.001M Na_2MoO_4	-180	1.33×10^{-4}	-65.15

0.1M HCl + 0.075M Na_2MoO_4	-320	$9.65x10^{-6}$	-13.22

B)

Solution	E_c (mV *vs.* Ag/AgCl, sat)	i_{corr} (A/cm^2)	E_{pp} (mV)
0.1M HCl	-313	$2.0x10^{-5}$	-164.1
0.1M HCl + 0.001M Na_2MoO_4	-190	$6.75x10^{-4}$	-98.16
0.1M HCl + 0.075M Na_2MoO_4	-294	$3.55x10^{-6}$	-79.24

3.2.2 R_p vs. time measurements

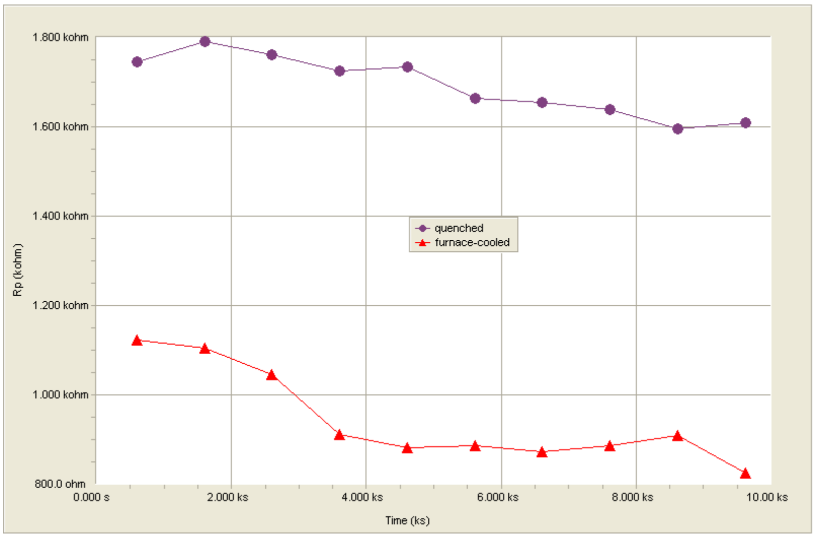

Figure 4. R_p *vs. t* of 304SS in 0.1M HCl at 50 ^{o}C.

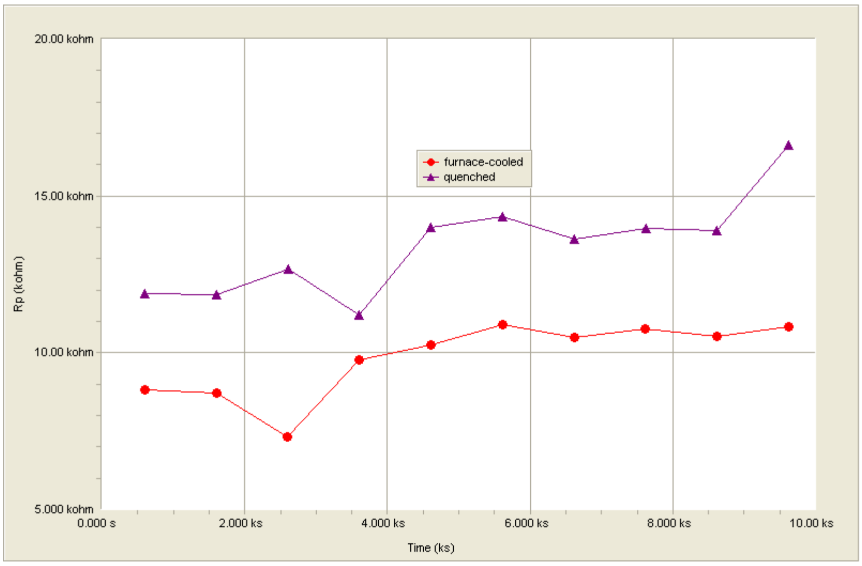

Figure 4b. *Rp vs. t* of 304SS in 0.1M HCl+0.001M Na_2MoO_4 at 50 ^{o}C.

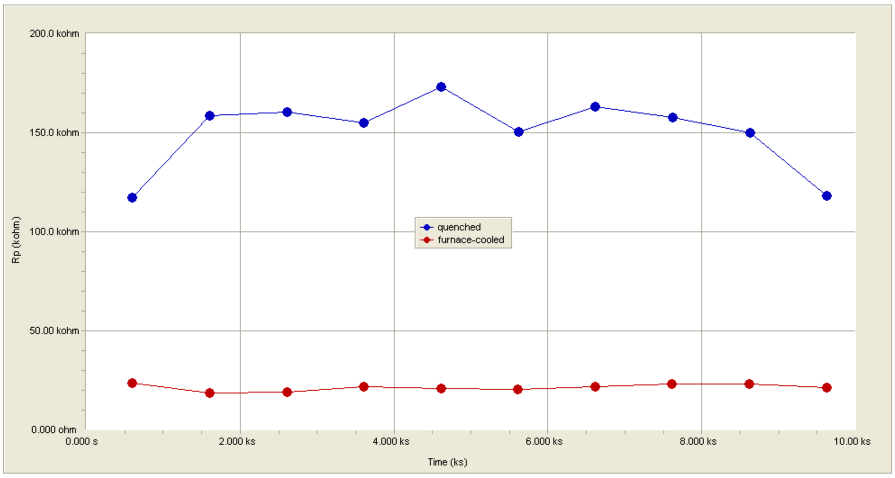

Figure 4c. *Rp vs. t* of 304SS in 0.1M HCl+0.075M Na_2MoO_4 at 50 ^oC.

Table 5. Polarization resistance data for 304SS tested in 0.1M HCl as a function of molybdate ions concentrations tested at 50 ^oC.

Solution	R_p (kΩ-cm^2, quenched, from Rp vs. t curves)	R_p (kΩ-cm^2, furnace-cooled, from Rp vs. t curves)	R_p (kΩ-cm^2, quenched, from cyclic polarization)	R_p (kΩ-cm^2, furnace-cooled, from cyclic polarization)
0.1M HCl	0.536	0.577	0.643	0.504
0.1M HCl + 0.001M Na_2MoO_4	4.24	2.83	0.377	0.313
0.1M HCl + 0.075M Na_2MoO_4	47.6	6.77	3.928	1.611

Figure 4 (a, b, and c) shows the R_p *vs. t* measurements for both steels in 0.1M HCl in the absence and presence of molybdate ions at different concentrations at 50 ^oC. The two steels show similar trends to testing at 22 ^oC. However, when both steels were tested under same conditions, the R_p values obtained at 50 ^oC (Table 5) are lower than that obtained at 22 ^oC (Table 3), which indicate the two steels are more susceptible to general corrosion at 50 ^oC.

From the results that summarized and discussed above, it is fair to conclude that molybdate ions are more effective in enhancing both general corrosion resistance (higher R_p values) and pitting corrosion resistance (higher $E_{pp.}$values); of both steels at 22 ^oC than 50 ^oC, with the non-sensitized steels showing better resistance to pitting corrosion at both temperatures.

In order to further investigate the effects of the addition of molybdate ions on both the general corrosion and the pitting corrosion, potentiostatic polarizations were performed at $E_{applied}$ = -250 mV (in the active potential range where the influence of molybdate ions, if any, should be more profound) and at $E_{applied}$ = +400 mV (in the pitting region). Tests were conducted in 0.1M HCl both in the absence and presence of molybdate ions addition for the non-sensitized steel at 22 ^oC.

Figure 5 shows current density vs. time for non-sensitized steel under $E_{app.}$ = -250 mV. The current switches from anodic to cathodic current with the addition of 0.01M Na_2MoO_4. While in the absence of molybdate the current density *vs.* time showed a typical behavior of a decreasing anodic current density over time before stabilizing, the addition of molybdate ions resulted in a sharp drop in the current density and a switch from anodic to cathodic behavior.

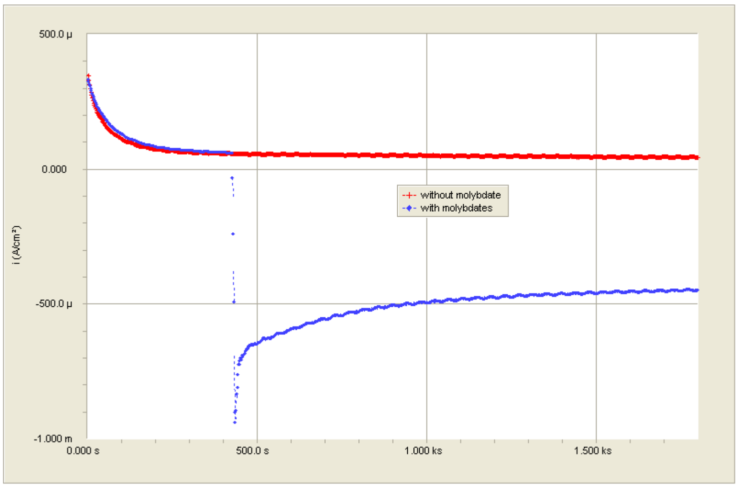

Figure 5. Potentiostatic polarization curves of non-sensitized 304SS in 0.1M HCl in the absence and presence of molybdate ions at $E_{applied}$ = -250 mV *vs.* Ag/AgCl, sat. $[MoO_4^{2-}]$= 0.01M.

The increase is sharp and accompanied by a spike as shown in the Figure 5. Such sudden drop and switch from anodic to cathodic currents can be attributed to film formation on the electrode surface and as a result making the film more passive or more protective shifting the potential in the noble direction. Such shifting of E_c to a value higher than $E_{applied.}$, resulted in making $E_{applied.}$ cathodic with respect to the E_c. One possible reaction is the reduction of Mo(VI) to Mo(IV) [12]:

$$MoO_4^{2-} + 4H^+ + 2e \rightarrow MoO_2 + 2H_2O$$

The fact that the shift in current density on the injection of molybdate ions is sharp and with a spike indicate a reduction reaction on the steel's surface. Similar behavior was reported by Iannuzzi and Frankel who investigated the effect of vanadates ions on the corrosion of aluminum alloy AA2024-T3 in NaCl solutions [18].

Figure 6 shows the poteniostatic polarization curves of non-sensitized 304SS tested in 0.1M HCl, both in the absence and presence of molybdate ions, at $E_{applied.}$= +400 mV, in the pitting region, where stable pits are expected to initiate and grow. It is clear from the current transient that in the absence of molybdates, pits formation and growth is dominant as evident from the increase in both the current densities and current transients over time, while pitting is significantly reduced as evident from the drop in current density and in the current fluctuation over time when 0.01M Na_2MoO_4 are added.

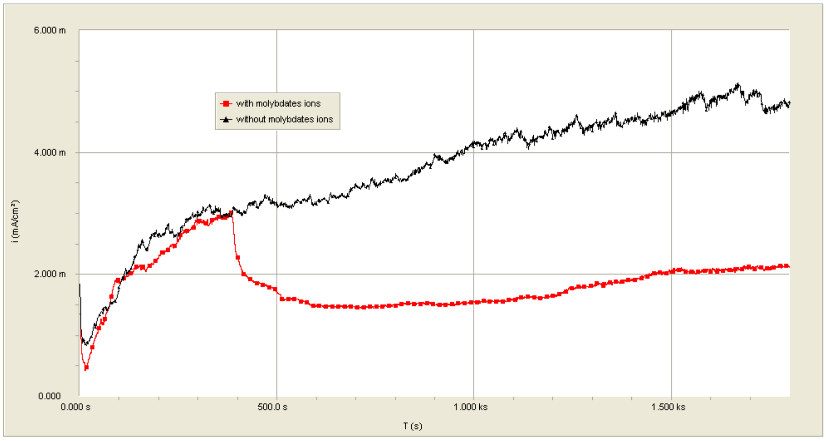

Figure 6. Potentiostatic polarization curves of non-sensitized 304SS in 0.1M HCl in the absence and presence of molybdate ions at $E_{applied}$ = +400 mV *vs.* (Ag/AgCl, sat). Note: molybdate ions were added at about the 400s mark, $[MoO_4^{2-}]$= 0.01M.

4. CONCLUSION

The following conclusions can be made:

1. The quenched (non-sensitized) steel shows better pitting corrosion resistance than the furnace-cooled (sensitized) steel when tested in 0.1M HCl at 22 °C and 50 °C.
2. Very low concentrations of molybdate ions have no effect or even detrimental effect on the corrosion resistance of both non-sensitized and sensitized 304SS in 0.1M HCl tested at 22 °C and 50 °C.
3. Resistance to general corrosion and pitting corrosion of both steels improves significantly in 0.1M HCl when relatively high concentrations of molybdate ions are used (0.075M).
4. Molybdate ions are more effective in increasing resistance to pitting at 22 °C than 50 °C for both steels.
5. Molybdate ions are more effective in enhancing pitting corrosion resistance in the non-sensitized steel than the sensitized steel at both 22 °C and 50 °C.
6. It is proposed that molybdate ions enhance resistance to pitting by lowering the current densities in the active and passive ranges to a critical values in which below these values, metastable pits, if exist, fail to grow into stable ones.

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