

Electrochemical Corrosion Behavior of C-250 Maraging Steel Grade in Sulfuric Acid Pickling Solution

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The effect of changing the time of immersion and temperature on the electrochemical corrosion behavior of C-250 maraging steel grade in 1.0 M H₂SO₄ pickling solution was reported. The study was carried out using potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) investigations. It was found that the increase of immersion time from 10 min to 1 h, 2 h, and 24 h increases the corrosion of the C-250 maraging steel in the acid solution through increasing the corrosion current and corrosion rate, while decreasing the corrosion resistance as was indicated by the polarization measurements. This effect was found to further increase with increasing the temperature of the sulfuric acid solution from 20° to 50 °C due to the increase of surface reactivity with temperature. The results obtained by EIS measurements confirmed those ones obtained by potentiodynamic polarization that C-250 maraging steel suffers sever uniform corrosion increases with elongating the immersion time and raising the solution temperature.

Keywords: Acid corrosion; Maraging steel; Electrochemical Impedance Spectroscopy; Polarization

1. INTRODUCTION

The ultra-high strength maraging steels have been developed mainly for many tooling applications including missile and rocket motor cases, aircraft, aerospace, wind tunnel models, landing gear components, high performance shafting, gears, fasteners, and nuclear and gas turbine applications [1-4]. Maraging alloy is a low carbon steel that classically contains about 18 wt% Ni and substantial amounts of Co and Mo, together with small additions of Ti [5]. The low carbon content makes maraging steels have good machinability [6]. However and depending on the demands dedicated by

the application, the composition of these steels can be modified [7]. Maraging steels are characterized by their higher modulus of elasticity, lower thermal expansion coefficient, high strength, moderate toughness, and good weldability as compared with conventional alloy steels [8-10]. Moreover, it has high thermal conductivity, which reduces surface temperature during thermal loading and lowers thermal stresses.

Maraging steels have been developed as alternative materials to conventional quenched and tempered steels for advanced technologies such as aerospace, nuclear and gas turbine applications. The ultra-high strength of maraging steels results from the precipitation of intermetallic compounds during the aging process; as example the strengthening of 18 wt.% Ni (18Ni) maraging steels results from the combined presence of Ni_3Ti and Fe_2Mo or Fe_7Mo_6 precipitates [8,11,12]. It has been reported [12] that the formation of Ni_3Ti takes place rapidly due to the fast diffusion of Ti atoms.

Several industries employ acid solutions for cleaning, pickling, descaling, and acidizing processes by which steels come in contact with acids. The corrosion of maraging steels in acidic solutions results mainly during the acid cleaning, where the formed scales and corrosion products on the surface cause a negative effect to the performance of the steel equipment. A search of the literature reveals only a few reports on the corrosion studies of 18 Ni 250 grade maraging steel, which is entirely in martensitic phase. Bellanger and Rameau [13] have studied the effect of slightly acidic pH in the absence and presence of chloride ions in radioactive water on the corrosion of maraging steel and have reported that corrosion behavior of maraging steel at the corrosion potential depends on pH and intermediates remaining on the maraging steel surface in the active region favoring the passivity. The effect of carbonate ions in a slightly alkaline medium on the corrosion of maraging steel was studied by G. Bellanger [14]. Maraging steels were found to be less susceptible to hydrogen embrittlement than common high-strength steels owing to significantly low diffusion of hydrogen in them [15]. Poornima et al. [4] have studied the corrosion behavior of 18 Ni 250 grade maraging steel in a phosphoric acid medium and reported that the corrosion rate of the annealed sample is less than that of the aged sample.

In the present work, the corrosion behavior of C-250 grade maraging steel after its immersion for 10 min, 1 h, 2 h, and 24 h in 1.0 M H_2SO_4 pickling solution was investigated. The work was also extended to report the corrosion behavior of this steel at temperatures of 20 °C, 30 °C, 40 °C, and 50 °C for the acid solution. To achieve our objectives we employed potentiodynamic polarization and electrochemical impedance spectroscopy techniques.

2. EXPERIMENTAL DETAILS

2.1. Materials and Chemicals

The material employed in this study was a maraging steel C-250 grade (18Ni250) coupon with the chemical compositions of 0.03 wt. % C, 7.50 wt. % Co, 4.80 wt. % Mo, 18.0 wt. % Ni, 0.4 wt. % Ti, 0.4 wt. % Al, and the balance was Fe. For the electrochemical measurements, the steel rods with dimensions of 1×1×0.5 cm were cut from the delivered steel sheet. A stock solution of 1.0 M sulfuric

acid was prepared from the concentrated acid solution (96%) that was purchased from Merck and was used as received.

2.2. Electrochemical cell

A conventional three-electrode cell accommodates for 200 mL solution was employed in this study to perform the electrochemical measurements. The C-250 maraging steel, Ag/AgCl (in saturated KCl), and a platinum foil were used as the working, reference, and counter electrode, respectively. One face of the steel working electrode was drilled and welded with copper wire and was then embedded into two-component epoxy resin and mounted in a glass holder. Where, the other face was left to be polished and exposed to the acid solution. Prior to each experimental measurement, the exposed surface of the steel electrode (of area 1.0 cm^2) was wet ground with silicon carbide abrasive papers up to 1000 grit, rinsed with ethanol and air dried.

2.3. Electrochemical measurement techniques

Electrochemical experiments were performed using an Autolab Potentiostat (PGSTAT20, computer controlled) operated by the general purpose electrochemical software (GPES) version 4.9. The potentiodynamic polarization curves were obtained by scanning the potential in the forward direction against the Ag/AgCl reference electrode and at a scan rate of 0.001 V/s . The electrochemical impedance spectroscopy (EIS) tests were performed at corrosion potentials over a frequency range of 100 kHz to 100 mHz , with an ac wave of $\pm 5\text{ mV}$ peak-to-peak overlaid on a dc bias potential, and the impedance data were collected using Powersine software at a rate of 10 points per decade change in frequency. All measurements were recorded at the free corrosion potential that was obtained after immersing on a new steel surface of the working electrode after its immersion in a fresh solution of $1.0\text{ M H}_2\text{SO}_4$ for 10 min, 1 h, 2 h, and 24 h. Every experiment was repeated for at least three times to ensure the reliability and reproducibility of the obtained data.

3. RESULTS AND DISCUSSION

3.1. Potentiodynamic polarization measurements

The Potentiodynamic polarization curves obtained for C-250 maraging steel electrodes after their immersion in $1.0\text{ M H}_2\text{SO}_4$ solutions for (1) 10 min, (2) 1 h, (3) 4 h and (4) 24 h at room temperature are shown in Fig. 1. The values of the cathodic (β_c) and anodic (β_a) Tafel slopes, corrosion current density (j_{Corr}), corrosion potential (E_{Corr}), and polarization resistance (R_p) obtained from the curves shown in Fig. 1 are listed in Table 1. The values of β_c and β_a were determined according to our previous study [16,17]. The values of j_{Corr} and E_{Corr} were obtained from the extrapolation of anodic and cathodic Tafel lines located next to the linearized current regions. From the slope analysis of the linear polarization curves in the vicinity of E_{Corr} , the values of R_p in the acid

solution were obtained [18-20]. It has been reported [9,10,18] that the cathodic reaction of steel in acid solutions is the evolution of hydrogen;



The electrons consumed by this reaction are produced due to the anodic dissolution of iron, which is the base metal in the maraging steel alloy according to the following reaction [9,10];



The produced ferrous cations, Fe^{2+} , further oxidized to ferric cations, Fe^{3+} , under the influence of the aggressiveness action of the sulfuric acid and with increasing the applied potential in the less negative direction as follows;

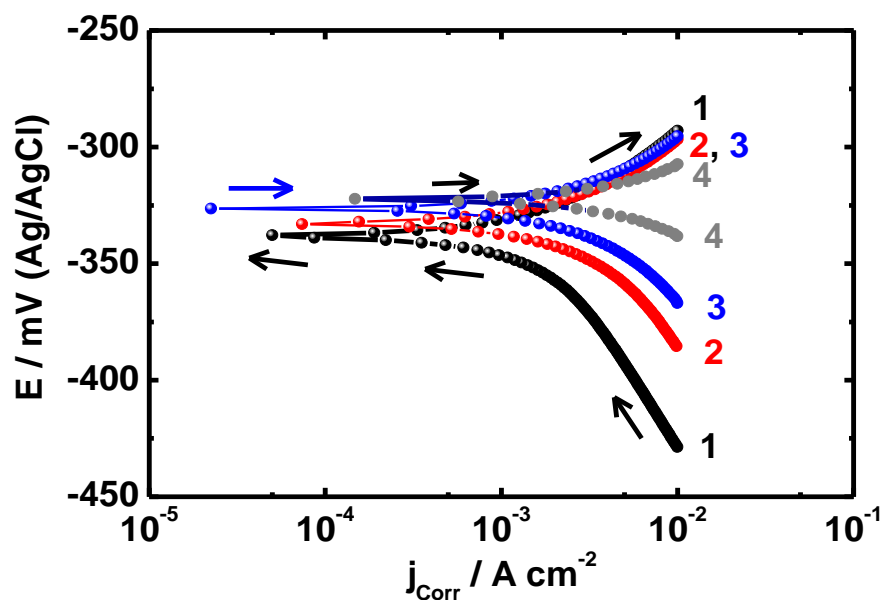


Figure 1. Potentiodynamic polarization curves obtained for C-250 maraging steel electrodes after their immersion in 1.0 M H_2SO_4 solutions for (1) 10 min, (2) 1 h, (3) 4 h and (4) 24 h at room temperature.

Table 1. Potentiodynamic polarization parameters obtained for C-250 maraging steel after its immersion in 1.0 M H_2SO_4 solution for different immersions at room temperature.

Time	Corrosion parameters				
	β_c (mV/dec ⁻¹)	β_a (mV/dec ⁻¹)	E_{Corr} (mV vs. Ag/AgCl)	j_{Corr} ($\mu\text{A cm}^{-2}$)	R_p ($\Omega \text{ cm}^2$)
10 min	25.74	38.07	-338	620.35	10.75
1.0 h	19.61	29.83	-333	703.24	6.39
2.0 h	18.19	26.04	-326	926.98	5.02
24 h	14.36	20.32	-295	1188.9	3.07

It is clearly seen from Fig. 1 that the increase of immersion time increases the anodic, cathodic and corrosion currents as well as shifts the corrosion potential toward the less negative values. The values of the parameters listed in Table 1 confirmed that the corrosion of C-250 maraging steel increases with increasing the time of immersion. Where, the value of j_{Corr} , which is directly proportional with the corrosion rate, significantly increased by elongating the immersion time of the steel in the acid solution before measurement from 10 min to 1 h and 2 h and further to 24 h. This effect was found to decrease the values of R_p and thus increase the dissolution of the maraging steel. This is due to the increased corrosiveness attack of the sulfuric acid towards the steel, which makes its surface always fresh and active and thus corrodes easily and rapidly.

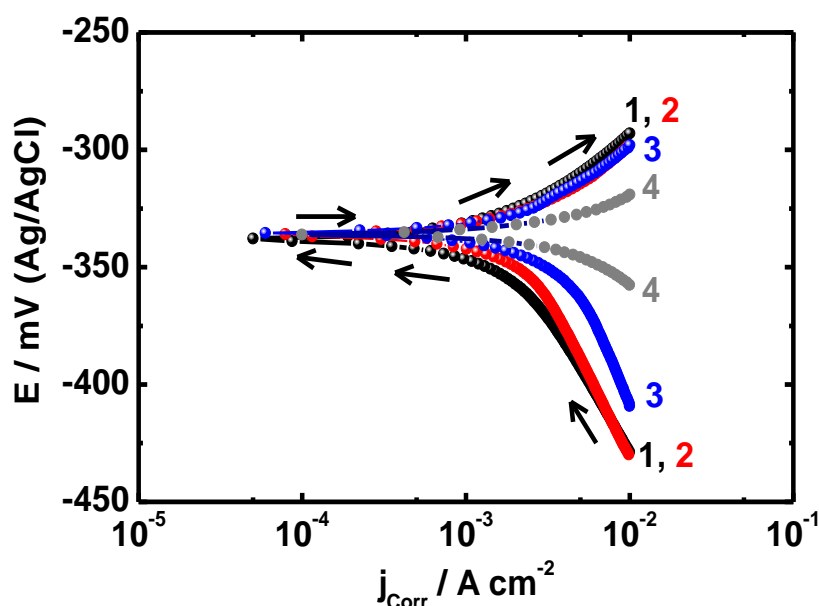


Figure 2. Potentiodynamic polarization curves obtained for C-250 maraging steel electrodes after their immersion for 10 min in 1.0 M H_2SO_4 solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C and (4) 50 °C, respectively.

Table 2. Potentiodynamic polarization parameters obtained for C-250 maraging steel after its immersion in 1.0 M H_2SO_4 solution for 10 min at different solution temperatures.

Temperature	Corrosion parameters				
	β_c (mV/dec ⁻¹)	β_a (mV/dec ⁻¹)	E_{Corr} (mV vs. Ag/AgCl)	j_{Corr} ($\mu\text{A cm}^{-2}$)	R_p (Ωcm^2)
20 °C	25.74	38.07	-338	620.35	10.75
30 °C	21.65	26.63	-334	885.19	5.85
40 °C	12.92	13.59	-332	930.35	2.91
50 °C	9.37	8.71	-330	988.15	2.10

In order to report the effect of changing the temperature of the sulfuric acid on the corrosion behavior of the C-250 maraging steel, potentiodynamic polarization measurements were carried out. Fig. 2 shows the potentiodynamic polarization curves obtained for C-250 maraging steel electrodes after their immersion for 10 min in 1.0 M H_2SO_4 solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C and (4) 50 °C, respectively. The corrosion parameters that were calculated from the obtained polarization curves are listed in Table 2. It can be seen from Fig. 2 and Table 2 that the increase of temperature increases the corrosion of the C-250 maraging steel through increasing the anodic and cathodic currents. This effect also highly increased the values of j_{Corr} and significantly decreased R_p values and shifted E_{Corr} towards the less negative values. Moreover, the little change of β_c and β_a values listed in Table 2 with increasing temperature of the acid solution proves that there was a little change in the mechanism of corrosion of the steel with increasing temperature. This is because the increase of temperature increases the reactivity of the surface of the steel and thus increases its dissolution under the effect of the corrosiveness action of the acid solution and the increase of the applied potential in the less negative direction.

3.3. Electrochemical impedance spectroscopy (EIS) measurements

The EIS Nyquist plots obtained for C-250 maraging steel electrodes after their immersion in 1.0 M H_2SO_4 solutions for (1) 10 min, (2) 1.0 h, (3) 4.0 h and (4) 24 h, respectively at room temperature are shown in Fig. 3. The obtained spectra showed one single distorted semicircle for all immersion times. At this condition, the deviation from the ideal semicircle is generally attributed to the frequency dispersion [21], as well as to the inhomogeneities of the surface and mass transport resistant [22]. It is obvious that the size of the diameter of the semicircle decreased with increasing time. The capacitance loop intersects the real axis at higher and lower frequencies.

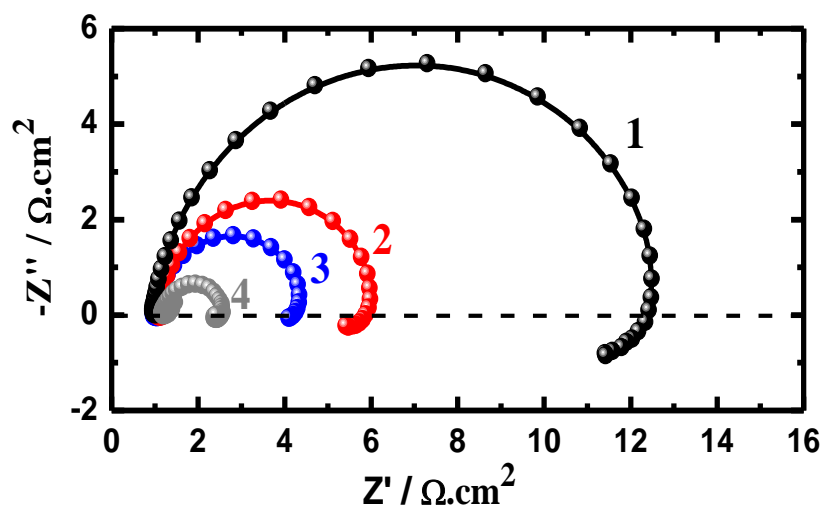


Figure 3. Typical Nyquist plots obtained for C-250 maraging steel electrodes after their immersion in 1.0 M H_2SO_4 solutions for (1) 10 min, (2) 1 h, (3) 4 h and (4) 24 h at room temperature.

Impedance behaviour can be well explained by pure electric models that could verify and enable to calculate numerical values corresponding to the physical and chemical properties of electrochemical system under examination [23]. The simple equivalent circuit that fit to many electrochemical systems consisting of a parallel combination of a double layer capacitance (C_{dl}) and the charge transfer resistance (R_p) corresponding to the corrosion reaction at metal/electrolyte interface and the solution resistance (R_s) between the working and reference electrode [24, 25]. To reduce the effects due to surface irregularities of metal, constant phase element (CPE) is introduced into the circuit instead of a pure double layer capacitance which gives more accurate fit [26]. The Nyquist plots represented in Fig. 3 were analyzed by best fitting to the equivalent circuit model shown in Fig. 4 and the values of the symbols of this circuit are listed in Table 3. The parameters of our circuit can be thus defined according to usual convention, as follows; R_s represents the solution resistance, R_p is the polarization resistance and can also be defined as the charge transfer resistance, and Q is the constant phase elements (CPEs). The value of R_p is a measure of electron transfer across the exposed area of the metal surface and it is inversely proportional to rate of corrosion [27]. At high frequency end the intercept corresponds to the solution resistance (R_s) and at lower frequency end corresponds to the sum of R_s and R_p .

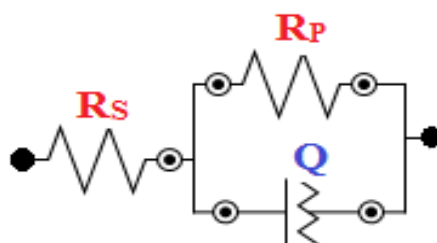


Figure 4. The equivalent circuit model used to fit the EIS experimental data shown in Fig. 3.

Table 3. EIS parameters obtained for maraging steel electrodes after their immersion in 1.0 M H_2SO_4 solutions 10 min, 1.0 h, 2.0 h and 24 h at room temperature.

Time	EIS parameters			
	$R_s (\Omega \text{ cm}^2)$	CPE (mMho)	n	$R_p (\Omega \text{ cm}^2)$
10 min	1.00	0.99	0.94	12.0
1.0 h	1.10	4.25	0.95	5.00
2.0 h	0.98	13.51	0.96	3.60
24 h	1.51	230.00	0.97	0.70

The Q (CPEs) represents a double layer capacitor (C_{dl}) because the value of its component, n , was very close to unity, which was expected to cover up the charged steel surfaces [9,10,18]. The CPE

has been reported [24] to vary depending on the value of its component n , where it may be resistance or capacitance or inductance. The impedance of CPE in the case of maraging steel in sulfuric acid solutions can be expressed as following;

$$Z_{CPE} = 1 / Y_0 (j\omega)^n \quad (4)$$

Where, Y_0 is the magnitude of CPE, n is the exponent (phase shift), ω is the angular frequency and j is the imaginary unit. It is clearly seen from Fig. 3 and Table 3 that the increase of immersion time decreases the diameter of the semicircle and the values of R_s and R_p as well as increases the values of CPEs. The decrease of the values of R_s and R_p indicates that the increase of immersion time increases the corrosion of maraging steel. This is because of the continuous dissolution of the steel under the aggressiveness action of the corrosive acid solution, which does not allow the formation of oxide films or corrosion product layers on the surface.

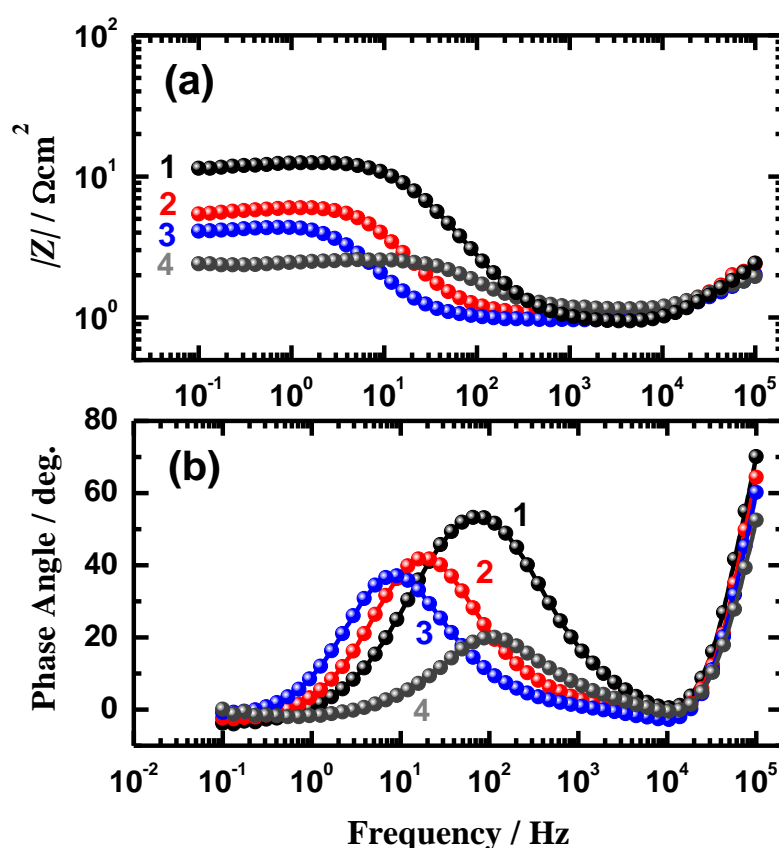


Figure 5. (a) Bode impedance and (b) Bode phase angle plots obtained for C-250 maraging steel electrodes after their immersion in 1.0 M H₂SO₄ solutions for (1) 10 min, (2) 1 h, (3) 4 h and (4) 24 h at room temperature.

The Bode impedance of the interface (a) and the Bode phase angle (b) plots for the maraging steel after its immersion for (1) 10 min, (2) 1.0 h, (3) 2.0 h, and (4) 24 h in 1.0 M H₂SO₄ solutions are shown respectively in Fig. 5. It seen from the figure that the increase of immersion time decreased the absolute impedance values across the whole range of the applied frequency as well as decreased the

maximum degree of the phase angle. It has been reported [28] that the decrease of the impedance values and the decrease of the maximum degree of the phase angle at the low frequency region result from the sever dissolution of the material's surface. This means that the increase of the immersion time from 10 min to 24 h decreases the corrosion resistance of the steel in H_2SO_4 solution through increasing its surface dissolution.

The effect of increasing temperature of the sulfuric acid solution on the corrosion behavior of C-250 maraging steel was also investigated using EIS technique. Typical Nyquist plots obtained for C-250 maraging steel electrodes after their immersion for 10 min in 1.0 M H_2SO_4 solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C and (4) 50 °C, respectively are shown in Fig. 6. The Bode impedance ($|Z|$) and Bode phase angle (ϕ) for the maraging steel after its immersion for 10 min in the acid solution at the different temperatures are also shown in Fig. 7(a) and Fig. 7(b), respectively. These EIS data were also best fitted to the equivalent circuit model that is shown in Fig. 4. The values of the parameters obtained from the fitted data for the maraging steel in sulfuric acid solutions after different temperatures are listed in Table 4.

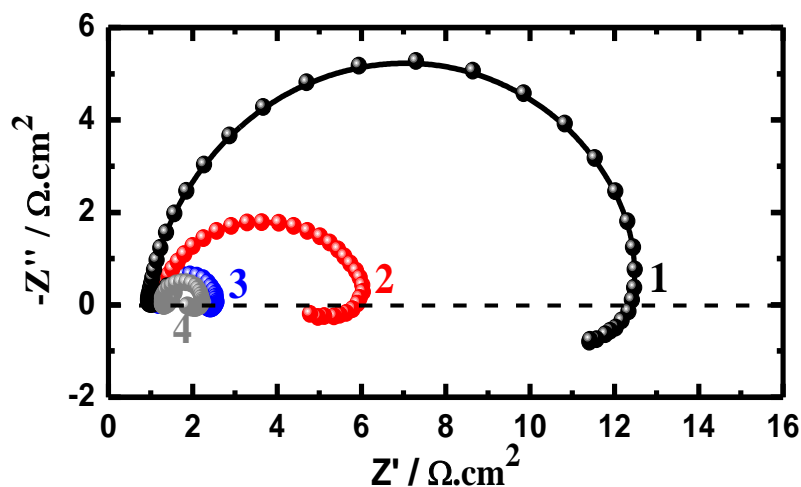


Figure 6. Typical Nyquist plots obtained for C-250 maraging steel electrodes after their immersion for 10 min in 1.0 M H_2SO_4 solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C and (4) 50 °C, respectively.

Table 4. EIS parameters obtained for C-250 maraging steel after its immersion in 1.0 M H_2SO_4 solution for 10 min at different solution temperatures.

Temperature	EIS parameters			
	R_s ($\Omega \text{ cm}^2$)	CPE (mMho)	n	R_p ($\Omega \text{ cm}^2$)
20 °C	1.00	0.99	0.94	12.0
30 °C	1.15	1.84	0.83	5.00
40 °C	1.23	1.97	0.85	2.50
50 °C	1.14	2.43	0.91	1.40

It is seen from Fig. 6 that the maraging steel recorded only one semicircle, whose diameter decreases with increasing the temperature of the acid solution Table 4 also indicated that the increase of temperature increased the values of R_s and R_p , while decreased the value of CPE. The value of CPE with its component, n , close to unity at all temperatures represents a double layer capacitor. The Bode impedance of the interface, Fig. 7(a) decreases with temperature, particularly at the low frequency values. The increase of temperature also led to decreasing the maximum phase angle as shown in Fig. 7(b). The EIS data thus confirm that the increase of the acid solution temperature from 20 °C to 50 °C decreases the resistance of the C-250 maraging steel against corrosion through increasing the dissolution of its surface. This is because the increase of temperature increases the severity of the reaction between the acid solution and the steel surface, which gets activated and susceptible to severe dissolution by the increase of the temperature. The EIS results also confirm the data obtained by polarization measurements that the increase of immersion time from 10 min to 24 as well as the increase of solution temperature increase the corrosion of the investigated steel grade in 1.0 M H_2SO_4 solutions. The corrosion resistance calculated from EIS results shows the same trend as those obtained from polarization measurements. The difference in corrosion resistance of the two methods may be due to the different surface status of the electrode in two measurements [29].

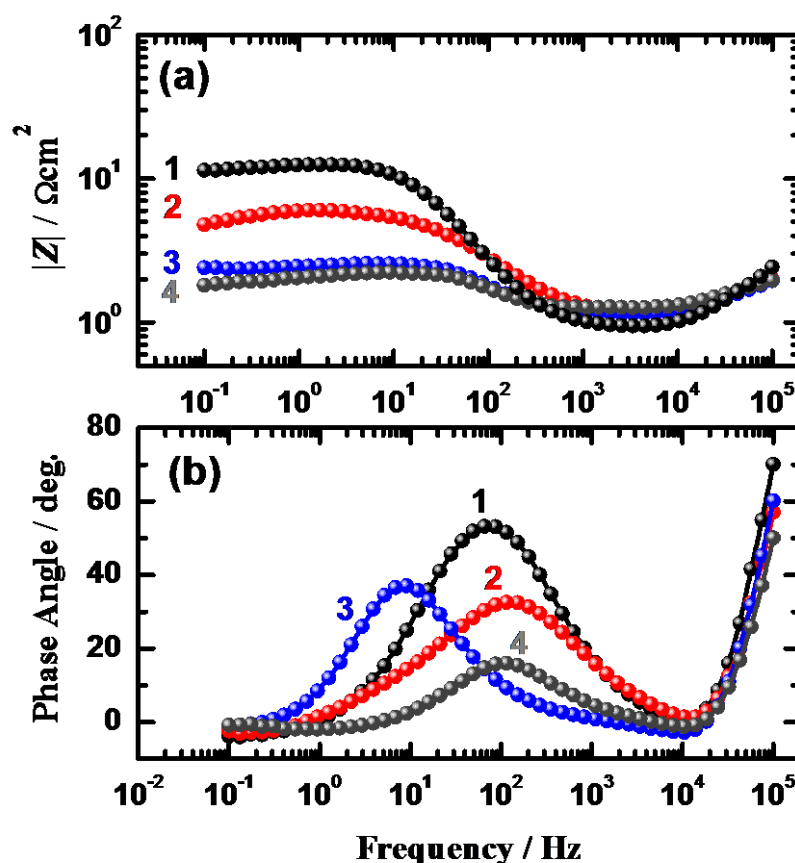


Figure 7. Bode impedance (a) and Bode phase angle (b) plots obtained for C-250 maraging steel electrodes after their immersion for 10 min in 1.0 M H_2SO_4 solutions at (1) 20 °C, (2) 30 °C, (3) 40 °C and (4) 50 °C, respectively.

4. CONCLUSIONS

The electrochemical behavior of the conventional C-250 maraging steel grade was investigated after its immersion for different exposure periods of time, namely 10 min, 60 min, 120 min and 24 h in 1.0 M H₂SO₄ pickling solutions. The work was extended to report the effect of increasing the acid solution temperature between 20 °C and 50 °C the anodic dissolution of the steel. Potentiodynamic polarization and electrochemical impedance measurements showed that the increase of immersion time increases the corrosion of steel in the acid solution. The increased time of contact increases the attack of the acid solution on steel and leads to the continuous dissolution of its surface. This was indicated by the increase of the corrosion current and the decrease of corrosion resistance for steel with time. It was also found that the increase of temperature of the sulfuric acid solution from lower temperatures to 50 °C increases the corrosion of C-250 maraging steel through increasing the reactivity of its surface that in turn creates more active centers. All results were in good agreement with each other showed clearly that the increase of immersion time as well as the increase of temperature increase the corrosion of of C-250 maraging steel in 1.0 M H₂SO₄ pickling solutions.

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