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Asiful Hossain Seikh Mohammad Rezaul Karim

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Corrosion inhibitory effect of thiourea on recrystallized E-34 microalloyed steels in acidic media

Asiful Hossain Sheikh and Mohammad Rezaul Karim

Centre of Excellence for Research in Engineering Materials, Advanced Manufacturing Institutes, King Saud University, Riyadh, Saudi Arabia

Abstract

Purpose – The purpose of this paper is to evaluate the effect of thiourea (TU) on corrosion resistance property for rolled and recrystallized E-34 microalloyed steels by using electrochemical polarization techniques.

Design/methodology/approach – To perceive the effect of TU on the corrosion inhibition efficiency, various concentrations of TU (from 1×10^{-4} to 1×10^{-2} M) and different temperatures (20, 30 and 400°C) in 1N sulfuric acid are used.

Findings – It is found that TU has significant inhibition effect on corrosion process. Moreover, it reveals that both the inhibitor concentrations and temperatures have a strong influence on the corrosion prevention efficiency of inhibitor. Thermodynamics studies confirm that the inhibitor adsorption follows the Langmuir adsorption isotherm model.

Originality/value – To the best of the authors' knowledge, it is the first work that has been disclosed the corrosion inhibitory effect of TU for recrystallized E-34 microalloyed steels in acidic media.

Keywords Corrosion, Inhibitors, Electrochemistry

Paper type Research paper

1. Introduction

Microalloyed steel finds wide application in the car bodies and other engineered parts because of its high strength and ductility. Precise grain-sized microstructure is the underlying reason for these properties. Microalloyed steel has a ferritic matrix similar to that of "mild" steel but has a fine-grained structure. Alloying additions of niobium and titanium at the micro level bring this refinement in the microstructure. The corrosion behavior of the steel and the effect of microstructure on such behavior is still a field that is open for investigation to correlate the metallurgical condition with its corrosion behavior. The corrosion of steel is considered to be a major industrial problem that costs hundreds of billions of dollars (Oluwad and Agbaje, 2007). The corrosion of structural elements is a major issue for the chemical processing industry, due to the service environment. Numerous industries use acid solutions for cleaning, pickling, descaling and acidizing procedures, during which steels are exposed to acidic conditions. Several methods have been developed for preventing or reducing corrosion, which include metal coating, inorganic coating, painting and the use of chemical inhibitors. Corrosion inhibitors are needed in the oil, fertilizer, metallurgical and other industries, where contact between

metal and the aggressive medium is necessary (Eddy *et al.*, 2009a, 2009b). The use of inhibitors in acid solutions is one of the most practical methods for protecting metals against corrosion (Franklin *et al.*, 2010; Jacob and Parameswaran, 2010). Among alternative corrosion inhibitors, organic compounds containing hetero atoms such as N, O, S and multiple bonds in their molecules have been proven as effective compounds that can reduce the dissolution rate of metals and alloys in acid media (Krim *et al.*, 2008; Quraishi and Shukla, 2009; Satpati and Ravindran, 2008). It has been reported that organic compounds containing both nitrogen and sulfur have excellent corrosion inhibitory characteristics, compared with other compounds that contain only nitrogen or sulfur (Agrawal *et al.*, 2007; Amin *et al.*, 2007; Ashassi-Sorkhabi *et al.*, 2007). Thiourea (TU) has been used as a corrosion inhibitor for plain carbon (mild) steel in acidic solutions (Agrawal and Nambodhiri, 1990; Ahmed and Abdel-Hakam, 1989). It has been reported that without changing other microconstituents, grain refinement of ferritic microalloyed steels can be achieved by repeated quenching at a temperature just below the lower critical temperature, i.e. in the ferritic region (Song *et al.*, 2006). However, less attention has been paid to the combined effect of TU inhibition and repeated recrystallization of microalloyed steels under heat

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treatment conditions. Therefore, in this study, the effects of TU inhibition and repeated recrystallization under heat treatment conditions upon the corrosion resistance properties of E-34 microalloyed steel were investigated to evaluate the process and determine if the metallurgical condition had any effect on the corrosion response.

2. Experimental details

2.1 Materials preparation

The material used for this study is commonly known as Nb-bearing commercial microalloyed steel of E-34 grade with chemical composition as follows: C = 0.12 per cent, Mn = 1.0 per cent, S = 0.025 per cent, P = 0.025 per cent, Si = 10 per cent, Al = 0.02 per cent, Nb = 0.08 per cent and iron (balance). The steel sheet was mechanically press-cut to form coupons with dimensions of $10 \times 10 \times 5$ cm. The specimens were repeatedly heat treated to 600°C in a muffle furnace coupled with a proportional temperature controller with an accuracy level of $\pm 20^\circ\text{C}$ and then quenched in ice water. For the corrosion studies, an insulated copper wire was secured to one of the surfaces of each steel specimen with solder at a low temperature. The specimens were mounted in epoxy resin such that only the opposite flat surface to that which the copper wire was connected would be contacted with the corrosive solution. The surface of the mounted specimens, as received (designated later as AR), after first quench (Q1), after second quench (Q2) and after third quench (Q3) were polished as per standard metallographic practice to obtain a mirror finish, washed with double distilled water, degreased with acetone and dried before immersing in the corrosion medium.

2.2 Solution preparation

Standard 1N sulfuric acid solution was prepared by diluting analytical grade 98 per cent sulfuric acid using double distilled water. All experiments were carried out using a calibrated thermostat at temperatures of 20, 30 and 40°C (± 0.5). The inhibitive action of TU on the corrosion of microalloyed steels in 1N H_2SO_4 solution was studied by introducing different concentrations (from 1×10^{-4} to 1×10^{-2} M) of the inhibitor into the solution at the three test temperatures.

2.3 Experimental procedure

A conventional three-electrode assembly with microalloyed steel strips as the working electrode, a saturated calomel electrode (SCE) as the reference electrode and a graphite rod as the counter electrode were used for corrosion measurements. The electrochemical cell was cleaned and then washed with distilled water; 500 mL of the electrolyte solution was used in the cell for each individual run for the polarization measurements. Polarization of the working electrode, whether anode or cathode, was recorded in volts with reference to SCE for various values of current density in the absence or presence of various concentrations of inhibitor with three different temperatures (20, 30 and 40°C). For each run, the specimen surface was ground and freshly polished, washed, degreased in ethanol and dried in warm air. The freshly polished electrodes (specimens) were placed into the electrochemical cell and pre-exposed to the test solution to attain a steady state at zero current potential.

3. Results

The linear polarization behavior for the as-received and heat-treated microalloyed E-34 steels at three different temperature in 1N H_2SO_4 solution without and with the addition of TU was recorded over a potential range between -0.76 and -0.55 V versus SCE. The best straight line through the linear polarization points was calculated, and its intersection with the zero current horizontal provided the accurate E_{corr} values, as shown in Figure 1. The results of all specimens (as received and repeatedly quenched) without and with the addition of TU at three different temperatures were confirmed via same techniques and are shown in Figures 2–4.

The galvanostatic anodic and cathodic polarization curves were drawn from the current (i in mA) and potential (E in volts) values derived from the polarization experiments, the current density was calculated for all specimens using the area exposed to the electrolyte. Anodic and cathodic polarization curves (E vs. $\log I$) were plotted, and the best regression line through the Tafel region was plotted and extrapolated. Linear regression equations for the straight lines were obtained. It is clear from the Figure 5 that the best straight line through the linear polarization points was drawn, and the values of corrosion current (I_{corr}) and corrosion potential (E_{corr}) for the as-received E-34 steel were obtained from the extrapolation of anodic (β_a) and cathodic (β_c) Tafel lines located next to the linearized current regions. The values of β_a and β_c (not reported) were calculated from the straight line in the anodic and cathodic branches, respectively. It was found that the values of β_a and β_c were close to each other but with opposite sign; that is, β_a was positive and β_c was negative. Polarization diagrams for all specimens (as received and repeatedly quenched), without and with the addition of TU, were obtained (several examples are shown in Figures 6–8). The calculated E_{corr} and I_{corr} values from the polarization diagrams for all of the specimens are given in Tables I and II.

4. Discussions

4.1 Effect of the inhibitor concentration and temperature

From the polarization diagrams (Figures 6–8), it is evident that, at every temperature, all the polarization curves shifted toward lower values of I_{corr} in proportion to the

Figure 1 Linear polarization of E-34 microalloyed steels as-received samples (using without inhibitor)

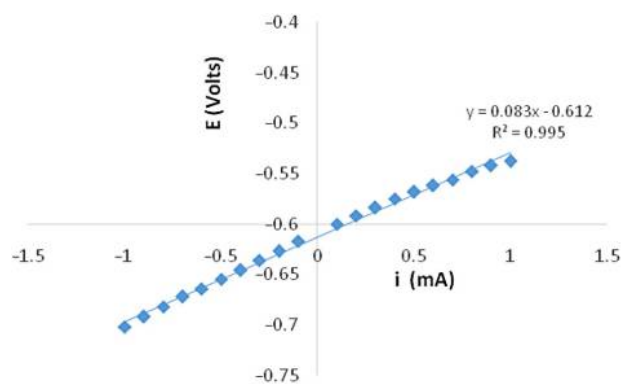


Figure 2 Linear polarization of E-34 as-received samples at 20°C

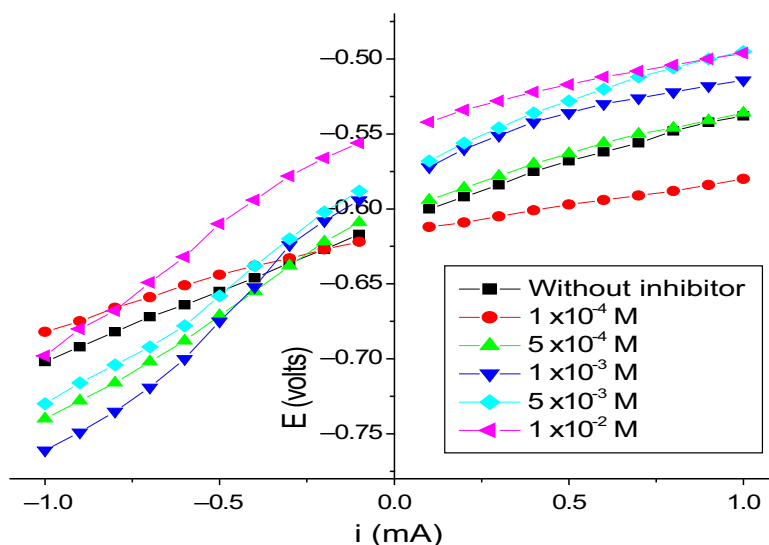


Figure 3 Linear polarization of E-34 samples after second quench at 30°C

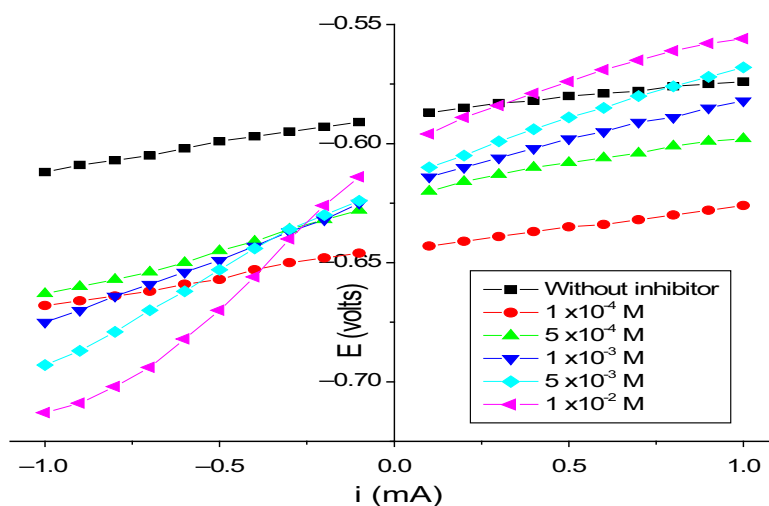


Figure 4 Linear polarization of E-34 samples after third quench at 40°C

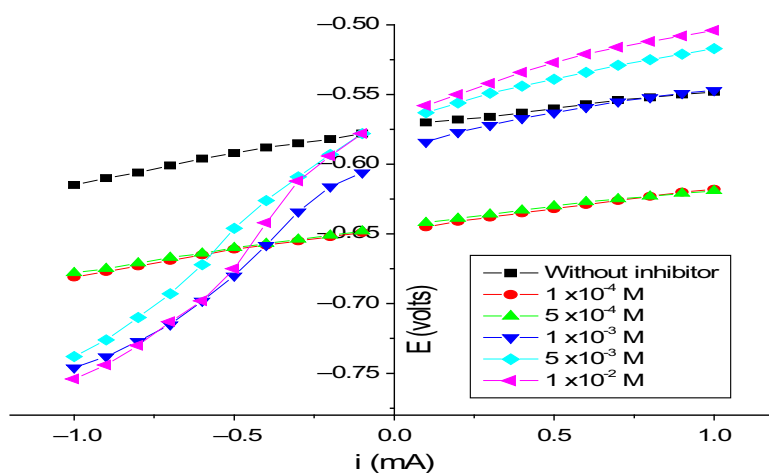
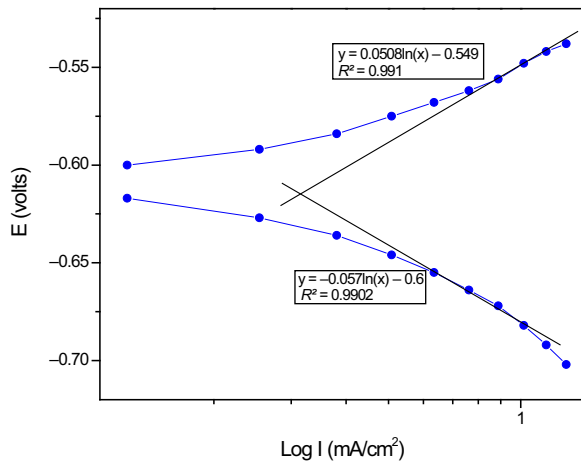
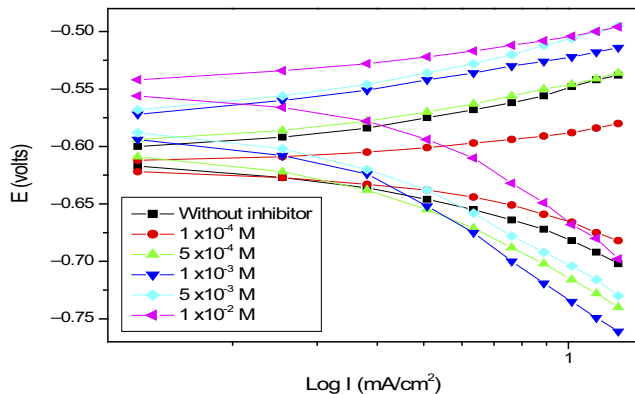
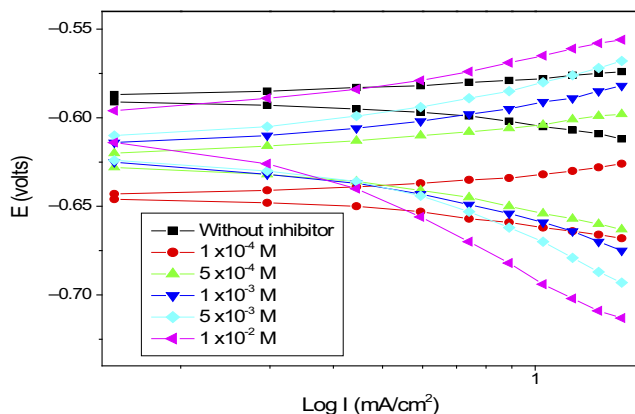
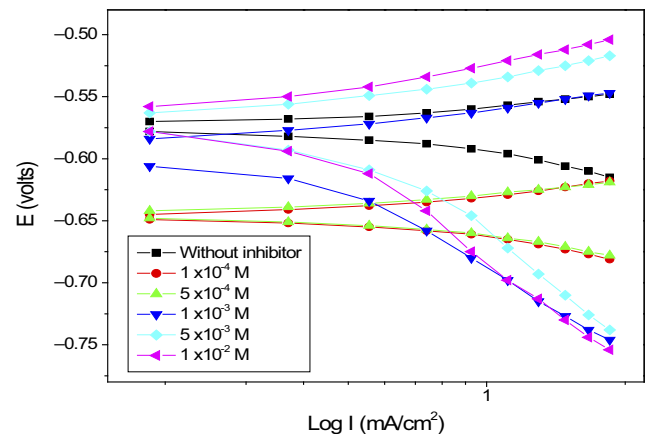


Figure 5 Polarization diagram of E-34 as-received samples (using without inhibitor)**Figure 6** Polarization diagram of E-34 as received samples at 20°C**Figure 7** Polarization diagram of E-34 samples after second quench at 30°C

inhibitor added. Also, E_{corr} values of the E-34 steel in inhibitor-containing solution shifted toward more noble values with increased TU inhibitor concentration. The addition of TU did not affect the cathodic Tafel region, which was assumed to be an effect of extrapolating the cathodic Tafel region. At higher temperatures and TU concentrations, the limiting current density region

Figure 8 Polarization diagram of E-34 samples after third quench at 40°C**Table I** E_{corr} values of E-34 microalloyed steels

Concentrations (M)	Temperature (°C)		
	20	30	40
As received			
Without inhibitor	-0.608	-0.569	-0.598
1×10^{-4}	-0.616	-0.617	-0.646
5×10^{-4}	-0.600	-0.605	-0.636
1×10^{-3}	-0.582	-0.582	-0.621
5×10^{-3}	-0.576	-0.578	-0.597
1×10^{-2}	-0.548	-0.576	-0.590
After first quench			
Without inhibitor	-0.583	-0.577	-0.599
1×10^{-4}	-0.618	-0.598	-0.64
5×10^{-4}	-0.584	-0.572	-0.624
1×10^{-3}	-0.556	-0.555	-0.602
5×10^{-3}	-0.544	-0.552	-0.587
1×10^{-2}	-0.538	-0.545	-0.585
After second quench			
Without inhibitor	-0.582	-0.589	-0.578
1×10^{-4}	-0.626	-0.645	-0.646
5×10^{-4}	-0.604	-0.624	-0.626
1×10^{-3}	-0.574	-0.620	-0.596
5×10^{-3}	-0.558	-0.615	-0.590
1×10^{-2}	-0.541	-0.605	-0.574
After third quench			
Without inhibitor	-0.578	-0.584	-0.574
1×10^{-4}	-0.614	-0.640	-0.647
5×10^{-4}	-0.596	-0.625	-0.645
1×10^{-3}	-0.568	-0.580	-0.595
5×10^{-3}	-0.554	-0.542	-0.570
1×10^{-2}	-0.526	-0.538	-0.568

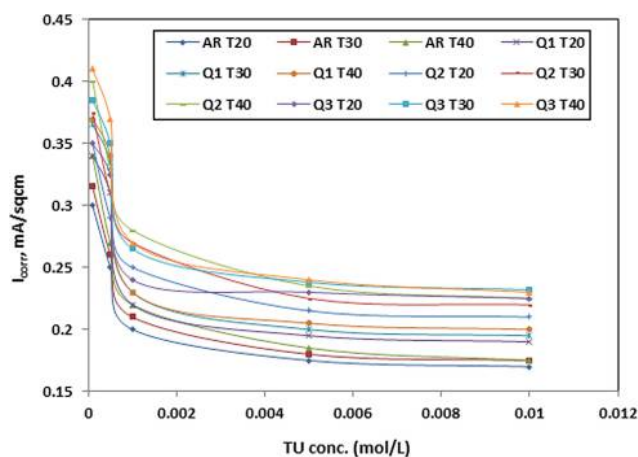
overshadowed the Tafel region, due to increase of TU adsorption that resulted from decreased hydrogen ion mobility to the cathode. The cathodic reaction, therefore, was significantly retarded, and the cathodic Tafel slope could not be drawn. The anodic Tafel slope, however, was

Table II I_{corr} values of E-34 microalloyed steels

Concentrations (M)	Temperature ($^{\circ}\text{C}$)		
	20	30	40
As received			
Without inhibitor	0.32	0.34	0.36
1×10^{-4}	0.30	0.315	0.34
5×10^{-4}	0.25	0.26	0.27
1×10^{-3}	0.20	0.21	0.22
5×10^{-3}	0.175	0.18	0.185
1×10^{-2}	0.17	0.175	0.175
After first quench			
Without inhibitor	0.36	0.39	0.40
1×10^{-4}	0.34	0.365	0.37
5×10^{-4}	0.31	0.335	0.34
1×10^{-3}	0.22	0.23	0.23
5×10^{-3}	0.195	0.2	0.205
1×10^{-2}	0.19	0.195	0.2
After second quench			
Without inhibitor	0.37	0.40	0.43
1×10^{-4}	0.35	0.375	0.4
5×10^{-4}	0.29	0.31	0.33
1×10^{-3}	0.25	0.27	0.28
5×10^{-3}	0.215	0.225	0.235
1×10^{-2}	0.21	0.22	0.225
After third quench			
Without inhibitor	0.38	0.42	0.45
1×10^{-4}	0.35	0.385	0.41
5×10^{-4}	0.325	0.35	0.37
1×10^{-3}	0.24	0.265	0.27
5×10^{-3}	0.23	0.238	0.24
1×10^{-2}	0.225	0.232	0.23

clear and was nearly the same for all concentrations. The polarization properties of the repeatedly recrystallized samples were the same as for those of the as-received samples of each grade. All of the polarization curves shifted toward lower I_{corr} values in proportion to the TU concentration and E_{corr} values in the solution containing inhibitor shifted toward more noble values as the inhibitor concentration increased (Tables I and II). For samples quenched three times, the E_{corr} for all samples with and without TU shifted to more noble values due to the repeated recrystallization, indicating that the addition of TU affected mainly the anodic process. This observation was consistent with other findings that for mild steel corrosion inhibition by TU, its derivative effect is on the anodic process only (Shetty *et al.*, 2007). The I_{corr} values of the third quenched samples in the uninhibited solution were found 0.38, 0.42 and 0.45 and for the same samples under 1×10^{-2} M of TU solution were 0.225, 0.232 and 0.230 at 20, 30 and 40°C , respectively (Table II). Thus, due to an increase in TU concentration, the I_{corr} values decreased for quenched steel at all the three temperatures. The same results were observed for the samples recrystallized twice. Corrosion rates as function of TU concentration are shown in Figure 9.

Figure 9 Corrosion rate as a function of TU concentration



4.2 Adsorption isotherm and corrosion inhibition mechanism

The efficiency of an inhibitor is expressed as:

$$\text{Inhibitor efficiency (\%)} = 100 \times \theta$$

where θ is the surface coverage and can be expressed by (Odiongenyi *et al.*, 2009):

$$\theta = (I_0 - I)/I_0$$

where I_0 is the corrosion rate of the uninhibited system (i.e. without inhibitor) and I is the corrosion rate of the inhibited system (i.e. with inhibitor). The values of inhibitor efficiency (%) are given in Table III.

All repeatedly quenched samples at a particular temperature exhibited a decrease in the corrosion current (I_{corr}) with the increases in the TU concentration. This was correlated with the increasing degree of surface coverage. The inhibitor efficiency of E-34 for as-received and repeated recrystallized samples was increased with higher TU concentrations for all three temperatures (Table III). The degree of surface coverage increases with an increase in temperature for the as-received microalloyed steel, and there was an appreciable degree of protection (even at very low inhibitor concentrations). This finding strongly supports the idea that the TU inhibits the corrosion process through surface adsorption. The results from Table III also indicate that there was no change in the inhibitor efficiency of TU due to repeated quenching. The inhibitor efficiency values for the highest concentration of TU were nearly the same for the as-received and repeatedly recrystallized samples. Thus, repeated recrystallization of the E-34 microalloyed steels to achieve grain refinement had little effect on the efficiency of corrosion inhibition by TU.

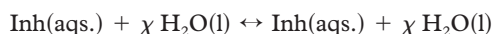
The trends for the E_{corr} , I_{corr} and inhibitor efficiency values were the same for samples quenched once, twice and three times (Tables I–III), but they reached maximum values for the samples after the third quench. It was anticipated that repeated recrystallization might increase the proportion of ferritic microalloyed steel and therefore the effectiveness of the inhibition mechanism; however, this did not occur (Agrawal *et al.*, 2007). Inhibitor efficiency increased with an increase in

Table III Inhibitor efficiency (%) of E-34 microalloyed steels

Concentrations (M)	Temperature (°C)		
	20	30	40
As received			
1×10^{-4}	6.25	7.35	8.33
5×10^{-4}	21.87	23.53	25.00
1×10^{-3}	37.50	28.24	38.89
5×10^{-3}	45.31	47.06	48.61
1×10^{-2}	46.87	48.53	51.39
After first quench			
1×10^{-4}	5.55	6.41	7.5
5×10^{-4}	13.88	14.11	15.00
1×10^{-3}	38.88	41.02	42.50
5×10^{-3}	45.83	48.27	48.75
1×10^{-2}	47.22	50.00	50.00
After second quench			
1×10^{-4}	5.41	6.25	6.97
5×10^{-4}	28.62	22.50	23.25
1×10^{-3}	23.43	32.50	34.88
5×10^{-3}	41.89	43.75	45.35
1×10^{-2}	43.24	45.00	47.67
After third quench			
1×10^{-4}	7.89	8.33	8.89
5×10^{-4}	14.47	16.67	17.78
1×10^{-3}	36.84	36.91	40.00
5×10^{-3}	39.47	43.33	46.67
1×10^{-2}	40.79	44.76	48.89

the TU concentration, but it reached a maximum at 1×10^{-2} M for all the heat treatments.

Organic corrosion inhibitors are known to decrease metal dissolution via adsorption on the metal/corrosive interface to form a protective film that separates the metal surface from the corrosive medium. The adsorption route usually is regarded as a substitution process between the organic inhibitor in the aqueous solution ($\text{Inh}[\text{aq.}]$) and water molecules adsorbed at the metal surface ($\text{H}_2\text{O}[\text{l}]$) as follows (Mu *et al.*, 2004):



where, χ represents the number of water molecules replaced by one molecule of adsorbed inhibitor. The adsorption bond strength is dependent on the composition of the metal, corrosive, inhibitor structure, concentration and orientation as well as temperature (Kissi *et al.*, 2006). Basic information on the interaction between the inhibitor and alloy surface can be provided by the adsorption isotherm. The surface coverage (θ) values were evaluated graphically by fitting to a suitable adsorption isotherm. The different types of adsorption isotherm equations that might govern the adsorption processes are:

$f(\theta) = \{\theta/(1 - \theta)\} \times \{\theta + n(1 - \theta)^{n-1}/n^n\}$ as per the Bockris-Swink model (Bockris and Swinkel, 1964),

$f(\theta) = \theta/\exp(n - 1)(1 - \theta)^n$ as per the Flory-Huggins model (Abiola, 2005) or

$\theta = K \cdot C_{\text{inh}}/(1 + K \cdot C_{\text{inh}})$ and $C_{\text{inh}}/\theta = C_{\text{inh}} + 1/K$ as per the Langmuir model (Alberty and Silbey, 1998). In the above equations, (n) is an integer and (C_{inh}) represents inhibitor concentration. The data were tested in each model systematically, but failed to fit either the Bockris-Swink isotherm model or the Flory-Huggins isotherm model. The plots of C_{inh}/θ versus C_{inh} showed straight lines for the as-received and quenched samples at three different temperatures, which clearly demonstrate that the adsorption process followed the Langmuir adsorption isotherm model (Figure 10). The results are consistent with other findings that TU-derived corrosion inhibition of mild steel corrosion obeys the Langmuir adsorption isotherm model (Azim *et al.*, 1998).

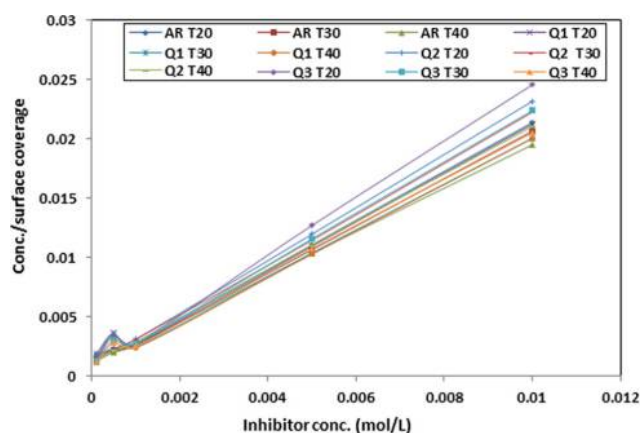
The free energy of adsorption ($\Delta G_{\text{ads}}^\circ$) at different temperatures was calculated from the Langmuir adsorption isotherm curve of C_{inh}/θ versus C_{inh} . The intercept ($1/K$) of the straight line (where, K represents the adsorption coefficient), which is temperature dependent and is related to the Gibb's free energy ($\Delta G_{\text{ads}}^\circ$) value, is a key factor to change the enthalpy ($\Delta H_{\text{ads}}^\circ$) of the process (Abdallah, 2004):

$$K = \exp(\Delta G_{\text{ads}}^\circ/RT) \text{ and } \Delta G_{\text{ads}}^\circ = \Delta H_{\text{ads}}^\circ - T\Delta S_{\text{ads}}^\circ$$

(from Gibb-Helmholtz equation)

Table IV shows the $-\Delta G_{\text{ads}}^\circ$ values of as-received E-34 steels, following the first, second and third quenches at 20, 30 and 40°C. The free energy of adsorption had negative values for all the as-received and quenched steels. The increase in solution temperature increased the free energy change ($-\Delta G_{\text{ads}}^\circ$) values. The low and negative values of $\Delta G_{\text{ads}}^\circ$ indicate the spontaneous adsorption of inhibitors on the metal surface (Ajmal *et al.*, 2000). The values of $\Delta H_{\text{ads}}^\circ$ and $\Delta S_{\text{ads}}^\circ$ of the E-34 microalloyed steels are given in Table V. The enthalpy

Figure 10 Langmuir's adsorption isotherm plots

Table IV Free energy ($-\Delta G^\circ$ KJ/Mole) values of E-34 microalloyed steels

Temperature °C	AR	Q1	Q2	Q3
20	-16.595	-15.682	-16.383	-16.383
30	-17.402	-16.38	-16.942	-16.942
40	-17.976	-17.293	-17.728	-17.728

Table V Enthalpy and entropy changes in E-34 microalloyed steels

Samples	$\Delta H^\circ_{\text{ads}}$	$\Delta S^\circ_{\text{ads}}$
AR	−15.235	0.0696
Q1	−14.035	0.0805
Q2	−15	0.0672
Q3	−15	0.0672

change of the adsorption process was negative ($\Delta H^\circ_{\text{ads}} < 0$); that is, adsorption is an exothermic reaction as reported by others for mild steel (Paul *et al.*, 2012).

5. Conclusions

Tests were carried out to evaluate the effectiveness of TU in reducing the corrosion rate of E-34 microalloyed steel at various solution temperatures of 20, 30 and 40°C and to establish if refining the grain size had any effect on the inhibition efficiency of the TU. An increase in the TU concentration decreases the corrosion rate in repeatedly quenched microalloyed steel at different temperatures. However, the trend of the efficiency values was almost the same for the as-received and for the single-, double- and triple-quenched samples, although the values reached a maximum for the triple-quenched sample. The inhibition process of TU for E-34 microalloyed steels was confirmed to be an exothermic reaction and followed the Langmuir adsorption isotherm model.

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Corresponding author

Mohammad Rezaul Karim can be contacted at: mkarim@ksu.edu.sa