Effect of heat treatment on hydrogen permeation of API 5L X60 steel in soil solution

Efecto del tratamiento térmico en la permeación de hidrógeno del acero API 5L X60 en solución de suelo

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This work deepens the study of the influence of steel microstructure on hydrogen permeation. The evaluated steel was subjected to a quenching heat treatment, favoring the formation of phases such as bainite and acicular ferrite. These phases increase the mechanical properties and increase the reversible traps (dislocations), which promote hydrogen absorption/release. This mechanism occurs at the metal-electrolyte interface and favors increasing the service life of the steels. It is of vital importance to deepen the knowledge of the influence of the microstructure on the susceptibility to hydrogen damage. With a heat treatment with accessible and simple costs for the industry we can obtain good results in the behavior of hydrogen diffusion in the tested steel. The microstructural changes due to quenching heat treatment have a multidisciplinary approach to understand the changes in mechanical properties, susceptibility to hydrogen damage and corrosion rate. For this purpose, a classical electrochemical technique such as electrochemical hydrogen permeation is used to obtain information related to the microstructure and the behavior of hydrogen diffusion in the volume of the steel. Steels are the most widely used material due to their mechanical properties and resistance to corrosion in the different environments to which they are subjected. Hence the interest in improving their properties to extend their useful life. The quenching heat treatment modifies the diffusion of hydrogen through the steel. The bainite and acicular ferrite phases promote the formation of reversible hydrogen traps.

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Abstract

Hydrogen permeation tests were carried out on API 5L X60 steel specimens under three conditions: base material and heat-treated specimens at 1050 °C for 15 and 20 minutes, then quenched in water. These specimens were subjected to hydrogen permeation tests in CAM soil solution, representative of the state of Campeche. The heat treatment modified the microstructure present in the steel and therefore the behavior of the hydrogen permeation curves. The bainite and acicular ferrite phases present in the heat-treated steel acted as reversible traps, due to their large number of dislocations. The diffusion coefficient and the number of reversible traps present in the volume of the steel were determined.

Objetive	Methodology	Contributions
Determine the influence of microstructure on hydrogen permeation, in conditions representative of the state of Campeche, Mexico.	Sample preparation and heat treatment. Characterization Microstructure and electrochemistry Results	Understanding the influence of microstructure on hydrogen permeation. Simulating soil conditions allows this absorption phenomenon to be studied more realistically.

Hydrogen, Heat treatment, Microstructure

Resumen

Se realizaron pruebas de permeación de hidrógeno en probetas de acero API 5L X60 bajo tres condiciones: material base y probetas tratadas térmicamente a 1050 °C durante 15 y 20 minutos, posteriormente templadas en agua. Estas probetas fueron sometidas a pruebas de permeación de hidrógeno en solución de suelo CAM, representativa del estado de Campeche. El tratamiento térmico modificó la microestructura presente en el acero y por ende el comportamiento de las curvas de permeación de hidrógeno.Las fases bainita y ferrita acicular presentes en el acero tratado térmicamente actuaron como trampas reversibles, debido a su gran número de dislocaciones. Se determinó el coeficiente de difusión y el número de trampas reversibles presentes en el volumen del acero.

Objetivo	Metodología	Contribuciones
Determinar la influencia de la microestructura en la permeación del hidrógeno en condiciones representativas del estado de Campeche, México.	Preparación de muestras y tratamiento térmico Caracterización Microestructural y Electroquímica Resultados	Entendimiento de la influencia de la microestructura sobre la permeación del hidrógeno La simulación de las condiciones del suelo permite estudiar este fenómeno de absorción de forma más realista.

Hidrógeno, Tratamiento térmico, Microestructura

Introduction

Low-strength alloy steels (HSLA) are good mechanical properties of strength, toughness and weldability due to the thermo-mechanical work performed during manufacture and their low carbon content. The different environments to which these steels are subjected range from acidic (swamp) and basic (soil) media, these hinder the prolonged use of HSLA steels due to susceptibility to corrosion and hydrogen damage.

One of the methods that minimize the loss of material by corrosion is cathodic protection, this has the indirect effect of evolving hydrogen on the surface of steel, a part of the evolved hydrogen enters inside as an atom, hydrogen is stored in reversible and irreversible traps.

Reversible traps are sites where the energy needed to bond is small this facilitates the entry and exit of hydrogen atoms, on the contrary, irreversible traps have a high binding energy this makes their exit from the inside metal difficult. The presence of hydrogen affects the mechanical properties of strength and toughness. The formation and presence of microstructural defects accelerates the effects of hydrogen damage.

Heat treatments are an effective method to improve mechanical properties and corrosion resistance. HSLA steels with low carbon (C) and Niobium (Nb), can be heat treated by hardening, the low carbon quantity minimizes the formation of martensite and the presence of Nb does not allow a large increase in average grain size.

This work studies the influence of microstructure and the effect of heat treatment on API 5L X60 micro-alloy steels on hydrogen diffusion. In addition to the effect of the constant cathodic potential on the evolutionary dynamics of atomic hydrogen in steels with and without heat treatment. The diffusion coefficient of the untreated and heat-treated steels was determined.

Methodology

API 5L X60 steel with ferrite as the main constituent was used, in addition to perlite bands (MCLMB). This steel was cut into strips of 150 mm x 20 mm x 11 mm, then subjected to 1050 °C for 15 (MCLTT15) and 30 (MCLTT30) minutes, immediately quenched in water.

The microstructure of the heat-treated test pieces was bainite and acicular ferrite, the latter being highly sub-structured and equiaxiated which is formed in continuous cooling by mixing diffusion and stresses which give the steels a high tenacity[1,2].

For the hydrogen permeation tests, steel with and without heat treatment was milled to 1 mm in thickness, with an effective working area of 1 cm^2. The input side of the hydrogen was sanded with silicon carbide (CS) paper from #100 to #1200 and the output side was sanded with CS from #100 to #2000, then polished with 1 μm diamond paste, this was electrochemically coated with palladium in order to minimize the base hydrogen permeation current density.

Permeation tests were carried out in a double cell, one for the generation or electrochemical charge of hydrogen and the other for the oxidation of hydrogen. The solution used for the electrochemical charge was CAMP (pH=8.1), the chemical composition of the soil-simulating solution is shown in Table 1.

Box 1

Table 1

Chemical composition of the CAM solution

Compound	$MgSO_4*7H_2O$	$CaCl_2*H_2O$	KNO_3	$CaCO_3$	$NaHCO_3$
Quantity (g/l)	0.0251	0.0215	0.050	0.202	0.823

The CAM solution is representative of the soil in the state of Campeche. A constant cathode potential of -950 mV vs SCE was applied to the input side allowing hydrogen generation and diffusion within the test pieces. On the output side, 0.2 molar solution of NaOH was used at a constant anode potential of 170 mV vs SCE, this potential was sufficient for the formation of a stable passive layer. Prior to permeation tests, high purity argon was bubbled to remove dissolved oxygen from the solution in order to prevent surface oxidation. The permeation current was recorded by means of a Solartron Potentiostat/Galvanostat and the reduction current was recorded with a Uicorr Potentiostat. All tests were conducted at 25°C 1.

Analysis of the permeation current

In Figure 1, the characteristic parameters that can be obtained from the hydrogen permeation curve are shown.

Box 2

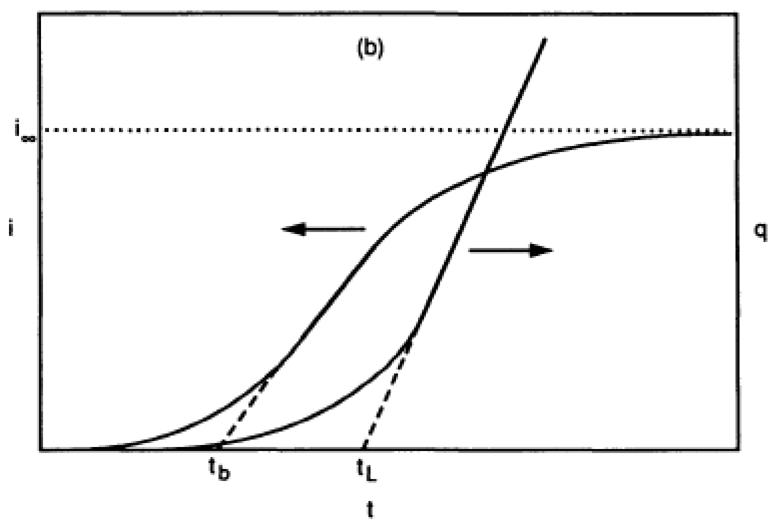

Figure 1

Variation in the anode current over time for a membrane with a constant flow on the electrochemical charge side

Source: 3, 10, 11.

The flow of hydrogen $J_H\left(\frac{molH}{m^2s}\right)$, through the steel was measured by the steady-state permeation current density $\left(\frac{i_p^\infty}{nF}\right)$ [3, 10,11]:

$$J_H = \frac{i_p^\infty}{nF} \quad [1]$$

Where n is the number of electrons transferred and F is the Faraday constant. The rate of hydrogen permeation $\left(\frac{molH}{m^2s}\right)$, is defined by[3, 10,11]:

$$J_H L = \frac{i_p^\infty}{nF} \quad [2]$$

Where L is the thickness of the test piece. The effective diffusivity of hydrogen D_{eff}, can be calculated by[3, 10,11]:

$$D_{eff} = \frac{L^2}{6t_L} \quad [3]$$

Where the time of backtracking t_L, is the point on the hydrogen permeation curve where $i_t = 0.63 i_p^\infty$. If the surface hydrogen is in thermodynamic equilibrium with the sub-surface hydrogen, the apparent hydrogen solubility (C_{app}), is [3, 10,11]:

$$C_{app} = \frac{J_H L}{D_{eff}} \quad [4]$$

In addition, the density of hydrogen traps can be estimated according to the following equation:

$$N_T = \frac{C_0}{3}\left(\frac{D_1}{D_{eff}} - 1\right) \quad [5]$$

Where N_T is the number of hydrogen trap sites per unit volume and D_1 is the diffusion coefficient in the crystal lattice for α-Fe is 1.28 x 10-4 cm2s-1[4,5].

Results

Analysis of the cathode current associated with the hydrogen evolution process

Figure 2 shows the I_{cath} of MCLMB, MCLTT15 and MCLTT30 in CAMP solution at potential -950 mV vs SCE.

At a cathodic potential of -950 mV vs SCE, the Volmer reaction is favored:

$$H_2O + e^- \leftrightarrow H_{ad} + OH^- \quad [6]$$

H_{ad} atoms can be absorbed by steel:

$$H_{ad} \leftrightarrow H_{ab} \quad [7]$$

This input mechanism is given with water molecules and proceed in alkaline and neutral media[6,7]. Curve 1 has the lowest I_{cath} values compared to heat-treated steels. In general, the I_{cath} values tend to increase from 45 μAcm^{-2} to 12 μAcm^{-2}, the I_{cath} values are similar to those obtained by the test pieces in NS4 solution. For hardened and tempered steels, the stress corrosion mechanism appears to be dominated by hydrogen embrittlement[8,9].

Box 3

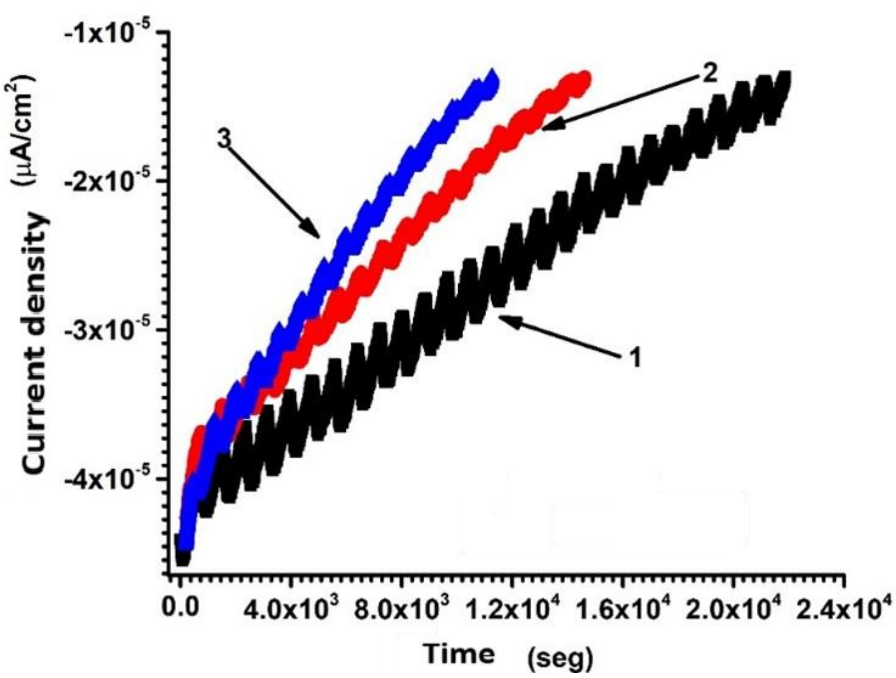

Figure 2
Cathode current density in 1-MCLMB, 2-MCLTT15 and 3-MCLTT30 steels in CAM solution
Source [*Own elaboration*]

Analysis of hydrogen permeation stream in CAM solution

Figure 3 shows the hydrogen permeation curves of API 5L X60 steel specimens with and without non-conventional heat treatment at cathodic potential (170 mV vs SCE), in standard CAM solution. Curves 1, 2 and 3 are MCLMB, MCLTT30 and MCLTT15 respectively.

Box 4

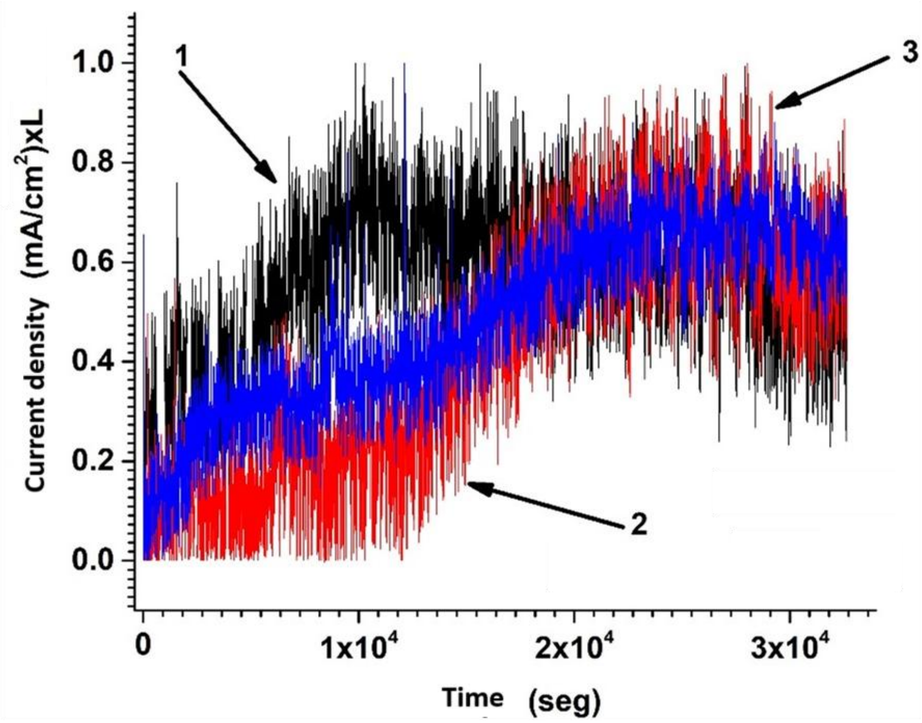

Figura 3

Hydrogen permeation curve of the test pieces with and without non-conventional heat treatment in CAM solution

Source [*Own elaboration*

Liang, O. et al.,[10, 11 13] tells us that in solutions simulating deoxygenated soil with alkaline pH there is an electrical reduction of the solution itself, as well as a drop in ohmic potential, which brings more negative potentials during hydrogen reduction.

The permeation current density of steel with and without unconventional heat treatment shows a great similarity for the three tested conditions.

La i_∞, gradually increases to a constant average value of 0.826 μAcm-2, 0.672 μAcm-2 and 0.817 μAcm-2 for the MCLMB, MCLTT15 and MCLTT30 test pieces in CAM solution. The results show that the i_∞, of 8.27 x 10^{-7} was the highest and is for the MCLMB test piece. The stationary current densities of MCLTT15 and MCLTT30 were 6.73 x 10^{-7} and 8.17 x 10^{-7}, respectively.

Box 5

Table 2

Parameters associated with hydrogen permeation in steel with and without non-conventional heat treatment in CAM solution

Solution CAM	i_∞ (μAcm^{-2})	t_L (seg)	J_∞ $(molcm^2s^{-1})$	D_{eff} (cm^2s^{-1})	C_0 $(molcm^{-3})$	N_T (cm^3)
MCLMB	8.26e-7	3977	8.52e-12	5.69e-9	1.74e-5	7.95e22
MCLTT15	6.73e-7	12887	6.97e-12	1.63e-9	5.73e-5	1.32e24
MCLTT30	8.17e-7	8959	8.47e-12	2.16e-9	5.13e-5	1.17e24

Table 2 presents the main average parameters associated with the hydrogen permeation curves in CAM solution at 170 mV vs SCE. The highest value of the D_{eff} is given in the MCLTTMB test piece, followed by the MCLTT30 test piece and finally the MCLTT15 test piece. This shows that microstructural changes due to unconventional heat treatment have no great influence on the behavior of hydrogen permeation[12] in CAM solution.

Conclusions

Heat treatment of tempering modifies the microstructure generating bainite and acicular ferrite from ferrite and perlite. The phases present after heat treatment increase the properties of the material in arrival condition. The microstructure favors the formation of reversible traps (dislocations mainly), which modify the behavior in hydrogen diffusion.

Declarations

Conflict of interest

The authors declare that they have no conflict of interest. They have no financial interests or personal relationships that could have influenced this book.

Authors' contribution

Calan-Canche, Damian: Main author of research topic, contribution to methodology and analysis of results.

Pérez-Montejo, Salatiel: Review of the research methodology.

Gonzalez-Sanchez, Jorge: Contribution to the subject of research, review of methodology and analysis of results.

Flores-Chan, J. E.: Analysis of results.

Availability of data and materials

The data and materials used in this study are available.

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