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EVALUATION OF HYDROGEN-INDUCED CRACKING IN ASTM A 36 STEEL SAMPLES IN AN ACID MEDIUM AND SUBJECTED TO UNIAXIAL STRESS

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ABSTRACT

The work of reconditioning or "workover" in Colombian oil producing wells has as main function to increase flow capacity and production flow. For this, operations with acid stimulation treatments are implemented, including acid washes to well production pipes to dissolve scale and mineral deposits that reduce and obstruct the effective flow area and generate failure due to hydrogen-induced cracking. HCl, is one of the most commonly used acids in these processes, which is why it will be used in this study to determine its reaction in the mechanical properties of an ASTM A 36 steel. Two concentrations of HCl will be used as corrosive and contributor medium hydrogen to steel at 30 ° C, atmospheric pressure, and 5 periods of different immersion times; to determine the conditions that favor the development of cracks and their effect on the failure of the material. Once the immersion times have passed, the test pieces will be subjected to tension and Charpy tests, analyzing them microscopically to perform the failure analysis. At the end of the study, the results obtained will show when the steel and tabular plates failed, and the variations in mechanical properties will be plotted.

Keywords: Hydrogen embrittlement, hydrogen induced cracking, hydrochloric acid, one-way tensile stress.

1. INTRODUCTION

In the petroleum extraction industry, treatments are performed to in oil wells in order to avoid incrustations and the presence of “scale”, such treatments are performed with chemical agents like hydrochloric acid (HCl) that, if not washed properly, can cause damage to the pipeline. Sometimes the washing baths made after the application of the acid are not effective enough and there are remnants of acid in the well, which can lead to later hydrogen embrittlement problems in the system [1]. Additionally, the well products (oil, gas) can cause hydrogen embrittlement problems [2, 3]. The problem to be studied is what effect does hydrogen have on the embrittlement of the ASTM A36 steel due to the contribution of hydrogen from HCl. Therefore, the question is: Is the concentration of HCl diluted in water detrimental to the behavior of the material in its service condition due to the presence of hydrogen?

With the information obtained in the tests, it is expected to perform tabulations and graphs on the Stress-Strain of each test, and their variation respecting the curves of the untested steel to determine values that can serve as a guide for decisions in the field.

1.1 Hydrogen Embrittlement

The hydrogen embrittlement is a diffusive phenomenon caused by the adsorption of molecular hydrogen (H_2) by the material, which can occur in irons, steels, titanium, aluminum or nickel-cobalt alloys (superalloys) [4] and its consequent dissociation in atomic form (H) within the crystalline structure, causing deformation and therefore a process of embrittlement. Embrittlement is a function of hydrogen concentration, temperature, time, internal forces and the type of material [5, 6]. For steels, it also depends on the class of steel, its microstructure and the interaction of its defects with hydrogen [7]. Hydrogen embrittlement (HE) is classified into environmental hydrogen embrittlement (HEA) and internal hydrogen embrittlement (FHI). Hydrogen can be generated from a corrosive reaction or by its presence in an atmosphere, hydrogen solution (FHA) or by its absorption during component manufacturing processes such as the manufacture of structures and welds during the assembly or manufacturing process (FHI) [8]. In this article, the phenomenon of FHA is investigated.

According to API 6A “Wellheads and Christmas Tree Equipment Specifications”, [9] the equipment is designed with materials that meet the requirements described in Tables 3, 5, and 8. According to these tables, A36 steel due to its characteristics would be in the similar range of materials used for general services (AA-BB) and acid services (DD-EE). In the same way, due to their range of tensile strength (58000-80000 psi), they would meet the 36K and 45K material approval requirements. Likewise, the chemical composition of A36 would be in the range of elements described in table 8 of API 6A. For this, and for the ease of obtaining A36 steel, this steel was used as test material.

Once the was test carried out (container with aqueous solution of HCl in two concentrations and test specimens immersed at certain times for subsequent tension and Charpy tests) the influence of hydrogen adsorption on the properties of yield stress, tensile stress, ductility was recorded as well as the toughness of ASTM A 36 steel and its relationship to the formation of fissures and/or cracks.

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2. MATERIALS AND METHODS

Choice of Material: Why an A 36 Steel as Test Material?

For the present study, a section of ASTM A36 steel sheet measuring 305 mm^2 by a 9 mm thick area was cut. 14 plates, 25.4 mm wide by 305 mm long by 9 mm thickness were cut by using a mechanical machine tool. Subsequently, they were turned, machined and polished according to the dimensions established in the ASTM A 370 specification "Standard test methods and definitions for mechanical tests on steel products" [10]. The specimens cut from the steel section were distributed in 2 sets of 5 plates and a plate called specimen "0", which was not subjected to any chemical attack and served as a reference in relation to the specimens immersed in HCl at concentrations of 7.5 % and 15%.

Table-1 shows the distribution according to immersion of each set of test specimens in the two concentrations of HCl.

Table-1. Specimen identification for tensile and Charpy tests.

% HCl in Aqueous Solution	Immersion time (h)	Plate Tension Identification	% HCl in Aqueous Solution	Plate Tension Identification
	12	1A		1B
	24	2A		2B
	48	3A		3B
15%	60	4A	7.5%	4B
	120	5A		5B
		Charpy Plate Identification		Charpy Plate Identification
		A15		B7.5

Source: The Author

Test Conditions: Why Concentrations of 7.5% and 15% of HCl for the Tests?

HCl is commonly used in stimulation fluids used during reconditioning in some oil wells [11]. Acidification treatments include the use of HCl to remove scale or shale (such as carbonates) from the producing formation to be stimulated. Several acids can be implemented for these tasks, starting with the most commonly used, HCl with concentrations ranging from a 7% to 28% [12]. Likewise, in well stimulation, HCl is used in concentrations of 15% by weight; however, this acid concentration can vary between 5% and 35%. HCl has the ability to dissolve clays, dolomites and other carbonates. The Overflush is implemented for the displacement of the stimulation treatment; therefore, it provides push to the acid so that it can reach damaged areas and those of low permeability. About 25 GALs are injected per foot of sand with a composition of 5% -10% HCl, 2% ammonium chloride solution, clean and filtered kerosene, diesel, oil or crude. Acid solutions at different concentrations or diffusion by cathode medium have been used to assess the influence of hydrogen on the embrittlement of low carbon steels [13-17].

Test Conditions: Why Temperature of ± 30 ° C for the Test?

According to API 571 "Corrosion and materials" [18], the HE effect occurs at temperatures from ranging from environmental to approximately 149 ° C. The effect decreases with increasing temperature, HE is not likely to occur above 71 ° C to 81 ° C. The value of 30 ° was taken as a reference value, since it is an average temperature of the wellheads in cleaning stages and without pressurization.

Equipment for Conducting the HE Test

To carry out this experiment, two glass beakers of 2000 ml each were used; two 0 ° -150 ° C digital thermometers; a variable temperature heating oven; 1622 ml of 7.5% HCl acid and 378 ml of water in one of the glasses and 811 ml of 15% HCl plus 1189 ml of water in the other; epoxy paint to cover the gripping area of the plate jaws to avoid contact with acid and embrittlement in these areas; two wooden lids to hold the plates on the beakers. In figure 1, the equipment used and the test plates are observed.

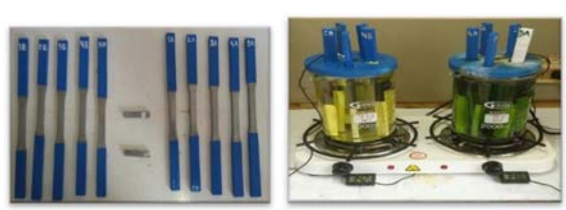

Figure 1. Elements used for HE testing and immersion of steel in HCl. Source: The Author.

One of the beakers contained a 7.5% aqueous HCl solution in which 5 plates (1B, 2B, 3B, 4B and 5B) were placed through the holes in the wooden lids. Likewise, in the other beaker containing 15% HCl aqueous solution, the remaining 5 plates (1A, 2A, 3A, 4A and 5A) were inserted through the lid. See figure 1. For each of the

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aqueous solutions (7.5% and 15%). The test began when introducing the first test specimens (5B and 5A) with the longest immersion time (120 hours). Subsequently, a test specimen was introduced into each aqueous solution until having all 5 test specimens for each HCl concentration and immersion times of 12, 24, 48, 60 and 120 hours.

Material Characterization

An elemental chemical analysis was performed using the spark source optical emission spectroscopy technique in the Quantron Magellan Q8 Spectrometer. The analysis was performed following the guidelines of ASTM E 415 "Standard Test Method for Analysis of Carbon and Low Alloy Steel by Optical Emission Vacuum Spectrometry" [19]. According to the ASTM A36 designation "Standard Specification for Structural Carbon Steel" [20] the chemical composition for this steel is that shown in table 2, which was compared with the chemical composition in the quality certificate and the results obtained in the laboratory, obtaining the same results.

The tension test was carried out in a Microtest em2/300/FR equipment. The stress and creep requirements of an ASTM A 36 steel are shown in Table-3, together with the results of the quality certificate and the stress test of specimen 0 (specimen without HCl chemical attacks). The results for tensile stress and creep shown in the quality certificate, as well as those obtained in the tensile test on specimen 0 performed in the laboratory, correspond to ASTM A36 steel.

Table-2. ASTM chemical composition vs laboratory results for an ASTM A 36 steel.

Element	Requirements ASTM A 36	Material Certificate	Laboratory
%C Maximum	0,25	0,1	0,138
%Mn	Not specified	0,55	0,28
%P Maximum	0,04	0,027	…..
%S Maximum	0,05	0,012	0,08
%Si Maximum	0,4	0,24	0,18

Source: The Author.

Table-3. ASTM A36 steel mechanical properties vs. laboratory tests.

Property	Requirements ASTM A 36 (psi)	Certificate (psi)	Laboratory (psi)
Stress tension (psi)	58000-80000	64.686,95	69773,34
Creep stress (psi)	36000 Min	45.396,90	45557,24

Source: The Author.

a) Metallography

Sections removed from A36 without exposure to HCl, in bakelite molds and attacked with 5% Nital, were evaluated in the LEICA model DVM 2500 optical microscope, obtaining the images shown in Figure-2. The samples were prepared according to ASTM E3 "Standard Guide for preparation of metallographic specimens" [21]. In the metallography's (Figure-2) at 100X, a steel matrix (gray color) with small non-metallic inclusions (black dots) is observed throughout the structure. These are metallic compounds (Fe, Mn, Al, Si) with the non-metals (0, S, C, H, N) that form separate phases. These inclusions, despite their small content, exert a significant effect on tensile strength, hardness, corrosion resistance, and fatigue. In the micrographs magnified at 1400 X and 1800 X, a microstructure typical of a hypoeutectoid carbon steel is observed, consisting of equiaxial ferrite (white areas) and pearlite (black areas), with grains of uniform distribution and random orientation, typical of the A36 of this study.

3. RESULTS

A. Mechanical Tests

After the immersion times at average temperatures of 30ºC; Tensile tests are performed on the 5 specimens that were immersed in 7.5% aqueous HCl solution and the 5 that were immersed in 15% HCl. Likewise, a stress test was carried out on the specimen that did not have contact with the acid called specimen 0. In tables 4 and 5 the values obtained for each specimen were recorded. For each tensile test, the universal machine emits a Stress-Deformation graph, where the force exerted in an equiaxial direction and the deformation that this produces in the area of reduced section is related; recording for each specimen (11 in total) the maximum force, breaking stress, elastic limit stress, deformation and maximum elongation.

Taking the stress curves for each set of 5 specimens for the two concentrations of HCl, the behavior

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of each stress curve respecting specimens' 0 curve is presented in a single graph.

Figure-3 shows the consolidation of the curves of 5 specimens immersed in 15% HCl up to 120 hours and subjected to stress testing, with the description of the type of failure. It is observed that the unit deformation in the specimens 3A and 5A was lower than the other specimens, this implies that after 48 hours in 15% HCl, the specimen 3A begins to present more plastic than elastic behavior, evidenced in a less pronounced curve in comparison with the curve of the specimen 0 and presenting a fracture more brittle than ductile, according to fractographic analysis. The specimen 5A presents a curve with a behavior much more susceptible to fracture, due to the fact that it was exposed to HCl for a longer time (120 hours).

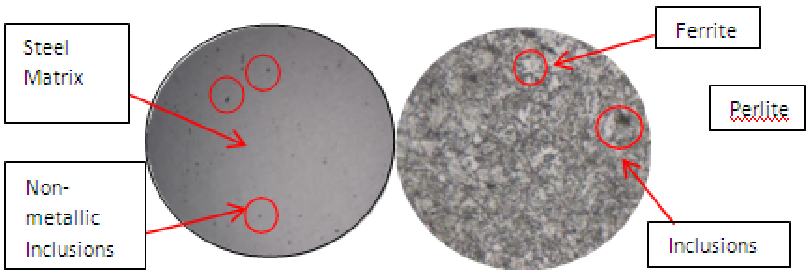

Figure-2. Steel Metallography's A36 a 100X (200um) 1400X (50 µm) y A 2800X (20 µm) Source: Author.

Table-4. Tension test results on plates immersed in 15% HCl.

Specimen	t(h)	Breakage (psi)	Elastic Limit (psi)	Elastic Module (psi)	Deformation %	Elongation (mm)
0	0	69773.39	45557.24	13196216.94	0.545	8.208
1A	12	67573.79	44782.99	7618662.55	0.789	7.354
2A	24	68284.27	43646.39	6631155.33	0.858	7.460
3A	48	66692.41	42681.74	9617284.55	0.645	5.643
4A	60	62737.05	40403.74	10570084.56	0.583	7.420
5A	120	52601.03	40135.42	17105532.81	0.483	3.024

Source: The Author.

Table-5. Tension test results on plates immersed in 7.5% HCl.

Specimen	t(h)	Breakage (psi)	Elastic Limit (psi)	Elastic Module (psi)	Deformation %	Elongation (mm)
0	0	69773.39	45557.24	13196216.94	0.545	8.208
1B	12	69906.62	45068.90	11713371.39	0.586	7.652
2B	24	66578.08	45411.54	16846403.03	0.471	6.992
3B	48	66743.95	43905.54	12627487.63	0.572	6.784
4B	60	66909.84	42399.60	9062695.18	0.670	6.817
5B	120	65225.16	42400.24	17430198.49	0.445	7.257

Source: The Author.

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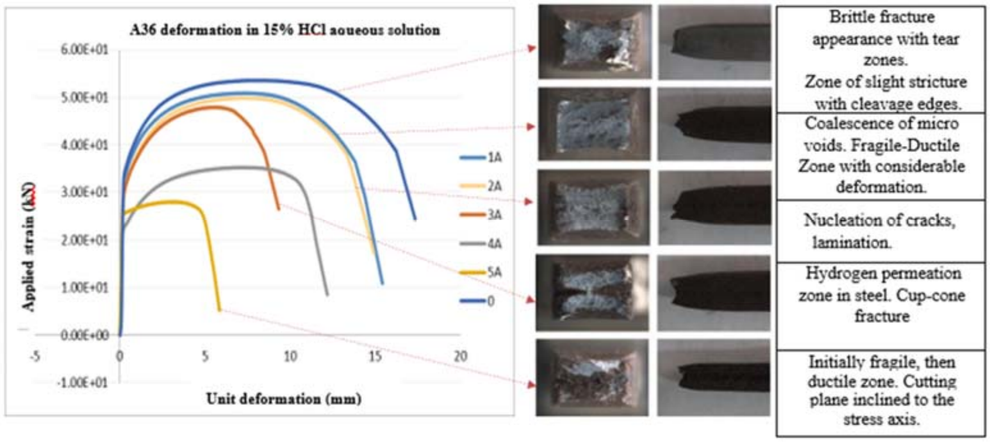

Figure-3. Behavior of A36 specimens in tension and Fractography tests respecting specimen 0. Immersion in 15% HCl.

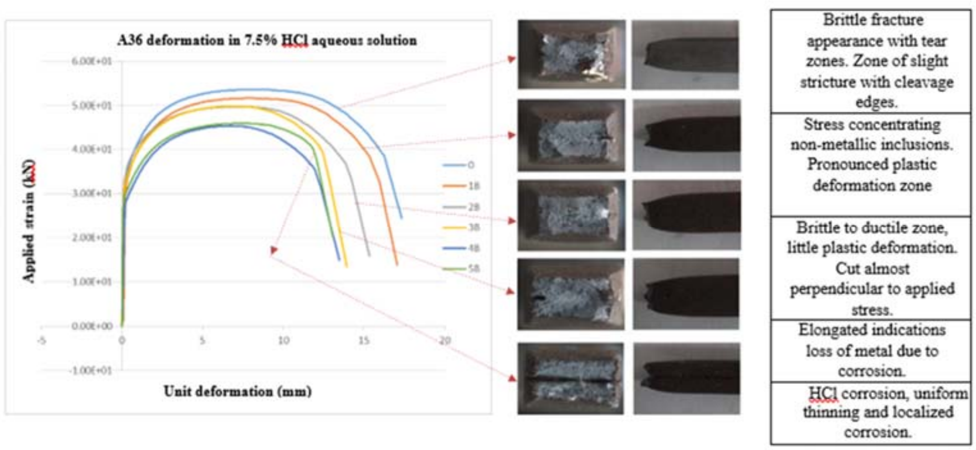

Figure-4. Behavior of A36 specimens in tension and Fractography tests, respecting specimen 0. Immersion in 7.5% HCl. Source: Author.

Figure-4 presents the curves for 5 A 36 steel specimens in 7.5% HCl brought to tension once the immersion times have passed.

They are compared with the stress-strain curve of specimen 0 (not exposed to the acid solution). The amount of elastic deformation that steel can withstand is therefore small during elastic deformation. In this way, the specimens in 7.5% HCl, despite the decrease in tensile strength and elastic limit, do not present values below the stresses allowed for ASTM A36 (Tension, minimum 58,000 psi; minimum creep 36,000 psi). The fractographic description of the surface is presented in the same figure once the specimens have failed.

Engineering Stress (Breaking Stress)

For the 5 specimens immersed for up to 120 hours in 15% HCl, the stress in A36 after 12, 24, 48, 60 and 120 hours, compared to specimen 0, shows a decreasing behavior as the immersion time progresses, until a minimum breakage value of 52601.03 psi is obtained, a value 24.6% below the values obtained for breakage stress in the specimen without chemical attacks. Likewise, the specimens in 7.5% HCl present a considerable decrease of 4.58% in the value of resistance to tension in specimen 2B respecting specimen 0 at 20 hours of immersion; and then stabilize from 40 to 120 hours with a value of 6.52% below the value recorded for the stress in the specimen 0.

Elastic Limit Tension

The minimum value of the set of specimens in 15% HCl is recorded in specimen 5B (40135.420 psi), which compared to specimen 0 (45557.241 psi) represents a decrease of 11.90% in this property. In the curve generated for 15% HCl, the elastic limit of A36 decreases as the immersion time elapses, having its minimum point at 120 hours, with a value of 40 135.420 (psi). The minimum value for specimens in 7.5% HCl is observed in specimens 4B and 5B. The value in test piece 4B, after 60 hours of immersion represents 7.06% decrease respecting specimen 0. The elastic limit after 120 hours shows a decreasing behavior until a value of 42400.240 psi is obtained, which represents a decrease 7.4%.

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Elasticity Modulus

The behavior is linear in the specimens after 60 hours for the two concentrations, reaching their maximum values at 17105532.814 psi for test tube 5A after 120 hours of immersion in 15% HCl, and 17430198.486 psi for test tube B immersed 120 hours in aqueous 7.5% HCl solution.

Elongation Percentage

The elongation % in 15% HCl shows that the greatest reduction in area occurs in specimens 2A (14.92% at 24 hours) and 4A (11.84% at 60 hours). The lowest is presented in test tube 5A with 6.05%. Greater elongation, 15.30% in specimen 1B at 12 hours' immersion; minimum elongation in the specimen 13.57% in the specimen 3B after 48 hours of immersion in 7.5% HCl.

Area Reduction Percentage

Relatively constant behavior up to 48 hours of immersion in 15% HCl, where a significant decrease occurs to the lowest value in test piece 4A (50.43%) at 60 hours. Almost linear behavior in the reduction of the area reduction, reaching the minimum value in the specimen 5A (53.17%) after 120 hours of immersion in 7.5% HCl.

Charpy Test Results

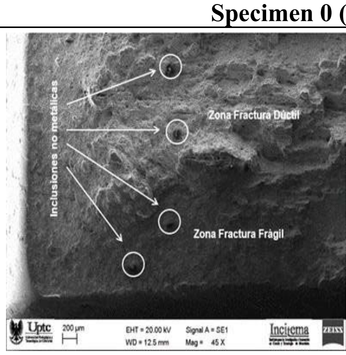
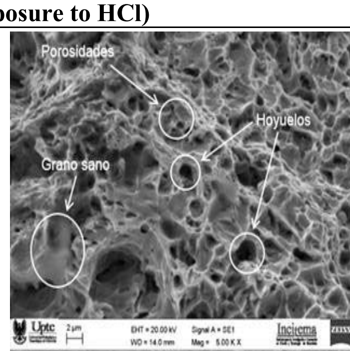
The absorbed energy is directly related to the toughness, evidencing in the test that the test piece immersed in 15% HCl aqueous solution lost more toughness value than the one obtained from the test piece immersed in 7.5% HCl.

Hardness Results

For immersion in 15% HCl the value decreases proportionally to a minimum of 166.737 V, which is a loss of toughness of 20.42% with respect to the value of the specimen 0. For HCl at 7.5% the loss of hardness is 12.76%, compared to the value of 209.567 V in specimen 0.

Scanning Electron Microscopy

Below is a description of the main metallographic findings:

Specimen 0 (No exposure to HCl)		Description
		Cross section of specimen 0 (without exposure to HCl) subjected to tension test. The material presents non-metallic inclusions typical of the manufacturing process. Breakage at 69,773 psi with a maximum yield stress of 45,557 psi. Presence of dimples and grains of healthy material.

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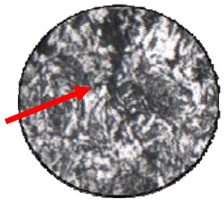
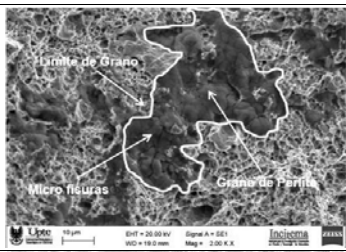
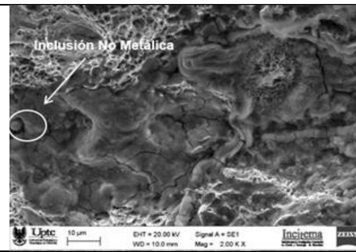
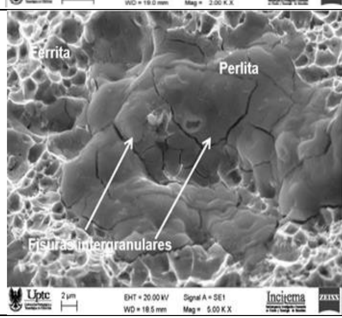
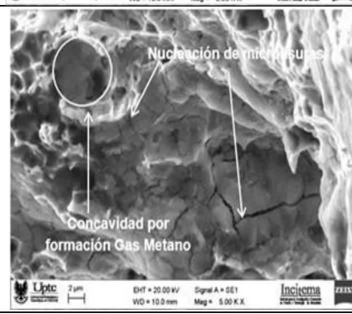
Immersion in HCl 15%	Immersion in HCl 7.5%	Description
		Formation of intergranular and staggered cracks typical of the HIC phenomenon. Brittle fracture showing signs of cleavage, where the fracture propagates through specific planes of the grain or intergranular fractures.
		The micrograph shows at 5.00 KX, pearlite grains embedded in a ferritic matrix, typical structure of low carbon steels. In the micrograph (left), the grain boundaries are clearly defined. Right non-metallic inclusion detail.
		Intergranular type cracking affecting only perlite. The ferrite has no initial cracking (left). H_2 diffuses into steel reacting with less thermodynamically stable carbides and forms methane gas bubbles at grain boundaries that can cause rapid coalescence failure of methane bubbles and contribute to the formation of microcracks.

4. CONCLUSIONS

The unit deformation for the two concentrations of HCl summarizes that the specimens 3A and 5A were lower than the other specimens, this implies that after 48 hours of immersion in 15% HCl, A 36 begins to present more plastic than elastic behavior and greater susceptibility to fracture, presenting a fracture more brittle than ductile, according to fractographic analysis.

The elasticity modulus in 7.5% HCl, present low values respecting specimen 0, which indicates that they are less rigid and easier to bend under load. Specimens 2B and 5B showed values above the reference of specimen 0, which indicates greater rigidity and resistance to elastic deformation. The Young's modulus loss of values begins to be relevant after 60 hours of immersion. For 15% HCl, with the same value of the specimen 0 in Young's modulus, values are observed to show a loss of this property as the immersion time progresses. Thus, the A 36 specimens at this concentration have values well below that of specimen 0, which indicates less rigidity and less resistance to deformation, even from 12 to 60 hours of immersion.

Conventional deformation, for 7.5% HCl there is a decrease in the percentage of elongation after 48 hours of immersion. Likewise, for the set of specimens immersed in 15% HCl, there is a considerable decrease in the % elongation after 120 hours of immersion.

In the reduction in area %, in the case of A 36, specimen 0 presented a % reduction in area of 66.02 %. From this value, the specimens immersed at 7.5% showed a decrease in ductility of 18.23%, as their plastic deformation was affected by the induction of H2. For the set of specimens immersed in 15% HCl, the decrease in ductility was observed in a loss of 22.44%, which is reflected in the lack of capacity of the material to recover its ductile zone, and fail in a brittle way.

The impact resistance results test show that the toughness is considerably diminished at the longer contact time with the aqueous solution as well as at the higher concentrations of HCl. For the hardness, a decrease is observed respecting specimen 0, up to 12.8% in the set of specimens immersed for up to 120 hours in 7.5% HCl. For specimens in 15% HCl, a decrease of 20.42% is recorded; this concentration of HCl being the one that most influences the decrease in the hardness of the A36 specimens tested.

Hydrogen-induced cracks inside the A36 have propagated through porous areas. From a local cracking produced by hydrogen embrittlement, the micro-cracks rapidly propagate the breakage making the material go from fragile to ductile.

As corrosion by HCl occurs in the areas surrounding the place where the specimens failed, it acts as a stress concentrator and an aid in the propagation of the crack and subsequent failure. Corrosion acts as a notch and the fracture does not start in the center of the section, but originates from the notch and spreads to the center of the section. Optically a rather opaque surface is appreciated. Observed at higher increases in the SEM, it is composed of small depressions or cavities, which are the result of the coalescence of microcavities or "voids", which are initiated by decohesion and flow of the material around non-metallic inclusions, porosities product of the reaction of H2 with C (methane gas bubbles) that produce a concentration of stresses and an increase in plastic flow.

As the deformation process progresses, the existing microcavities grow under the stress conditions at the end of the crack, until they are very close to each other. Finally, the walls or ligaments that separate them break, resulting in a surface fracture characterized by hemispherical or semi-ellipsoidal depressions "cavities" or "dimples". It can be concluded that the A36 specimens in aqueous HCl immersion fractured by a mixed mechanism of cleavage and coalescence of microcavities called quasi cleavage. Intergranular fracture is also present as a microstructural failure mechanism. The fracture is produced by the un-cohesion of the crystalline grains and the grain surface is observed smooth without revealing any type of morphological details or evidence of any plastic deformation. Hydrogen comes from the aqueous HCl solution used in the test, and as the concentration increases and the exposure time increases, the loss of mechanical properties increases. Hydrogen diffuses and accumulates in areas subject to local stresses and deformations, which explains the appearance of secondary cracks in the plane of the fracture and in the area adjacent to it.

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