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EVALUATION OF THE THRESHOLD LINEAR ELASTIC TOUGHNESS IN API 5CT P110 STEEL BY MEANS OF HYDROGEN ASSISTED FRACTURE TESTS

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Abstract. The interaction of materials used in the fabrication of equipment with the environments to which they are put into service frequently causes their degradation. The hydrogen embrittlement presents as a degradation process characterized by the nucleation and propagation of cracks in the materials, being classified as one of the most dangerous to structural integrity, since it can occur suddenly and difficult to perceive, resulting in catastrophic fracture. Therefore, it is necessary to make efforts to improve and develop methods and criteria for material selection, inspection and maintenance of equipment, where the operating conditions favors the occurrence of a environment assisted fracture. For this, fracture toughness tests were performed on API 5CT P110 steel, monitoring the crack growth by means of the alternating current potential drop technique. The tests were conducted in synthetic sea water solution under cathodic overprotection potential. The results showed a important decrease in the fracture toughness of the steel, occurring crack initiation and intergranular crack propagation.

Keywords: Hydrogen embrittlement, potential drop, fracture toughness.

1. INTRODUCTION

The use of fossil fuels, including oil and natural gas, are the predominant forms of energy used on the planet. Although the use of renewable energy will triple in the next 30 years, the world will still remain dependent on these fuels for at least 50% of the energy needed. In order to meet this demand, the oil companies have invested in offshore drilling, since the readily available reserves are gradually depleting.

The offshore exploration of oil trough wells located at great depths under the sea imposes to the materials of structures to severe conditions of temperature, pressure and the effect of corrosive substances, such as CO_2, H_2S, chlorides and organic acids, which participate in the process corrosive behavior of the casing wells. Therefore, it is essential to prevent corrosion of these materials.

The method used to prevent corrosion is called cathodic protection, however when used improperly it promotes the production of hydrogen, which can dissociate, diffuse in steel and generate embrittlement processes, where, together with the presence of corrosive substances, provoke the action of synergistic effects acting as a form of more lethal embrittlement (HARTT and CHU, 2005).

Among the materials used for oil prospecting, we highlight the API 5CT P110 steel, classified as High Strength and Low Alloy steel, used in well casings, drilling, completion and production operations. This steel is a reason for studies due to its high cost and its high susceptibility to hydrogen embrittlement when mainly subjected to cathodic protection in potentials of overprotection

Therefore, in order to achieve higher levels of reliability and safety in the applications of API 5CT P110 steel in the manufacture of pipelines for oil and gas exploration, and to define under what conditions this material can operate safely, a detailed evaluation of the properties and the behavior of this alloy in front of the hydrogen assisted fracture. These evaluation is highly relevant due that it can define the conditions in which the material can operate in safety. In addition, also help in the selection of materials, since it aids in the assessment of the structural integrity to prevent failure of components in service

2. EXPERIMENTAL PROCEDURE

The material used was the of API 5CT P110 steel. C(T) type specimens were used in hydrogen-assisted fracture toughness tests. An alternating current potential dropping equipment (CGM-7) was used to monitor crack initiation in the specimens. The hydrogen-assisted fracture toughness tests were done on two batteries, with three samples each, in order to identify an accurate crack propagation threshold.

The specimens were hydrogenated for 144 hours and remained immersed in synthetic sea water during the test prepared according to ASTM D1141-98 (2008), under potential overprotection of $-1300mV_{Ag/AgCl}$. The loading sequence was started with 5% of the fast fracture load, and for the other specimens 5% of the load obtained during the start of crack propagation was used. The fracture surfaces were analyzed using a scanning electron microscope.

3. RESULTS AND DISCUSSION

3.1 Determination of the beginning of crack growth

After calibration of potential drop equipment, it was possible to identify the-time in which crack initiation occurred through the crack growth curve, Fig.1 (point P_4):
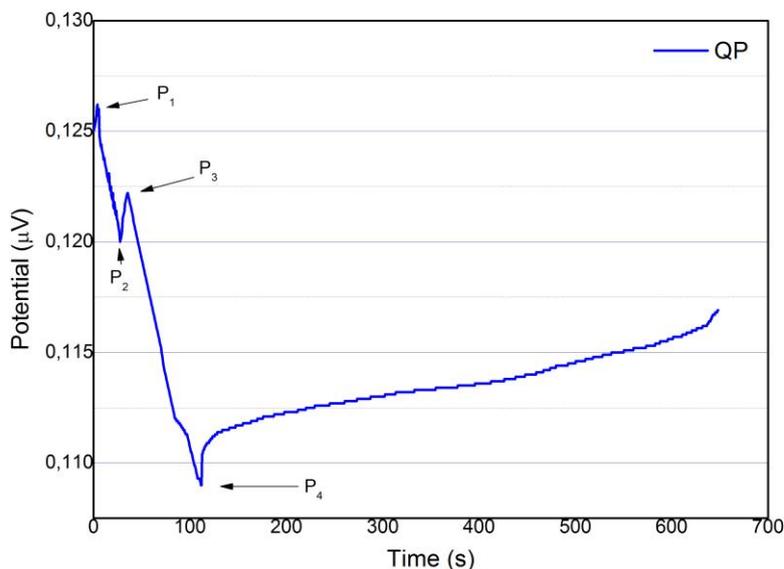

Figure 1. Crack growth curve

Initially, there is a potential decay, about $174\mu V.mm^{-1}$. Okumura et al. (1981) justifies this fact by the effect of plastic and elastic deformation on magnetic permeability and resistivity. The detection of crack initiation is influenced by the excitation frequency of the alternating current, starting at the lowest point of the potential drop, fully dependent on the frequency, due to the decrease in that value.

As can be observed, there is an increase in the potential drop due to elimination in the short circuit caused by the spacing of the pre-crack faces, being indicated by P_1. After a first decay occurs, point P_2, due initially to the elastic deformation at the crack tip, the end of this process is characterized by a slight increase in the potential drop, P_3. Next, the blunting of the crack tip occurs and the formation of a plastic region with the potential drop. Finally, the crack

growth starts when the potential value reaches the minimum point on the graph, P_4, increasing the value of the potential difference. Similar observations were reported by Okumura et al. (1981) and Gibson (1987).

3.2 Hydrogen-assisted fracture toughness tests

The results of the hydrogen-assisted fracture toughness tests, according standard ASTM F1624 (2012), are shown in tables I and II, for the samples tested in the first and second battery, respectivelly. The results are presented as values of K_{IHAC}* corresponding to provisional threshold hydrogen-assisted toughness, K_{IHAC}, obtained in each test. Also shown are the tensile values at the crack initiation time and the crack propagation start load. The total test time is equal to the sum of the time for hydrogen-charge on the specimen, 144 hours, with the time for the complete test.

Table 1. Results of the first battery of tests

	Total Test Time (Hours)	Load in steps (N)	Crack initiation strength (MPa)	Charging initiation (N)	K_{IHAC}* (MPa√m)	Number of passes
CP1	208	1300.00	138.49	10387.12	38.5	8
CP2	294	519.35	132.09	9906.75	36.7	19
CP3	302	495.33	133.95	10049.96	37.29	20

Table 2. Results of the second battery of tests

	Total Test Time (Hours)	Load in steps (N)	Crack initiation strength (MPa)	Charging initiation (N)	K_{IHAC}* (MPa√m)	Number of passes
CP4	206	1300.00	139.81	10485.00	38.92	8
CP5	293	524.25	133.14	9986.076	37.07	19
CP6	296	499.30	126.62	9497.00	35.25	19

As can be seen in table 2, in the second battery, the initiation load presented a decay with the reduction of the pass load, obtaining the minimum load of 9497N and K_{IHAC}* of 35.25 MPa√m. The reduction of the initiation load in the later tests, CP5 and CP6, in the same battery, can be attributed to the longer time in that the sample remained under the effect of the hydrogen generated in the cathodic protection system.

The threshold is calculated from the load of the last pass of the last specimen in each battery, with the pre-initiation load being adopted as the crack propagation threshold. The K_{IHAC} was determined from the average between the two values of threshold load obtained in the two batteries, being this value of 9,523.85 N, as shown in fig. 2.
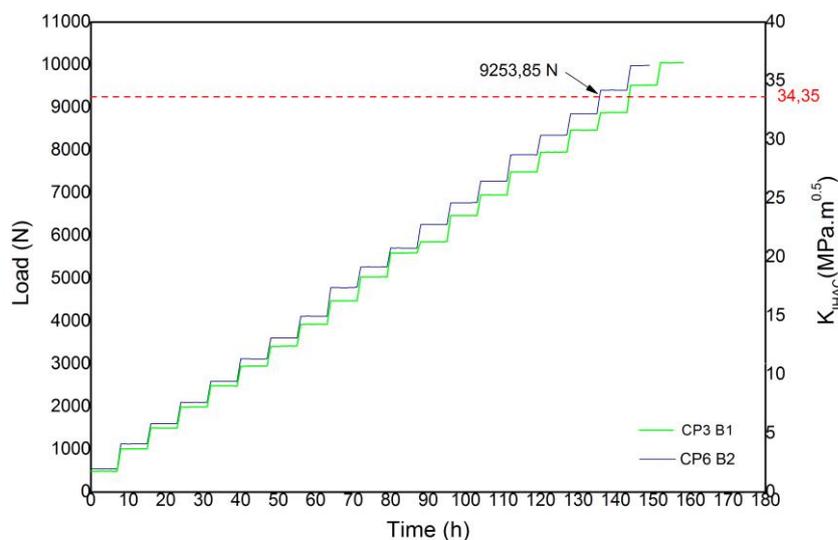
Figure 2. K_{IHAC} determined from the average between the values obtained in the two batteries

In recent work, Carrasco (2013) determined the average critical value of the CTOD (maximum load CTOD), that is, about δ_C= 0.4mm, for an average maximum load, $P_{MÁX}$ = 30,59 kN. The fracture initiation in CTOD test must be occurring for load values between 18 kN – 22 kN. As can be observed, there was a reduction of 53.74% of the load required to initiate the crack propagation in the hydrogen-assisted tests when compared to the average crack propagation start loading in the laboratory environment, around 20 kN This reduction in the crack propagation threshold in the hydrogen-assisted test is attributed to the deleterious effect of hydrogen produced by the cathodic protection, on the mechanical properties of the steel.

The cathodic protection plays an important role as a source of hydrogen, causing a superficial hydrogen charge in the steel and creating favorable conditions for the emergence of embrittlement phenomena, as studied recently by Sanchez et. al. (2016). Negative protection potential values tend to cause a higher hydrogen charge. The potential of -1300mV$_{Ag/AgCl}$ was used in this study, that is, a potential of overprotection, which intensifies the hydrogen embrittlement phenomena. The degradation caused by hydrogen embrittlement tends to be more intense as the concentration of this element increases. The potential of protection studied, guaranteed a large amount of hydrogen available for adsorption.

As a reference for the comparison of the obtained results, we mention the work of Dias (2009), in which the author verified a marked decrease in the fracture toughness assisted by the environment, showing a reduction of 80.31%. It obtained values of fracture toughness for the very reduced superduplex stainless steel. The results obtained by Fonseca (2010) show that it also studied API C110 steel, a great loss of tenacity for the material, approximately 150%, compared to the results obtained in tests carried out in the air at room temperature.

3.3 Validation of K-values by linear-elastic fracture mechanics as K_{IEAC}

During loading, at room temperature, in materials with significant tenacity, plastic deformation occurs in the region ahead of the fatigue crack tip causing bulging of the originally acute crack tip, with the consequent removal of the faces of the same, before the beginning of the crack propagation. This behavior is known as "blunting" of the crack tip. During blunting, the plastically deformed region that develops at the crack tip appears as a stretched area at the fracture surface (Anderson, 2005). In Fig. 03, the fracture surface of the API 5CT P110 steel tested at room temperature in laboratory environment. The fractographic analysis shows the fatigue pre-crack, the blunt zone at the crack tip (crack tip blinding) and the crack propagation zone (ductile micromechanism-dimples) during loading.
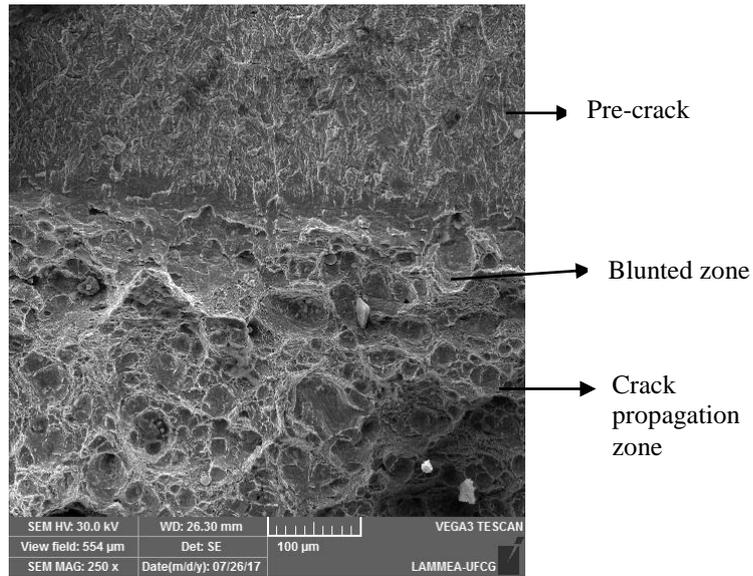
Figure 3. MEV Fracture surface of API 5CT P110 steel from the region of the fatigue crack tip in the air.

In the hydrogen-assisted tests, the absence of the stretched area in the fracture surface is observed (figures 4 and 5). This means that the onset of fracture occurred within the small-scale flow regime. Dias (2009) using the same methodology made valid the K_C values of steel fracture considering the mechanics of linear elastic fracture as K_{IEAC} values due to the absence of the stretched zone.
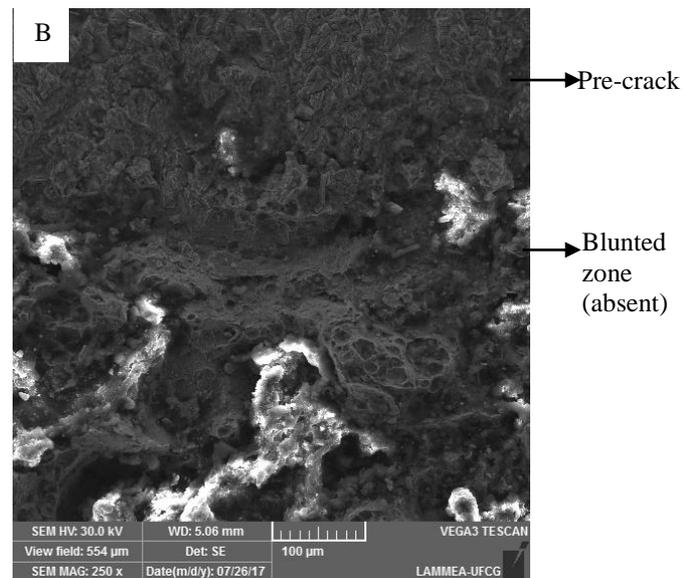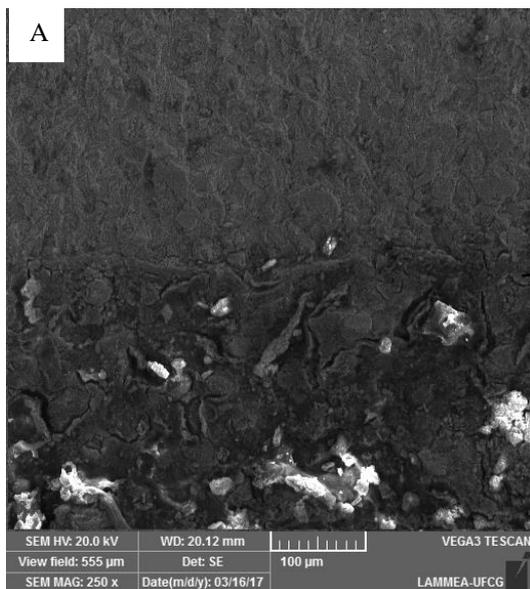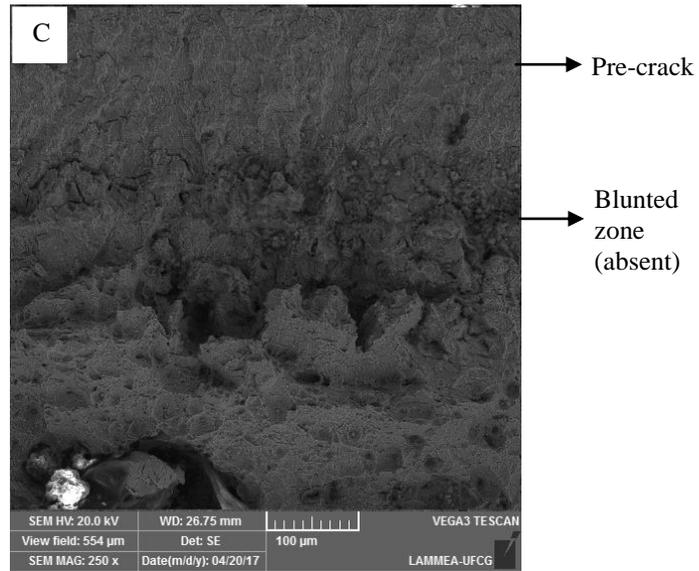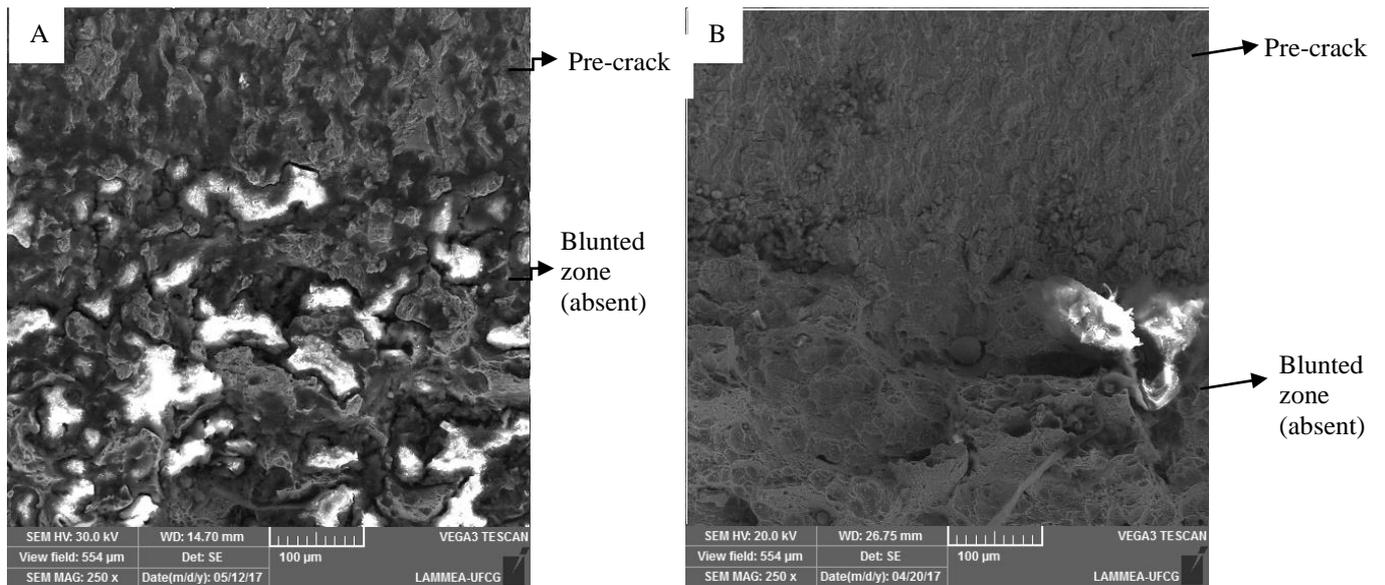
Figure 4. Fractographs of the region of the tip of the fatigue crack of the shows referring to the first battery of tests. (A) CP1. (B) CP2. (C) CP3.
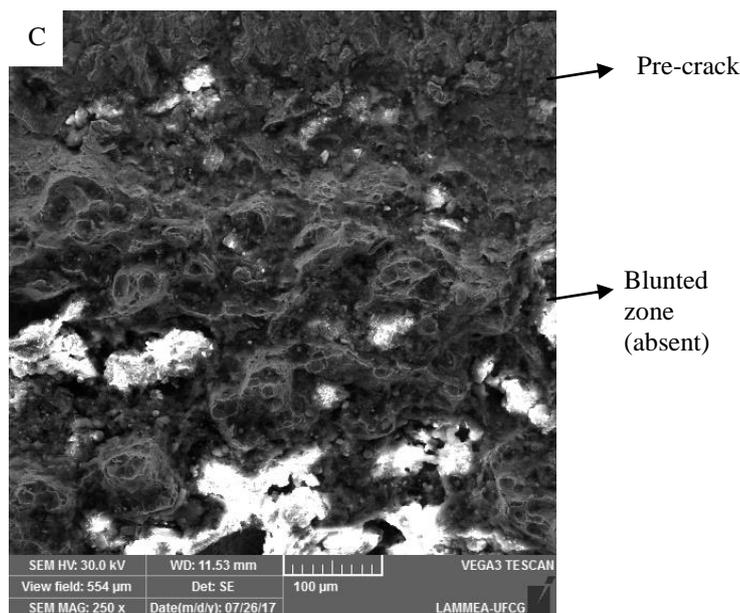
Figure 5. Fractographs of the region of the fatigue crack tip of the samples referring to the second battery of tests. (A) CP4. (B) CP5. (C) CP6

Due to the observed evidence, the-values obtained in the step loading tests can be validated as K_{IHAC} for the studied API 5CT P110 steel, charged with hydrogen in a cathodic protection system at the potential of $-1300mV_{Ag/AgCl}$, in a load control test.

4. CONCLUSIONS

The initiation of cracks occurs when the potential value reaches a minimum point, being noticeable only for low frequencies. The variation in the potential increases only when the faces of the pre-crack are removed, being evidenced the elimination of the short-circuit with consequent increase in the potential.

It was inferred that the fracture of the steel occurred in the elastic linear regime and that the K_I values were obtained through the step loading tests and validated as values of K_{IEAC} in the studied environment, named K_{IHAC}.

There was an expressive reduction in the crack propagation threshold of the steel when assisted by hydrogen due to the embrittlement by hydrogen. The K_{IHAC} value was determined to be 34.35 MPa.$\sqrt{m}$, corresponding to 46.26% of the initiation fracture toughness (in air) value obtained by Carrasco (2013).

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