A STUDY OF INTERNAL HYDROGEN EMBRITTLEMENT AND ENVIRONMENTAL HYDROGEN EMBRITTLEMENT OF API 5L X60 STEEL

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Abstract.. *The pipelines that are used in the petroleum industry transport gas and oil with content of sulfidric gas that intensifies the adsorption of hydrogen in the metallic surface provoking the absorption of hydrogen in the wall of the pipe, what can flow in a embrittlement process. Additionally during pipe production and welding processes can be given the conditions that facilitate the entrance and permanence of the hydrogen in the materials. That hydrogen can weaken most of the structural alloys and under certain conditions and depending on the source, one of those degradation phenomena denominated hydrogen embrittlement, can happen in the forms of Internal Hydrogen Embrittlement and Environmental Hydrogen Embrittlement. In this work an experiment was carry out for to the determination of the mechanical behavior of a HSLA API 5L X60 steel under tensile loading and environmental assisted conditions, with the purpose of studying the internal hydrogen embrittlement and the environmental hydrogen embrittlement in that steel. Potenciodinamic polarization tests were accomplished, where was determined the potential and current density for hydrogen generation in the metallic surface when subjects an of NaOH atmosphere. Starting from data of diffusivity and solubility of the hydrogen in that material, the hydrogenation of the specimens was accomplished in defined times by the solution of the Fick's 2nd law for the appropriate boundary conditions. The tests in specimens without hydrogen were compared with tests in hydrogenated specimens. The results showed changes in the mechanical behavior of the steel embrittled by environmental hydrogen and internal hydrogen, attributed to the presence of hydrogen in the material. A fractographic analysis revealed that the fracture mode is predominantly ductile for dimples in both environmental hydrogen embrittlement and internal hydrogen embrittlement cases, however, the quasi-cleavage fracture was observed in the steel embrittled by environmental hydrogen. Those results are in agreement with the expected effects and reported in the scientific literature for this type of steels.*

Keywords*: Hydrogen Embrittlement; Potenciodinamic polarization; HSLA API 5L X60 steel.*

1. INTRODUCTION

In the current technological way great interest exists in understanding the role of the hydrogen when interacting with metals and metallic alloys and of the damage produced by that interaction. In the industrial sectors affected for the hydrogen degradation, the petrochemical sector is one of the most prejudiced

The transport of oil and gas can induce failures in the pipeline systems, due to that those products commonly contains a wide variety of corrosive environments such as entrained water, CO_2 and H_2S. This last one intensifies the adsorption of hydrogen in the metallic surface, facilitating their absorption in the wall of the pipe, could generate a embrittlement process. Each year, many million of dollars are expended to replace or repair pipes that suffer Environmental-Assisted Cracking (EAC) processes, either Stress Corrosion Cracking or Hydrogen-Assisted Cracking. Inspection programs indicate that 25% of equipment failures in the petroleum refining industry are in some way associated with hydrogen damage (Bezerra *et al*., 1995).

The degradation of mechanical properties as a result of hydrogen interaction with metals and metallic alloys is manifested of diverse ways: blisters on the surface of the material or internal cracks due to *Hydrogen-Induced Cracking*, fisheyes due to *Hydrogen Attack*, failure in non-ferrous based materials structures for *Cracking from Hydride*

Formation, failure due to residual hydrogen in *Cracking from Precipitation of Internal Hydrogen* and catastrophic cracking of high strength steels due to *Hydrogen Embrittlement* (ASM, 1978).

Hydrogen Embrittlement is of particular interest in high strength steels, because their fracture resistance is largely affected by hydrogen concentration in trapping sites contained by their microstructure. It has been established that, as larger is the mechanical resistance of a steel, as larger is their susceptibility to that degradation (Interrante, 1982; Hardie *et al*., 2006). Additionally, the wide use of cathodic protection systems in high strength pipeline steels does increase the probability of Hydrogen Embrittlement (Cwiek, 2005).

The presence of atomic hydrogen in the metallic lattice under either residual or applied stresses affects the ductility and the strength of the steels negatively. Depending on the source, Hydrogen Embrittlement has been classified into two forms: Environmental Hydrogen Embrittlement (EHE) and Internal Hydrogen Embrittlement (IHE) (Tiwari *et al*., 2000; Symons, 2001).

EHE takes place through adsorption of molecular hydrogen generated in a hydrogenated atmosphere or during a corrosion reaction, and its absorption within the lattice after dissociation into atomic form. IHE, in contrast, takes place in the absence of a hydrogenated atmosphere and is brought about by hydrogen which has entered the lattice during processing or fabrication of steel, i.e., prior testing or service. That entrance is facilitated in the metallurgical process because the hydrogen solubility in the molten metal is much higher than when it is in the solid condition. Another source of hydrogen during the manufacturing process is the galvanizing process or flash pickling. Once into the lattice, hydrogen embrittles the steel over a period of time which is a function of concentration, temperature and state of stress within the matrix (Woodtli & Kieselbach, 2000; Eliaz, 2002).

High strength low alloy (HSLA) steels are widely used for structural applications to oil and gas transport where high internal pressures are expected. The work carried out in recent years to develop grades of pipeline steels with larger strength (API grades X60, X80 and X100) for use in oil and gas transport, too increase the probability of possible problems with hydrogen embrittlement, because it was verified that increasing strength levels tend to decrease the resistance of steels to hydrogen embrittlement (Hardie *et al*., 2006). According to Beidokhti *et al*. (2009) the high strength of pipeline steels generally involves low hydrogen embrittlement resistance, making critical the understanding of the susceptibility of these steels to hydrogen embrittlement for a safe selection and use in hydrogenated environments. Therefore, in order to ensure the pipeline steels performance, hydrogen degradation mechanisms should be identified, and should be carried out hydrogen permeability evaluations as well as hydrogen embrittlement susceptibility tests.

In the present work an experimental investigation was carried out on the mechanical behavior of a API 5L X60 steel under tensile loading and environmental assisted conditions, with the purpose of studying the response of these steel to IHE and to environmental hydrogen embrittlement.

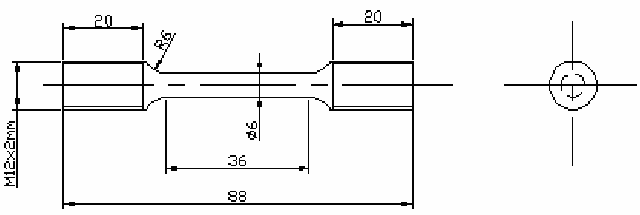
2. EXPERIMENTAL PROCEDURES

This study was performed using the API 5L X60 steel with chemical composition shown in Table 1. Specimens of this steel were hydrogenated and subjected to uniaxial tensile tests until fracture. To reproduce experimentally the two types of hydrogen embrittlement condition previously described, two different procedures for hydrogenation were employed. The specimens were extracted from the wall of a pipe already conformed and were machined in the direction of lamination. The geometry and dimensions of the specimens, named Type I and Type II, are shown in Fig. 1.

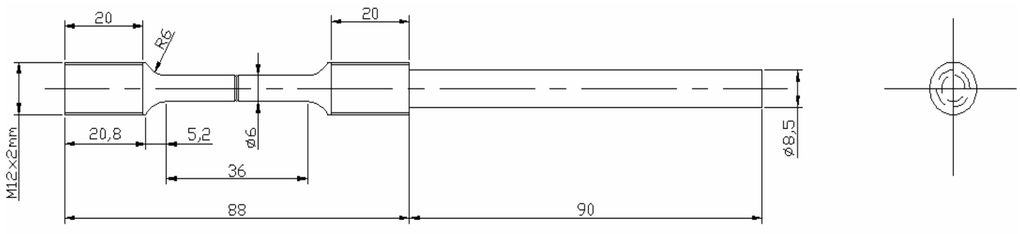
Table 1. Chemical composition of API 5L X60 steel (Albuquerque, 2004).

C%	Si%	Mn%	P%	S%	Mo%	V%	Al%	Nb%	Ti%	Cu%
0,12	0,27	1,48	0, 012	0, 008	0, 032	0, 048	0, 039	0, 041	0, 009	0, 006

Initially, it was carried out a potentiodynamic polarization test to determine the potential and current density for generation of hydrogen. After determining these parameters and from data of diffusivity and solubility of API 5L X60 steel (Vianna, 2005), the times were estimated for saturation of hydrogen inside the material.



(a)



(b)

Figure 1. Geometry and dimensions (in mm) of the specimens. (a) Type I to reproduce environmental hydrogen (EHE) and (b) Type II to reproduce internal hydrogen embrittlement (IHE).

To reproduce the EHE condition, the hydrogen was permeated through the Type I due to the gradient concentration between the surface and the inner alloy matrix. The hydrogen was produced on the specimen surface from potentiostatic condition utilizing a power supply (Icel-PS7000) at room temperature with the employ of a 0,1M NaOH solution. A Pt Fisher electrode was utilized as anode. The potential to produced hydrogen was obtained from the potentiodinamic polarization curve utilizing a three electrode cell and a PAR multichannel-potentiostat model VMP3. The load of hydrogen was performed for 23 h in absence of mechanical stress. After hydrogenation, the Type I were immersed in a copper sulfate solution to impede the hydrogen desorption. This way, the hydrogen concentration in the material was approximately uniform, as it happens in the material of the structures that work in hydrogenated atmospheres or in the presence of promoters of the formation of hydrogen, staying like this until the beginning of the tensile tests. This process was carried out at Electrochemical Engineering Laboratory - LEEq of UFCG.

On the other hand, to reproduce the IHE condition, the hydrogen was introduced cathodically at room temperature using a 0,1M NaOH solution and a same potential employed in EHE tests. The experimental setup was used according with the charging methodology proposed by Tiwari *et al*. (2000): a) employ of a Type II with a extended end portion, that forms the cathode of the electrolytic cell used in the hydrogen load; b) hydrogenation performed under tensile stress of 65% yield strength for 52 h to allow the hydrogen presence in the lattice above of solubility limit; c) stabilization of the hydrogen concentration in the material for 24 hours additional to the hydrogenation time, to make possible that at the end of that period most of the hydrogen introduced into specimens, above of the solubility limit, diffuse out of the lattice and that the residual hydrogen stays strongly bounded to the irreversible trapping sites of the steel. In this process, the hydrogen was generated in the extended end portion of specimen and the hydrogenation was accomplished by diffusion due to a concentration gradient and due to a stress gradient created by a notch present in the middle of specimen. It is believed that during internal hydrogen embrittlement, once the hydrogen enters in the lattice remains inactive until that the environmental conditions allow its transport to the stressed region. This hydrogenation under stress process was carried out in the fixture developed by Costa (2008) shown in Fig. 2.

The uniaxial tensile tests at room temperature under low strain rate were performed using a universal testing machine INSTRON model 5582, according to the recommendations of ASTM E8M standard. In the case of tests for EHE was utilized the Type I show in Fig. 2(a); in the case of test for IHE was utilized the Type II show in Fig. 2(b); in both the cases, the strain rate applied was 2.5×10^{-5} s^{-1}. For each condition were conducted three tests and obtained a average value of parameters. To evaluate the hydrogen influence in the fracture mode, the fracture surfaces of specimens were studied by scanning electron microscopy (SEM) at Center for Studies and Research in Materials – NEPEM/UFPB. This tensile tests and the IHE hydrogenation process it was carried out at Multidisciplinary Laboratory of Active Materials and Structures (LaMMEA) of UFCG.

Figure 2. Fixture used for the hydrogenation process in IHE (Costa, 2008)

3. RESULTS AND DISCUSSIONS

3.1. Potentiodynamic polarization

The potentiodynamic polarization curve is show in the Fig. 3. The curve analyzes shows the potential for hydrogen produced was -1,2V/SCE approximately.

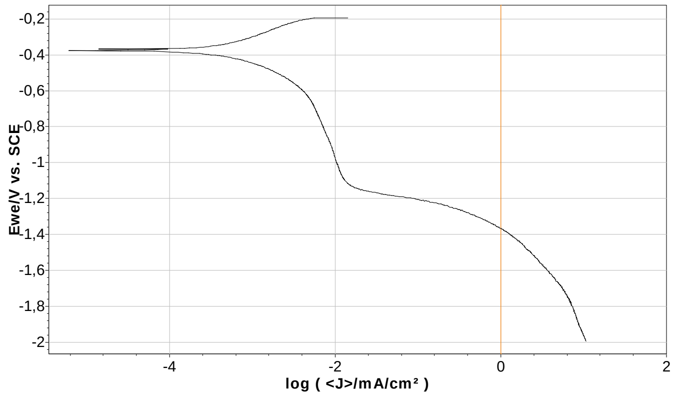


Figure 3. Potentiodynamic polarization for API 5L X60 steel.

These results are consistent with the reported by Vianna (2006).

3.2. Tensile tests

3.2.1. Environmental hydrogen embrittlement

Figure 4 show the stress-strain curves for non-hydrogenated and hydrogenated (for 23 hours) Type I. In them can be observed that the hydrogenated steel had an increase in ductility and a decrease in yield strength (σ_Y) and ultimate tensile strength (σ_{UTS}). This indicates that the steel in hydrogenated condition has a softening.

The effect of hydrogen on the deformation behavior of the material is increased ductility, which increases from an average value of 19.70% for the specimen without hydrogen, to 20.15% for specimen hydrogenated for 23h. In percentage terms, this represents an increase of approximately 2.28%, which is small. Additionally, is observed a decrease in the yield limit, of 7.99% in percentage terms (from 464.24 MPa in the condition of the steel as-received to 427.11 MPa in the hydrogenated steel) and 2.57% in the strength limit. The change in the strength limit is very small, but the change in the yield strength is relevant and evidence the softening suffered by the steel. It is likely that the results are subject to the time of hydrogenation. The Table 2 shown the summary of mechanical properties of API 5L X60 steel for Type I without and with hydrogen.

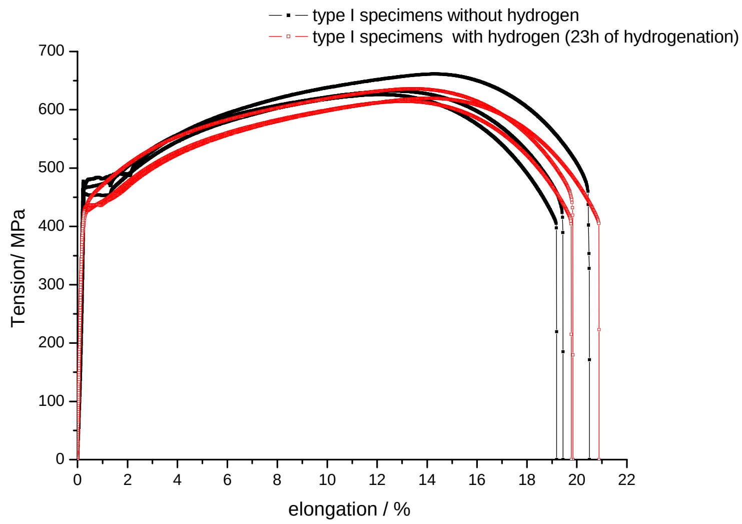


Figure 4. Stress–strain curves plotted for API 5L X60 steel Type I without hydrogen (black points) and with hydrogen charged for 23 h (red points).

Table 2. Mechanical properties of API 5L X60 steel for Type I specimens without hydrogen.

	Type I - without hydrogen	Type I - with hydrogen
σ_{YE} (MPa)	464.24 ± 14.55	427.11 ± 7.71
σ_{UTS} (MPa)	639.97 ± 18.89	623.50 ± 11.08
ε (%)	19.70 ± 0.69	20.15 ± 0.62
R.A. (%)	72.73 ± 0.82	72.66 ± 2.85
Effective modulus	213.77 ± 0.53	200.41 ± 15.33

The results obtained from the stress-strain curves of engineering are very similar to the results in the work of Vianna (2005), i.e. reduction in the limits of yield strength (σ_Y), ultimate tensile strength (σ_{UTS}) and slight increase in ductility. But these results aren't common for other API grades HSLA steels, as shown in the scientific literature for these materials.

The susceptibility of HSLA steels of diverse API specifications to effects of the hydrogen was investigated by many researchers: Hardie, *et al*. (2006) worked with HSLA X60, X80 and X100 grade steels, Trasatti *et. al*. (2005) in a X80 steel, Beidokhti *et al*. (2009) in a X70 steel, Cwiek (2005) in a X80 steel, Torres-Islas *et a*l. (2005) in a X70 steel, Fang *et al*. (2006) in a X70 steel, Vianna (2005) in a X60 steel, Domizzi *et al.* (2001) in eight steels, the X-60 steel includes, and others.

Trasatti *et al*. (2005) showed that if the steel is deformed under cathodic charging conditions, it has a high susceptibility to embrittlement by hydrogen, this in turn directly affecting the ductility of steel. Furthermore, the lower the strain rate greater the susceptibility to embrittlement by hydrogen. Hardie *et al*. (2006) showed that there is a distinct susceptibility to loss of ductility after hydrogen charging and this tends to increase with the level of resistance of steel depending of the current density charging of hydrogen. Cheng (2007) also showed the susceptibility to hydrogen embrittlement of X70 steel due to the decrease in ductility in tests at low strain rates.

In general, these works reported loss of the ductility in all of HSLA steels studied. In the work of Domizzi *et al*. (2001) no reference exists on the effect of the hydrogen in the ductility of the steel.

3.2.2. Internal hydrogen embrittlement

Figure 5 show the stress-strain curves for non-hydrogenated and hydrogenated (for 52 hours) Type II. In them can be observed that the hydrogenated steel had a loss in ductility and an increase in yield strength (σ_Y) and ultimate tensile strength (σ_{UTS}).

The effect of hydrogen on the deformation behavior of the material is the decrease in ductility (or notch ductility), which decreased from an average of 8.08% for Type II no-hydrogenated until 7.52% for Type II hydrogenated during 52h, behavior that is similar to that reported in the literature for steels in general. This loss of ductility is strongly associated with the presence of atomic hydrogen in the microstructure of the material and the way it was introduced in the material. In the case of this phenomenon can't be found articles about HSLA X60 steels. Studies on maraging steel and mild steel (Tiwari *et al*. 2000) indicated that the material on these conditions the hydrogenation suffers a loss of ductility followed by a hardening, evidenced by the increase in the effective modulus. In percentage terms, the value represents a decrease of approximately 6.93%. It is likely that the obtained value is subject to the method of hydrogenation, under tension, which allows a greater amount of hydrogen is trapped in irreversible trapping sites. The decrease in ductility obtained in this Type of hydrogenation is more relevant than the increase observed in the charging with hydrogen environment. Table 3 shown the summary of mechanical properties of API 5L X60 steel for Type II without and with hydrogen.

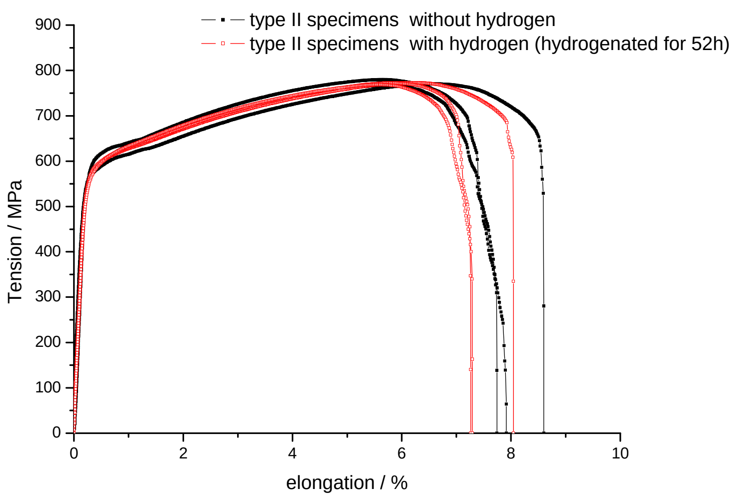


Figure 5. Stress–strain curves plotted for API 5L X60 steel Type II without hydrogen (black points) and with hydrogen charged for 52 h (red points).

Table 3. Mechanical properties (at the notch) of API 5L X60 steel for Type II in the as-received and hydrogenated conditions.

	Type II - without hydrogen	Type II - with hydrogen
σ_{UTS} (MPa)	772.39 ± 6.66	771.36 ± 2.37
ε (%)	8.08 ± 0.44	7.52 ± 0.43
Effective Modulus (GPa)	275.19 ± 22.39	259.71 ± 8.97

3.2. SEM fractographic characterization

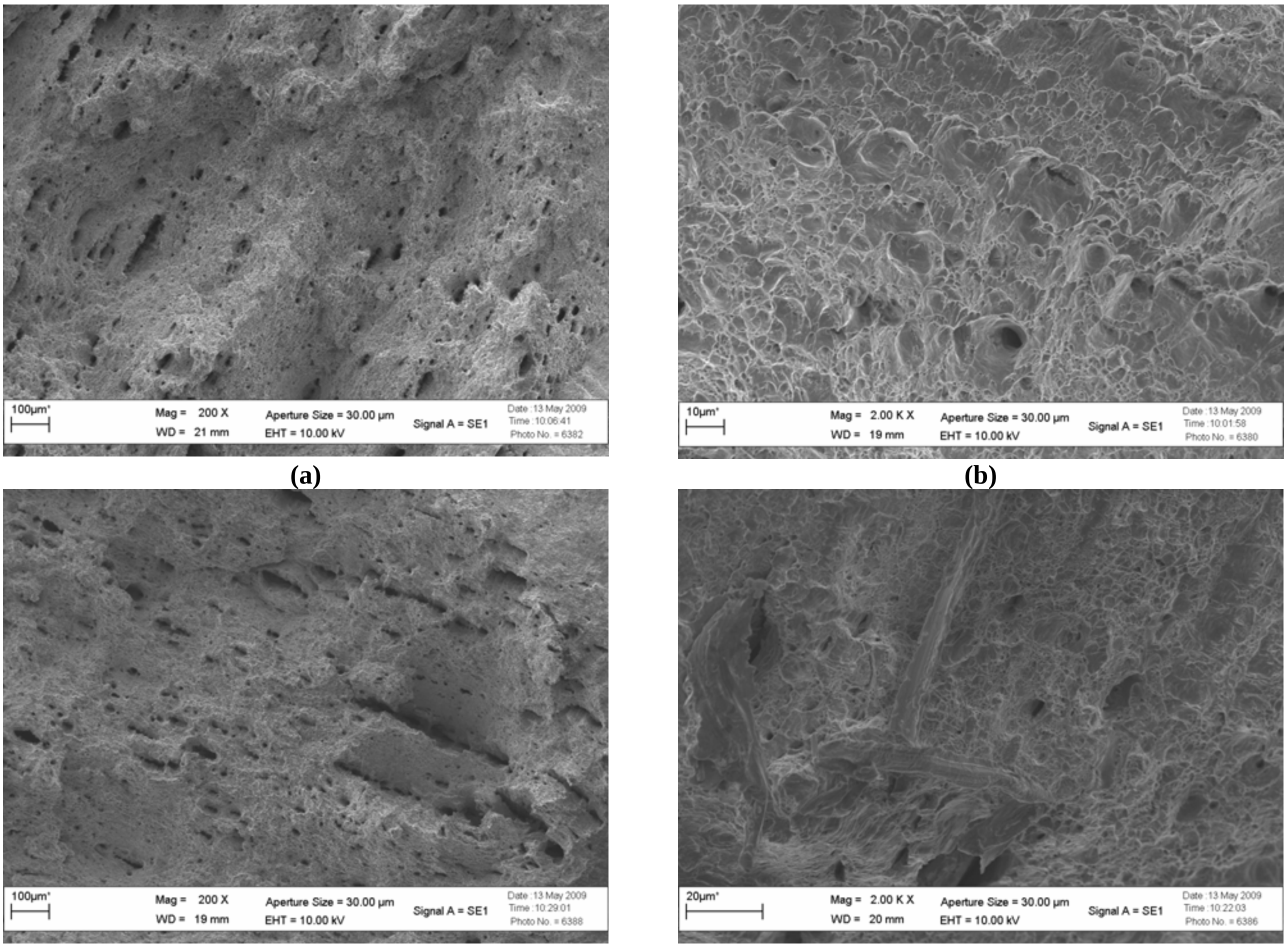
3.2.1. Environmental hydrogen embrittlement

Figures 6(a) and 6(b) show the images of the fracture surface of the Type I free hydrogen material. In them is observed that the mode of fracture is predominantly ductile by dimples rupture, which is characteristic of the fracture behavior of these steels in the test conditions.

Figures 6(c) and 6(d) show the images obtained for the Type I with the hydrogenated material, where it notes that the predominant mode of fracture is dimples rupture too, and the dimples found on the fracture surface are smaller size to those found in the material without hydrogen. The small dimples are formed by the activation of a large number of nucleation sites and adjacent microvoids coalesce before having the opportunity to grow to larger sizes.

This effect is caused by the presence of large amounts of hydrogen atoms in the material introduced in the hydrogenation process used, resulting in dimples that "break" formed before the formed in the free hydrogen material. That is why the hydrogenated material presents a larger number of dimples per unit area.

In Fig. 6(d) regions are observed with the morphology of the two types of fracture, ductile and cleavage, associated with the presence of quasi-cleavage fracture, characteristic of embrittled materials. Despite the fact that a condition for the existence of such behavior is that the plastic deformation is prevented, the overall effect of hydrogen on the behavior of hydrogenated specimens, given the type of hydrogenation used, was to cause the softening of the material, which was perceived by increasing the ductility. The microvoids increase the plasticity due to the reduction of internal tension and the average number of pile-up dislocations (Vianna, 2005).



(a) **(b)**

(c) **(d)**

Figure 6. SEM of fracture surface of API 5L X60 steel Type I: (a) without hydrogen. 200X, (b) without hydrogen. 2000X; (c) with hydrogen. 200X and (d) with hydrogen. 2000X.

3.2.2. Internal hydrogen embritlement

Figures 7(a) and 7(b) show the images of the fracture surface of the Type II free hydrogen material. In them is possible to observe that the fracture mode is predominantly ductile by dimples rupture, which is characteristic of the fracture behavior of this steels in the test conditions.

Figures 7(c) and 7(d) show the images for Type II with the hydrogenated material, where is possible to observe that the predominant fracture mode is ductile.

The effects of hydrogen in the mode of fracture and the size of dimples are indicative of the fact that hydrogen influences the local plasticity. The results of fractographic analysis allow associate effects of hydrogen with the small decrease in the ductility observed in the results of tensile tests shown in table 3.

The results obtained here suggest that the reason for the effects of the action of hydrogen comes from the accumulation of its high concentration within the matrix due to the type of hydrogenation used: it is known that a large amount of hydrogen (much higher than the concentration of equilibrium) can be retained at room temperature in the matrix of some alloys due to the presence of numerous structural defects that trap it. In steels, the main trapping sites are grain boundaries, secondary grain boundaries and some carbides (Tiwari *et al*., 2000).

Therefore, the effect of hydrogen in reducing the ductility for this type of hydrogenation can be attributed to the ability of API 5L X60 steel to dissolve large quantities of hydrogen by trapping, because it contains many elements of alloy like titanium and niobium that have a great affinity for hydrogen.

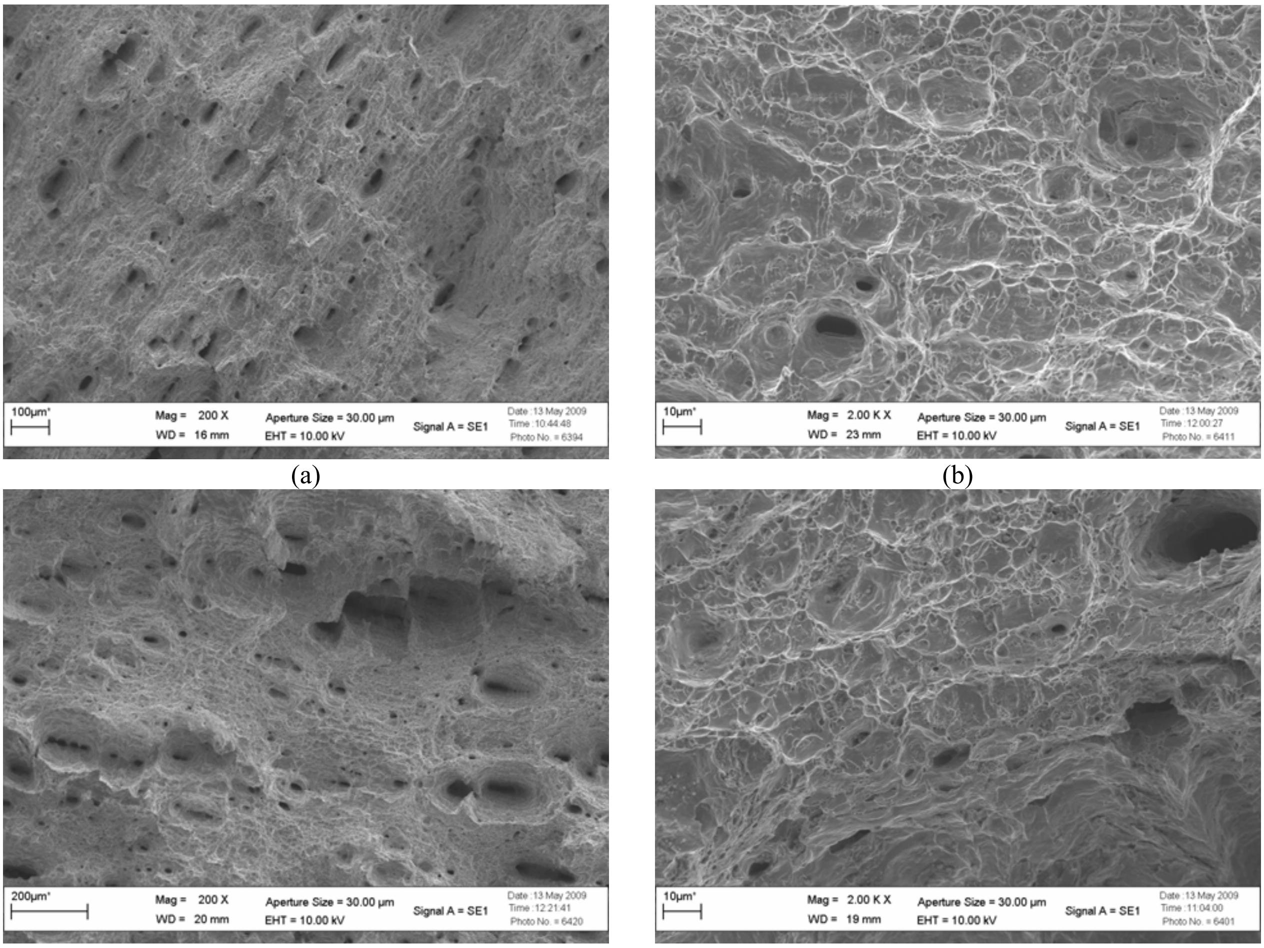


Figure 7. SEM of fracture surface of API 5L X60 steel Type II: (a) without hydrogen. 200X, (b) without hydrogen. 2000X, (c) with hydrogen. 200X and (d) with hydrogen. 2000X.

4. CONCLUSIONS

The morphology of the fracture surface of steel under the two types of hydrogen embrittlement, although predominantly ductile by dimples rupture, has important differences, such as the presence of regions with a quasi cleavage fracture and greater reduction in size of dimples observed in the environmental hydrogen embrittlement.

The mechanical behavior in tensile tests for the specimens tested are different due to the effect of hydrogen on the ductility, which was a slight increased in environmental hydrogen embrittlement. The variation in the ductility was more expressive in the internal hydrogen embrittlement.

The behavior of steel under the two different types of hydrogenation is different by aspect fracture, although predominantly ductile rupture with decreasing size of simples is much more evident in the environmental hydrogen embrittlement.

In the process of EHE was a decrease in yield strength evidencing the softening experienced by steel in the conditions of test.

5. ACKNOWLEDGEMENTS

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