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**Determination of Mechanical Properties of Materials by Small  
Punch and Other Miniature Testing  
Techniques**

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# Rate effects on the estimation of fracture toughness by Small Punch tests in hydrogen embrittlement scenarios

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**Abstract:** In this paper, different techniques to test notched Small Punch (SPT) samples for the estimation of the fracture properties in aggressive environments are studied, based on the comparison of the micromechanisms at different rates. Pre-embrittled samples subsequently tested in air at conventional rates (0.01 and 0.002 mm/s) are compared to embrittled ones tested in environment at the same rates (0.01 and 0.002 mm/s) and at a very slow rate (5E-5 mm/s); a set of samples tested in environment under static loads that produce very slow rates complete the experimental results. As a conclusion, is recommended to test SPT notched specimens in environment at very slow rates, of around E-6 mm/s, when characterizing in Hydrogen Embrittlement (HE) scenarios, in order to allow the interaction material-environment to govern the process.

**Keywords:** Small Punch Test; hydrogen embrittlement; punch rate, fracture toughness

## 1. Introduction

A critical aspect concerning high strength steels is their resistance to Stress Corrosion Cracking (SCC) and Hydrogen Embrittlement (HE) phenomena, both of which lead to degradation of the mechanical properties of these steels when facing aggressive environments [1,2]. The effect of hydrogen is especially significant in high-strength steels exposed to aqueous environments under cathodic protection (such as off-shore platforms) or those typical of H<sub>2</sub>S presence (as in gas transport pipelines). Both phenomena, HE and SCC, are similar, resulting in brittle failures in the presence of an aggressive environment and maintained stress. Both phenomena are dependent on the crack deformation rate, and may even disappear at very high rates, while at very slow strain rates hydrogen continues to exert an embrittling effect [3]. The recommendations presented by various research groups over the last few decades have been collected in the standard ISO 7539 [4]. It establishes requirements concerning specimen size and solicitation rate, but does not specifically define the procedure to follow in numerous applications. Also, there are particular situations where standards cannot be followed to perform characterizations on in-service components, mostly due to the impossibility of machining specimens fitting the dimensions, or mainly the thickness required. One of those situations is usually present in the welded joints of any type of structure.

To find a solution for these types of scenarios, among these alternative techniques, the Small Punch Test (SPT) is one of the most notables [5]. The SPT, based on punching a reduced dimensions plane specimen, allows the estimation of parameters such as the yield stress, ultimate tensile strength and fracture toughness of metallic materials with high reliability. Over the last years, some authors have proved the validity of the SPT when used in HE and SCC characterization [5-12]. In order to reproduce accurately the micromechanisms taking place in HE failures, as it is imposed by standardised environmental characterizations, the test rates should be very slow, or even quasi-static [5,11]. The ultimate research in the SPT field [11,12] indicates that tests performed in environment, under static load applied to the specimen, should be one suitable option to reproduce the micromechanisms of real subcritical processes, despite the time and samples requirements are higher.

In this paper a review of all possible SPT testing techniques and rates for its application to HE scenarios is carried out, from pre-embrittled samples tested in air at conventional rates to tests submerged in environment under static load, going by submerged tests at different punch rates from 0.01 mm/s to 5E-5 mm/s.

## 2. Materials and Methods

The material used in this study is a Cr-Ni-Mn high-strength steel, which is employed in the manufacture of large anchor chain links for off-shore platforms. It is obtained by quenching and tempering processes, which give the tempered martensite microstructure showed in Figure 1. This steel is received in the factory in bars, which are then forged to conform the links by bending forces.



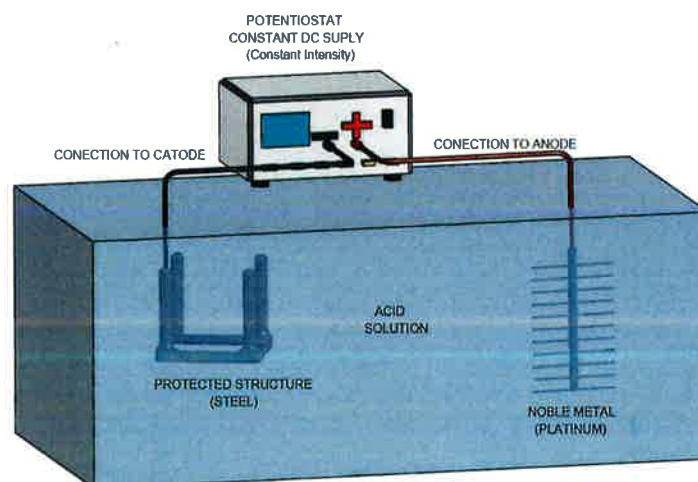
**Figure 1.** Microstructure of the steel used.

Compact specimens for fracture mechanics according to [13] and Small Punch notched samples according to [5,14] were obtained from a chain link for the subsequent labors. Also, in order to determine the mechanical behavior of the material as received [13,15,16] some other tensile and fracture specimens were machined to obtain the results shown in Table 1. The Hydrogen content of the material as received was measured by the hot extraction technique in a LecoR analyser, obtaining the value of 0,84 ppm showed in Table 1.

**Table 1.** Mechanical properties of the steel.

Parameter		Value
Yield Stress	(MPa)	920
Ultimate Stress	(MPa)	1015
Young's Modulus	(GPa)	205
R.O. parameter	N	14,5
R.O. parameter	$\alpha$	1,15
J0,2	(KN/m)	821
KJ0,2	(MPa*m <sup>1/2</sup> )	410
H2 content	(ppm)	0,84

An environmental condition known as cathodic charge (CC), or anodic polarization, has been employed in this study. It is used to protect against corrosion structures that operate in aggressive environments, or to reproduce local situations where a high amount of hydrogen is present. It causes substantial embrittlement on the steel by the action of the hydrogen going through and getting trapped in it.



**Figure 2.** Cathodic charge method. [5,11].

Figure 2 shows a set-up of the method used in this work [5,11]. It consists of the interconnection, via an acid electrolyte, of a noble material (platinum in this case) and the steel, which will be protected due to the fixed current interposed [1,17,18]. In this study, for the cathodic charge situations, an environmental condition in accordance [7,10,11,17,18,19] was proposed, consisting of an 1N H<sub>2</sub>SO<sub>4</sub> solution in distilled water containing 10 drops of CS<sub>2</sub> and 10mg of As<sub>2</sub>O<sub>3</sub> dissolved per liter of dissolution. The solution of As<sub>2</sub>O<sub>3</sub> was prepared using Pessouyre's method [17,19]. A platinum grid was used as an anode. The PH was controlled in the range 0,65 - 0,80 during the tests and at room temperature 20°C - 25°C. An embrittlement level of 5mA/cm<sup>2</sup> was employed. The Hydrogen content of the material resulting from its exposition to this environment was 5,86 ppm (vs 0,84 ppm as received).

The SPT, that is in a pre-standard state, has been successfully employed in the evaluation of tensile [20] and fracture [21] properties of different materials. Because of its reduced dimensions and simplicity, this technique has been applied to characterize embrittlement situation on steels, such as the evolution of materials properties with neutron irradiation [22], the brittle-ductile transition temperature of metallic materials [23], or environmental embrittlement [5-12]. A schematic of the device used in this work for the performance of these tests is represented in Figure 3; during the test the force and the punch displacement were registered continuously.

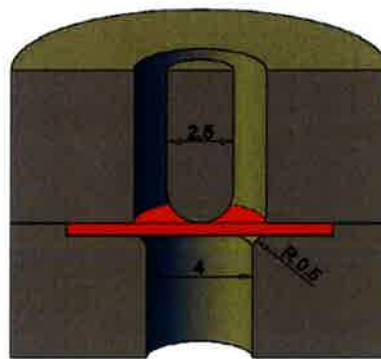


Figure 3. SPT device and samples used.

From SPT tests at standard rates, curves like the ones shown in Figures 4.a and 4.b [24] are obtained. In brittle materials or embrittlement situations (Figure 4.b), the membrane stretching (zone III) does not exist, moving from a yielding plastic behaviour directly to the final plastic instability. It can be observed that while in ductile situations the specimen rupture surface has a semicircular shape and its deflection is higher meanwhile (figure 4.a), in brittle scenarios the breaking typology is a star (figure 4.b) and the specimen deflection lower, so the energy under the register is also lower [5].

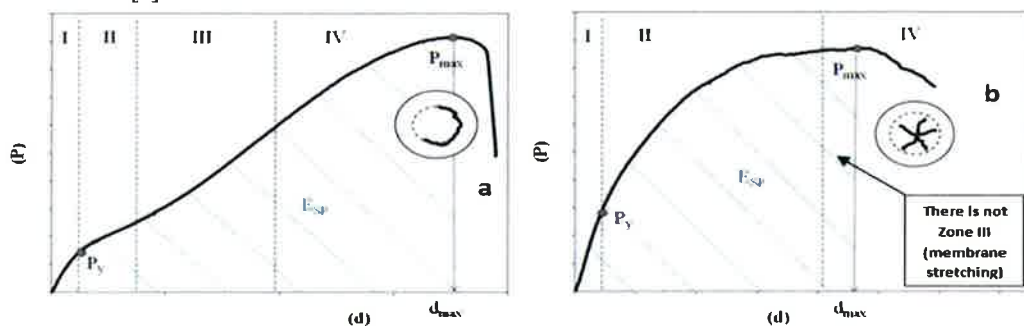


Figure 4. Schematic of SPT curves at conventional rates [24]; (a) ductile materials; (b) brittle scenarios.

When characterizing materials in HE situations the testing rate is an important parameter to take in account [3,5,12], as far as it will govern the micromechanism taking place. In environmental subcritical processes very low rates, or even static tests, are commonly employed [4], being the Slow Strain Rate Tests (SSRT) and the tests under constant load the most widely used [25] for conventional characterizations. By using these testing conditions, hydrogen will have enough time not just to diffuse from reversible traps to the new cracking areas subsequently generated during the test, but also to escape from irreversible traps helped by plastic deformation and diffuse to the new cracking areas [26,27]. The ultimate research for the Small Punch test in HE characterizations [5,11,12] advises that static load tests, or very low punch rates (Slow Small Punch Test, SSPT), should be used, in order to



allow hydrogen to cause all of its embrittling power; in the same way as advised for standardized tests. In [11] the punch displacement vs time SPT curve resulting from static load tests in environment (d-t register) was studied, resulting in the three zones shown in figure 5.

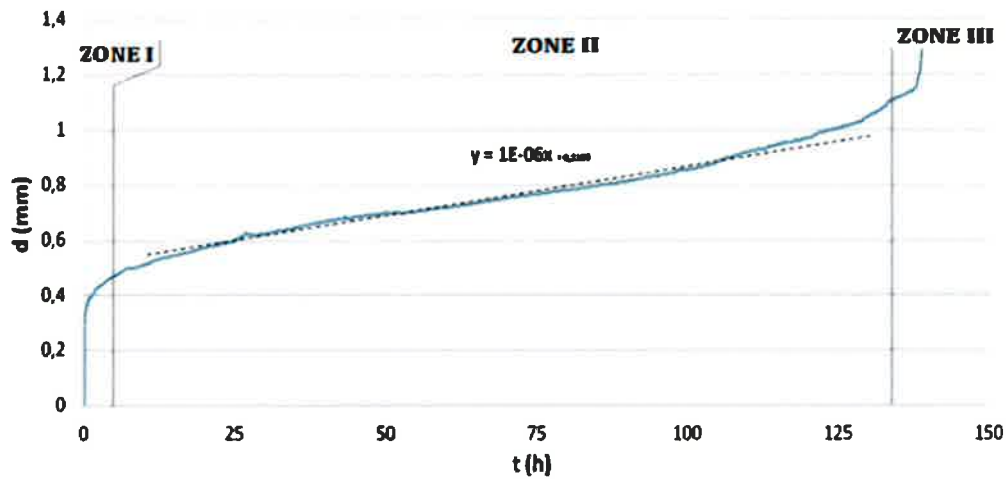


Figure 5. Displacement-time curve for static load tests in environment [12].

- Zone I consists of the punch indentation and settlement.
- In zone II a quasi-constant punch rate takes place, caused by the variation on the flexibility of the system produced by an increasing cracking in the specimen in both radial and thickness directions.
- Finally, in zone III the damage level of the system is so high that the punching load can't be supported anymore and the specimen leads to final instability and fails.

In a first attempt fracture mechanics tests were carried out in the environmental condition described (cathodic polarization at a level of  $5\text{mA/cm}^2$ ) in order to determine the micromechanisms of fracture by SEM images, as well as the  $K_{IEAC}$  value. Prior to the test, the specimens were subjected to hydrogen absorption by exposing them for 48 hours to the same environment and aggressiveness conditions as the test itself, that was performed subsequently by applying the corresponding loading rate using a slow strain rate machine. Two loading rates were employed in order to study their effect, one test was performed at  $6E-9$  m/s of constant solicitation rate and another 10 times faster, at  $6E-8$  m/s, following the recommendations of the Standard ISO-7539 [4]. The methodology proposed by ASTM E-1820 [13] was employed for the  $K_{IEAC}$  value calculation. Figure 6 shows a test while being carried out.

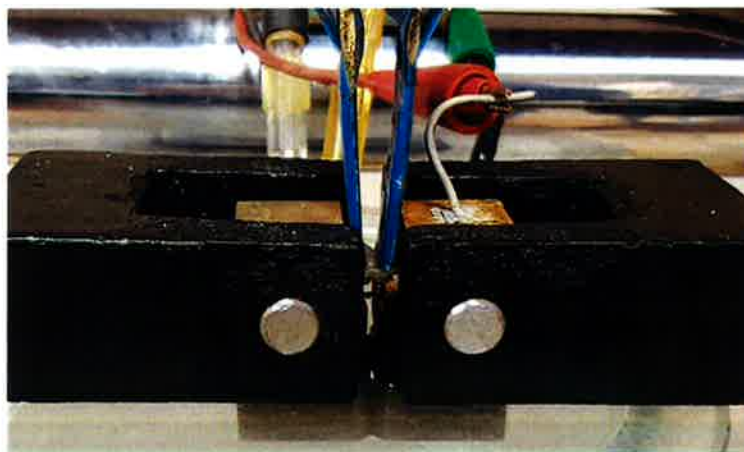
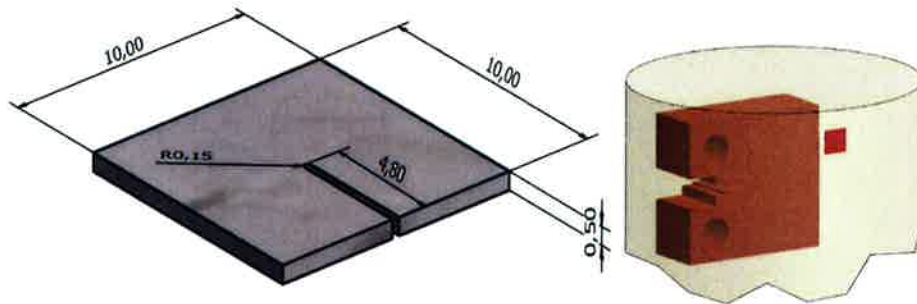


Figure 6. Fracture mechanics test carried out.

The sample geometry employed for SPT, according to [5,14,28,29], is presented on Figure 7, it consists on a plane  $10\text{mm} \times 10\text{mm}$  of section and  $0.5 \pm 0.01\text{mm}$  of thickness including a lateral notch machined by wire electro-



erosion of 0,15 mm radius. The orientation of the notches in SPT and C(T) samples was the same, in order to reproduce the same material orientation in both cases.



**Figure 7.** SPT notched samples employed and its orientation compared to C(T)'s one.

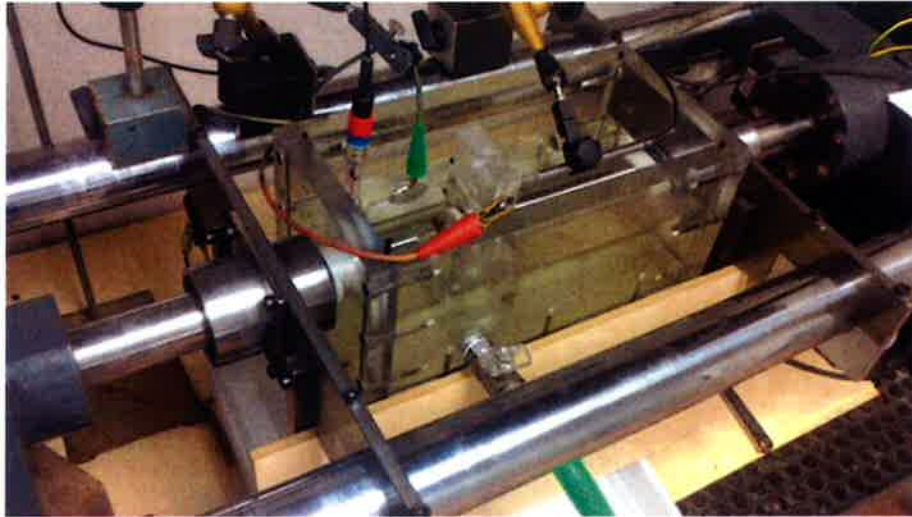
Prior to the test, the specimens were subjected to hydrogen charging by exposing them for 2 hours to the corresponding environment, a period of time considered sufficient for a proper and complete diffusion of the hydrogen inside the material [5], then the mechanical testing was applied. In the case of the SPT, four mechanical testing conditions were employed in order to produce the different punch rates on the sample to be studied:

- Pre-embrittled samples tested in air at the conventional 0.01mm/s punch rate recommended by [14,21] and widely employed by authors, and another rate of 0.002 mm/s, five times slower, in order to compare their effect. For this purpose, the samples were charged, as shown in Figure 8, and immediately extracted, dried and tested in air environment in an electric machine.



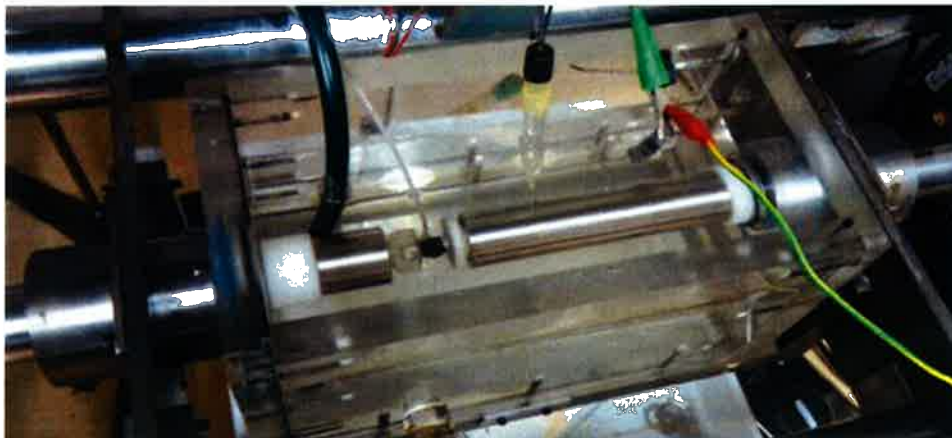
**Figure 8.** SPT pre-embrittled specimens during its hydrogen absorption.

- Embrittled samples tested in continuous exposition to the environment at the conventional rate of 0.01 mm/s, and five times slower of 0.002 mm/s. For this purpose, the samples were charged and tested in a device was specifically designed, built and patented [30] for this purpose that is presented in Figure 9; in this case the punching is applied in the horizontal direction.



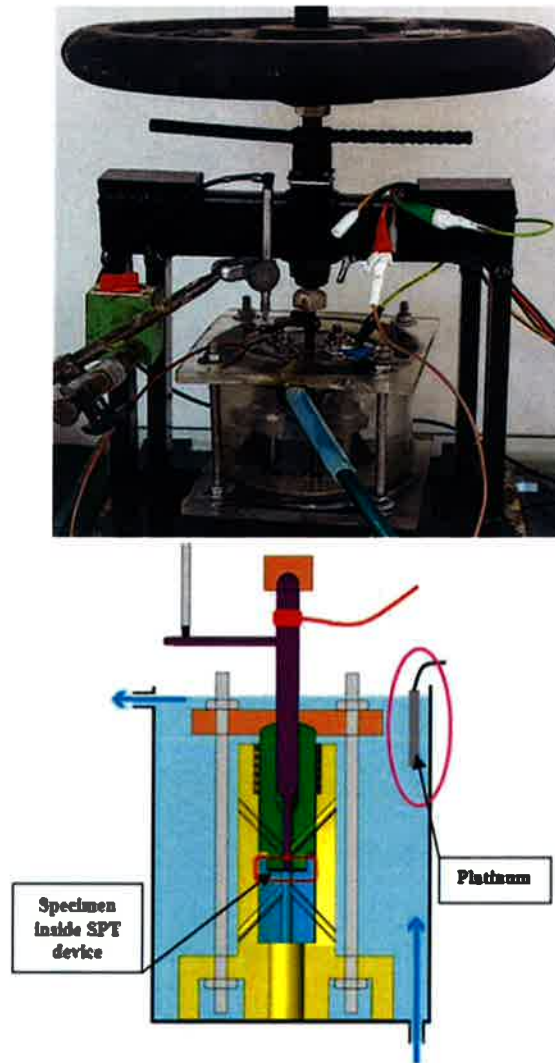
**Figure 9.** SPT specimen while being tested in environment in the device designed.

- Embrittled samples tested in continuous exposition to the environment at a very slow rate, 500 times lower than the conventional rate, of  $5E-5$  mm/s that has been previously employed by some authors [5,6,9,10]. These tests were carried out in the device presented in Figure 9, a detail is shown in Figure 10.



**Figure 10.** SPT device for testing samples submerged in environment.

- Embrittled samples tested in continuous exposition to the environment under static loads. A set of samples were tested using decreasing imposed loads, which produced decreasing punch rates in the zone II of the curve, up to that load that was not enough to produce any cracking departing from the edge of the notch. After embrittling, the load was softly applied by an endless screw system on the specimen subjected to the environment. For the purpose of this test, the experimental device presented in Figure 11 was designed and built.

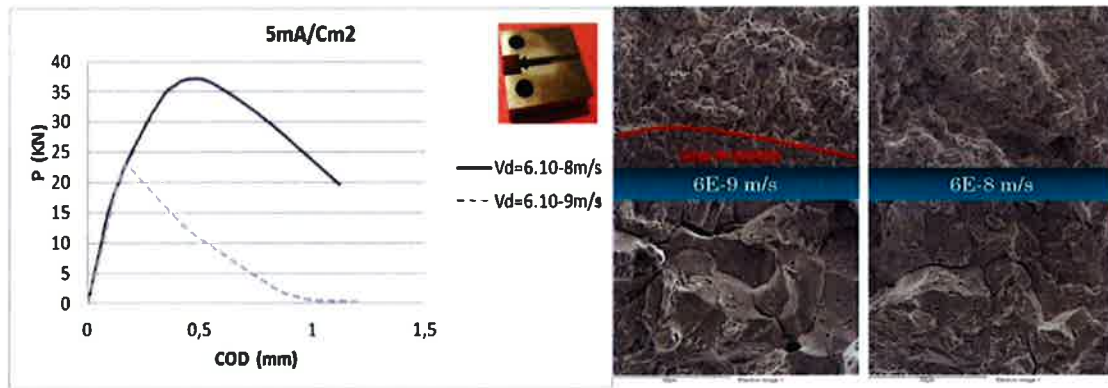


**Figure 11.** Experimental device and schematic for performing SPT tests in environment under static load.

### 3. Results

#### 3.1. Standard Fracture Mechanics tests on C(T) specimens at slow rates

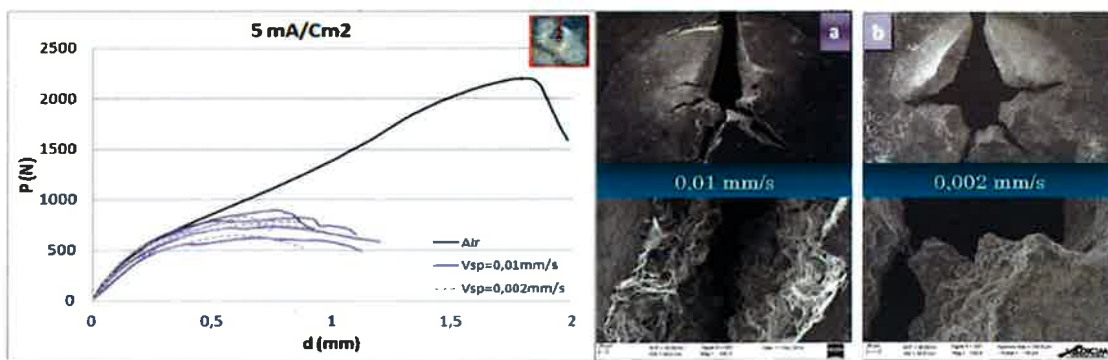
Figure 12 shows the load-COD registers and the fractographic images obtained from the C(T) samples tested in environment. By applying [13,16] values of  $K_{IEAC}=32.62\text{MPa}\cdot\text{m}^{1/2}$  and  $K_{IEAC}=30.08\text{MPa}\cdot\text{m}^{1/2}$  were obtained from the samples tested at  $6\text{E}-8$  and  $6\text{E}-9$  m/s respectively. The curves shape tends to lower maximum loads and COD values (lower energy) when the testing rate is slower. There is just a slight influence on the fracture toughness due to a similar crack initiation micromechanism, as presented in the fractography, but the sample at the lower rate develops more brittle processes during propagation, which explains such a mechanical difference between both curves. The slower the rate, the higher enough the time for Hydrogen trapped in the material to get activated and diffuse to the new cracking areas, allowing all the embrittling capacity of the environment. A clear environmental effect can be appreciated in the material, as a result of brittle fracture in a mixed mode of transgranularity and grain boundaries separation by cracking. It can also be observed a slightly more brittle cracked system in the most aggressive situation.



**Figure 12.** Experimental curves and fractographic images from conventional fracture mechanics tests.

### 3.2. SPT tests on pre-embrittled samples tested in air at conventional rates of 0.01 and 0.002 mm/s

Figure 13 presents the curves and fractography from the SPT tests performed on pre-embrittled samples tested in air at conventional rates (0.01 and 0.002 mm/s). The register from an SPT test of the material as received is superposed for comparison (black line). It can be observed that the exposition to the environment caused an important embrittlement in the material traduced in loss of mechanical properties. The shape of the curve, that was the typical form a ductile material (black line) is now of a total brittle typology. Comparing the curves from tests at 0.01 and 0.002 mm/s, as well as its fractography, a clear difference cannot be found.

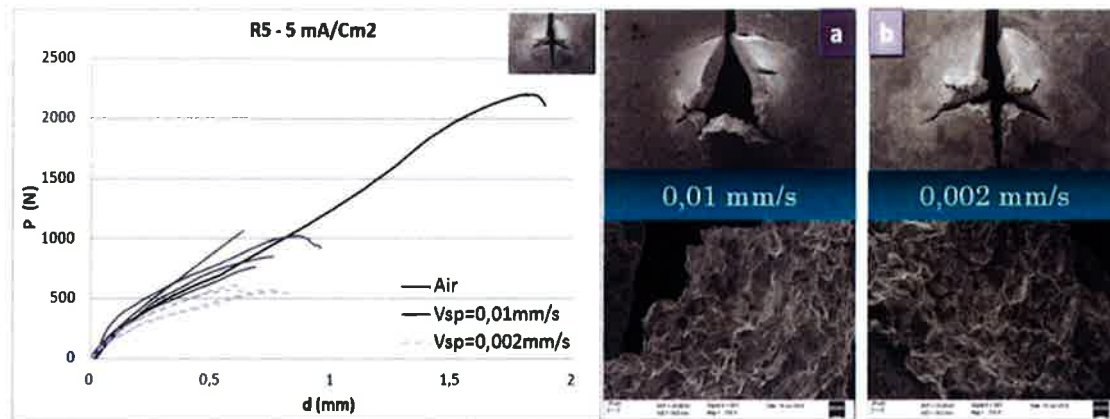


**Figure 13.** SPT curves and fractographic images from pre-embrittled SPT samples tested in air at conventional rates. SPT curve of as-received SPT test is superposed for comparison (in black).

### 3.3. SPT tests on embrittled samples tested in environment at conventional rates of 0.01 and 0.002 mm/s

Figure 13 presents the curves and fractography from the SPT tests performed on embrittled samples tested in environment at conventional rates (0.01 and 0.002 mm/s). The register from an SPT test of the material as received is superposed for comparison (black line). In this case, the environment produced again an important embrittlement, showing the SPT curves a typical brittle shape and a decrease of properties compared to the as received curve (black line). Regarding the fractography for this case, a mixed mode can be observed for both rates (0.01 and 0.002 mm/s), but slight differences can be found between them. The lower punch rate, 0.002 mm/s, shows a more brittle system that presents a slightly higher transgranularity and grain boundaries separation than 0.01 mm/s sample, and it is nearly as brittle as the one presented by fracture mechanics tests (Figure 12).

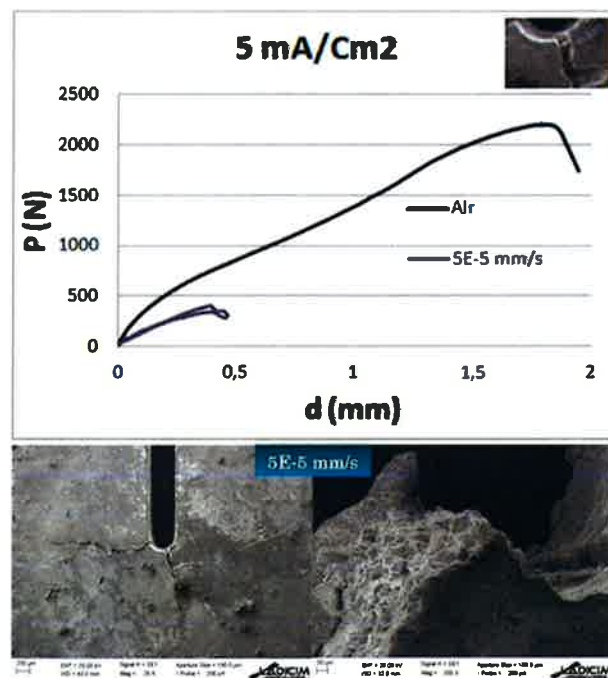




**Figure 14.** SPT curves and fractographic images from embrittled SPT samples tested in environment at conventional rates. SPT curve of as-received SPT test is superposed for comparison (in black).

#### 3.4. SPT tests on embrittled samples tested in environment at very low rate of $5E-5\text{mm/s}$

Figure 15 presents the curves and fractography from the SPT tests performed on embrittled samples tested in environment at a very low rate proposed by [6] ( $5E-5\text{ mm/s}$ ). The register from an SPT test of the material as received is superposed for comparison (black line).



**Figure 15.** SPT curves and fractographic images from embrittled SPT tested in environment at very slow rate.

The registers exhibit once more a brittle typology, that is enhanced by the very slow rate ( $5E-5\text{ mm/s}$ ), which gives the Hydrogen time enough to develop all of its embrittling and damaging power to the microstructure. From the fractographic images, it can be stated that a clearly brittle pattern in a mixed mode is presented, showing transgranularity and grain boundaries separation in a similar magnitude to the one presented on conventional fracture mechanics tests on C(T) samples (Figure 12).

### 3.5. SPT tests on submerged samples tested in environment under static load

Figure 16 shows the registers displacement-time, and presents the macrographic pictures of the samples tested by this methodology. The imposed loads produced a punch rate developed by the flexibility variation of the cracked sample when developing new cracking areas, due to its exposition to the combination of aggressive environment and applied load, as stated in [11]. The two highest loads produced the failure in 5 minutes and less than 4 hours developing rates around E-4 and E-5 mm/s. The rest of the samples reached a quasi-stable zone II of cracks evolution, resulting this in rates in the range of E-6 to E-7 mm/s. Due to the conclusion obtained from Figure 15, where the micromechanism shown seemed to be equivalent to the one presented for conventional fracture mechanics tests, no fractographic analysis were carried out.

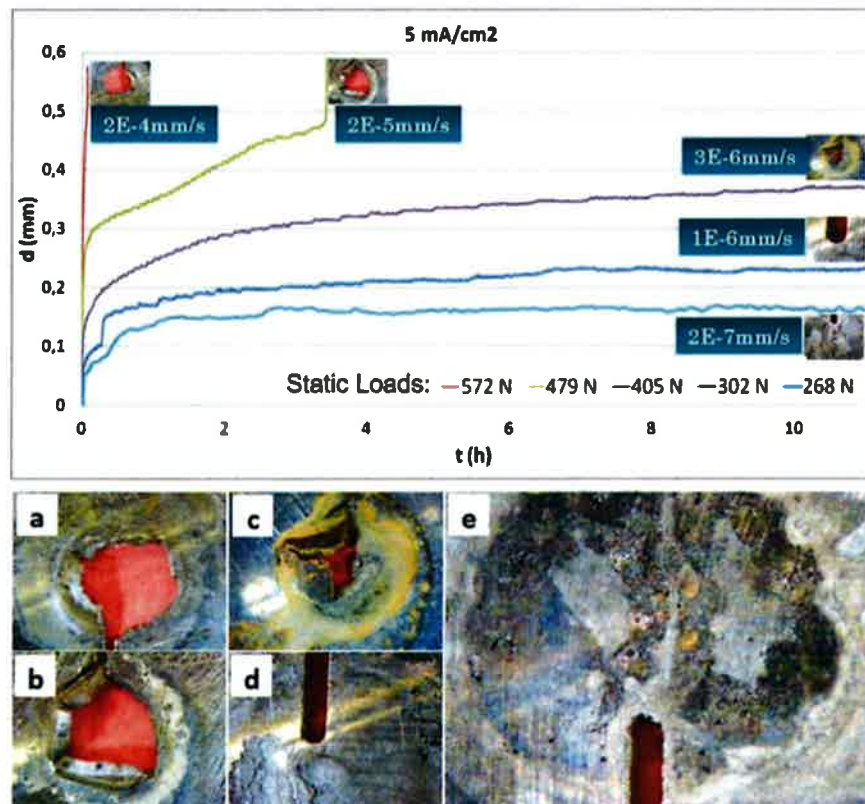


Figure 16. Displacement-time curves and macrographs from embrittled SPT samples tested in environment under static loads.

## 4. Discussion

A brittle fracture mixed mode can be observed for pre-embrittled samples at both rates (0.01 and 0.002 mm/s), but its intensity seems to be less brittle than the cracked system presented on Figure 12 for conventional fracture mechanics tests. There is a competition between two effects taking place. On the one hand, the lower the punch rate is, the more time given to the trapped hydrogen to diffuse to the new cracking areas and its close zones of plasticity causing its embrittling effect. But on the other hand, the lower the rate is, the more time takes the test to be performed, so a higher quantity of Hydrogen can diffuse out of the sample due to its reduces thickness (0.5mm), not being able to cause any embrittlement any more. Ergo, the profit by lowering the punch rate is compensated by the diffusion out of the sample; this dual effect will be in function of the material microstructure and hydrogen trapping net [31].

Even if is clear that the SPT pre-embrittled samples are capable of reproducing HE situations, the aforementioned fact points clearly that a more accurate way to reproduce the micromechanisms from conventional fracture mechanics tests is needed. A way to solve this situation was performing SPT tests on samples exposed to a continuous source of Hydrogen, by submerging them in the environment. Despite the 0.01 mm/s SPT curves don't differ much from the SPT pre-embrittled samples (Figure 13) and tested in environment ones (Figure 14), for tests in

environment a clearer difference between both rates (0.01 and 0.002 mm/s) can be appreciated (Figure 14). Slower punch rate curves (0.002 mm/s) show a clear reduction of their mechanical properties (lower maximum load and displacement) from 0.01 mm/s ones, being also placed lower than their homologous at 0.002 mm/s pre-embrittled curves (comparing Figures 13 and 14). Thus, it can be stated that carrying out the test submerged in the environment has a clear influence. It seems that submerging the embrittled samples during the test, and reducing the punch rate in order to allow the Hydrogen time enough to diffuse to the cracking plastic zones, is the way to reproduce the micromechanisms taking place during conventional fracture mechanics test accurately.

In a further step, SPT embrittled samples were tested in environment at a very low rate (5E-5 mm/s). At the first look at the graphs, its mechanical response results poorer than the samples tested in environment at conventional rates and a lot poorer than the SPT test of the material as received (Figure 15). The registers exhibit once more a brittle typology, that is enhanced by the very slow rate (5E-5 mm/s), which gives the Hydrogen time enough to develop all of its embrittling and damaging power to the microstructure. From the fractographic images, it can be stated that a clearly brittle pattern in a mixed mode is presented, showing transgranularity and grain boundaries separation in a similar magnitude to the one presented on conventional fracture mechanics tests on C(T) samples (comparing Figure 15 and Figure 12). Thus, the rate imposed by this methodology (5E-5 mm/s) can reproduce the same micromechanisms taking place during conventional fracture mechanics tests.

In order to study the effects for lower values of the rate, submerged SPT tests in environment under static load were performed. A set of samples was tested using decreasing imposed loads, which produced decreasing punch rates in the quasi-constant punch displacement zone (zone II from Figure 5), up to the load that was not enough to produce any cracking departing from the edge of the notch. As presented in Figure 16, the two highest loads produced the failure in 5 minutes and less than 4 hours developing rates around E-4 and E-5 mm/s, but the rest of the samples reached a quasi-stable zone II of cracks evolution, resulting this in rates in the range of E-6 to E-7 mm/s. For the punch rates developed of E-6 to E-7 mm/s, Hydrogen will have time enough to cause all of its embrittling capacity, so micromechanisms will be alike to those from Figure 15.

Thus, it can be stated that the static load SPT test is an appropriate methodology to reproduce HE situations. Allowing the system to be auto-cracked by the load imposed, it is assured the application of a rate slow enough to produce the HE micromechanisms present in real scenarios. On the other hand, the disadvantage of this method is the need to test several samples, up to finding the one that does not produce any cracking from the edge of the notch of the specimen.

## 5. Conclusions

A mechanical analysis of the curves together with a fractographic study of the micromechanisms presented by the samples tested at different rates was carried out. Pre-embrittled SPT notched samples were tested in air at conventional rates of 0.001 and 0.002 mm/s, and embrittled samples were tested in environment from rates of 0.01 mm/s to static load tests, that reached up to E-7 mm/s. From this study, it is concluded that:

- Very slow rates are necessary to perform SPT characterizations in environment.
- This makes necessary the continuous exposition of the sample to the embrittling environment, i.e. being submerged during the hole test.
- Tests at rates of 5E-5 mm/s, or lower, showed similar micromechanisms than those from conventional fracture mechanics tests on C(T) samples for the same environment.
- Static load tests developed punch rates of around E-6 to E-7 mm/s, as a result of the system's flexibility variation assisted by the environment. Even if this technique seemed suitable, it demands an excessive amount of material and time, as it needs to tests a set of samples for each environmental condition. Tests in environment at punch rates around E-6 mm/s seem to be a more suitable option, ergo rates 10,000 times slower than the conventionally used for SPT tests in air [14,32].

As a general conclusion, to perform SPT applications to HE scenarios, is recommended to employ embrittled notched samples tested completely submerged in the environment at a range of rates of around E-6 mm/s. By doing so, it will be assured to reproduce the micromechanisms taking place during real processed.

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