Available online at www.sciencedirect.com

ScienceDirect

Procedia Structural Integrity 28 (2020) 188–199

Structural Integrity Procedia

www.elsevier.com/locate/procedia

1st Virtual European Conference on Fracture

The role of the testing rate on Small Punch tests for the estimation of fracture toughness in hydrogen embrittlement

B. Arroyo[a,*], J.A. Álvarez[a], F. Gutiérrez-Solana[a], R. Lacalle[a,b], S. Cicero[a]

[a]*LADICIM University of Cantabria, Avenida de los Castros s/n, Santander 39005, Spain*
[b] *INESCO INGENIEROS, CDTUC Módulo 9, Avda. Los Castros 44, 39005, Santander, Cantabria, Spain*

Abstract

In this paper, different techniques to test notched Small Punch (SPT) samples in fracture conditions in aggressive environments are studied, based on the comparison of the micromechanisms at different rates. Pre-embrittled samples subsequently tested in air at rates conventionally employed (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 a set of constant loads that produce very slow rates completes the experimental results. As a conclusion, it 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;

* Corresponding author. Tel.: +34-942-201-837
E-mail address: arroyob@unican.es

1. Introduction

Stress Corrosion Cracking (SCC) and Hydrogen Embrittlement (HE) phenomena lead to degradation of the mechanical properties of high-strength steels when facing aggressive environments (Hamilton, J.M. 2011). The effect of hydrogen is significant when exposed to aqueous environments such as off-shore cathodic protection systems, or

10.1016/j.prostr.2020.10.024

H_2S presence typical from oil&gas pipelines. Both phenomena result in brittle failures in the presence of an aggressive environment and maintained stress and are dependent on the crack deformation rate: may even disappear at very high rates while at very slow ones hydrogen can show an strong embrittling effect (Johannes Rehrl et al. 2014).

Standards, such as ISO 7539 (ISO 7539:2011.), establish requirements for specimen sizes and testing rates but does not specifically define the procedure to follow in numerous applications. There are situations, such as welded joints, characterizations of in-service components, or thin elements, where it is not possible to machine specimens fitting the dimensions required by the aforementioned standards. To solve this issue the miniature test family was developed, being the Small Punch Test (SPT) one of the most employed techniques. The SPT consists of punching a small plane specimen up to failure while the load and punch displacement are registered, it has been applied to estimate the yield stress, ultimate tensile strength and fracture toughness of metallic materials with high reliability (Martínez-Pañeda E et al. 2016).

Recently, some authors have proved the validity of the SPT when used in HE and SCC characterizations (Arroyo B. et al. 2016, García T.E. et al. 2016, Arroyo B. et al. 2017). In order to reproduce the micromechanisms taking place in HE failures accurately, SPT testing rates should be very slow, or even quasi-static (ISO 7539:2011). In this paper, a review of all possible SPT testing techniques and a wide range of rates for its application to HE scenarios is carried out, form pre-embrittled samples tested in air at conventional rates to tests in environment at different punch rates from 0.01 mm/s up to constant load (E-7 mm/s).

2. The Small Punch Test

The Small Punch Test developed in the early 80's (Manahan, M.P. et al. 1981) it allows to characterize metallic materials when the amount to obtain samples is very reduced. There is a European Code of Practice, CWA 15627, edited by CEN in 2007 (CWA 15627, 2007), based on which a European Standard is in revision process (EN Standard Working Draft WI, 2018). It has been successfully employed in the evaluation of tensile (Eskner M. et al. 1995) and fracture (Lacalle R. et al. 2012) properties of different materials. Also, has been applied to characterize embrittlement situation on steels, such as the evolution of materials properties with neutron irradiation (Finarelly D. et al. 2004), the brittle-ductile transition temperature of metallic materials (Kim M.C. et al. 2005), or environmental embrittlement (Arroyo B. et al. 2016, García T.E. et al. 2016, Arroyo B. et al. 2017).

SPT consists of punching a plane specimen of small dimensions deforming it until fracture, a schematic of the device used for the performance of these tests is represented in Figure 1. During the test the force and the punch displacement are registered continuously, obtaining curves like the ones shown in Figure 2 (Arroyo B. et al. 2017) divided in 4 zones.

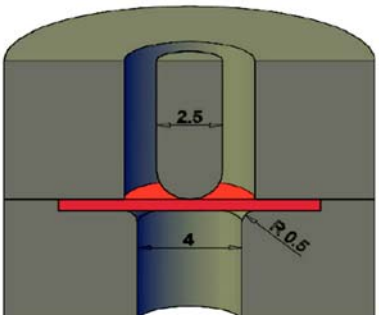

Figure 1. SPT device and sample used; dimensions in milimiters (mm).

Zone I, or elastic region, is the result of the superposition of the punch indentation and the elastic behavior as a plate of the specimen. Zone II, after the first convexity change, consists of a generalized plate yielding of the specimen. Zone III, after the second convexity change of the curve, gets deformations are concentrated in certain regions of the specimen and the behavior of the sample changes from plate to membrane. Finally, zone IV indicates the beginning of the plastic instability, and the appearance of cracks that will lead to the final specimen fracture after the maximum force of the test is exceeded.

It must be pointed that in brittle materials, or embrittlement situations, the membrane stretching (zone III) does not exist, going from a yielding plastic behavior directly to the final plastic instability. Also, while in ductile situations the specimen rupture surface has a semicircular shape and its deflection is higher (figure 2.a), in brittle scenarios the breaking typology is a star-type (figure 2.b) and the specimen deflection lower, so the energy under the curve is also lower (Arroyo B. et al. 2016).

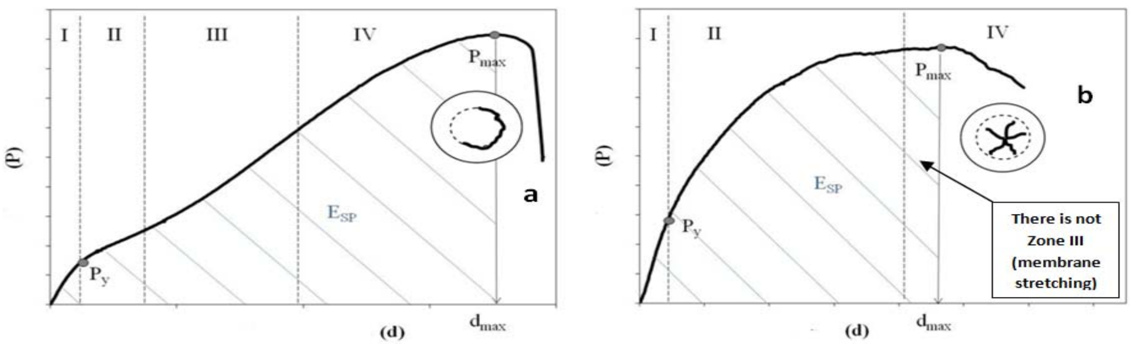

Figure 2. Schematic of SPT force-displacement curves; a) ductile materials; b) brittle materials

In environmental characterizations the testing rate is an important parameter to take into account (Johannes Rehrl et al. 2014, Arroyo B. et al. 2017), as far as it will govern the micromechanism taking place; very low rates, or even constant tests, are commonly employed (ISO 7539:2011). By using these testing conditions, hydrogen will have enough time not just to diffuse form reversible tramps to the new cracking areas subsequently generated during the test, but also to escape form irreversible tramps helped by plastic deformation and diffuse to the new cracking areas (Pressouyre G.M. et al. 1981).

The ultimate research for the SPT in HE characterizations (Arroyo B. et al. 2017) advises to use tests under constant load or very slow punch rates. When using SPT under constant load, the punch displacement vs time is registered, resulting in curves like the one shown in in Figure 3, where three zones can be distinguished. Zone I consists of 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 of the system is so high that the punching load cannot be supported anymore and the specimen leads to final instability and fails.

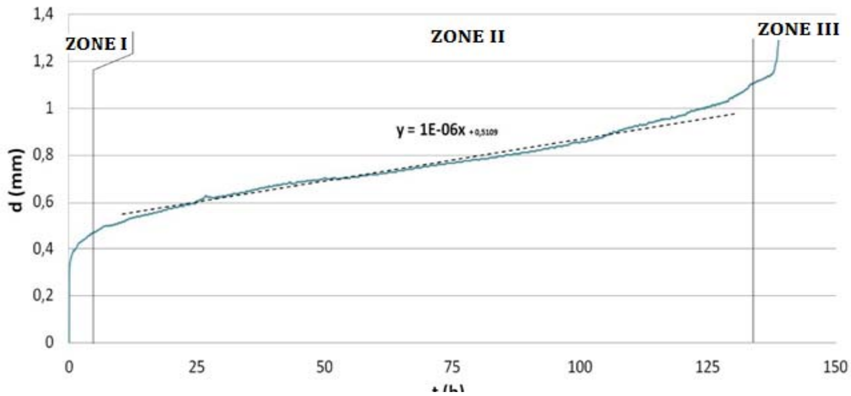

Figure 3. SPT displacement-time curve for constant load tests in environment (7).

3. Material and environment employed

3.1. Material

A Cr-Ni-Mn high-strength steel was employed, it is used for manufacturing large anchor chain links for off-shore platforms. It is obtained in bars by quenching and tempering processes, and then forged to conform the links by bending forces. It has a tempered martensite microstructure (Figure 4) and a chemical composition mainly consisting of 0.23% C, 1.05% Cr, 1.08% Ni, 1.25% Mn, 0.51% Mo, 0.24% Si, 0.25% Cu and 0.10% V (% weight). Its hydrogen content as received was measured and equal to 0.84 ppm, and its main tensile parameters are σ_y = 920 MPa, σ_u = 1015 MPa and E = 205 GPa, while its fracture parameter $J_{0.2}$ = 821 kN/m.

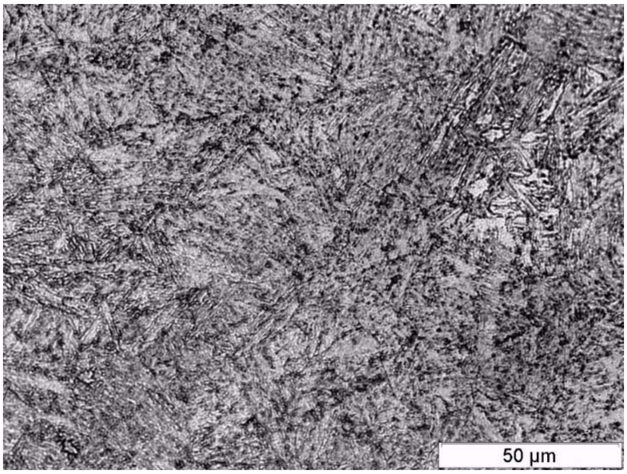

Figure 4. Microstructure of the steel used in the R-L plane.

3.2. Simulation of Hydrogen Embrittlement (HE)

Cathodic charge (CC), known also as anodic polarization, has been employed in this study. It is mainly used 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. A level of 5mA/cm^2 was employed, as done in previous works through the last decades (Arroyo B. et al. 2016, García T.E. et al. 2016, Arroyo B. et al. 2017, Gutiérrez-Solana F. et al. 1995, Álvarez J.A. et al. 1998).

Figure 5 shows a set-up of the method used in this work (Arroyo B. et al. 2017). 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 (Hamilton, J.M. 2011, Álvarez J.A. et al. 1998, Álvarez J.A. et al. 1998). In this study, an environmental condition in accordance (García T.E. et al. 2016, Arroyo B. et al. 2017, Pressouyre G.M. et al. 1981) was proposed, consisting of an 1N H_2SO_4 solution in distilled water containing 10 drops of CS_2 and 10mg of As_2O_3 dissolved per liter of dissolution; the solution of As_2O_3 was prepared using Pressouyre's method (Álvarez J.A. et al. 1998). 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 20ºC - 25ºC. The hydrogen content of the steel was measured, obtaining 5.45 ppm (vs 0.84 ppm in as received state).

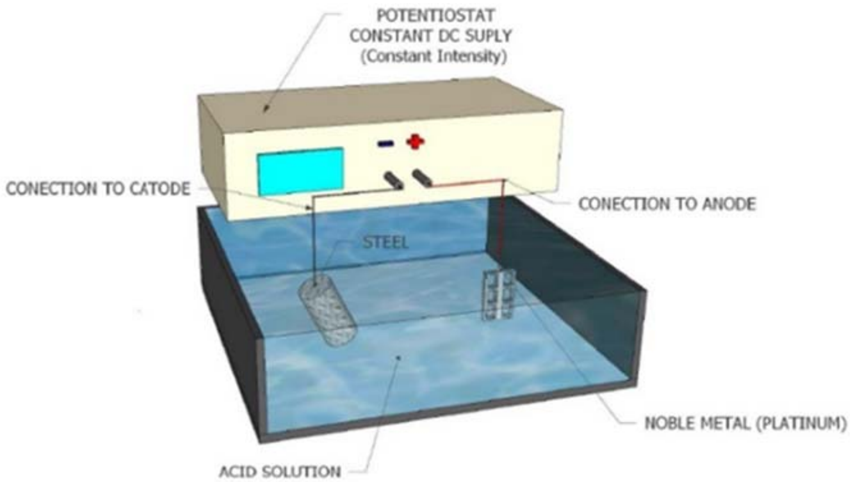

Figure 5. Cathodic charge method. (7).

4. Experimental methodology

4.1. SPT samples employed

The sample geometry employed for SPT, according to (Arroyo B. et al. 2017, EN Standard Working Draft WI, 2018), is presented on Figure 6, it consists on a plane 10mmx10mm of section and 0.5±0.01mm of thickness including a lateral notch machined by wire electro-erosion of 0.15 mm radius.

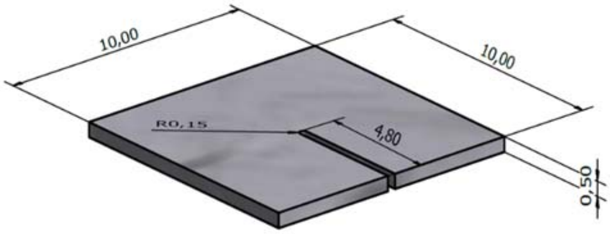

Figure 6. SPT notched samples employed.

Prior to the test, the specimens were subjected to hydrogen charging by exposing them for 2 hours in the same environmental condition previously described. This amount of time, proposed by (Arroyo B. et al. 2016, Arroyo B. et al. 2017) assured a proper and complete diffusion of the hydrogen inside the material up to the saturation of the 0.5mm thickness.

After the charge, the following three mechanical testing conditions were employed, in order to produce the different punch rates on the sample to be studied.

4.2. Embrittled samples tested in air

The samples were charged in the environment (Figure 7) and immediately extracted, dried and tested in air environment in an electric machine. Two rates were employed, the regular one of 0.01 mm/s, in the range of (EN Standard Working Draft WI, 2018) recommendations, and another one of 0.002 mm/s, five times slower in order to compare their effects. A total of 8 samples were tested; 4 samples at 0.01 mm/s and 4 at 0.002 mm/s.

In order to obtain H_2 contents, another 8 samples (4 for each rate) were precharged together with the aforementioned ones and left in air exposition during the mechanical tests to reproduce the diffusion during the tests (that lasted 5 and 15 minutes for 0.01 mm/s and 4 at 0.002 mm/s respectively). Contents of 4.86 ppm and 4.15 ppm were obtained.

Figure 7. SPT pre-embrittled specimens during its hydrogen absrotion.

4.3. Embrittled samples tested in environment under different punch rates

The samples were charged in the environment and tested in continuous exposition to it. A device was specifically designed and built for this purpose that is presented in Figure 8; in this case, the punching is applied in the horizontal direction. The range of rates employed in order to study their effects were the conventional one of 0.01 mm/s and another five times slower of 0.002 mm/s (as in the previous case), a third very slow rate (500 times slower than the conventional rate) of 5E-5 mm/s, that has been previously employed by some authors (Arroyo B. et al. 2016, García T.E.. et al. 2016, Arroyo B. et al. 2017), was also included. A total of 10 samples were tested: 4 at 0.01 mm/s, 4 at 0.002 mm/s and 2 at 5E-5 mm/s due to the amount of time required for each test. The H_2 content in this case remained constant in 5.45 ppm during the whole test.

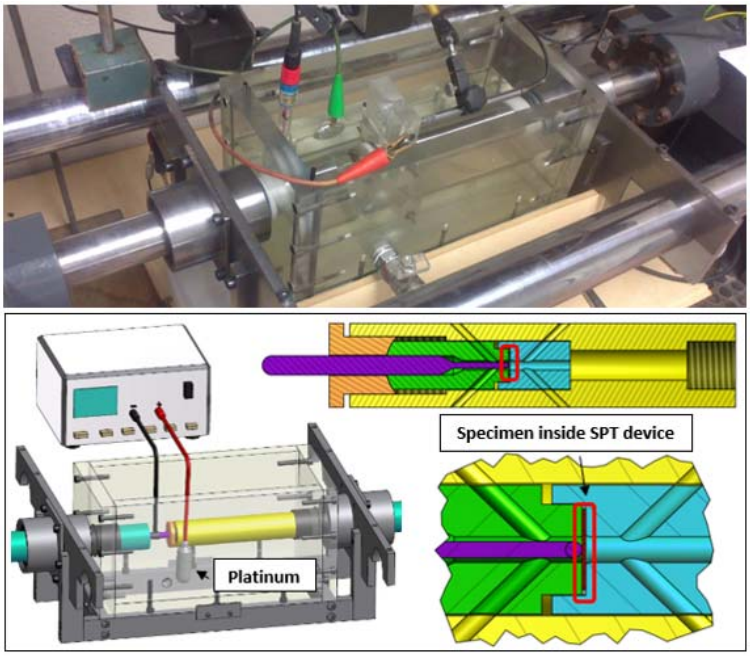

Figure 8. Experimental device for performing SPT tests in environment at very slow rates; real picture during a test and sketch.

4.4. Embrittled samples tested in environment under sustained constant load

The samples were charged in the environment and tested in continuous exposition to it; The device presented in Figure 9 was specifically designed and built for this task. A set of 5 samples, one per each constant load used, were tested using decreasing imposed constant 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. Again the H_2 content in this case remained constant in 5.45 ppm during the whole test.

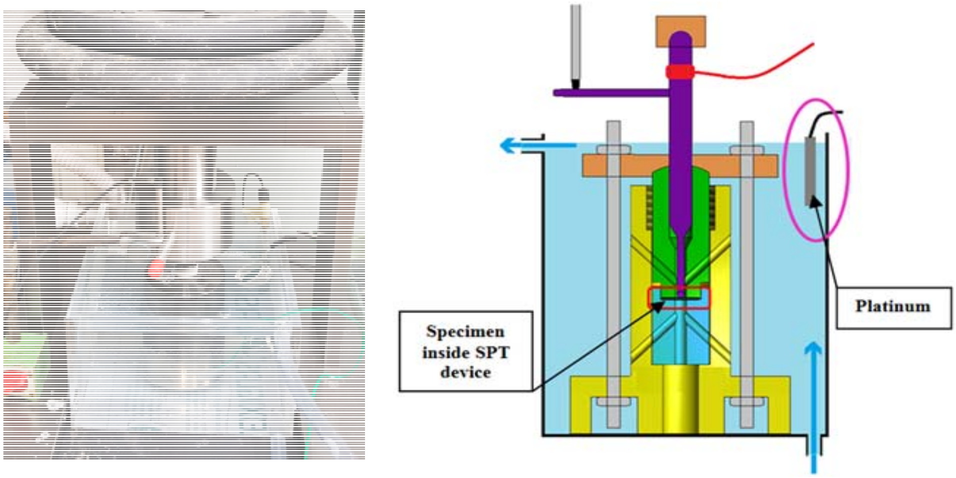

Figure 9. Experimental device for performing SPT tests in environment under constant load; real picture during a test and sketch.

5. Experimental results and discussion

5.1. Embrittled samples tested in air

Figures 10 and 11 and Table 1 present the curves, values of load and displacement and fractography form the SPT tests performed on 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 a loss of mechanical properties. The shape of the curve, that was the typical form a ductile material (black line) shows here a completely 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. In Figure 11, a semi-brittle slightly transgranular fracture mode can be observed for both rates (0.01 and 0.002 mm/s), without finding any important difference between them.

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 (Pressouyre G.M. et al. 1981).

Even if is clear that the SPT embrittled samples tested in air are capable of reproducing HE situations, a more accurate that avoids diffusion out is necessary, as can be for instance performing the test in environment.

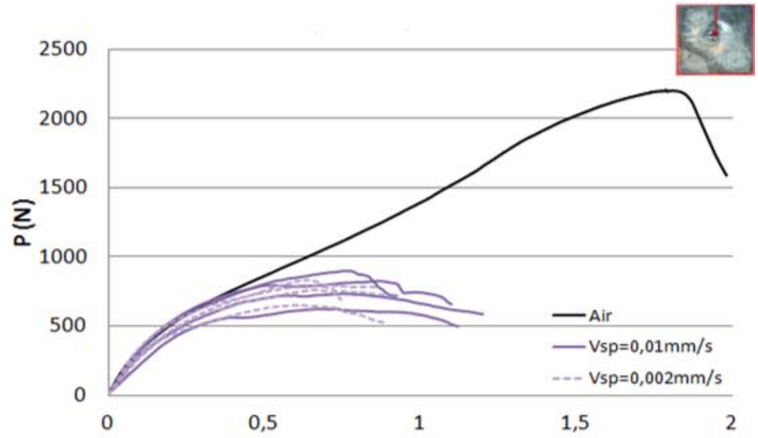

Figure 10. Load-displacement curves from embrittled SPT tested in air at conventional rates; SPT as received test superposed.

Table 1. Values of maximum loads and its corresponding displacements from embrittled SPT tested in air at conventional rates.

Punch rate	Max load (N)		Max displacement (mm)	
0.01 mm/s	732	**768 N** (mean)	0.75	**0.77 mm** (mean)
	820		0.87	
	901		0.76	
	618		0.68	
0.002 mm/s	760	**759 N** (mean)	0.66	**0.65 mm** (mean)
	797		0.68	
	831		0.64	
	648		0.62	

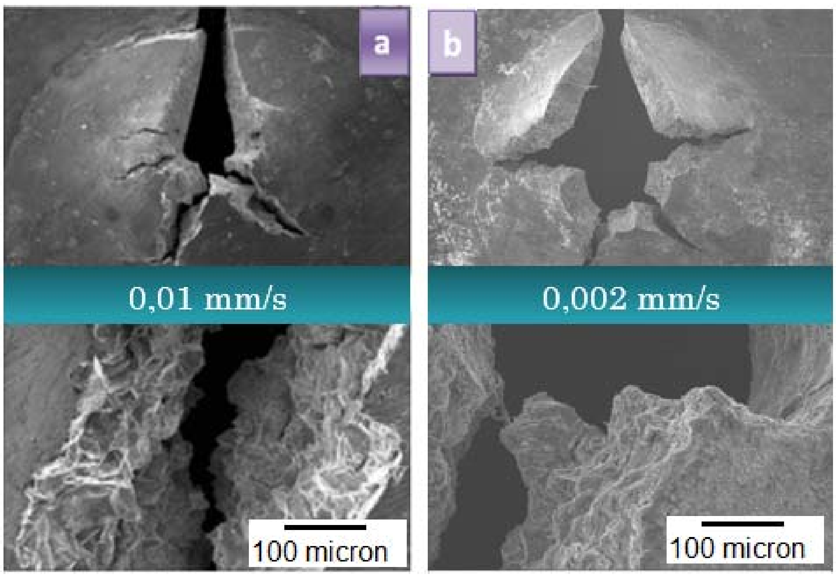

Figure 11. Fractographic images from embrittled SPT tested in air at conventional rates

5.2. Embrittled samples tested in environment under different punch rates

Figures 12 and 13 and Table 2 present the curves, values of load and displacement and fractography form the SPT tests performed on embrittled samples tested in environment at the conventional rate of 0.01 mm/s, another 10 times slower of 0.002 mm/s, and another very slow one (500 slower than conventional) of 5E-5 mm/s. Again, the register form an SPT test of the material as received is superposed for comparison (black line).

It can be seen the embrittlement produced by the environment, showing the curves a typical brittle shape and a decrease of mechanical properties (maximum load and its displacement) when compared to the as-received curve in air. Also, the effect of the punch rate is noticeable, showing the curves lower maximum loads and displacements the lower the punch rate is, this means that the punch rate reduction allows the environment to cause a higher embrittling effect.

If the curves at 0.01 and 0.002 mm/s rates in environment (Figure 12) are compared to their homologous embrittled and tested in air (Figure 10), the 0.01 mm/s ones don't differ much (5.45 ppm vs 5.45 to 4.86 ppm diffusion during the test). Nevertheless, when comparing the 0.002 mm/s rate ones (5.45 ppm vs 5.45 to 4.15 ppm diffusion during the test) a clearer reduction of maximum load and displacement can be appreciated when the samples are tested in environment.

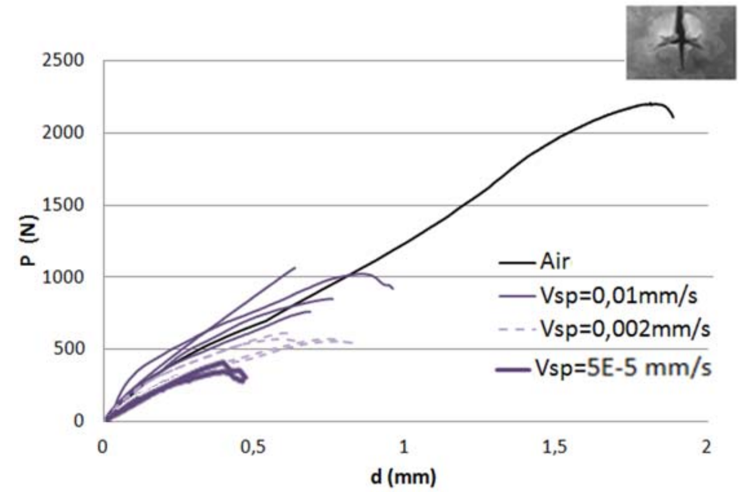

Figure 12. Load-displacement curves from embrittled SPT tested in environment at different rates; SPT as received test superposed.

Table 2. Values of maximum loads and its corresponding displacements from embrittled SPT tested in environment at different rates.

Punch rate	Max load (N)		Max displacement (mm)	
0.01 mm/s	848	**924N** (mean)	0.71	**0.70 mm** (mean)
	757		0.66	
	1065		0.63	
	1025		0.78	
0.002 mm/s	555	**556 N** (mean)	0.47	**0.58 mm** (mean)
	551		0.58	
	569		0.56	
	549		0.71	
5E-5 mm/s	401	**369 N** (mean)	0.37	**0.35 mm** (mean)
	337		0.33	

The aforementioned facts have their correspondence in the fractography (Figure 11 vs Figure 13). A combined brittle transgranular and intergranular fracture mode can be observed for both rates when tests are performed in environment, which is slightly more brittle at 0.002 mm/s; fact that was not happening in embrittled samples tested in air (Figure 10), where the lower rate of 0.002 mm/s produced less brittle mechanisms. Finally, the samples tested in environment at the lowest rate (5E-5 mm/s) showed the most brittle pattern of all in a mixed mode with transgranularity and grain boundaries separation; the macrography shows that the sample did not have practically any deformation before the crack departed from the notch tip (while in the 0.01 and 0.002 mm/s cases did).

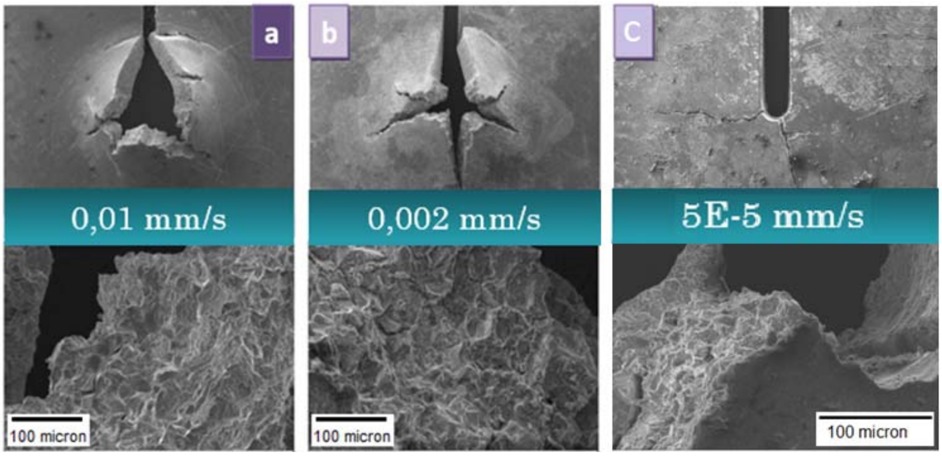

Figure 13. Fractographic images from embrittled SPT tested in environment at different rates.

5.3. Embrittled samples tested in environment under sustained constant load

In the previous sections, it was proved that performing the tests in environment after embrittling the samples, and reducing the punch rate several orders of magnitude, allows the environment to cause a higher embrittling effect. In order to study how low the punch rate should be in order to cause all of its damaging potential, a set of static SPT test in environment under constant load was performed. A set of samples were tested using decreasing imposed constant loads, which produced decreasing punch rates in the zone II of the curve, up to the load that was not enough to produce any cracking departing from the edge of the notch. Figure 14 shows the registers displacement-time, Table 3 the values of loads and displacements in each case, and Figure 15 presents the macrographic pictures of the samples tested by this methodology.

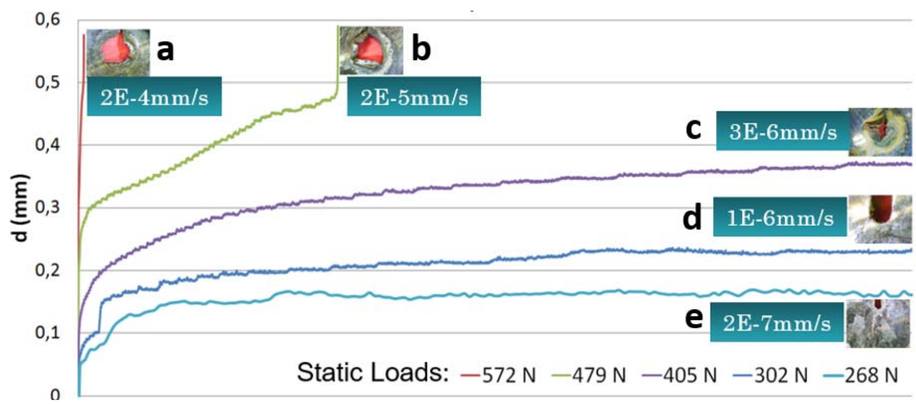

Figure 14. Displacement-time curves from embrittled SPT samples tested in environment under constant loads.

Table 3. Values of maximum loads and its corresponding displacements from embrittled SPT tested in environment under constant loads.

Punch rate	Load (N)	Initiation displacement (mm)
2E-4 mm/s	572	0.31
2E-5 mm/s	479	0.30
3E-6 mm/s	405	0.29
1E-6 mm/s	302	0.15
2E-7 mm/s	**268** (lowest, sample "e")	**0.11** (lowest, sample "e")

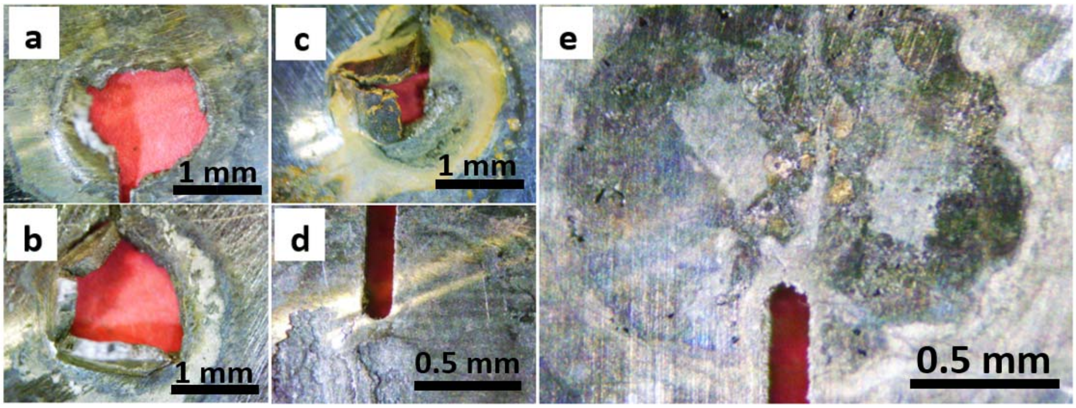

Figure 15. Macrographys from embrittled SPT samples tested in environment under static loads ("e" didn't developped macroscopic cracks).

The highest loads ("a" and "b") lead to failure relatively fast, producing rates in the range of E-4 to E-5 mm/s, which were similar conditions to the tests at the lowest rate in the previous section (5E-5 mm/s).

Samples "c" and "d" produced rates in the order of magnitude of E-6 mm/s for quasi-stable zone II of cracks evolution, while the lowest load (sample "e") showed a rate one order of magnitude lower: E-7 mm/s. In sample "c" an important crack that drilled the sample when it collapsed (as can be appreciated), while in sample "d" a crack starting from the edge of the notch was found, stopping test prior to the sample's collapse. Sample "e" did not show any macroscopic crack with its onset of the edge of the notch, although a subcritical cracking on the sample's thickness could be taking place according to the literature (Arroyo B. et al. 2017).

This means that the constant load imposed must be around the threshold, so it can be concluded that the threshold will take place in rates around E-6 to E-7 mm/s (more exactly under loads that cause this rates in zone II). This punch rates will give the environment time enough to cause all of its embrittling capacity.

6. Conclusions and future work

SPT embrittled samples tested in air at conventional rates around 0.01 mm/s are able to show environmental effects of HE, but don't allow the whole of its embrittling effects. Slower rates, as the 0.002 mm/s studied, do not solve this issue either as the benefits of a higher time to the allow the environment to reach the crack tip plasticity zones and apply a bigger embrittling capacity is countered by the higher time for H_2 to diffuse outside the sample; there is a competition between both effects.

SPT embrittled samples tested in continuous exposition to the environment partially solve the aforementioned issue, as the same rates than in the samples tested in air (0.01 and 0.002 mm/s) showed a higher embrittling effect of the environment, which was verified in the load-punch displacement curves and in the maximum load and displacement values. It was also proved that the reduction of the punch rate several orders of magnitude while testing in continuous exposition allows higher embrittlement effects; the results of the 5E-5 mm/s punch rate tests exhibited more brittle mechanisms and maximum load and displacement values than 0.01 and 0.002 mm/s. This proves that embrittled samples tested in environment at very slow rates are the most suitable option to show all the environment's embrittling effects.

It can be stated that constant load SPT tests in environment are appropriate to reproduce HE situations, as the system will be auto-cracked by the load imposed and the application of a rate slow enough assured. The punch rates generated near the threshold were found to be around E-6 to E-7 mm/s. On the other hand, the disadvantage of this method is the need to test several samples to find the one that does not produce any cracking from the edge of the notch of the specimen, which can take plenty of time.

In order to palliate this, embrittled SPT samples tested in environment under very slow rates, around E-6 to E-7, can be a faster alternative, which should be deeply studied and compared to constant load tests as a future work, in order to find the simplest and fastest possible way to apply SPT tests to environmental characterizations.

References

Hamilton, J.M., 2011. "The challenges of Deep-Water Artic Development", International Journal of Offshore and Polar Engineering, 21 (4), pp. 241-247.

Johannes Rehrl, Klemenes Mraczek, Andreas Pichler, Ewald Werner, 2014. "Mechanical properties and fracture behavior of hydrogen charged AHSS/UHSS grades at high- and low strain rate tests", Materials Science & Engineering A, 590, pp. 360-367.

ISO 7539:2011. Parts 1 to 9 "Corrosion of metals and alloys"

Martínez-Pañeda E., García T.E., Rodrígez C., 2016. "Fracture toughness characterization through notched small punch test specimens", Materials Science and Engineering A, vol. 657, pp. 422-430.

Arroyo B., Álvarez J.A., Lacalle R., 2016. "Study of the energy for embrittlement damage initiation by SPT means. Estimation of KEAC in aggressive environments and rate considerations", Theoretical and Applied Fracture Mechanics, 86, pp. 61-68.

García T.E., Arroyo B., Rodríguez C., Belzunce F.J., Álvarez J.A., 2016. "Small punch test methodologies for the analysis of the hydrogen embrittlement of structural steels", Theoretical and Applied Fracture Mechanics, 86, pp. 89-100

Arroyo B., Álvarez J.A., Lacalle R., Uribe C., García T.E., Rodríguez C., 2017. "Analysis of key factors of hydrogen environmental assisted cracking evaluation by small punch test on medium and high strength steels", Materials Science and Engineering A, 691, pp. 180-194.

Manahan, M.P., Argon, A.S., Harling, O.K., 1981. "The development of a miniaturized disk bend test for the determination of post irradiation mechanical properties", Journal of Nuclear Materials, 103 & 104, pp. 1545-1550.

CWA 15627, 2007. "Small Punch test method for metallic materials, Part A: Code of practice for Small Punch creep testing, Part B: Code of practice for Small Punch testing for tensile and fracture behavior", Documents of CEN WS21, Brussels.

EN Standard Working Draft WI, 2018. "Metallic materials - Small punch test method" Documents of ECISS/TC 101, AFNOR.

Eskner M., Sandstrom R., 1995. "Mechanical property using the small punch test", Journal of Testing and Evaluation, vol 32, Nº 4, pp. 282-289.

Lacalle R., Álvarez J.A., Arroyo B., Gutiérrez-Solana F., 2012. "Methodology for fracture toughness estimation based on the use of Small punch notched specimens and the CTOD concept", The 2nd International Conference SSTT 2012, Conference Proceedings.

Finarelly D., Roedig M., Carsughi F., 2004. "Small Punch Tests on Austenitic and Martensitic Steels Irradiated in a Spallation Environment with 530 MeV Protons", Journal of Nuclear Materials 328, pp. 146-150.

Kim M.C., Oh Y.J., Lee B.S., 2005. "Evaluation of ductile-brittle transition temperature before and after neutron irradiation for RPV steels using Small Punch tests" Nuclear Engineering and Design 235, pp. 1799-1805.

Pressouyre G.M., Bernstein I.M., 1981. "An example of the effect of hydrogen trapping on hydrogen embrittlement", Metallurgical transactions, vol. 12, nº A, pp. 835-844.

Gutiérrez-Solana F., Álvarez J.A., González J., Brass A., Chêne J., Coudreuse L., Renaudin C., Astiz M., Belzunce J., 1995. "Stress corrosion cracking on weldable micro-alloyed steels", Contract No 7210-KB/327 Final Report, EUR 17249 EN (1992-1995).

Álvarez J.A., Gutiérrez-Solana F., 1998. "An elastic-plastic fracture mechanics-based methodology to characterize cracking behavior and its applications to environmental assisted processes", Nuclear engineering and design, vol. 188, pp. 185-202.

Álvarez J.A., Gutiérrez-Solana F., 1998. "Hydrogen Induced Cracking Processes in Structural Microalloyed Steels. Characterization and Modelling", Materials Science Forum, vols. 284-286, pp.303-310.