Using Small Punch tests in environment under static load for fracture toughness estimation in hydrogen embrittlement

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Abstract. In this paper, the response of three medium and high-strength steels to hydrogen embrittlement is analyzed by means of the quasi-non-destructive test known as the Small Punch Test (SPT). SPT tests on notched specimens under static load are carried out, applying Lacaclle's methodology to estimate the fracture toughness for crack initiation, comparing the results to K_{IEAC} fracture toughness obtained from C(T) precracked specimens tested in the same environment; SPT showed good correlation to standard tests. A novel expression was proposed to define the parameter $K_{IEAC\text{-}SP}$ as the suitable one to estimate the fracture toughness for crack initiation in hydrogen embrittlement conditions by Small Punch means, obtaining good accuracy in its estimations. Finally, Slow Rate Small Punch Tests (SRSPT) are proposed as a more efficient alternative, introducing an order of magnitude for the adequate rate to be employed.

1. Introduction

A critical aspect concerning mid and high strength steels is their resistance to Stress Corrosion Cracking (SCC) and Hydrogen Embrittlement (HE). The recommendations presented by various research groups over the last few decades have been collected in the standard ISO 7539 [1], but there are particular situations where standards cannot be followed characterize in-service components, due to the impossibility of machining specimens fitting the dimensions. The Small Punch Test (SPT) is one of the most notables solutions, which estimates parameters such as the yield stress, ultimate tensile strength and fracture toughness of metallic materials with high reliability. Recently the validity of the SPT when used in HE and SCC has been proved, being faster and easier and cheaper to perform than standard tests [2,3,4,5]; the ultimate research, which is applied to this work, consists on SPT tests performed in environment under static load [6]. The aim of this work is to estimate the crack initiation toughness of the material in embrittlement conditions, $K_{IEAC\text{-}SP}$, caused by a liquid aggressive environment.

2. Materials and environments employed

2.1. Three materials employed

- A quenched and tempered Cr-Ni-Mn high-strength, which has a tempered martensite microstructure. It is employed in the manufacture of large anchor chains for off-shore platforms.
- A rolled X80 medium-strength steel, which has a ferritic-pearlitic microsctructure. This steel is commonly employed for the manufacture of gas and petroleum pipes at low temperatures.

- A weldable thermo-mechanically treated S420 medium-strength steel, which has a ferritic-pearlitic microsctructure. It is mainly used in the construction of pressure vessels.

2.2. Environment for Hydrogen Embrittlement (HE) simulation

An environmental condition known as cathodic charge, or anodic polarization, was employed, it causes substantial embrittlement on the steel by the action of the hydrogen going through and getting trapped in it. It consists of the interconnection, via an acid electrolyte, of a noble material (platinum in this case) and the steel, which will protected due to the fixed current interposed. In this study an environmental condition, previously used [6,7] to reproduce cathodic charge situations, was proposed. It is 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 Pessouyre's method [7]. 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 ℃ - 25 ℃. An embrittlement level of 5mA/cm^2 was employed.

3. The Small Punch Test

The quasi-non-destructive SPT technique allows to test in-service structures since the extraction of a sample does not compromise the component's integrity. It has a European Code of Practise, CWA 15627, edited by CEN in 2007 [8]. It has been successfully employed in the evaluation of tensile [9], fracture [9,10] and creep properties, and it was recently applied to HE scenarios [5,6]. The SPT consists of punching a plane specimen of small dimensions deforming it until fracture; during the test the force as well as the displacement made by the punch are registered continuously.

When characterizing materials in HE situations the testing rate is an important parameter to take in account in order to reproduce the subcritical processes taking place [11]. The ultimate research in the SPT field [6, 11] advises to perform static load tests in order to allow hydrogen to cause all of its embrittling power. In this type of testing three zones can be distinguished in the punch displacement vs time register (d-t); zone I: indentation and settlement, zone II: quasi-constant punch rate caused by the flexibility variation of the system produced by an increasing cracking in both radial and thickness directions of the specimen, and zone III: final and rupture.

4. Experimental Methodology

4.1. Standard validation fracture mechanics K_{IEAC} determination tests

Fracture mechanics tests were carried out in order to determine K_{IEAC} for each of the materials in the aforementioned environmental condition (cathodic polarization at a level of 5mA/cm^2). B=25mm C(T) specimens according to [12] were employed, being precracked up to a/w=0.53. The specimens were subjected to hydrogen absorption during 48 hours, and the test was performed subsequently by applying 6E-9 m/s of constant solicitation rate; recommendations of Standard ISO-7539 [1] were followed submerging the samples up to the crack tip. The methodology proposed by ASTM E-1820 [12] was employed for the K_{IEAC} value calculation.

4.2. Static load SPT tests for crack initiation toughness in environment determination, $K_{Jth\text{-}EAC}$

SPT notched samples were used to determine the material toughness necessary for a crack initiation from the edge of the notch, $K_{Ith\text{-}EAC}$. The environment employed was cathodic polarization at a level of 5mA/cm^2. Prior to the test, the specimens were subjected to hydrogen charging during 2 hours [6, 11]; subsequently the load was softly applied by an endless screw system on the specimen subjected to the environment. For each material several SPT notched samples were tested using decreasing loads, which produced decreasing punch rates in the zone II of the curve [6] (figure 1-right), up to that load that was not enough to produce any cracking departing from the edge of the notch. The sample geometry employed, according to [8,10,11], presented on figure 1-left, was consisting on a plate 10mmx10mm of section and 0.5±0.01mm of thickness including a lateral notch machined by wire electro-erosion of 0,15 mm of radius. The orientation of the notches in SPT and C(T) samples for validation tests was the same.

From each sample the material toughness for crack initiation from the edge of the notch, K_{Jth}, was calculated using Lacalle's methodology [10], which is based on the CTOD parameter for crack initiation on a SPT notched sample, δ_{SPi}, and allows to calculate using the expression (1) the initiation toughness J_{Ic}. Then, using the expression (2) the toughness values in terms of J_{Ic} can be transformed into terms of K_{Jth}. In (1), d_n is a parameter tabulated in [13] (figure 2-top) which is function of the material properties; δ_{SPi} can be obtained in function of the punch displacement that causes the crack initiation form the edge of the notch, d_i, using the graph proposed in [9] (figure 2-right).

$$J_{Ic} = \frac{\delta_{SPi} \cdot S_y}{d_n} \qquad + \qquad K_{Jth} = \sqrt{J_{Ic} \cdot E} \qquad (1) + (2)$$

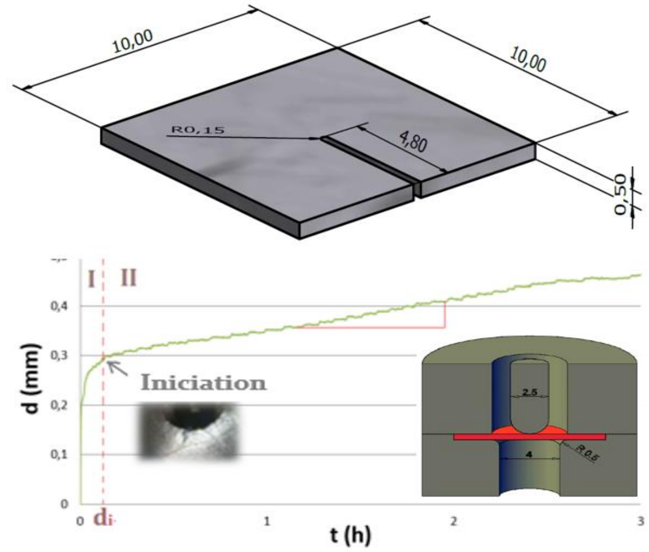


Figure 1. Top: SPT notched samples employed; **Bottom:** Determination of the punch displacement, di, for crack initiation between zones I and II.

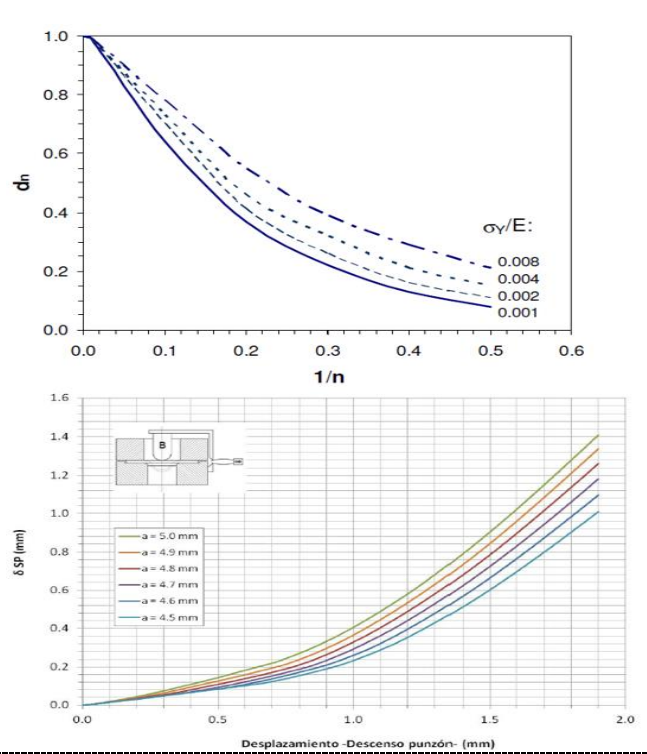


Figure 2. Top: Determination of d_n [13]; **Bottom:** δsp in function of punch displacement, d [9].

5. Experimental results

5.1. Determination of K_{IEAC} by standard fracture mechanics tests

Figure 3 shows the results of the fracture mechanics tests performed on C(T) specimens in environment.

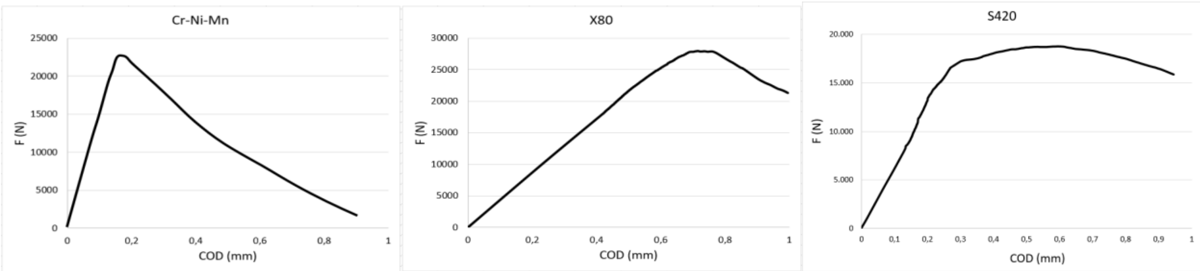


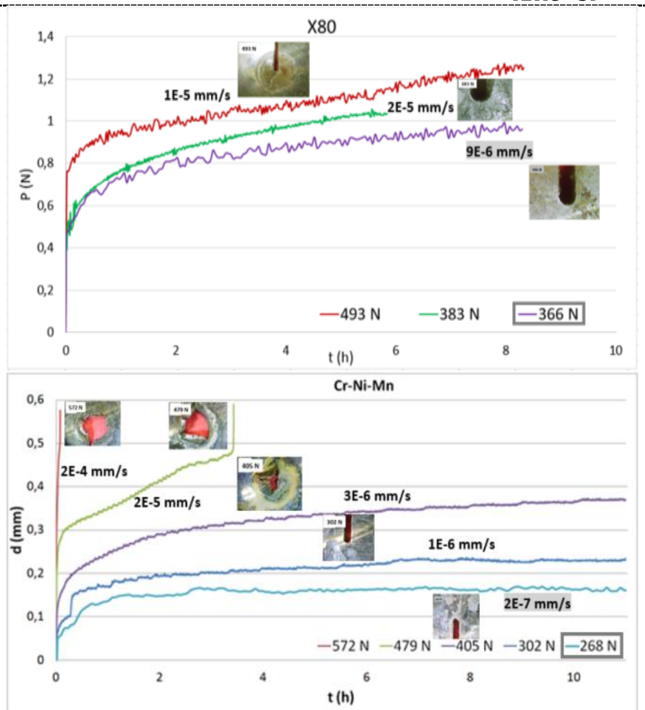
Figure 3. P-COD registers of the standard test performed on the three steels to obtain K_{IEAC}.

5.2. Determination of $K_{Jth-EAC}$ by SPT static tests in environment

In figure 4 the d-t registers from the SPT tests performed on the three materials studied are presented; accompanying them the punch rates developed by the system in the zone II and pictures of the crack initiation area of the samples are shown. In table 1 the values of $K_{Jth-EAC}$ for a specific material and environment obtained by SPT means, defined as a lower bound (sample where propagation didn't take place), are presented and compared to the results of K_{IEAC} obtained from the standard tests in environment.

It can be observed that in all cases the SPT values are higher (around the double) of their homologous K_{IEAC} obtained from standard tests. The relationship shown in equation (3) is proposed to obtain an initiation fracture toughness parameter by Small Punch means, $K_{IEAC-SP}$, as an optimization to Lacalle's methodology previously exposed [9,10] for embrittlement scenarios. Looking for the best fit for (3), showed in figure 5, a value of 0.498 was found for α, obtaining the values of $K_{IEAC-SP}$ showed in the penultimate column of table 1.

$$K_{IEAC-SP} = \alpha \cdot K_{Jth-EAC} \quad (3)$$



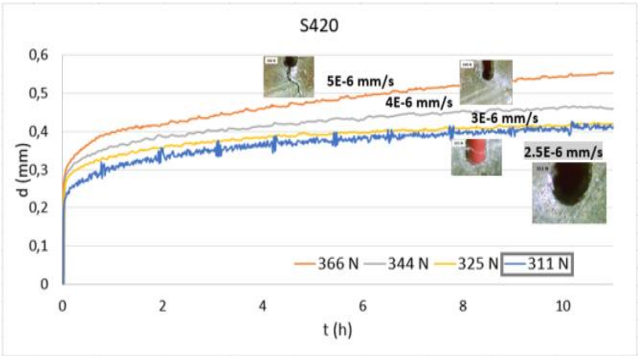


Figure 4. Punch displacement vs time (d-t) registers of the SPT static load tests in environment performed on the three steels; **Top:** Cr-Ni-Mn steel; **Mid:** X80 steel; **Bottom:** S420 steel;

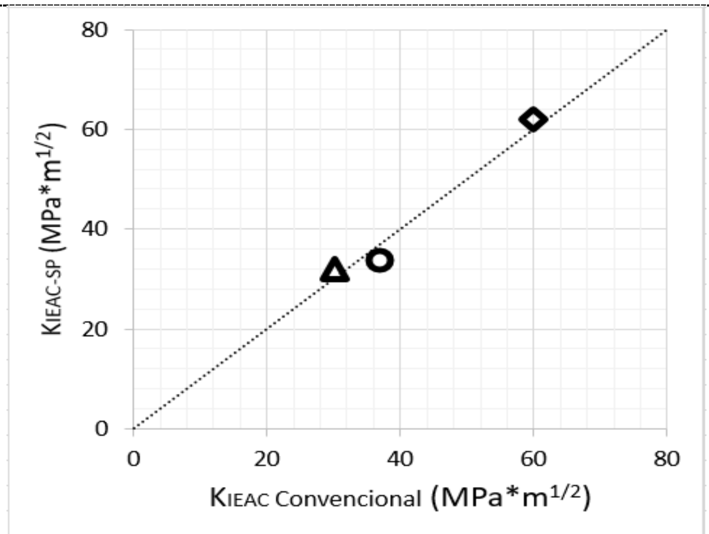


Figure 5. K_{IEAC} results from C(T) vs K_{EAC-SP} SPT technique proposed results for the materials studied.

Table 1. $K_{Ini-EAC}$ results obtained from SPT static load tests vs K_{IEAC} results from C(T) standard tests.

Material	Load (N)	di (mm)	δ_{spi} (mm) (fig. 2)	J_{Ic} (kN/m) (expr.(1))	**K_{Jth} (MPa$\sqrt{m}$) (expr.(2))**	**$K_{IEAC-SP}$ (MPa$\sqrt{m}$) (expr.(3))**	**K_{IEAC} (MPa$\sqrt{m}$)**
Cr-Ni-Mn	268	0.110	0.021	20.13	**64.23**	**31.96**	**30.08**
X80	366	0.479	0.111	73.378	**124.13**	**61.76**	**59.89**
S420	311	0.231	0.039	21.76	**67.60**	**33.63**	**36.92**

5.3. Discussion about toughness estimation and punch rates

The tests where propagation did not take place were just the ones employed in each material to define K_{EAC-SP}; in this cases the punch rates developed were around 1E-6 or 1E-7 mm/s (4 orders of magnitude lower than usually employed in air). Based on this a more efficient way of testing may be to perform Slow Rate Small Punch Tests (SRSPT) of this order of magnitude. An upper bound for SPT testing rates should be defined in order to reproduce the subcritical micromecanisms where the interaction between a given material and environment governs the process; this will be a faster and easier testing method.

6. Conclusions and future work

6.1. Conclusions

A methodology to estimate the fracture toughness for crack initiation using notched Small Punch samples was proposed. It is based on Lacalle's research, using the CTOD parameter for crack initiation, and includes a lineal expression (to correlate its results to the validation tests on C(T)'s) to obtain the parameter $K_{IEAC-SP}$ that showed good accuracy.

Finally, the performance of Slow Rate Small Punch Tests (SRSPT) was proposed as a more efficient way of testing to determine the aforementioned parameter, but the first step in this research will be finding the appropriate rates, that should be in the order or magnitude of 1E-6 or 1E-7 mm/s.

6.2. Future work

On the one hand, the methodology to estimate fracture toughness by SPT tests in environment proposed should be extended and validated in a wider range of materials and embrittlement conditions. On the other hand, a suitable upper bound for SRSPT punch rates should be found. It could be the one that produces in the edge of the notch the same CTOD growing rate taking place, for instance, in a C(T) pre-cracked specimen during a fracture mechanics test in environment at a slow rate, reproducing like this in both cases the same plasticity conditions in the crack tip.

7. References

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