USE OF THE SMALL PUNCH TEST UNDER STATIC LOAD IN HYDROGEN EMBRITTLEMENT SITUATIONS. ANALYSIS OF THE TEST CURVE.

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RESUMEN

En este trabajo se analiza un acero de alta resistencia bajo escenarios de fragilización por hidrógeno mediante los últimos avances en el empleo de las técnicas Small Punch en escenarios frágiles. En una primera etapa se fundamenta porqué el ensayo Small Punch (SPT) puede ser una alternativa simple y económica cuando el empleo de ensayos normalizados en ambiente no es viable.

A continuación se llevan a cabo ensayos SPT sumergidos bajo carga estática sobre probetas previamente cargadas en hidrógeno. Con ello se comprueba que esta tipología de ensayos SPT da como resultado curvas de que comparten similitudes con las del Small Punch Creep Test, la cual es analizada en profundidad. A partir de ello se lleva a cabo una análisis de la evolución de la probeta a lo largo del ensayo, y se propone un posible camino de trabajo futuro para la determinación de la velocidad de ensayo SPT idónea en condiciones de fragilización por hidrógeno.

PALABRAS CLAVE: Ensayo Small Punch bajo carga estática; Fragilización por hidrógeno;

ABSTRACT

In this work, a high strength steel is analyzed under hydrogen embrittlement scenarios, employing the latest advances in the use of Small Punch techniques in brittle scenarios. In a first stage it is analyzed why the Small Punch (SPT) test can be a simple and economical alternative method when the use of normalized tests in environment is not viable.

Subsequently, SPT tests carried out under static load using pre-charged specimens in hydrogen. It is shown that this type of SPT test give as a result curves that share similarities with those of the Small Punch Creep Test, which is analyzed in depth. From this, an analysis of the evolution of the specimen is carried out throughout the test, and a possible way for the determination of the suitable SPT punch rate in hydrogen embrittlement scenarios is proposed as a future work.

KEYWORDS: Small Punch Test under static load; Hydrogen embrittlement;

1. INTRODUCTION

A critical aspect concerning high strength steels working in these installations 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 [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) [3] or those typical of H_2S 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 7539-9 [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, the miniature test family was developed, which uses specimen sizes much smaller than those required by standard tests. Among these alternative techniques, the Small Punch Test is one of the most notables. The SPT is based on punching a reduced dimension plane specimen, which allows parameters such as the yield stress, ultimate tensile strength and even fracture toughness of metallic materials to be estimated with high reliability [5]. Over the last years some authors have proved the validity of the SPT when used in HE and SCC characterization [5÷11], having the advantage of being faster and easier to perform than standard tests. The main objectives of this work are:

- To propose an experimental methodology to perform characterizations in hydrogen embrittlement scenarios using the small punch test under static load, the specimen being pre-charged and submerged in the environment during the whole test. Also, some result- processing techniques employed in standard stress corrosion cracking tests and in both conventional creep and small punch creep tests will be described and used in this work.

- To provide an interpretation of the measures obtained from the test (punch displacement vs. time) identifying the main parameters extracted from it and comparing them to those defined by experimental standardized procedures.

The whole study is completed with hydrogen content measurements as a function of the microestructural features in order to explain the behavior of the steels in hydrogen embrittlement scenarios.

2. MATERIALS AND CONSIDERATIONS

2.1. Materials

For this work a Cr-Ni-Mn high-strength R5 grade steel was used, which is employed in the manufacture of large anchor chain links for off-shore platforms. It is obtained by means of quenching and tempering processes, acquiring the tempered martensite microstructure presented in Figure 1. This steel is received in the factory in bars (80 to 150mm of diameter), which are then forged to conform the links by bending forces. Its main mechanical properties were obtained, which are shown in Table 1.

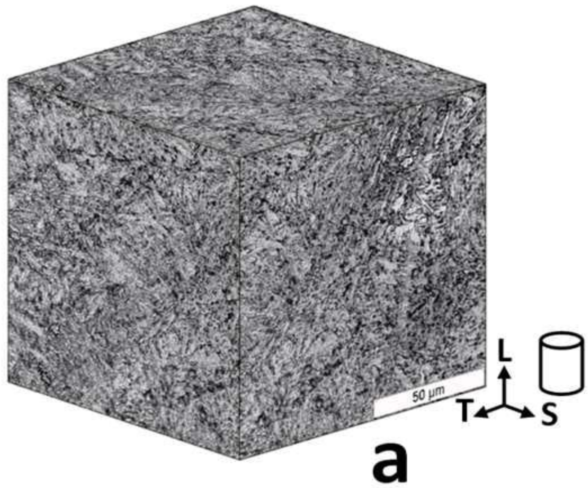

Figure 1. Cr-Ni-Mn steel microstrucure.

Table 1. Cr-Ni-Mn steel mechanical properties in air.

PARAMETER		Value
Yield Stress (MPa)		920
Ultimate Stress (MPa)		1015
Young's Modulus (GPa)		205
Ramber-Osgood Parameters	n	14,5
	α	1,15
$J_{0,2}$ (KN/m)		821
$K_{J0,2}$ (MPa*m$^{1/2}$)		410
Hardness (HBW30)		352

2.2. Simulating hydrogen embrittlement

An environmental condition known as cathodic charge, 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 huge 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 shows a set-up of the method used in this work.

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 [1]. In this study, for the cathodic charge situations, an environmental condition in accordance with [12,8,11,13] 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 Pessouyre's method [25]. 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. An embrittlement level of 5mA/cm^2 was employed.

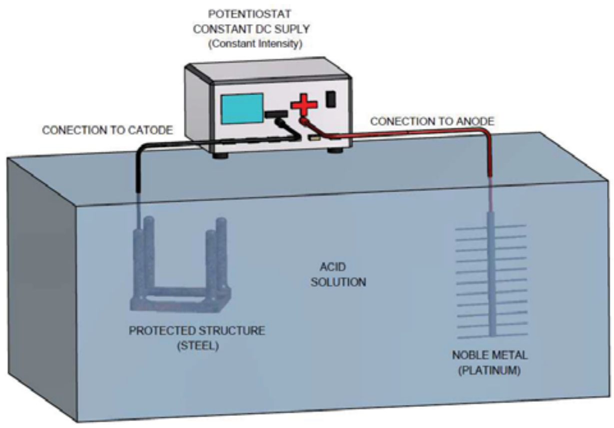

Figure 2. Schematic of the cathodic charge method.

2.3. The Small Punch test

The SPT can test in-service structures [15,16,17], since the extraction of a sample with such a small amount of material required for the Small Punch Test does not compromise the integrity of the component. It has been successfully employed in the evaluation of the tensile [15,18,19] and fracture [15,19] properties of different materials. Over the last few years, many groups have developed creep behavior models [20÷22]. This technique has been applied to characterize embrittlement processes in steels, such as the evolution of material properties with neutron or proton irradiation [23,24], the brittle-ductile transition temperature of metallic materials [28], stress corrosion cracking scenarios [5,6], or, more recently, environmental embrittlement [7÷11].

The Small Punch Test consists of punching a plane specimen of small dimensions and deforming it until fracture. A schematic of the device and samples used for the performance of these tests is represented in Figure 3. The typical shape of the registers obtained from SPT tests is presented in Figure 4.

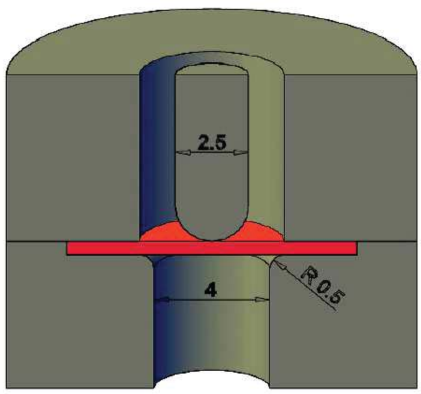

Figure 3. Schematic of SPT device and samples.

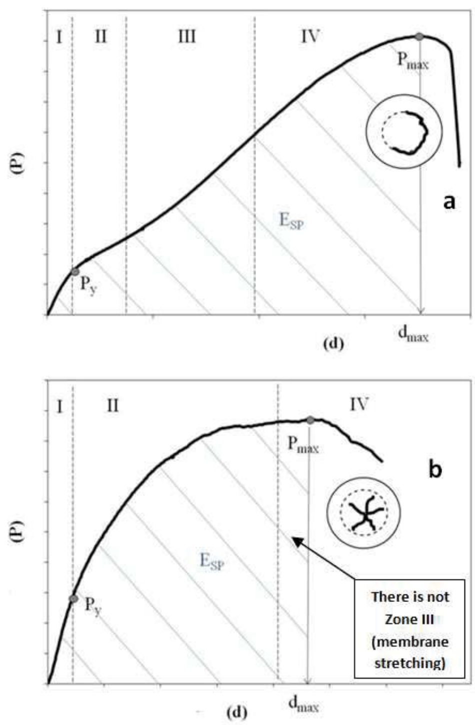

Figure 4. Typical SPT curves. Extracted from [14]; a) Ductile materials; b) Brittle behaviour.

2.3. SPT considerations in aggressive environment

Standards for conventional environmental characterizations [29,30,4,31] require the tests to be performed at very low (quasi static) solicitation rates, while the specimen is totally submerged. This ensures a steady state of hydrogen embrittlement [12,32,33,34].

The latest research shows that the small punch test is a useful tool when applied to the estimation of steel properties in hydrogen embrittlement and stress corrosion cracking scenarios. However, SPT estimations could be conservative [7] due to the punch rate conventionally used [16], that in the most aggressive environments is too high to allow the hydrogen damaging capability to act completely in the new surfaces of the crack tip in generation. In order to obtain more accurate results, it would be useful to reproduce the same micromechanisms that take place during fracture mechanics tests. To fulfill this recommendation, recent publications have pointed to the advantages of performing the SPT at a very low constant rate or static load tests [5,6] while submerging the specimen during the whole process [8÷11].

3. EXPERIMENTAL METHODOLOGY

In a cathodic charge environment under a 5mA/cm^2 level, seven SPT specimens, obtained from the straight zone of a Ø120mm chain link, were tested. The specimens were machined in accordance with [15,16], in an orientation such that the plane faces were transversal to the longitudinal axes of the components (cylindrical). Their specific dimensions were 10mmx10mm of section and 0.5±0.01mm of thickness. The test plan carried out is presented in Table 2.

Table 2. SPT test plan

LOAD (N)	RESSULT
883	**tr**; Vsp
667	**tr**; Vsp
608	**tr**; Vsp
583	**tr**; Vsp
534	**tr**; Vsp
509	**tr**; Vsp
461	**tr**; Vsp

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 [12]. Subsequently, the load was softly applied by an endless screw system on the specimen subjected to the environment. The test was considered over when the sample failure occurred, thus allowing both the time up to failure (tr) and the punch rate (Vsp) to be obtained. For the purpose of this test, an experimental device was designed and built, as presented in Figures 5 and 6.

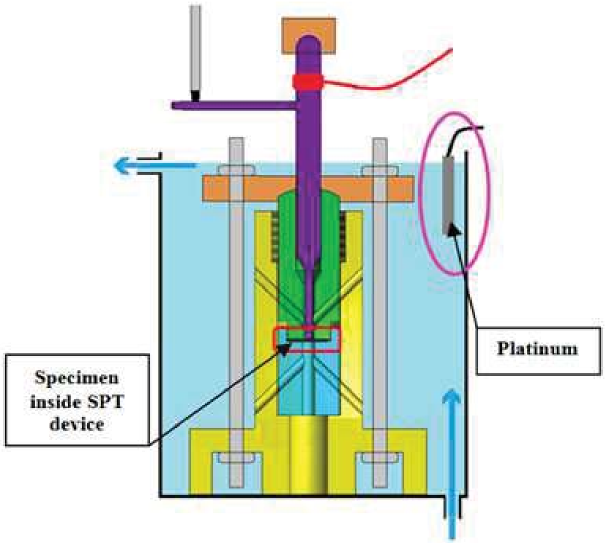

Figure 5. Schematic of SPT static load device.

Figure 6. Device during a test

4. RESSULTS AND DISCUSSION

Figure 7 presents the curves registered from the SPT tests carried out. In all the cases, the shape of the curves clearly resembles those obtained from creep tests, both conventional or small punch creep tests [21,22].

In these curves three zones can be distinguished: The first one, of the first indentation on the sample and load settlement combined with crack incubation, in which an important punch displacement takes places in a short time; the second zone, after the first turning, at which a quasi-constant punch rate takes place while cracks grow; the third zone, after the second turning, in which the cracks have grown to such an extent that the rupture of the specimen becomes imminent, causing again an important punch displacement in a short time and the total drilling of the specimen. The second part, as happens in creep scenarios [21,22], is the most representative and could be characterized by a steady displacement rate V_{sp}, which is the key parameter to be analyzed. In figure 7 this displacement rate, V_{sp}, is presented for each case, calculated by a linear fit of punch displacement and time in the second zone (between the first and second turnings of the curve).

In all the tested samples, fractographic images were obtained of the opposite side to the punch application. Figure 8 shows images obtained from two representative conditions [15,7]. Figure 8a shows a mainly brittle transgranular fracture [2,35,36,12], corresponding to the test under 667N. This trend is present in all the tests carried out except the one under 461N, which was stopped before the specimen rupture, in which emerging cracks can be observed.

The test under 461N (lower load applied) was stopped before the rupture of the specimen, after 278 hours had elapsed (almost 12 days). The fractographic analysis carried out on this specimen shows evident damage displayed as cracks, despite surpassing a long period of time without reaching the final rupture. As shown in Figure 8b, cracks appeared in the specimen which had the typical star shape stated in the bibliography [15,7,10,11], which means that the failure was predisposed to take place. For this reasons the time elapsed was used as the lower bound of the time to rupture in this case. When observing the lower part of Figure 13b, smaller cracks can be seen on the specimen surface revealing the grain boundaries, which means that the type of surfaces of the cracks will probably be of the same type as the rest of specimens tested.

In order to analyze the process and micromechanisms that take place during the second stage, where a quasi-constant punch rate takes place, some interrupted tests were carried out.

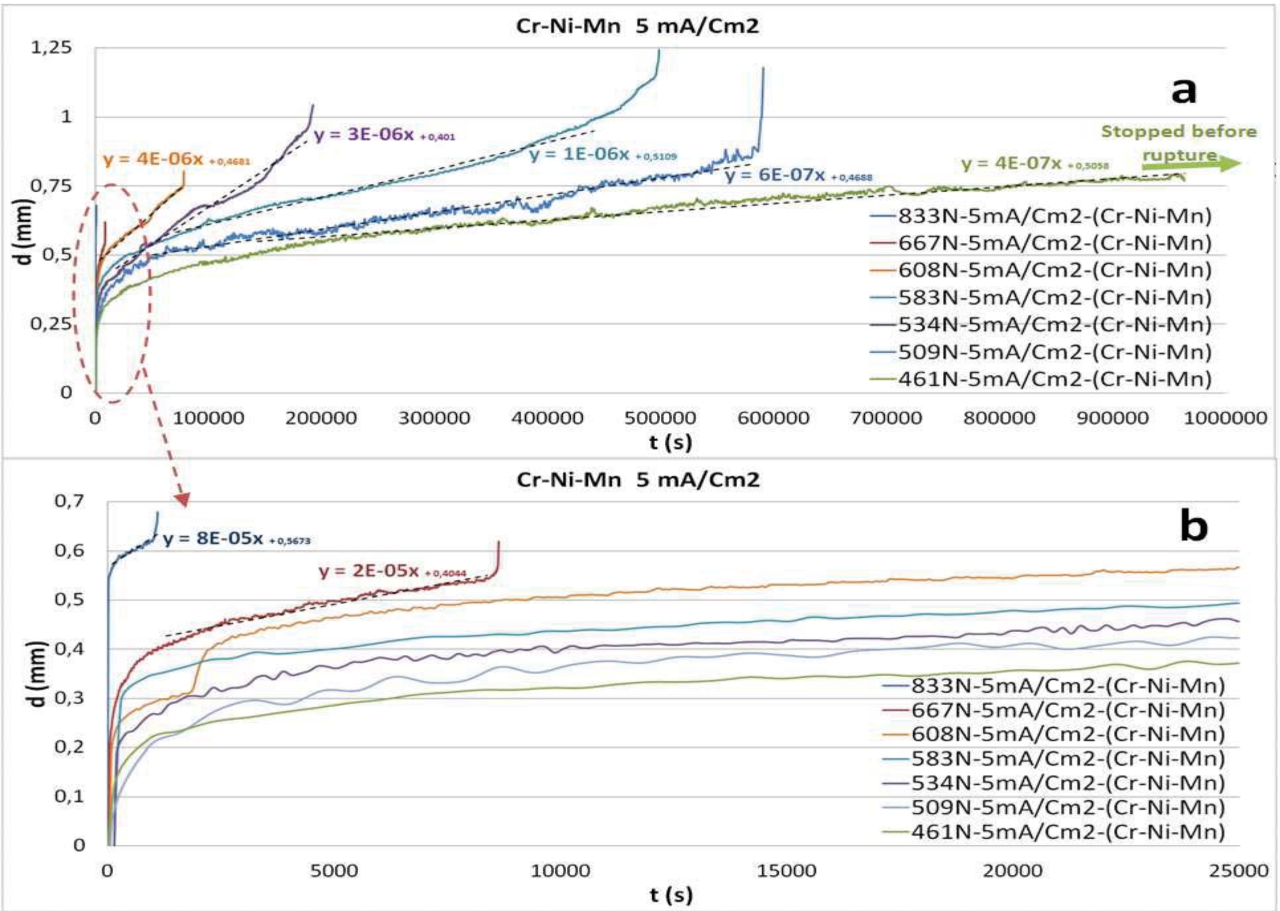

Figure 7. a) SPT curves performed. **b)** Zoom of those curves with lowest times to rupture.

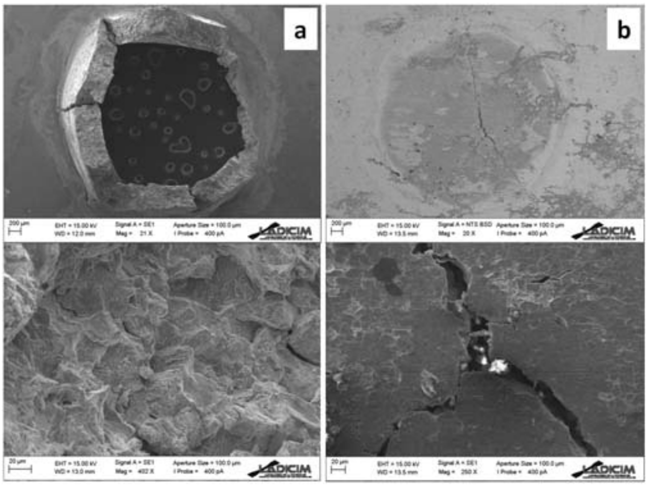

Figure 8. Fractographies under static SPT tests. a) Test under 667 N. b) Test under 461N.

The test under 5 mA/cm^2 and 608 N of load was selected thanks to its short duration, thus offering operational simplicity. As can be observed in figure 9, seven interrupted tests were performed under this condition, comprising times from 45 minutes (crack initiation) to 21 hours and 30 minutes (imminent rupture of the specimen).

The first sample was stopped right at the first turning of the curve, after 45 minutes of test, where it can be considered that the second zone of the curve starts. In the SEM image, a tiny emerging crack can be observed on the lower face of the specimen (most stressed part).

In the following tests, comprising times from 3 to 16 hours, it can be observed how primary cracks start growing in a star shape from the center to the periphery, and obviously through the specimen thickness, while the specimen bends pushed by the punch. Cracks grow successively until they reach the border sides of the test area (4mm of diameter), conforming the typical brittle star shape, so that, when they have grown sufficiently, this leads to a general bending of the lips of the specimen in contact with the clamp, generating a circumferential crack of 4mm of diameter (see Figure 16 at 21 hours of test). There are also secondary micro-cracks present in the specimen, as the ones presented in Figure 10, that take place in the interfaces between the grains of material causing detachment between them.

Finally, a sample was stopped just before its rupture (21 hours 30 minutes), when the curve was entering the last turning, which leads to the imminent drilling of the specimen by the punch.

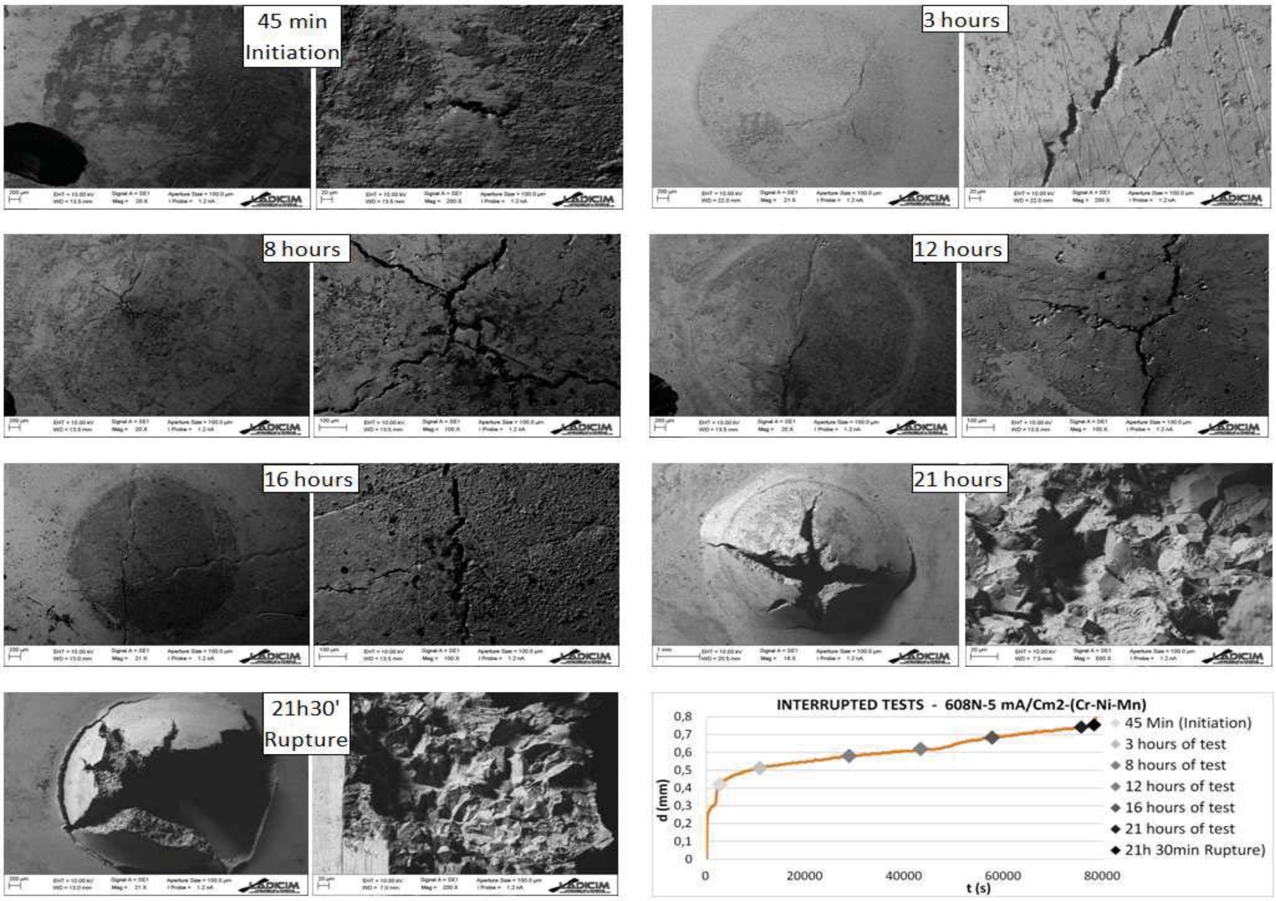

Figure 9. Set of interrupted static SPT tests at different stages (times of test) under 608 N.

In this case, it can be observed how several radial cracks have evolved up to the borders of the test zone, while others were on their way to doing this, until they were large enough to generate a circumferential crack at the contact between the clamp and the specimen. It can even be observed how one of the sectors between two of the main cracks has been pushed out and is already missing.

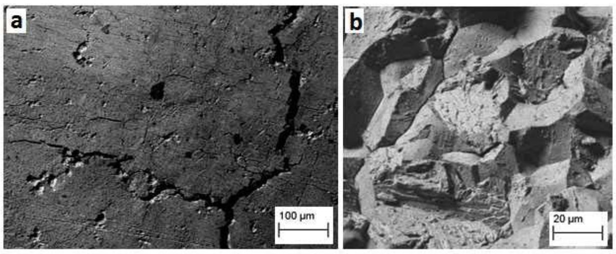

*Figure 10. Details of the secondary cracks present on the interrupted static SPT tests under 608 N. **a)** Primary cracks observed on the external face of the specimen (most stressed zone). **b)** Secondary cracks observed on the interfaces between the grains of material causing detachment between them.*

The results analyzed show that a crack propagation process takes place in the second zone modifying the sample's flexibility, and therefore the punch position, its displacement rate being constant. A similar analysis was made of all the tests performed, showing the relationship between the applied load and the time to failure, which is strongly dependent on the displacement rate. In Figure 11 the whole set of results obtained is presented. For each test, the time to rupture versus the force applied is represented, showing how the lower the force applied, the higher the time to rupture. The trend described has been plotted using an exponential fit.

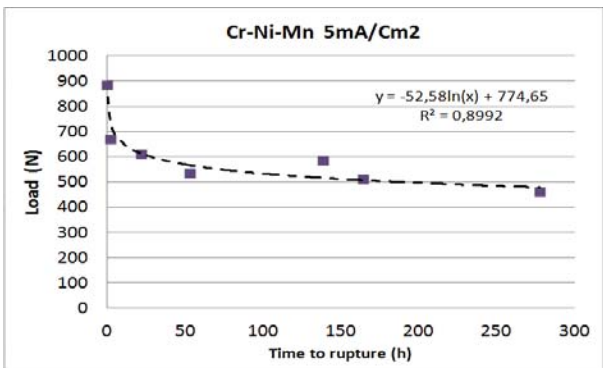

Figure 10. Representation of the load applied versus time to rupture under static SPT tests.

This behavior is present in other type of tests where the environment governs the behavior, such as standard stress corrosion cracking tests [33], or creep tests, both conventional or small punch creep tests [21,22]. In these scenarios, the time to rupture increases when the effort applied is progressively decreased, up to a threshold effort at which the time to rupture tends to infinity, so that for lower values the failure does not take place. Regarding this, it can be stated that the Small Punch test is a tool which can be used to predict a parameter of failure in embrittlement scenarios, such as the threshold SPT load, or to estimate the threshold stress in the material.

Below, in Table 4, the results of the whole SPT experimental campaign are summarized. For each test, the time to rupture, t_r, and the punch rate in the quasi-constant zone, V_{sp}, are indicated.

Table 4. Results obtained from SPT tests.

LOAD (N)	tr (h)	Vsp (mm/s)
883	**0.30**	**$8.0 \cdot 10^{-5}$**
667	**2.40**	**$2.0 \cdot 10^{-5}$**
608	**21.86**	**$4.0 \cdot 10^{-6}$**
583	**138.89**	**$1.0 \cdot 10^{-6}$**
534	**53.40**	**$3.0 \cdot 10^{-6}$**
509	**164.27**	**$6.0 \cdot 10^{-7}$**
461	**>277.78**	**$4.0 \cdot 10^{-7}$**

5. CONCLUSSIONS AND FUTURE WORK

In this paper, the Small Punch Test has been re-validated as a method for characterizing materials in embrittlement scenarios; some basic guidelines to perform this characterization have also been established. A high-strength Cr-Ni-Mn steel has been studied, characterizing it in an aggressive environment by Small Punch tests under static load

In a first step, conventional standardized hydrogen embrittlement and stress corrosion cracking testing techniques have been analyzed, underlining their weaknesses, such as the long period of time required, the inherent difficulties in undertaking the experimental set-ups, and mainly the impossibility of performing the characterization of particular zones such as welded joints or thin in-service components. Thus, the small punch test was proposed as a simple and economic alternative when it is not possible to perform standard tests in adverse environmental situations.

Subsequently, an experimental methodology to perform characterizations in hydrogen embrittlement scenarios using the small punch test has been described. It was proposed for this aim to use SPT under static load tests with the specimen pre-charged and submerged in the environment during the whole test. Also some other result processing techniques widely used in standard stress corrosion cracking tests, and both conventional creep and small punch creep tests, were shown to be adequate.

Based on the experimental results it was proved that:

- The small punch curve under static load in embrittlement environments has the same shape as the one that takes place during both conventional and small punch creep tests, having three zones. The first zone governs the indentation and settlement of the load, the second steady state zone governs the crack propagation and has a quasi-constant punch rate and the third zone governs the final stability and rupture of the specimen.

- The above curve shape is repeated for different loads under the same environment, showing lower punch rates and higher times of rupture when the load applied decreases. This behavior allows a threshold load to be defined, when the time to rupture tends to infinity, because of an environment-load combination that is not high enough to cause damage in the specimen, so that the failure will never occur.

- Interrupted tests were carried out on the steady state zone in order to find out what produces a constant punch rate. The presence of primary radial cracks combined with secondary micro-cracks was observed in grain boundaries growing in the specimen, leading to a continuous displacement rate in the sample. Finally, when the cracks have grown sufficiently, a circumferential crack at the contact between the clamp and the specimen is generated as a result of the bending of the specimen in that zone, immediately prior to the final failure.

The next step in this line of research will be the estimation of a very slow, but non-static, punch rate. It is proposed to employ static load SPT tests to reach asymptotic Load-t_r curves (this should include more tests, materials and environments), determining the average punch rate in the second region as the suitable to perform non-static, but very slow punch rate, SPT test in environment.

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