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Optimization of Step Times for ASTM F1624 Methodology Applied to Small Punch Tests in Aggressive Environments

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Abstract: Threshold stress in aggressive environments is usually determined by tests under constant load, which are very time-consuming, so the incremental loading technique published in the ASTM F1624 standard was created to solve this issue. This approach has been recently applied to Small Punch tests, but it requires an optimization of the incremental step times, which is carried out in this work. Three medium- and high-strength quenched and tempered steels of 35, 50 and 60 HRC are exposed to three different cathodic polarization environments of 1, 5 and 10 mA/cm^2 in 1N H_2SO_4 acid electrolyte with a Platinum anode, studying in each case three different step durations of one-quarter, one-sixth and one-eighth of the ones indicated in ASTM F1624. Optimal step times for Small Punch tests are derived from this work as one-sixth of the ones recommended in ASTM F1624 for tensile specimens, which are 20 min and 40 min for steps 1–10 and 11–20, respectively, in the case of $33 \leq$ HRC < 45 steels, 10 min and 20 min for steps 1–10, 11–20 in the case of $45 \leq$ HRC < 55 steels, and 10 min for steps 1–20 in the case of HRC $\geq$ 55 steels.

Keywords: threshold stress; small punch test; constant load steps

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1. Introduction

Currently, high-strength steels are widely employed in high-responsibility industrial applications, thanks to their higher properties, such as tensile strength, hardness and yield strength. This is the case in the automotive, oil and gas, power plants or aerospace industries, where components tend to be more and more durable and lighter while maintaining their properties [1–3].

The disadvantage of using high-strength steels is their higher susceptibility to environmental damages, such as stress corrosion cracking or hydrogen embrittlement [4], as they contain higher amounts of alloying elements than regular carbon steels, finer microstructure and less carbon [5–8]. So, its employment leads to the need for more tests to control their susceptibility to aggressive environments, with the scope of better assessing their behavior during their life in service [9,10].

The stress limit that these high-strength steels can withstand before failing in aggressive environments is known as threshold stress, and it depends on the combination of the alloy and the specific environment. To obtain the value of the threshold stress, the ISO 7539 [11] and the ASTM E1681 [12] standards are usually used. They are based on slow-strain-rate tests [10,13] and on tests under constant load, generally employing cylindrical specimens, which have the disadvantage of taking a very long time (up to 10,000 h per test [12,14,15]), and a certain number of specimens to reach enough accuracy [16,17].

Time consumption may be a scarce good on many occasions, on the one hand because of the need for fast results in order to make engineering decisions, and on the other hand because results can sometimes be less accurate due to the difficulty in the assurance of constant environmental conditions during the whole test [9]; in the same way, testing a higher number of samples may induce higher accuracy, but will also mean more time consumption.

These disadvantages can be solved by applying the ASTM F1624 [18] instead, which allows a very interesting reduction in the test duration when obtaining the threshold stress. While ASTM F1624 [18] and ISO 7539 [11], or ASTM E1681 [12], have similarities in the type of sample proposed, cylindrical in both cases, their philosophy is totally different. The classical ISO 7539 [11] and ASTM E1681 [12] employ a set of specimens tested under maintained loads, waiting for the load that does not produce failure, considered the threshold, which can lead to tests up to 10^4 h; another option is a single-specimen test at very slow rates ($\varepsilon = 10^{-5}$ s^{-1}). The newer methodology from ASTM F1624 [18], which is just valid for steels with 33HRC or harder, consists of applying constant load steps, incrementing its load until the failure of the specimen takes place, minimizing the experimental campaigns to only a few days and a minimum of three specimens tested in the environment (two of them with a difference of 5%, which gives accuracy to the result).

In many cases, it is difficult to obtain standard-size specimens, generally in the range of 80–200 mm long and Ø6–12 mm, such as local welded joints or in-service components. In those scenarios, the SPT (Small Punch test) is a very interesting alternative which can be used to estimate threshold stresses in aggressive environments, such as a hydrogen embrittlement environment [14,15].

The SPT, developed in the 1980s and recently standardized, is a quasi-non-destructive test that consists of punching a small plate-shaped specimen until its fracture (see Figure 1), while parameters such as the load applied or the deflection of the most stressed face of the specimens are registered. Its capacity to be applied to aggressive scenarios has been proved during the last decade [14,15], with the most convenient methodology determined to be the one consisting of applying incremental constant loads according to the step loading technique presented in ASTM F1624 [18].

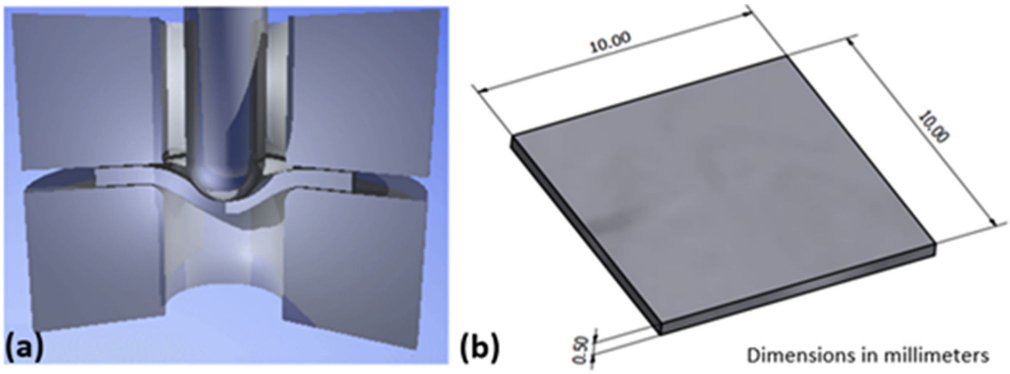

Figure 1. (**a**) Schematic of Small Punch test device; (**b**) SPT specimen geometry.

When applying SPT for the determination of threshold stress, it makes sense to reduce the step durations based on the fact that the hydrogen diffusion time to reach a steady state of saturation in the whole sample must be much shorter in an SPT specimen 0.5 mm thick than in a standard cylindrical specimen with ø6–12 mm, taking into account that diffusion time is proportional to the square of the thickness [19]. But it has also been proven that the real time necessary to embrittle SPT specimens, around 2 h according to the literature [14,15], is higher than the one theoretically estimated from calculations if using the hydrogen diffusion coefficient in iron [20].

The first publications that use SPT for this scope [14,15] have proven the methodology to obtain threshold stress in aggressive environments using convenient arbitrary times. The present paper investigates different step durations and carries out an optimization for each of the three steels' hardness ranges considered by ASTM F1624 [18]: $33 \leq$ HRC < 45, $45 \leq$ HRC < 55, and HRC ≥ 55. With all this, Section 2 provides a description of the materials used in this research and the methods followed for the analyses, Section 3 presents and discusses the obtained results, and Section 4 provides the corresponding conclusions.

2. Materials and Methods

2.1. ASTM F1624 Method

The methodology described in ASTM F-1624 [18] is a quick method to obtain the threshold stress or the threshold load to start subcritical crack growth in aggressive environments in the case of medium- to high-strength steels. First of all, a tensile test must be performed in air according to ASTM E8 [21] to obtain P_{FFS} (Fast Fracture Load). Afterwards, the step protocol to be applied is defined based on this Fast Fracture Load, fixing 20 step sequences of $P_{FFS}/20$ load each, until the sample fails, obtaining at this moment the $P_{th\text{-}1}$ load. The next sequence leads to $P_{th\text{-}2}$, $P_{th\text{-}3}$, … $P_{th\text{-}n}$. Each new sequence is determined by increasing the threshold value obtained in the prior one by 10% (1.1 $P_{th\text{-}(n\text{-}1)}$) and dividing this load again in 20 steps. A minimum of 3 sequences is required, and the final threshold load is obtained when the difference between two sequences threshold loads is lower than 5%. Figure 2 [18] summarizes the process (presenting an example where the threshold is reached after 4 sequences).

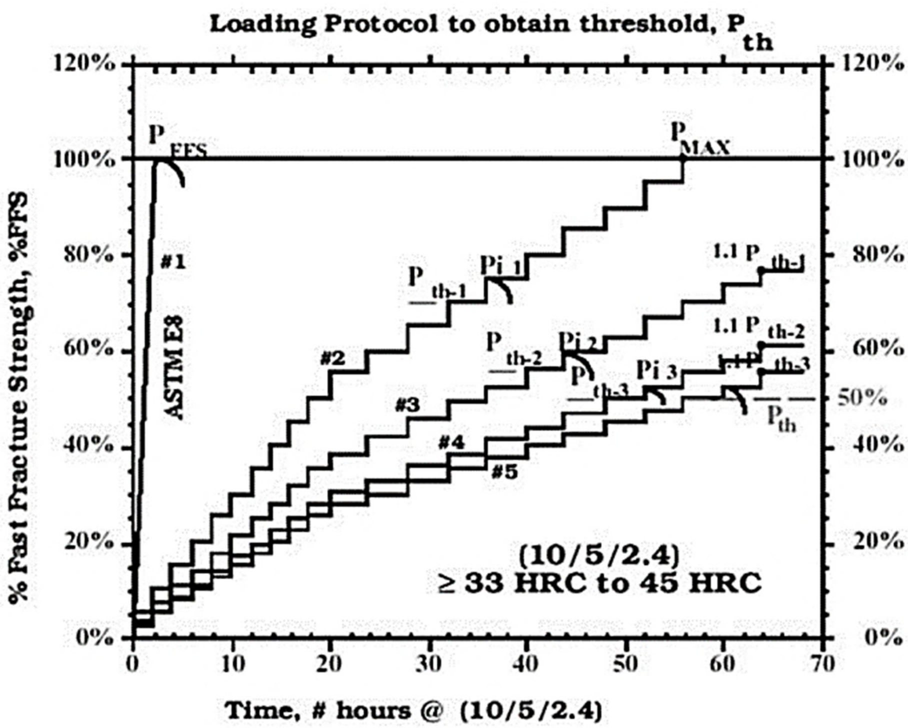

Figure 2. Schematic of the ASTM-F1624 methodology for a 33–44 HRC steel; extracted from [18].

The step duration times, so-called step load protocols, are a function of the steel hardness, as indicated in Table 1. In the lowest and medium ranges of hardness, 33 ≤ HRC < 45 and 45 ≤ HRC < 55, steps 1 to 10 have a certain duration, while steps 11 to 20 have double duration. This has the aim of allowing all the possible environmental effects in the areas of the specimen under plasticization to be close to the threshold, obtained by the effect of lower solicitation rates than at the initial steps where elasticity governs the process. In the highest hardness range, HRC ≥ 55, all 20 steps last the same as these high-strength steels are more affected by aggressive environments, which means lower solicitation rates in harder steels, as widely stated in the literature for SPT [20] and also for conventional tests [22].

Table 1. Step load profiles based on the steel hardness, according to [18].

Hardness	Step	Step Load	Step Time (h)	Profile Code [18]
$33 \leq$ HRC < 45	1–10 11–20	5% of P_{FFS}	2 4	(10/5/2.4)
$45 \leq$ HRC < 55	1–10 11–20	5% of P_{FFS}	1 2	(10/5/1.2)
HRC ≥ 55	1–20	5% of P_{FFS}	1	(10/5/1)

2.2. Application of Step Loading Technique from ASTM F1624 [18] to Small Punch Test

When applying the step loading methodology from ASTM F1624 [18] to SPT, the same standard must be followed as explained in the previous epigraph with the only difference of adapting the step durations [14,15]. It is to be considered that, prior to carrying out each step profile, the Small Punch specimen must be immersed in the environment for enough time to ensure complete embrittlement: in [14,15], 2 h is proposed, as usually employed in SPT environmental characterizations [22].

Considering, as aforementioned, that the diffusion time is proportional to the square of the thickness [19], the required conditions of an SPT specimen 0.5 mm thick versus a standard cylindrical ø6–12 mm one will be much shorter. In practice, it has been found that the embrittlement time of 2 h in SPT specimens is approximately 6 times shorter than that commonly used for ø6–12 mm cylindrical tensile specimens, where it takes around 12 h. Therefore, for SPT specimens, it is logical to also think of step durations around 6 times shorter than the ones proposed in [18] for standard specimens, which were the ones proposed in [14,15] for the first experiences as a reasonable and convenient choice.

Based on this, various times are tested in the present paper in the order of magnitude around 6 times shorter than standard specimens, but half and double times are also explored, meaning 1/4, 1/6 and 1/8 times the ones considered in ASTM F1624 [18]. So, depending on the steel hardness ranges, the experimental plan shown in Table 2 is carried out in order to find the optimal (shortest possible) step durations to obtain the Small Punch threshold load.

Table 2. Step times investigated in the present work.

Hardness	Step	Proposal #1 (min)	Proposal #2 (min)	Proposal #3 (min)
$33 \leq$ HRC < 45	1–10 11–20	30′ 60′	20′ 40′	15′ 30′
$45 \leq$ HRC < 55	1–10 11–20	15′ 30′	10′ 20′	7′ 15′
HRC ≥ 55	1–20	15′	10′	7′

To determine the optimal step durations, the method proposed here consists of comparing the threshold loads obtained in each case. Whenever the threshold load for shorter steps remains in a range of ±5% versus previous steps duration, then it can be reduced. If the threshold load deviates more than ±5% versus the previous step duration (usually increasing its value), then the step duration used is too short and the embrittling effect cannot be completed. Finally, the optimal step duration is chosen as the one which allows the threshold load to remain constant in each steel hardness range.

2.3. Materials and Environment Employed

Three heat-treated steels, one from each of the hardness ranges considered in [18], are employed in this work, with 35 HRC, 50 HRC and 60 HRC, respectively. All of them are obtained by quenching and tempering. The 35 HRC one is a thermomechanically treated TMCR 420 steel which is microalloyed and weldable and has a ferritic–pearlitic microstructure, containing 0.08%C. The 50 HRC and 60 HRC ones were obtained by quenching and tempering processes with Uddeholm Arne-treatable steel with just the aforementioned hardness requirement, resulting in a tempered martensitic microstructure in both cases, containing 0.95%C.

The aggressive environment used for the experiment consisted of an acid liquid solution 1N of H_2SO_4 in distilled water, prepared according to the Pressouyre method [19] with additions of As_2O_3 and CS_2, and cathodic polarization between a Platinum electrode and the steel specimen. This method is frequently found in the literature (i.e., [14–16,22]). To simulate different levels of aggressiveness, 1, 5 and 10 mA/cm^2 were applied on the submerged specimen, meaning there was a total of 9 different scenarios combining the various hardness levels and environments. Figure 3 shows an image of the experimental setup.

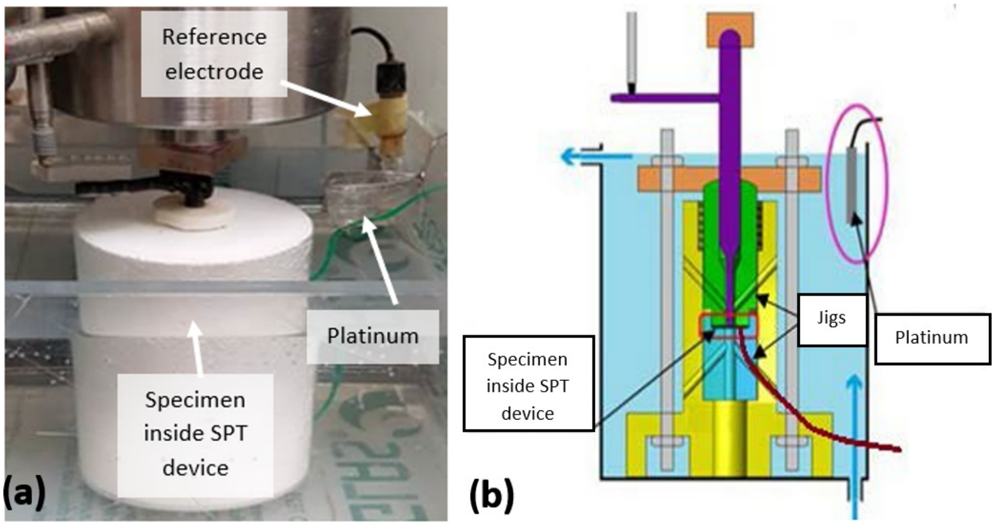

Figure 3. (**a**) Experimental setup to carry out the SPT tests while embrittling the sample; (**b**) schematic of its working principle.

3. Results

All the results from the experimental campaign are detailed in Tables 3–5, which collect the threshold load results from each P_{th} step. As an illustrative example of this, and in order not to extend the present paper too much, Figures 4–6 present the results obtained for some of the different combinations of steel and aggressiveness studied: 35 HRC material in the environment of 1 mA/cm^2, 50 HRC material at 1 mA/cm^2, and 60 HRC at 10 mA/cm^2. The whole set of results are plotted in a summarized way in the bar graphs shown in Figures 7–9 (same as in Tables 3–5). In each case, the different step durations investigated are plotted in different colors for one-eighth, one-sixth and one-quarter of ASTM F1624 [18] times; the SPT in air according to EN10371 [23] is also shown in grey. For 35 HRC, pink, light green and dark green correspond to 15′–30′ (one-eighth), 20′–40′ (one-sixth) and 30′–60′ (one-quarter), respectively. For 50 HRC, blue, red and green correspond to 7′–15′ (one-eighth), 10′–20′ (one-sixth) and 15′–30′ (one-quarter), respectively. For 60 HRC, red, pink and green correspond to 7′ (one-eighth), 10′ (one-sixth) and 15′ (one-quarter), respectively. The different threshold loads obtained are marked with a circle indication.

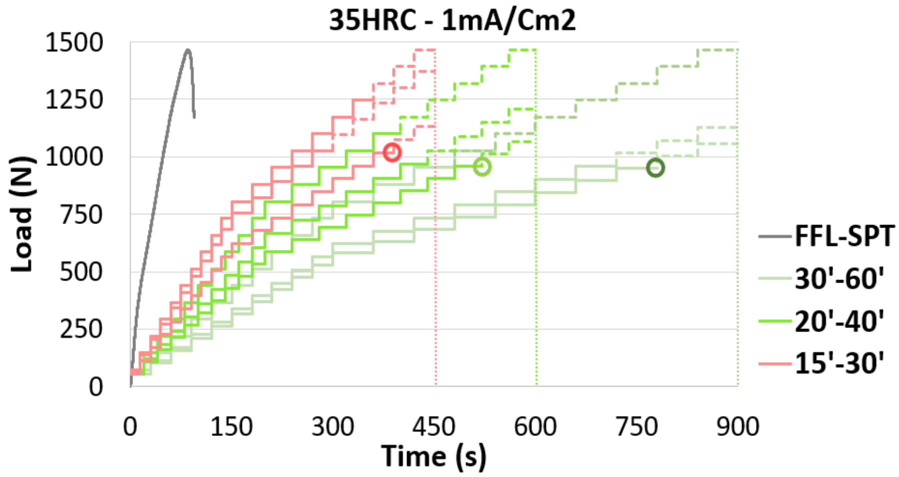

Figure 4. Step durations under study for 35 HRC steel in 1 mA/cm^2 environment.

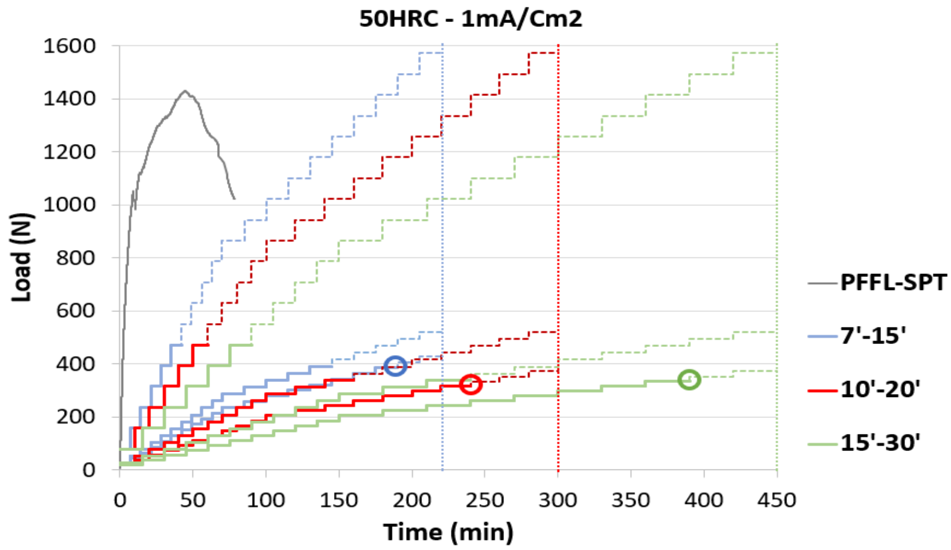

Figure 5. Step durations under study for 50 HRC steel in 1 mA/cm^2 environment.

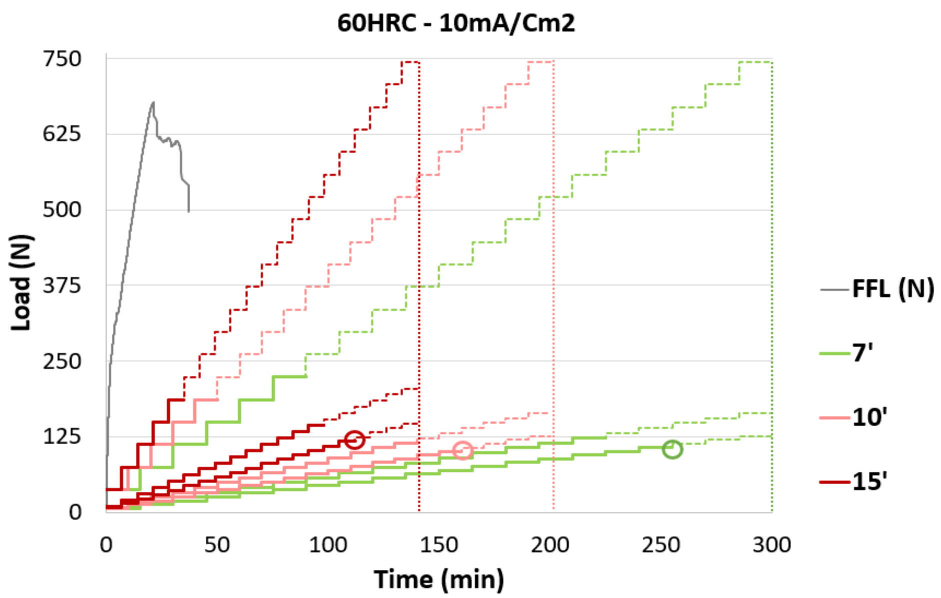

Figure 6. Step durations under study for 60 HRC steel in 10 mA/cm^2 environment.

Tables 3–5 below present all the results for the three steels under analysis, which are then also summarized in Figures 7–9.

In all the cases, the shortest step times (one-eighth of ASTM F-1624 [18]) show threshold values clearly higher than the ones obtained for the other two step times under study (one-sixth and one-quarter of ASTM F-1624 [18]). This means that the shorter times option gives rise to a load rate (or deformation rate) in the specimens too fast to reproduce the environmental effect, which will cause overestimation of the threshold value and compromise safety, so this option will be discarded.

In the case of the other two step times, very close values are present in all the cases. The percentage difference when comparing the threshold values obtained takes as a base the situation of one-sixth of the ASTM F1624, finding that the threshold values obtained differences smaller than 5% between one-sixth and one-quarter of the ASTM F-1624 [18]. This fact makes it possible to state that the shortest times that do not produce a significant variation in the threshold, thus being safe, are one-sixth the time recommended in ASTM F1624 for tensile specimens: 20 min and 40 min for steps 1–10 and 11–20, respectively, in the case of $33 \leq$ HRC < 45 steels, 10 min and 20 min for steps 1–10 and 11–20 in the case of $45 \leq$ HRC < 55 steels, and 10 min for steps 1–20 in the case of HRC $\geq$ 55 steels.

Table 3. Results for the 35 HRC steel under the different step durations investigated.

Material and Environment	Steps Duration	$P_{FFL\text{-}SPT}$ (N)	$P_{th\text{-}1}$ (N)	$P_{th\text{-}2}$ (N)	$P_{th\text{-}3}$ (N)	Difference
35 HRC 1 mA/cm^2	15′–30′	1466	1246	1028	1018	+6%
	20′–40′	1466	1099	968	958	-
	30′–60′	1466	1026	959	950	−1%
35 HRC 5 mA/cm^2	15′–30′	1466	953	786	778	+16%
	20′–40′	1466	880	677	671	-
	30′–60′	1466	880	677	671	0%
35 HRC 10 mA/cm^2	15′–30′	1466	880	774	766	+25%
	20′–40′	1466	806	621	615	-
	30′–60′	1466	806	621	615	0%

Table 4. Results for the 50 HRC steel under the different step durations investigated.

Material and Environment	Steps Duration	$P_{FFL\text{-}SPT}$ (N)	$P_{th\text{-}1}$ (N)	$P_{th\text{-}2}$ (N)	$P_{th\text{-}3}$ (N)	Difference
50 HRC 1 mA/cm^2	7′–15′	1428	471	389	385	+22%
	10′–20′	1428	471	324	321	-
	15′–30′	1428	471	324	321	0%
50 HRC 5 mA/cm^2	7′–15′	1428	393	346	342	+18%
	10′–20′	1428	314	294	291	-
	15′–30′	1428	314	294	291	0%
50 HRC 10 mA/cm^2	7′–15′	1428	393	302	299	+18%
	10′–20′	1428	314	259	257	-
	15′–30′	1428	314	259	257	0%

Table 5. Results for the 60 HRC steel under the different step durations investigated.

Material and Environment	Steps Duration	$P_{FFL\text{-}SPT}$ (N)	$P_{th\text{-}1}$ (N)	$P_{th\text{-}2}$ (N)	$P_{th\text{-}3}$ (N)	Difference
60 HRC 1 mA/cm^2	7′	677	223	197	195	+7%
	10′	677	223	184	182	-
	15′	677	223	184	182	0%
60 HRC 5 mA/cm^2	7′	677	223	160	158	+8%
	10′	677	186	143	142	-
	15′	677	149	147	146	+3%
60 HRC 10 mA/cm^2	7′	677	186	133	132	+16%
	10′	677	149	115	114	-
	15′	677	149	115	114	0%

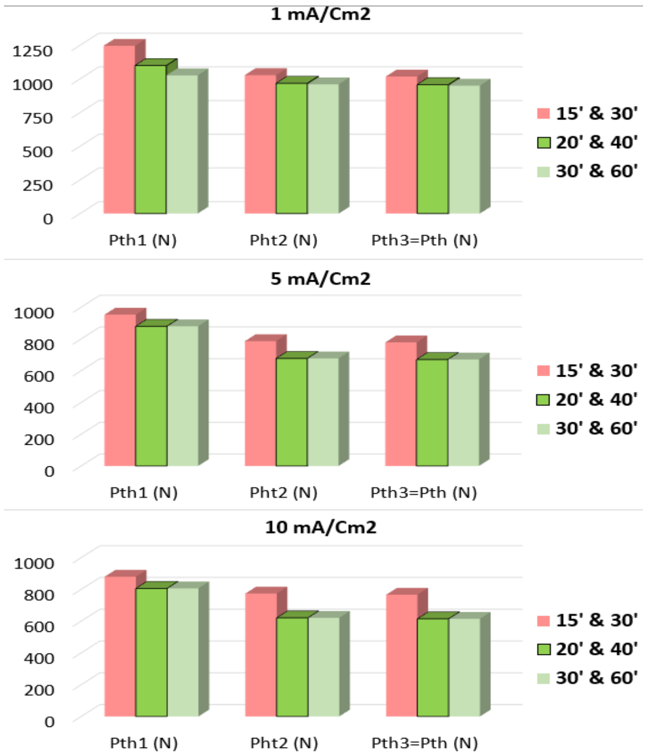

Figure 7. Threshold loads obtained under different step durations for the example case of 35 HRC steel in 1, 5 and 10 mA/cm^2 environments.

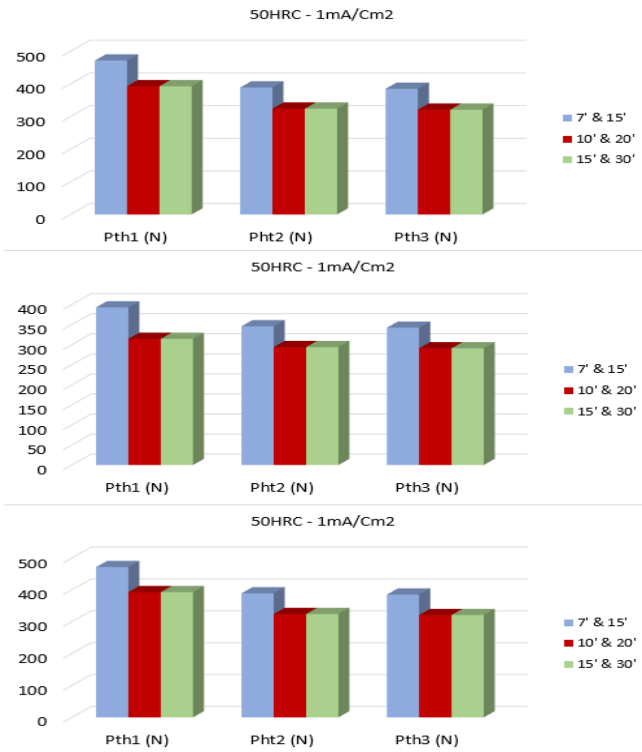

Figure 8. Threshold loads obtained under different step durations for the example case of 50 HRC steel in 1, 5 and 10 mA/cm^2 environments.

Figure 9. Threshold loads obtained under different step durations for the example case of 60 HRC steel in 1, 5 and 10 mA/cm^2 environments.

Figure 10 shows an example of the 35 HRC steel after the test at 10 mA/cm^2 under step times of 20 and 40 min (for steps 1–10 and 11–20), presenting SEM images of how the punch trespassed it (left) at the moment of the failure, as well as a micro image of its cross-section (right); in this case, the typical embrittled micromechanism discussed in the literature for SPT tested under a constant load in aggressive environments [14] can be observed. A transgranular fracture and secondary cracking present as a decohesion across the thickness can be observed, which mark some of the grain boundaries. It is important to note that this effect is already present in the least hard of the steels studied (35 HRC), and it is increased in the hardest steels (50 HRC and 60 HRC), more affectable by aggressive environments. This fact allows us to state that the micromechanisms present in SPT step loading tests under the proposed times are the correct ones for environmental characterizations for the three hardness ranges proposed in ASTM F1624, that is to say for steels 33HRC or harder.

After a preliminary analysis of the results shown above, in all the scenarios (combination of material hardness and aggressive environment), convergence happens after three-step sequences. In all the cases, P_{th1} was much lower than $P_{FFL,}$ as well as notably higher than P_{th2} and P_{th3}, which are very close together. This characteristic was also found in the tests according to ASTM F1624 of 35 HRC material in the same environments [14,15] (50 HRC and 60 HRC have not been tested yet).

The aforementioned behavior is one of the bases the step loading methodology relies on to be an accurate and robust technique. On the one hand, while in each subsequent step profile, the load is reduced and the application of 20 steps is maintained, it will imply a solicitation rate reduction in each one of them. On the other hand, when two step profiles have the same or a very similar (<5% difference) threshold load, it will mean that the rate developed on them is slow enough to allow the environment–material interaction to rule the process, implying that slower rates will not derive any difference in the threshold value. Finally, performing two step profiles with less than a 5% difference in the threshold value will mean in practice repeating the same test, which gives robustness to the technique, making the obtained value as accurate as possible in environmental scenarios.

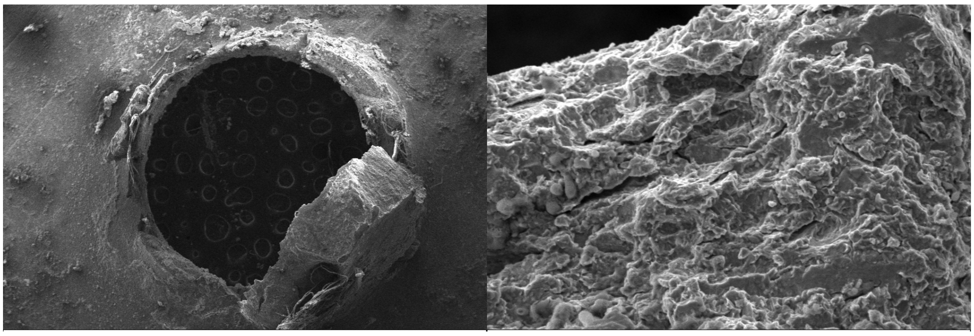

Figure 10. Example of SEM images corresponding to an SPT specimen of 35 HRC steel after being tested at 10 mA/cm^2 for step times of 20 and 40 min (for steps 1–10 and 11–20). Macrography (**left**) and cross-section (**right**).

In this context, when reducing the step times (one-quarter, one-sixth and one-eighth of ASTM F1624 ones), the effect will be similar. The target is to see which are the shorter times for the step profiles that derive the same threshold value as longer ones. This will be the borderline beyond which the application of shorter times will derive higher threshold non-valid values, because they would have been obtained from the domain where just the environment–material interaction rules the process.

The shortest step times that allow us to obtain a constant threshold load were in every case one-sixth of the ones recommended in ASTM F1624 for tensile specimens, which are 20 min and 40 min for steps 1–10 and 11–20, respectively, in the case of $33 \leq$ HRC < 45 steels, 10 min and 20 min for steps 1–10 and 11–20 in the case of $45 \leq$ HRC < 55 steels, and 10 min for steps 1–20 in the case of HRC ≥ 55 steels. Table 6 gathers these suggestions for the different ranges of hardness.

Table 6. Optimal step load protocols for Small Punch tests based on steel hardness.

Hardness	Step	Step Load	Step Time (min)
$33 \leq$ HRC < 45	1–10 11–20	5% of $P_{FFS\text{-}SPT}$	20 40
$45 \leq$ HRC < 55	1–10 11–20	5% of $P_{FFS\text{-}SPT}$	10 20
HRC ≥ 55	1–20	5% of $P_{FFS\text{-}SPT}$	10

Therefore, it is proven that the optimal step durations are one-sixth of the ones indicated in the ASTM F1624 standard [18] for regular tensile specimens. This attends to the fact that hydrogen must pass through the specimen's thickness to embrittle it completely (in the case of Small Punch specimens, the thickness is two orders of magnitude lower than in standard tensile specimens, 0.5 mm vs. ø6–12 mm) and also to the fact that the hydrogen needs time to penetrate from the electrolyte to the metal atomic network of the steel.

4. Conclusions and Future Work

A new methodology is developed to obtain the threshold load with SPT specimens, based on the standard ASTM F1624 [18], which explains how to use constant load steps in aggressive environments, increasing them subsequently until the specimen fails. To apply this methodology, the duration of the steps has been optimized in this paper, with the optimal times found to be one-sixth of the ones indicated in the mentioned standard, which are 20 min and 40 min for steps 1–10 and 11–20, respectively, in the case of $33 \leq$ HRC < 45 steels, 10 min and 20 min for steps 1–10 and 11–20 in the case of $45 \leq$ HRC < 55 steels, and 10 min for steps 1–20 in the case of HRC ≥ 55 steels.

Future work will now imply working on empirical correlations in order to directly estimate the threshold stress just based on SPT tests, as proposed in [14] by the authors of the present work. For this purpose, it would be suitable to work on calibrating experimental correlations, such as the one presented in [14], or proposing more suitable ones, covering the whole range of hardness from ASTM F1624.

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Data Availability Statement: The data presented in this study are available upon request from the corresponding authors.

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