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Application of the incremental step loading technique to Small Punch Tests on S420 steel in acid environments

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Abstract

The Small Punch test has been recently used to estimate mechanical properties of steels in aggressive environments. This technique, very interesting when there is shortage of material, consists in using a small plane specimen and punch it until it fails. The type of tests normally used are under a constant load in an aggressive environment, with the target to determine the threshold stress. However, this is an inaccurate technique which takes time, as the tests are quite slow. In this paper, the Small Punch tests are combined with the step loading technique collected in the standard ASTM F1624 [1] to obtain the value of threshold stress of an S420 steel in a total time of approximately one week. The ASTM F1624 indicates how to apply constant load steps in hydrogen embrittlement environments, increasing them subsequently and adapting their duration until the specimen fails. The environment is created by means of cathodic polarization of cylindrical tensile specimens in an acid electrolyte. A batch of standard tests are performed to validate the methodology.

Keywords: Small Punch; ASTME F1624; Hydrogen Embrittlement; Threshold Stress;

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1. Introduction

A wide range of High strength steels has been developed over the last decades, mainly to provide service in Industrial and Energy facilities, where adverse environments are common. For example, off-shore environments were these steels are often cathodic protected, or gas transport pipelines where there is usually H2O, both scenarios producing EAC, degrading the Steel and leading in the worst cases to catastrophic structural failures. Therefore, there is a need of a control to check High Strength Steels working in aggressive environments.

Two of the standards most commonly used for EAC characterization are ASTM E1681 (ASTM E1681-03, 2013) and ISO 7539 (ISO 7539, 2011). Normally the test used are under constant load, to determine the threshold stress under which a failure will never occur, and/or Slow Strain Rate Tests, to determine fracture properties. The tests of Sustained-Load vs Time-to-Failure estimate the threshold as an upper bound which causes a fracture after some time when the sample is exposed in a certain environment or does not cause it. They are normally performed on cylindrical specimens but they have the disadvantage that they are inaccurate and require a big amount of time (one test can reach 10000 h, requiring up to 12 specimens).

In fact, the standard ASTME F1624-12 has been published to solve these disadvantages, by means of applying constant load incremental steps until the sample breaks, due to the action of both material and environment. It allows to accelerate the test, allowing to estimate the threshold stress in Environmentally Assisted Cracking for steels withing just one week (with a minimum of 3 specimens).

In other cases, the problem is that it is not possible to get samples with enough size, enough thickness or in the amount required by the aforementioned norms. This is, i.e., the case of welded joints. In these situations, the Small Punch Test plays an important role. Developed in 1980's, it is becoming a worldwide alternative to standard tests as it has been proved that the SPT allows the characterization of medium and high strength steels in aggressive environments. Indeed, a European Standard for SPT will be published in 2020 (ECISS/TC 101 AFNOR, 2018).

Based on the good perspectives obtained when proposed to implement ASTME F6124 for SPT, (Tao B. et al., 2013, García T.E. et al., 2015, García T.E. et al., 2016, Arroyo B. et al., 2017, Arroyo B. et al., 2018, Arroyo B. et al., 2019), in this paper the incremental step loading technique is applied to the Small Punch test to estimate tensile threshold stress of S420 medium strength steel in hydrogen embrittlement environments, created by CP in an acid electrolyte. The methodology is validated using standard tests according to ASTM F1624 on cylindrical tensile specimens.

2. Materials and methodology

In this section, the S420 steel properties and the environment employed for the tests are described. The methodology of incremental step loading technique according to ASTM F1624 and the SPT tests are explained. Finally, the experimental program is detailed.

2.1. Material and Environment

The material employed in this work is is an S420, a thermomechanically treated medium strength steel, also known as TMCR 420 (BS EN 10225, 2009), potentially applied in pressure vessels, facilities for energy generation and the offshore industry. It's chemical composition as well as it's mechanical properties are shown in Table 1 and Table 2 respectively. The microstructure can be seen in Figure 1, showing a ferritic-pearlitic microstructure with grain size of 5-25 μm. To represent the aggressive environment and produce Hydrogen Embrittlement, cathodic polarization was used, imposing a fixed current density on the steel, which was connected to a platinum grid in an aqueous solution. For the aqueous solution, an acid electrolyte was prepared with 1N H_2SO_4 solution in distilled water with 10 mg of As_2O_3 and 10 drops of CS_2 per litre of dissolution according to Pressouryre's method (Pressouyre G.M. at al., 1981). The tests were done at room temperature, with three different current densities (1, 5 and 10 mA/cm2) and keeping the pH inside the range of 0.65 to 0.80.

Table 1. Chemical composition of S420 steel (wt %)

C	Si	S	P	Mn	Ni	Cr
0.08	0.28	0.001	0.012	1.44	0.03	0.02
Mo	**Cu**	**Al**	**V**	**Ti**	**Nb**	**Fe**
0.003	0.015	0.036	0.005	0.015	0.031	Rest

Table 2. Mechanical properties of S420 steel

E (GPa)	S_y (MPa)	S_u (MPa)	e_{max} (%)	K_{mat} ($MPa*m^{1/2}$)
206	447	547	13.9	356

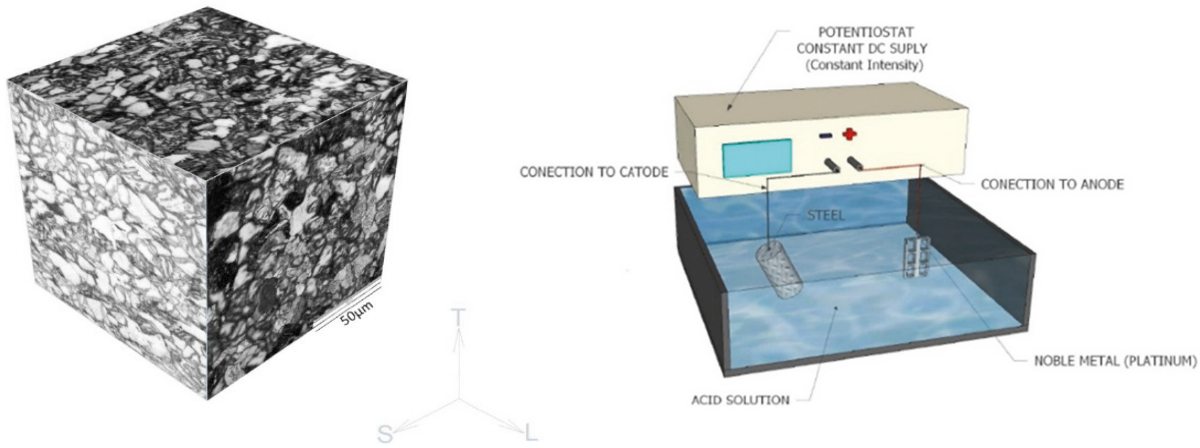

Figure 1. Microstructure of S420 steel (left) and schematic of the cathodic polarization (CP) set up (right).

2.2. ASTM F1624

This Standard explains the methodology to measure the load needed to start subcritical crack growth in steel in an aggressive environment. The load has to be increased progressively in subsequent steps after a certain time until the specimen's failure. Figure 2 represents the process schematically.

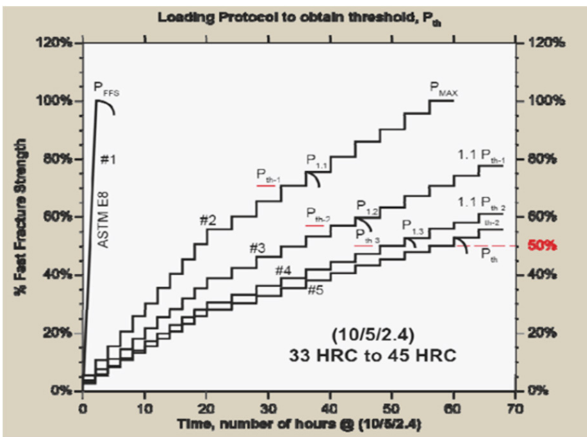

Figure 2. Example of loading protocol to obtain Pth in steels

First of all, a tensile test in air acc. to ASTM E8 (ASTM E8 / E8M 16a, 2016) has to be performed. It will serve as reference data, establishing the upper limit from which the steps protocol for the first test will be defined (P_{FFS}). Then the first test in environment is sequenced using P_{FFS} directly as P_{max}, and defining 20 steps with 5% load increment each. Therefore, after the specimen is precharged in the environment to create the embrittling effect, the subsequent load steps are performed up to the specimen rupture, and this load value is P_{th-1}.

The upper bound to plan the new test load profile is obtained increasing by 10% the threshold load obtained in the previous step protocol ($P_{th-n}=1.1*P_{th(n-1)}$). By reducing the maximum load profile the loading rate is also reduced increasing the accuracy of the result. The same protocol is repeated with at list a minimum of 3 sample protocols, until the threshold load obtained in two subsequent protocols differs less than 5 % ($P_{th-n}-P_{th-(n-1)} < 5\%$). The threshold load will be the one obtained in the last step protocol.

The duration of the loading steps is defined based on the hardness of the material. Steels with hardness value under 33HRC are not covered.

Table 3. Steps load profile depending on the hardness of the steel (ASTM F1624-12, 2018).

Hardness (HRC)	**Steps**	**Step force (% P_{max})**	**Step time (h)**	**Protocol code**
33 to < 45	1 to 10	5	2	(10/5/2,4)
	11 to 20	5	4	
> 45 to 54	1 to 10	5	1	(10/5/1,2)
	11 to 20	5	2	
> 54	1 to 20	5	1	(20/5/1)

2.3. Tensile specimen according to ASTME F1624

In order to validate the SPT methodology, tests on cylindrical specimens were performed following ASTM F1624 in the same cathodic polarization environment. The aim was to compare the threshold load obtained with its corresponding threshold stress, σ_{th} for the S420 steel material in the three different environments aforementioned. Cylindrical specimens with Ø6mm were prepared from an S420 plate in TL direction with the dimensions as indicated in Figure 3.

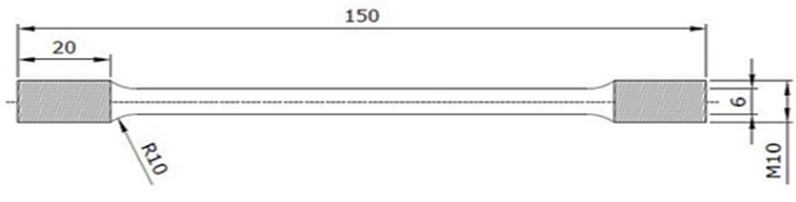

Figure 3. Schematic of the tensile specimens. Dimensions in mm.

In each environment, a specimen was tested in air in accordance with ASTM E8 (ASTM E8 / E8M 16a, 2016) to obtain P_{FFS}, afterwards used as P_{max} for the first step protocol. The following specimens were tested in environment following the steps protocol sequence until getting P_{th}. For the S420 steel, in the range of 35 to 44HRC, a total of 20 steps were performed: 10 steps of 2 h and 10 steps lasting 4 h; (10/5/2,4) protocol according to Table 3.

An electrolytic cell was specially designed to assure that the central part of the samples tested in environment were immersed inside the aqueous solution during the complete duration of the test, assuring a continuous recirculation inside the cell. Before each steps protocol, the specimens were exposed to the same aggressive environment conditions as during the test for 24 hours, enough time to allow hydrogen diffusion and produce hydrogen absorption (J.A. Álvarez et al., 1998).

2.4. SPT Step Loading Methodology

In previous works (Arroyo B. et al., 2019), it was exposed how the step loading technique could be implemented to the Small Punch Test. In the present paper the proposal is verified testing a susceptible to EAC material in three different environments and validating the results by comparing with the results obtained from tensile specimens tested according to ASTM F1624. The three different environments are 1, 5 and 10 ma/cm^2 in acid dissolution of H_2SO_4 in H_2O.

The step forward of the aforementioned work is that the goal at that time was to obtain the $P_{th\text{-}SPT}$ (SPT threshold load) preserving the general idea of ASTM F1624, but using shorter loading steps. Specifically, the steps were 6 times shorter than for the tensile samples: the step protocol (10/5/2,4) was used considering 10 steps of 20 min and 10 steps of 40 min, instead of 10 steps of 2 h and 10 steps more of 4 h. In the current work instead, the time of exposure was 2 h to allow a proper hydrogen diffusion, like recommended in the bibliography. Furthermore, the first test in air to get $P_{FFS\text{-}SPT}$ and $P_{max\text{-}SPT}$, was performed at a constant punch rate of 0.01 mm/s, following the European SPT standard working draft (ECISS/TC 101 AFNOR, 2018).

The SPT specimens were obtained from the S420 steel plate in the direction which allows to estimate tensile properties in TL orientation, ergo the thickness of the specimen had to be along T direction, perpendicular to the axis of the tensile specimens. Following the recommendations of several bibliography authors, (Tao B. et al., 2013, García T.E. et al., 2015, García T.E. et al., 2016, Arroyo B. et al., 2017, Arroyo B. et al., 2018, Arroyo B. et al., 2019), the SPT specimens dimension chosen was a square cross section of 10x10 mm^2 with 0.5±0.01 mm thickness as represented in Figure 4, which is equivalent to the dimension of Ø8 mm currently used in the European SPT standard draft (ECISS/TC 101 AFNOR, 2018). Grain size #2000 water sanding paper was used to finish the surface.

Also in Figure 4, the specifically designed device is shown. It consists on an electrolytic cell where the SPT specimen to be punched is embedded between two rigid jigs. Weights are used to apply the different loading steps, while the sample is immersed inside the aqueous solution, which is in continuous recirculation. To assure a complete electrical isolation, the jigs were built in insulant plastic material, the punch was coated with insulating varnish and the punch hemispherical head was made of ceramic material.

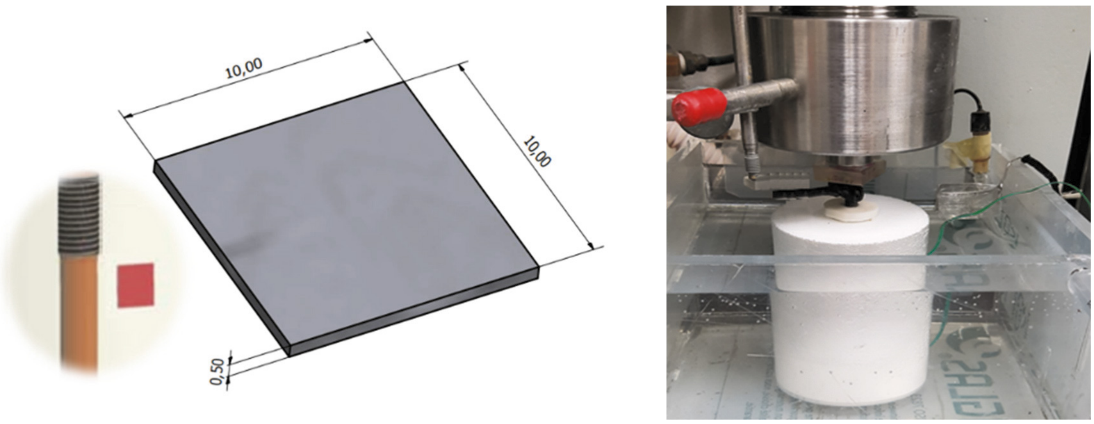

Figure 4. Schematic of the SPT specimens and its comparison to tensile specimens orientation (left), and detail of the SPT experimental set-up (right)

3. Results and discussion

Figure 5 presents the tests results. On the left are shown the results of the tensile tests according to ASTM F1624; each environment required three samples plus the test tensile ASTM E8 test to obtain P_{FFS}. On the right the results from the proposed SPT methodology can be seen; also 3 samples were required in each environment, plus the first test according to the European SPT standard working draft (ECISS/TC 101 AFNOR, 2018) to obtain $P_{FFS\text{-}SPT}$.

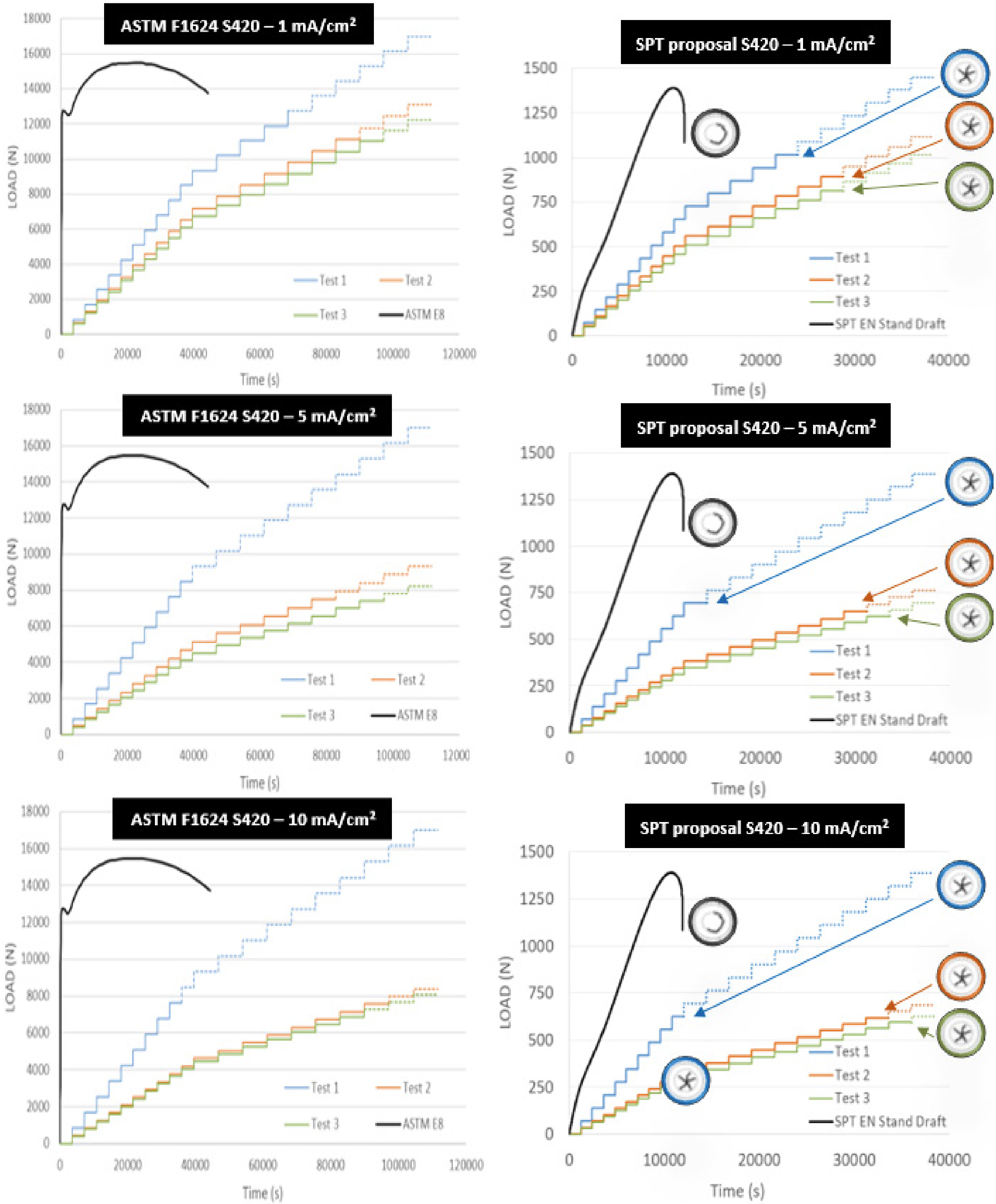

Figure 5. S420 steel load-time registers obtained when tested under CP; the dashed lines show the planned steps that did not take place after specimen failure. On the left are shown the results of applying ASTM F1624 and on the right the SPT proposal based on the step loading technique; TOP: 1mA/cm^2, CENTER: 5mA/cm^2, BOTTOM: 10mA/cm^2.

If we compare both techniques results, it is easily observed that the loading protocol had a similar trend and 3 samples were used for standard and for SPT cases, like stated in bibliography (Arroyo B. et al., 2019); the SPT proposal reflected the same behavior than the standard tests.

Table 4 organizes the previous results, converting the threshold load (P_{th}) into threshold stress (σ_{th}). Figure 6 plots the results to facilitate comparison, incorporating some fractographies in order to show that the micromechanisms in both techniques for the same environment are similar too. This allows to conclude that the SPT proposal is representative of the interaction between material and environment. Therefore, the SPT proposal is able to reproduce the same trends than the ASTM F1624 standard. Furthermore, the linear fit from P_{th}-σ_{th} in the three environment conditions shows a very good correlation coefficient.

Table 4. Results obtained by applying ASTM F1624 and the SPT to S420 steel under CP at 1, 5 and 10 mA/cm^2.

	ASTM F1624		**SPT Proposal**
	P_{th} (N)	σ_{th} (MPa)	$P_{th\text{-}SPT}$ (N)
Air	15445	547	1465
1 mA/cm^2	10729	379	812
5 mA/cm^2	7501	265	625
10 mA/cm^2	7282	258	594

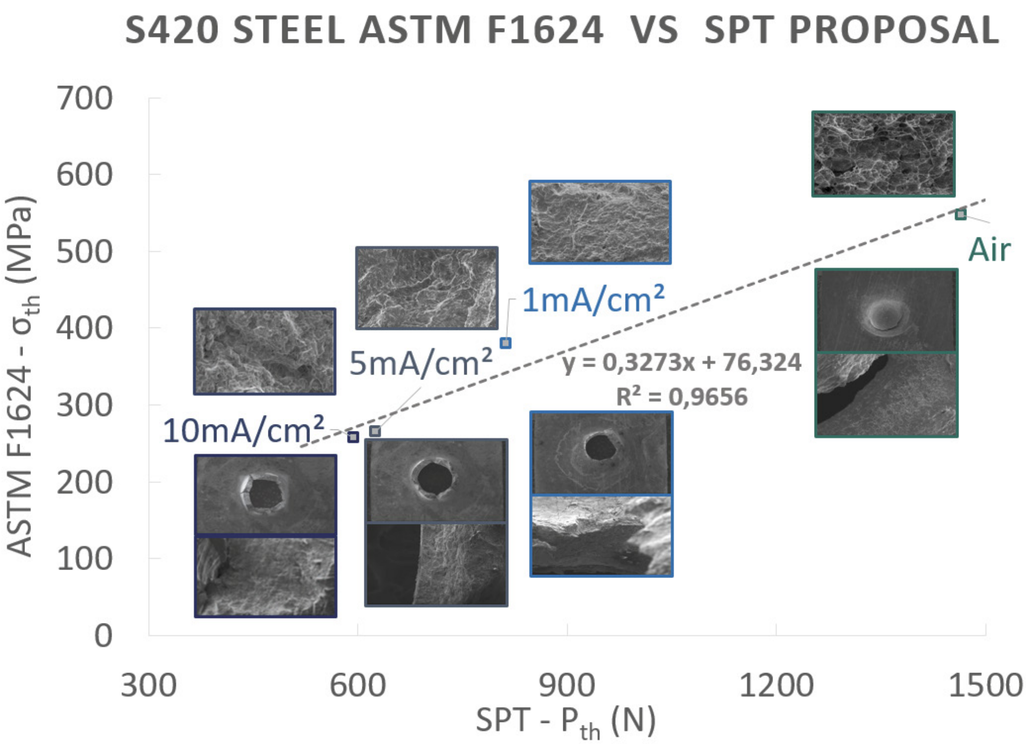

Figure 6. σ_{th} vs P_{th} results obtained applying ASTM F1624 and the SPT step loading technique to S420 steel under CP at 1, 5, and 10 mA/cm^2.

4. Conclusion

The exposed results validate the option to obtain the stress threshold in aggressive environments using the ASTM F1624 incremental step loading technique applied to the SPT.

In the present paper, this has been proved in 3 different embrittlement environments using cathodic polarization in an acid electrolyte, with an S420 medium strength steel.

As future investigations, the results should be extended to a wider range of materials and/or environments, and the steps duration might be reduced depending on the SPT punching rates.

5. Acknowledgements

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