Escuela Técnica Superior de Ingenieros de Caminos, Canales y Puertos.
UNIVERSIDAD DE CANTABRIA

VALIDATION OF THE INCREMENTAL STEP LOADING TECHNIQUE APPLICATION TO SMALL PUNCH TESTS IN AGGRESSIVE ENVIRONMENTS IN X80 STEEL

EIDEIC 2020 – International Meeting of Doctoral Students in Civil Engineering

Laura Andrea Calvo

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I. **Short research plan description**

II. State of achievement of competences

III. Example of a research developed in the Civil Doctorate

RESEARCH PLAN

- H2 affects materials, specially steel, modifying its mechanical properties and its structural behaviour.

- To develop steels with stable properties in H2 content environments, it is necessary to understand the mechanisms though which steel is affected by H2.

- The presence of H2 reduces the steel fracture toughness and increases the crack growth speed.

- Main targets:

  - To assess the suitability of current knowledge methods
  - To use that knowledge to improve the structural integrity evaluations

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COMPETENCE ASSESSMENT GUIDE FOR DOCTORAL STUDENTS

BASIC SKILLS	2. Science &Technique (bibliographic study)	3. Technology (tools and instruments)	4. Educational Activities (courses and seminars)	5. Results (publications)	6. Scientific Criticism (SWOT Analysis)	7. Work Plan	8. Mobility	9. Funding	10. Ethics
CB11 – Systematic understanding of a field of study and command of the skills and research methods related to the field.	X	X	X						
CB12 – Skill to conceive, design or create, implement and adopt a substantial process of research or creation.				X		X	X		
CB13 – Skill to contribute to the enlargement of the knowledge limits through an original research.				X					
CB14 -Skill to carry out a critical analysis and assessment and synthesis of new and complex ideas.					X				
CB15 – Skill to communicate with the academic and scientific community and with society in general about the scope of knowledge in the ways and languages of common use in the international scientific community.				X			X		
CB16 – Skill to encourage, in academic and professional contexts, the scientific, technological, social, artistic or cultural progress in a society based on knowledge.				X					X

CAPACITIES AND PERSONAL ABILITIES	2. Science &Technique (bibliographic study)	3. Technology (tools and instruments)	4. Educational Activities (courses and seminars)	5. Results (publications)	6. Scientific Criticism (SWOT Analysis)	7. Work Plan	8. Mobility	9. Funding	10. Ethics
CA01 – Cope in contexts in which there is little specific information.	X	X	X						
CA02 – Find the key questions to be answered to solve a complex problem.				X					
CA03 – Design, create, develop and undertake new and innovative projects in the knowledge scope.						X		X	
CA04 – Work both in teams and individually in an international or multidisciplinary context.			X				X		
CA05 – Integrate knowledges, face complexity and formulate judgements with limited information.	X	X	X						
CA06 – Intellectual criticism and defence of solutions.					X				

Compulsory multidisciplinary training: every doctoral student must have more than 80 hours of multidisciplinary training organized by the EDUC in two courses to be taken at the beginning (Basic Course) and the end (Advanced Course). All the related information is available in http://www.doctoradouniversidadcantabria.com/content/actividades-transversales

Favourable assessment of his yearly PI along the doctoral studies: the importance of the PI and its assessment is critical since on it the acquisition of competences CB11 and CB14 and the personal skills and capacities CA01, CA03 and CA06 directly depend, directly related to their sections 2, 3, 4, 6, 7 and 9.

International scientific publications: the publication of the results obtained in papers in international scientific journals helps the doctoral student to ensure the acquisition of competences CB12, CB13, CB15 and CB16 and the personal capacity and ability CA02.

International mobility: this condition is essential to achieve the personal capacity and ability CA04, completing the doctoral student training.

STATE OF ACHIEVEMENT OF COMPETENCES

Compulsory multidisciplinary training: transversal courses, seminaries, and others	
Favourable assessment of his yearly PI	
Participation on EIDEIC at least two times	
Research and development experience	
Publications at conferences	
Participation in international congress	
3 research papers published in journals of prestige with peer revision	
International and multidisciplinary experience	
Technology: tools and instruments	
Bibliographic study	
Thesis status (expected submission date is May 2022)	

STATE OF ACHIEVEMENT OF COMPETENCES

- Paper published in the 2019 Congress of the Spanish Fracture Group (GEF) *"Caracterización de la fragilización por hidrógeno mediante la aplicación de la técnica de escalones incrementales al ensayo small punch".* B.Arroyo, P. González, L. Andrea, J.A. Álvarez, R. Lacalle.

- Paper published in the Pressure Vessels & Piping Conference 2019 in San Antonio, TX, USA "*Application of the incremental step loading technique to small punch tests in hydrogen embrittlement*". B.Arroyo, P. González, L. Andrea, J.A. Álvarez, R. Lacalle.

- Paper published in the 5th Iberian Conference on Structural Integrity, Spain (IbCSI 2020) *"Application of the incremental step loading technique to X80 steel small punch tests in aggressive environments"*. B. Arroyo, L. Andrea, P. González, J.A. Álvarez, S. Cicero, A. Fernández, R. Lacalle.

- Paper published in the Pressure Vessels & Piping Conference 2020 in Minneapolis, Minnesota, USA *"Validation of the incremental step loading technique application to small punch tests in aggressive environments in X80 Steel".* B. Arroyo, L. Andrea, P. González, J.A. Álvarez, S. Cicero, A. Fernández, R. Lacalle

STATE OF ACHIEVEMENT OF COMPETENCES

- Paper in current revisión of the Metals indexed journal ISSN 2075-4701 (**JCR Q1** in Metallurgy & Metallurgical Engineering, current impact factor 2,259) *"Analysis of samples cleaning methods prior to Hydrogen content determination in steel".* B. Arroyo, L. Andrea, J.A. Álvarez, S. Cicero, R. Lacalle

- Plan to participate in SSTT 2020 6th International Small Sample Test Techniques Conference

STATE OF ACHIEVEMENT OF COMPETENCES

Compulsory multidisciplinary training: transversal courses, seminaries, and others	
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INTRODUCTION

- An aggressive environment degrades the material producing subcritic cracking, leading to brittle damage, very difficult to foresee.

- High Strength Steels are very susceptible.
  The anális of H_2 content is vital to evaluate the brittlement conditions.

- Threshold stress (σ_{th}) is the one below which failure will never take place. Above it a time-delayed fracture will occur in a certain environment (finite life).

- σ_{th} is usually obtained by **sustained-load vs time-to-failure** tests on cylindrical specimens.
  Main disadvantages:
  - The demand of big amount of time (requires around 12 samples, reaching 10000 h of test).
  - In some situations it is not possible to obtain samples of a sufficient size or thickness, to fit the requirements of ISO 7539 and ASTM E1681.

- ASTM F1624-12 is an accelerated method that consists on applying steps under constant incremental loads one after the other up to the specimen's rupture. It allows to estimate σ_{th} within one week by testing at least 3 specimens

- The SPT is a miniature test alternative and consists on punching a small plane specimen up to failure, and it can be used to estimate mechanical properties when there is shortage of material.

- **The incremental step loading technique is applied to the SPT to estimate σ_{th} of a HSS in H_2 embrittlement environment. Regular standard tests are carried out to validate the methodology.**

MATERIALS AND ENVIRONMENT EMPLOYED

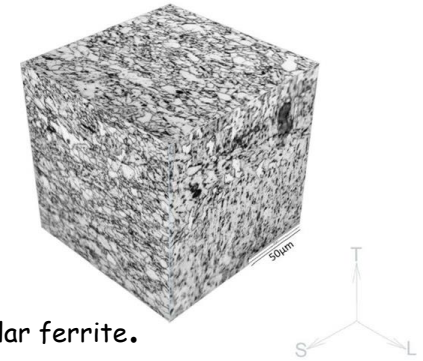
- **X80 material** is employed. It is a rolled HSS potentially applied in pipelines and offshore industry.

C	Si	S	P	Mn
0.07	0.18	<0.005	<0.005	1.83
Ni	**Mo**	**Cu**	**Al**	**Nb**
0.03	0.15	0.02	0.03	0.03

Chemical composition

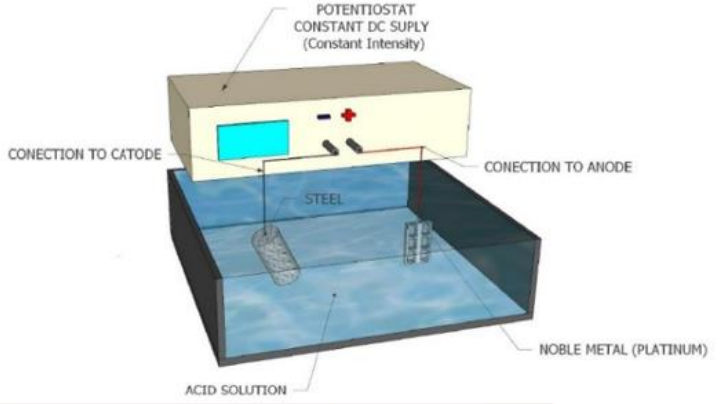
E (GPa)	S_y (MPa)	S_u (MPa)	e_{max} (%)	K_{mat} (MPa*$m^{1/2}$)
210	621	691	6.9	349

Mechanical properties



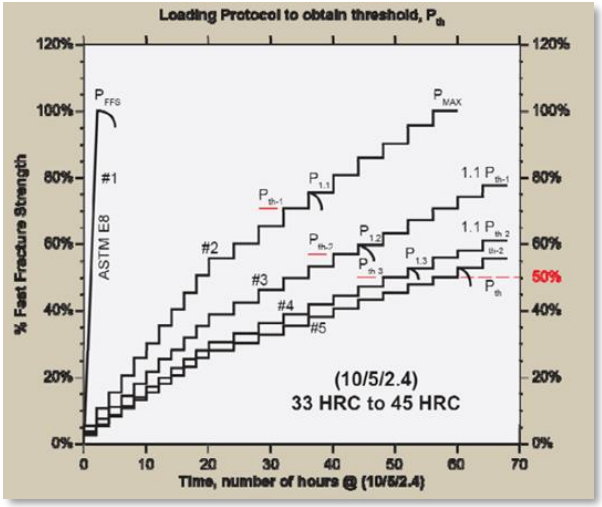
Ferritic microstructure with grain size range 5-15µm with some degenerated bainite/pearlite and no acicular ferrite.

- **Cathodic Polarization** technique is used to generate the H_2 Embrittlement aggressive environment.
- CP: imposing a fixed current density between the steel, connected to a platinum grid through an aqueous solution (acid electrolyte prepared acc. to Pressouyre's method)



INCREMENTAL STEP LOADING TECHNIQUE (ASTM F1624)

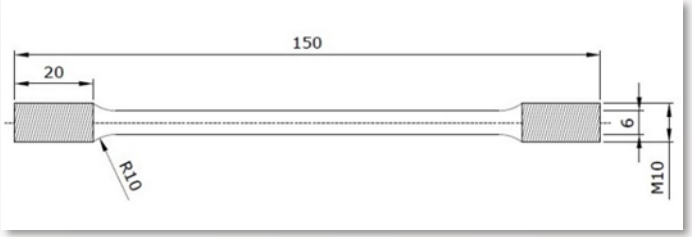
- This method gives the load to start a subcritical crack growth in aggressive environment.
- Consists on imposing load steps increased after a certain time up to the specimen rupture.
- First, a **tensile test in air** according ASTM E8 is performed, to establish the Fast Fracture Load, P_{FFS}.
- Then, the first test in environment is planned using **Pmax= P_{FFS}.**
  A total of 20 steps, each one having a 5% of Pmax increment.
- After precharging the specimen in the environment for embrittling, the steps take place. **The rupture load is Pth-1.**
- To plan the step load profile for the rest of tests: **Pth-n =1.1*Pth-(n-1).**
- This protocol is repeated until: **Pth-n - Pth-(n-1)< 5%**
- At least 3 samples should be performed.
- The duration of the load steps, depends on the hardness of each steel.



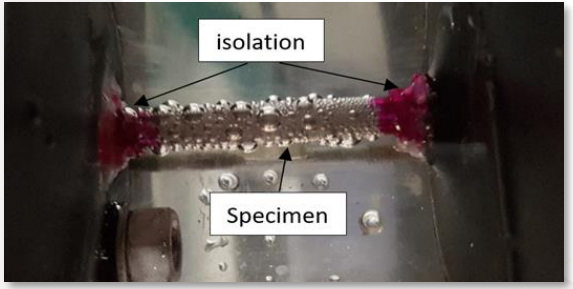
Hardness (HRC)	*Steps*	*Step force ($\%P_{max}$)*	*Step time (h)*	*Protocol code*
33 to <45	*1 to 10*	*5*	*2*	*(10/5/2,4)*
	11 to 20	*5*	*4*	
>45 to 54	*1 to 10*	*5*	*1*	*(10/5/1,2)*
	11 to 20	*5*	*2*	
>54	*1 to 20*	*5*	*1*	*(20/5/1)*

TENSILE SPECIMEN TESTED ACCORDING ASTM F1624

- To validate the method, tests on cylindrical specimens acc. to ASTM F1624 are carried out in the CP environment, to obtain Pth and its corresponding σth.
- Prior to start the specimens were exposed to the same environment during 24h for a proper H_2 diffusion.
- The tests were performed using an electrolytic cell designed to assure that the central part is completely immersed in the aqueous solution during the whole test.
- For each one of the 3 environments analyzed, Pth was obtained with the step loading technique.



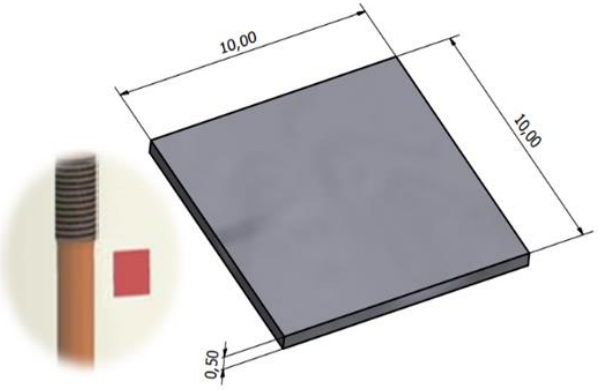
A set of Ø6mm cylindrical specimens were obtained from an X80 plate in TL orientation



The specimen areas coincident with the wall passages were coated with an insulating varnish.

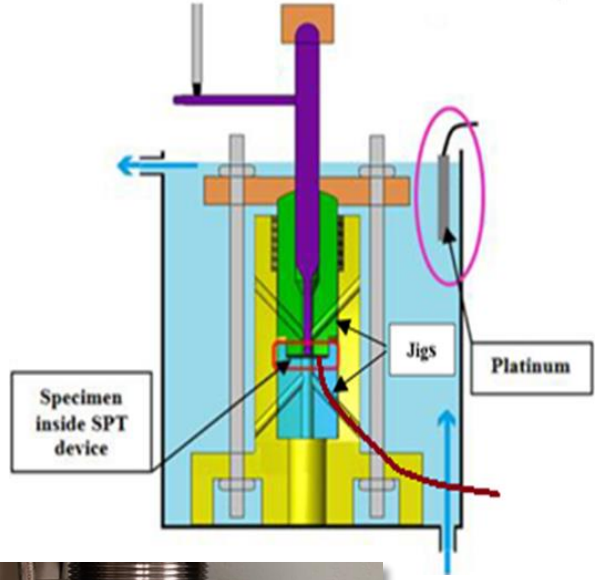
SPT STEP LOADING METHODOLOGY PROPOSED

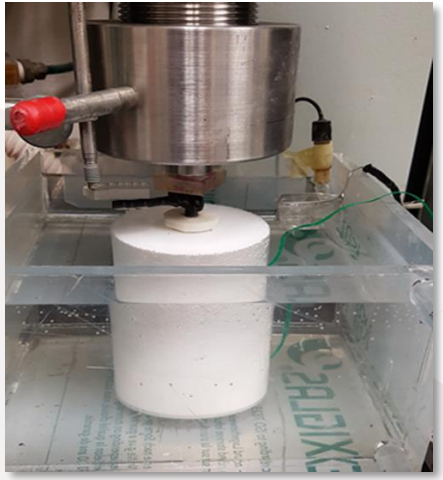
- The step loading technique is implemented to SPT testing X80 in three different environmental conditions (1, 5 and 10mA/cm2) and then compared to their homologous results obtained from tensile specimens tested following ASTM F1624.
- Steps durations 6 times shorter than those for tensile samples were chosen (min instead of h).
- For X80 steel with 35HRC step profile (10/5/2,4)
- The exposure time to the environment of the samples was 2 hours for a proper H_2 diffusion.
- The first test in air to obtain SPT fast fracture load, was at a constant punch rate of 0,01 mm/s according to the European SPT standard working draft.
- A set of X80 SPT specimens was obtained to estimate properties in TL orientation.



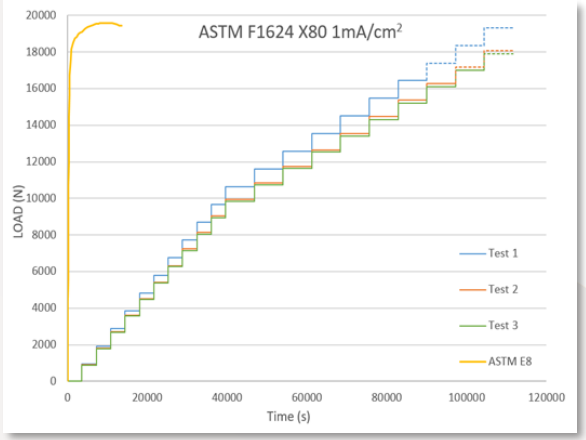
SPT STEP LOADING METHODOLOGY PROPOSED

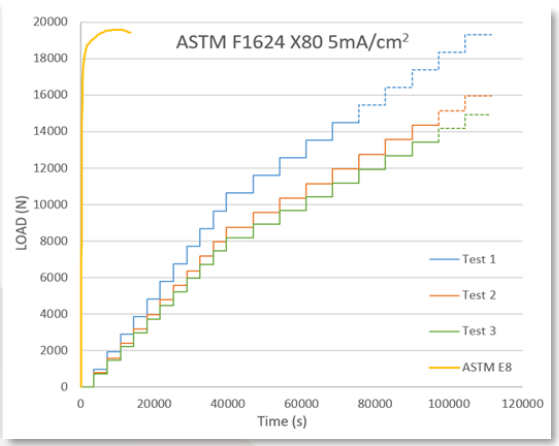
- The experimental device was designed and built for this purpose.
- It is an electrolytic cell where the SPT sample is embedded between to rigid jigs and punched.
- The loading steps are applied by the action of weights on the punch.
- The sample is completely immersed inside the aqueous solution during the whole test.
- To achieve a total electrical isolation of the process:
  - The punch was coated with insulating varnish
  - Its hemispherical head was made of ceramic material
  - The jigs were made of an insulant plastic material.

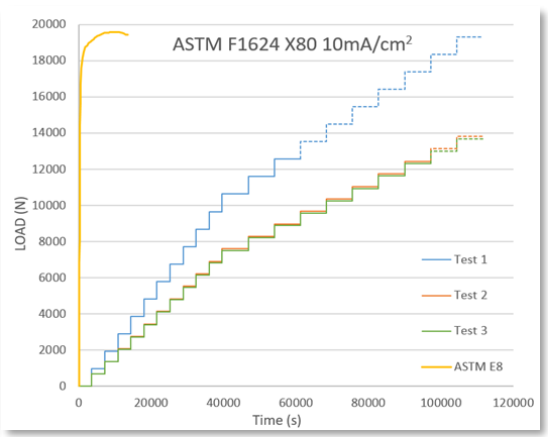




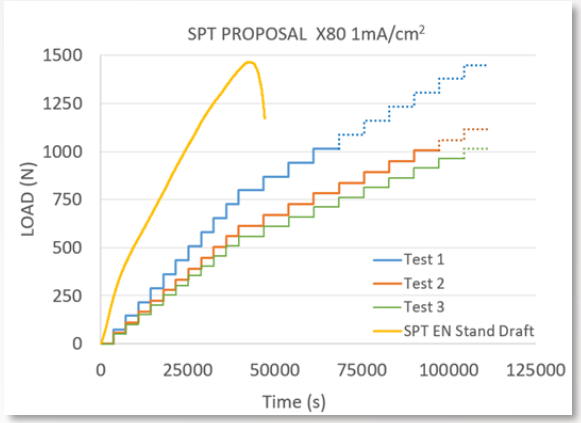
RESULTS

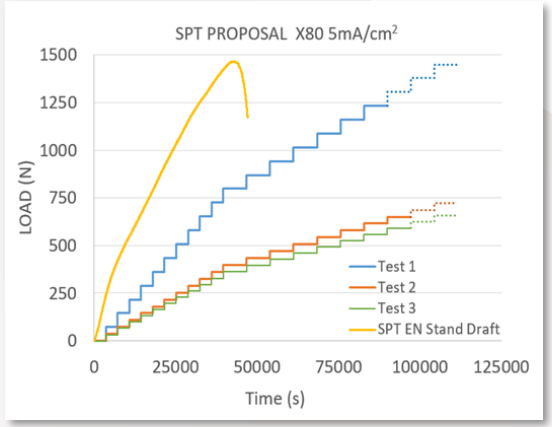


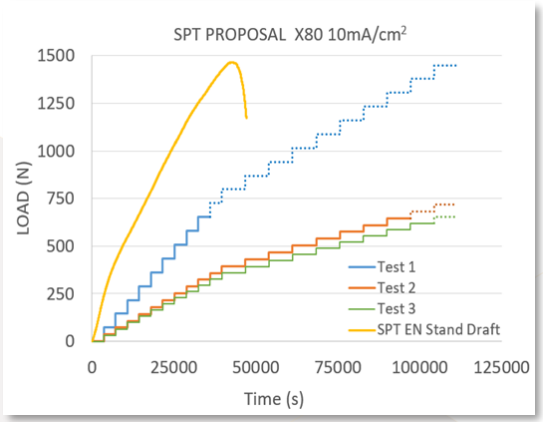




RESULTS WITH ASTM F1624





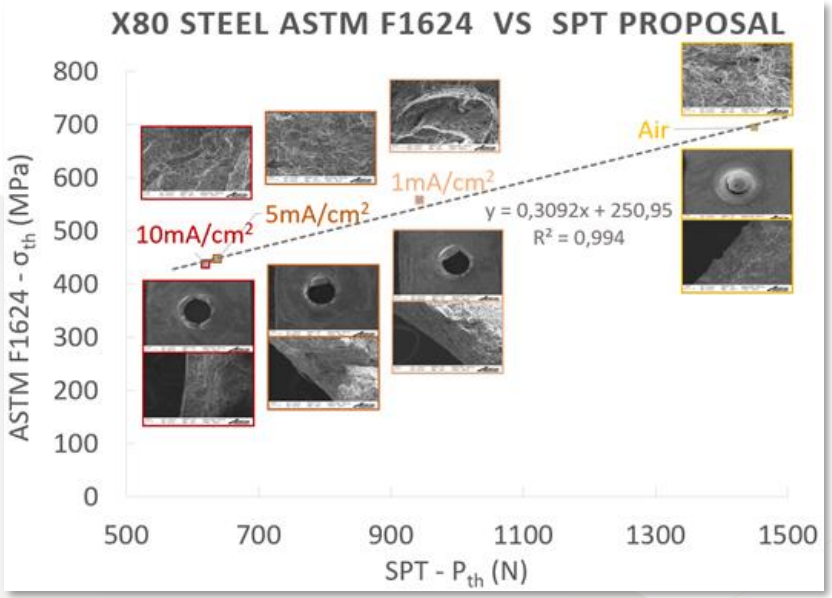


RESULTS WITH SPT

RESULTS

- In both cases the loading protocol had a similar trend.
- The fractographies from both methods show the same micromechanism taking place in both techniques for the same environmental condition.
- The proposed SPT technique is able to reproduce the same trends than the ASTM F1624.

	ATM F1624 [3]		***SPT Proposal*** [10]
	P_{th} *(N)*	σ_{th} *(MPa)*	$P_{th\text{-}SPT}$ *(N)*
Air	*19592*	***694***	***1450***
1 mA/cm²	*15723*	***556***	***943***
5 mA/cm²	*12623*	***447***	***637***
10 mA/cm²	*12315*	***436***	***620***



Pth, from tensile specimens is converted into σth and plotted into a graph **σth vs Pth-SPT**

CONCLUSIONS AND NEXT STEPS

- It is possible to estimate σ_{th} by using the incremental step loading techique from ASTM F1624 applied to SPT in aggressive environments.
- With the SPT method, duration of test is heavitly reduced.
- Amount of especimens and size of material needed is heavily reduced.
- The steps duration should be reduced in order to adapt it to SPT punching rates.
- This was proved for an X80 HSS in 3 different hydrogen embrittlement environments by cathodic polarization in an acid electrolyte.
- These results should be extended to a wider range of materials and environments.
- Steps duration can also be studied in detail for further improvement (reduction) of the testing time.

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