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

ANALISIS OF CLEANLINESS METHODS FOR DETERMINATION OF HIDROGEN CONTENT. EXAMPLE OF AN APPLIED CASE.

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I. **Short Introduction & Research Plan**

II. Example of a research developed in the Civil Doctorate

III. Publications and other researches in progress

Research Plan

- H_2 affects materials, specially Steel, modifying its mechanical properties and its structural behaviour.

- To develop steels with stable properties in high H_2 content environments it is necessary to understand the mechanisms for how it modifies the Steel.

- The presence of H_2 reduces the fracture tenacity 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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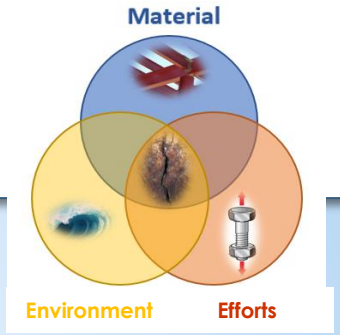
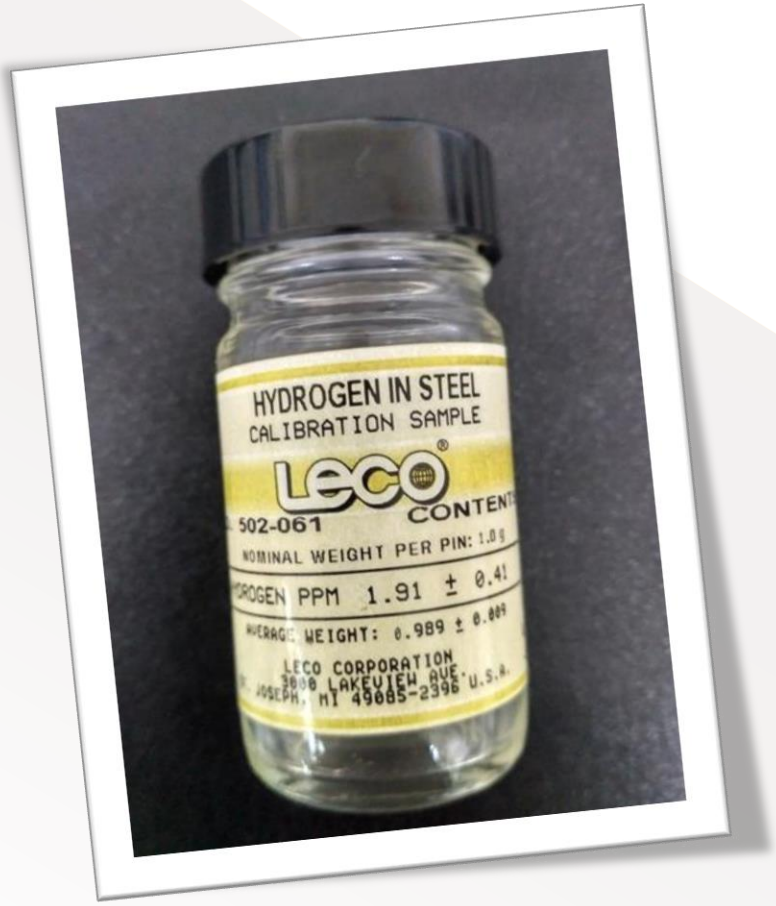
ANALISIS OF CLEANLINESS METHODS FOR DETERMINATION OF HIDROGEN CONTENT



- To understand the behaviour of Steel all three aspects affect.
- An aggressive environment degrades the material producing subcritic cracking which leads to brittle damage very difficult to foresee.
- The analysis of H_2 content in Steel is vital to evaluate the brittlement conditions.
- Before the anlysis, the most important task is the cleaning of the sample probes.
- Currently there is no agreement on which cleaning method is the best.
- The different cleaning methods in H2 charged conditions are compared to determine the most suitable one for HIC analysis.

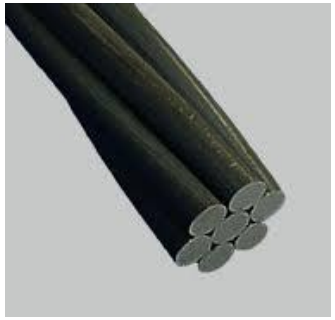
*HIC: Hydrogen Induced Cracking

PATTERN PINS



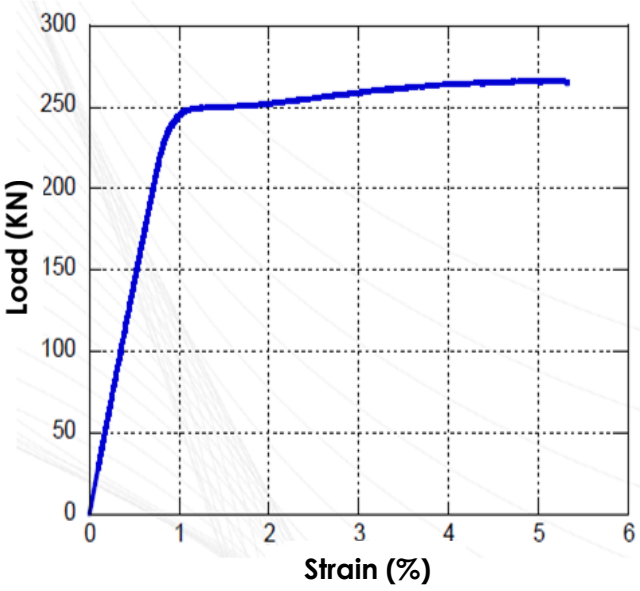
- Iron pins covered by a zinc layer
- Hydrogen content of 1,91 ±0,41 ppm certified by manufacturer
- Weight: 1g.

PRETENSION CORD STEEL



High strength steel from the central wire of a prestressing cord

Sample		UNE 36094
• **Elasticity Module, E**	202 ± 3 GPa	181,4 ÷ 208,7 GPa
• **Elastic limit at 0,1%, F_p 0,1**	242 ± 3 kN (1730 ± 24 MPa)	> 221 kN
• **Elastic limit at 0,2%, F_p 0,2**	248 ± 3 kN (1771 ± 21 MPa)	> 229 kN
• **Breaking load, F_m**	266 ± 3 kN (1901 ± 19 MPa)	260 ÷ 304 kN
• **Elongation under máximum load, A_{gt}**	5,19 ± 0,05 %	> 3,5 %
• **Ratio F_p 0,2/F_m**	0,93 ± 0,01	---



SAMPLE PREPARATION

Pattern pins

Cleaning

⇩

Analyzing

Uncharged samples

Oxide removal

⇩

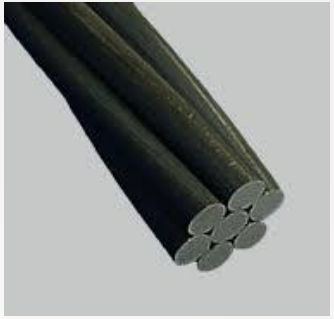
Cutting

⇩

Cleaning

⇩

Analyzing



Charged samples

Oxide removal

⇩

Cutting

⇩

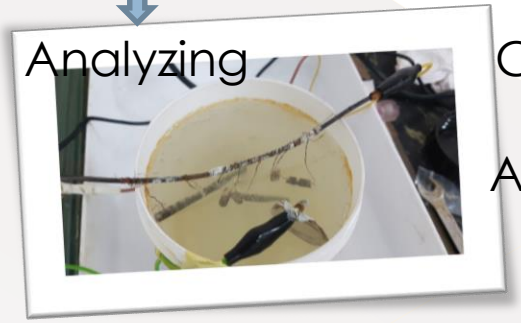
Charging

⇩

Cleaning

⇩

Analyzing



Charged and later discharged samples

Oxide removal

⇩

Cutting

⇩

Charging

⇩

Discharging

⇩

Cleaning

⇩

Analyzing

CLEANING METHODS

Simple method with acetone (ACETONE)	Degreasing the probe with alcohol
	Immerse in acetone at room temperature for a time no longer than 1 min.
	Dry with air at room temperature.
Complete method (ACETONE + ULTRASOUNDS + TRICHLOR)	Degreasing the probe with alcohol
	Immerse in acetone at room temperature for 8 min and apply ultrasounds.
	Immerse in trichlor for 1 min.
	Dry with air at room temperature.
Simple method with acetone and ultrasounds (ACETONE + ULTRASOUNDS)	Degreasing the probe with alcohol
	Immerse in acetone at room temperature for 8 min and apply ultrasounds.
	Dry with air at room temperature

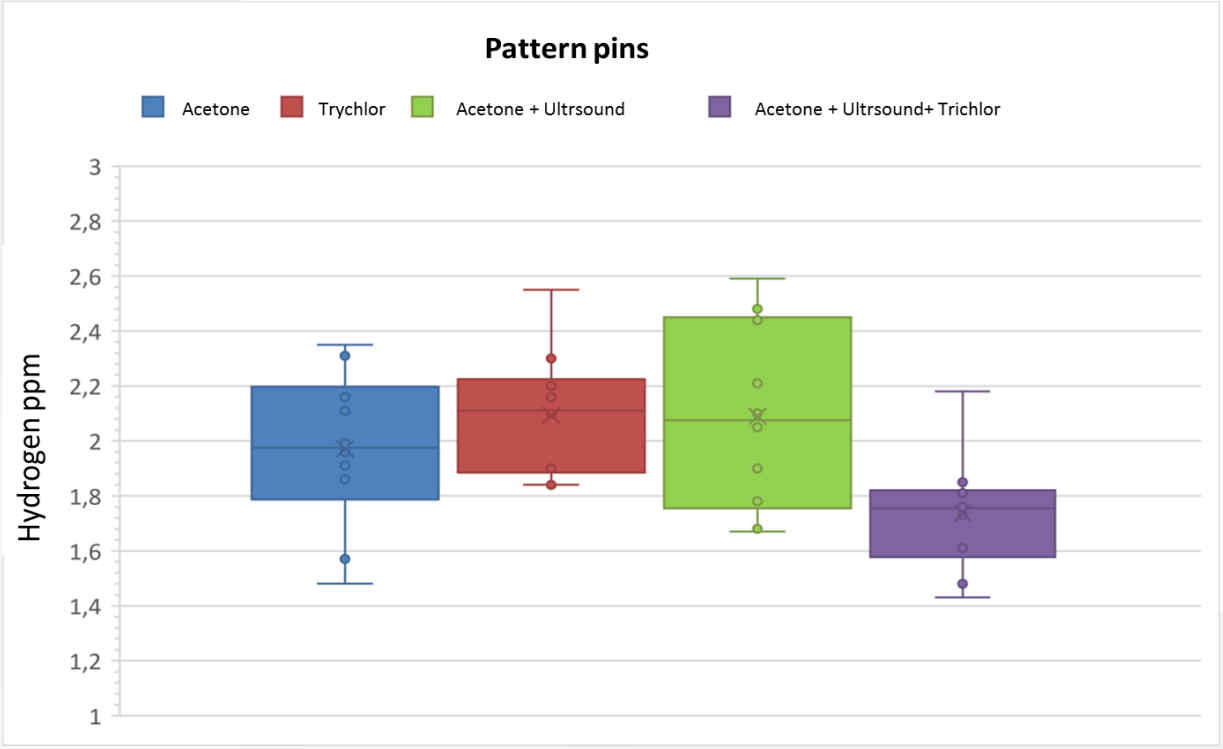
Simple method with trichlor (TRICHLOR)	Degreasing the probe with alcohol
	Immerse in trichor for 1 min.
	Dry with air at room temperature
Method C-3.5 from ASTM G1 (HCL DISSOLUTION)	Degreasing the probe with alcohol
	Immerse for 10 min. In a room temperature solution of 500 ml of hydrochloric acid (HCl, sp gr. 1.19), 3,5 g tetramine hexamethylene and wáter to get 1l
	Dry with air at room temperature

EQUIPMENT USED

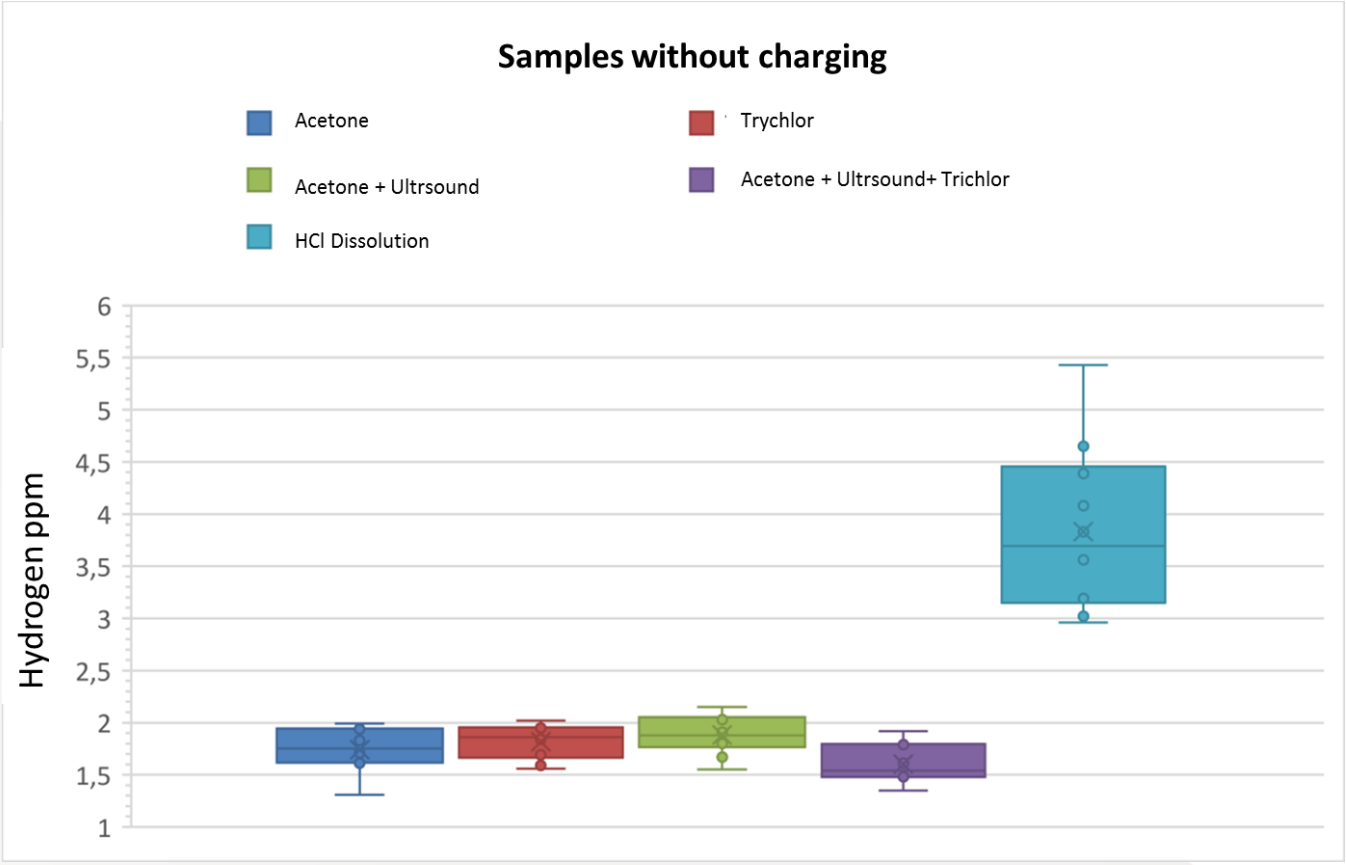
LECO analyzer model RH-402

LECO induction oven model HF-402

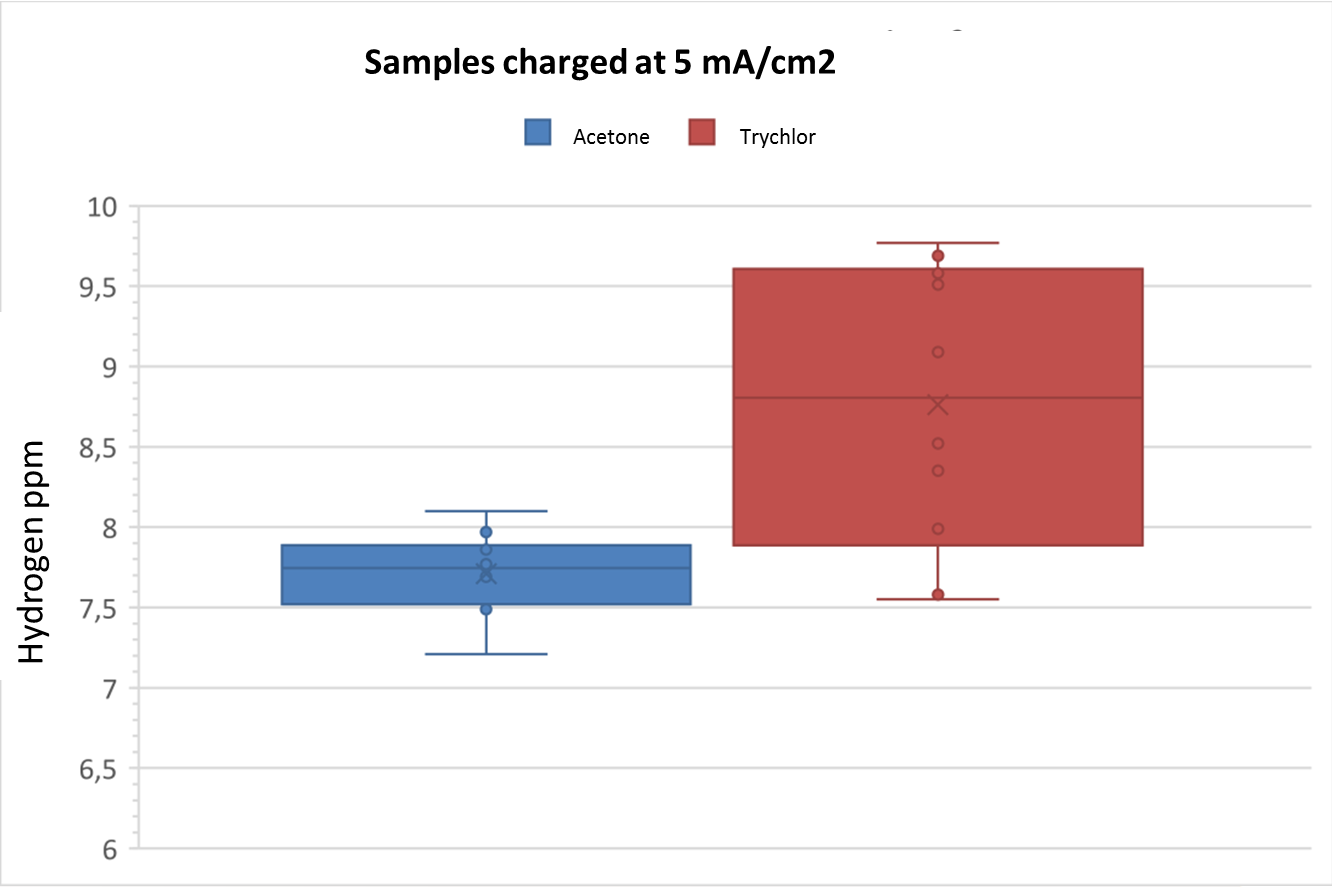
RESULTS ANALYSIS



- Values close to certified values
- The complete method shows lower values indicating the other methods may introduce some H_2 in the sample
- Best results given by Acetone and Trychlor simple methods

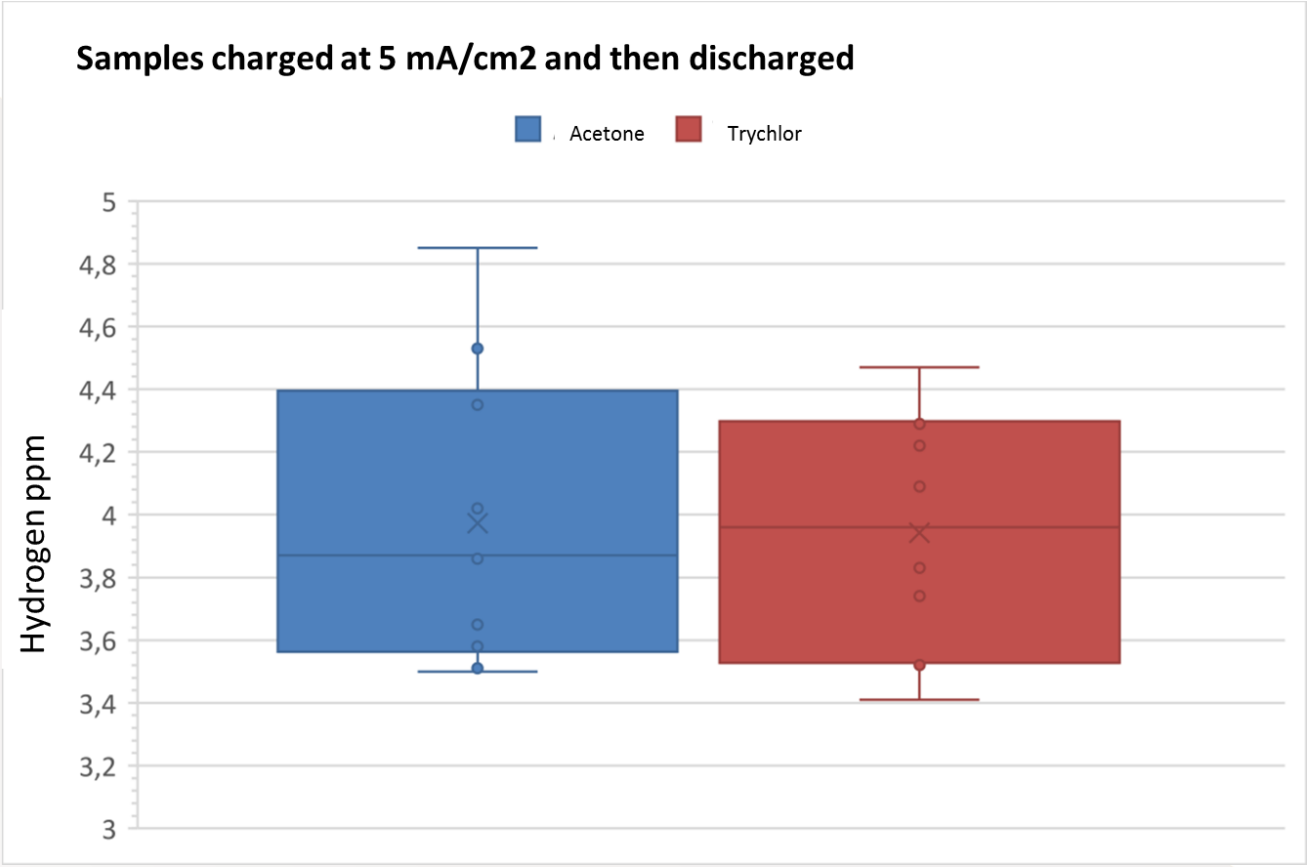


- The simple methods show similar mean and dispersion values
- Again the complete method shows values lower than the rest.
- The HCl dissolution results are around 2 ppm higher, meaning it introduces important amounts of H_2



- With higher H_2 content, part of it diffusible, the simple method with trichlor shows values higher than the simple one with acetone.

- It meansthat trychlor interacts with the diffusion conditions of H_2 in a ferritic matrix charged, allowing hydrogen to diffuse inside, giving inaccurate results.

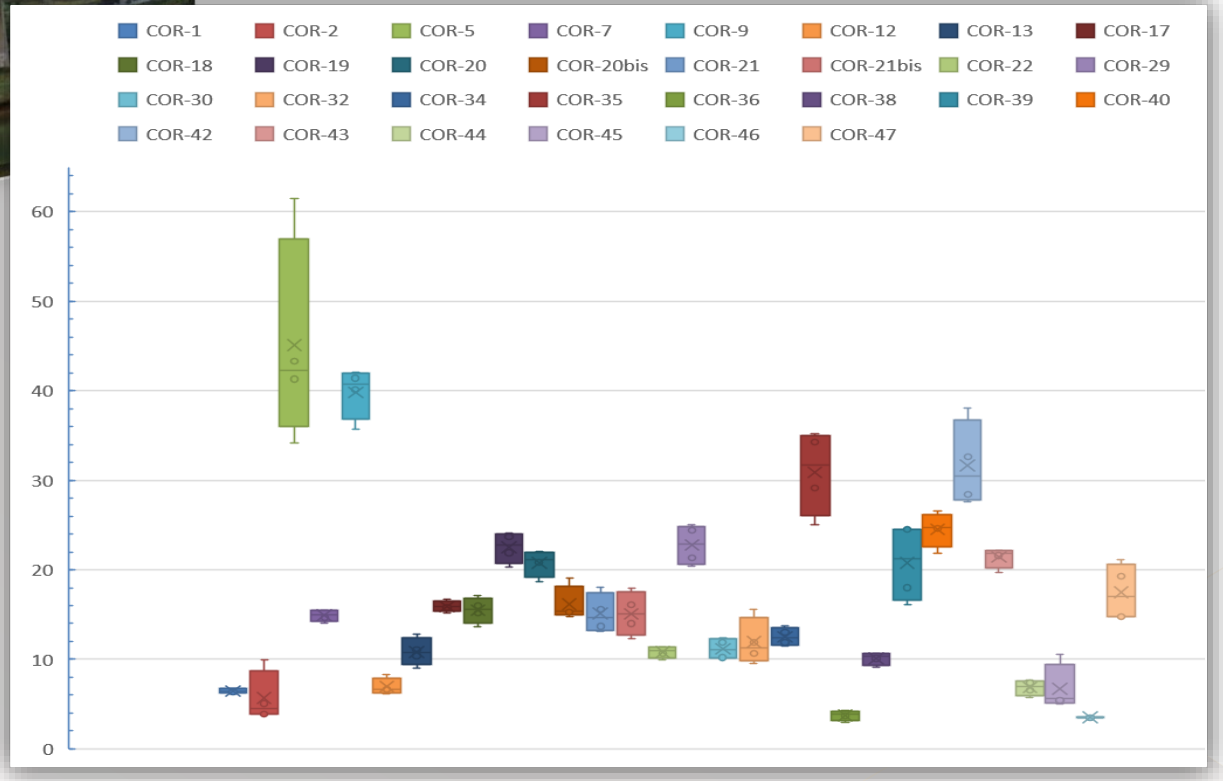


- As the hydrogen content is now lower and deeply trapped, the results of both methods are equivalent.
- The simple trichlor method gives higher values when the hydrogen content is important and a big part of it is diffusible.

CONCLUSIONS

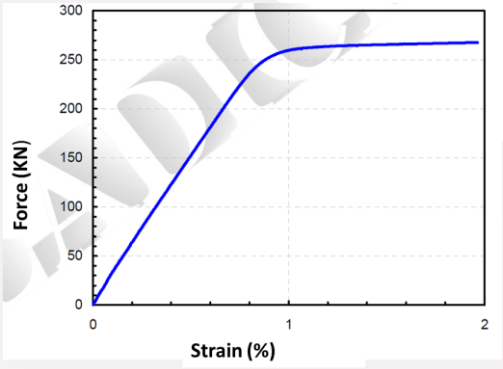
- The ultrasounds methods are not recommended due to longer times.
- The method C-3.5 fromASTM G1 introduces important amounts of H_2.
- With highly trapped H_2, the results of all four methods were quite similar
- When the H content is high and big part diffusible (immediately after charging) cleaning with trichloroethylene interacts offering wrong results.
- **The simple method with acetone is the most practical and reliable one.**

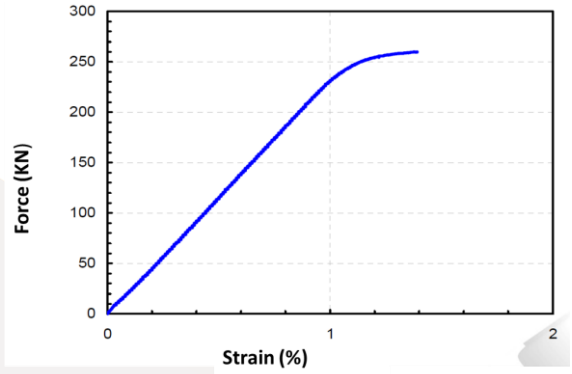
LA ARENA SUSPENDERS BRIDGE

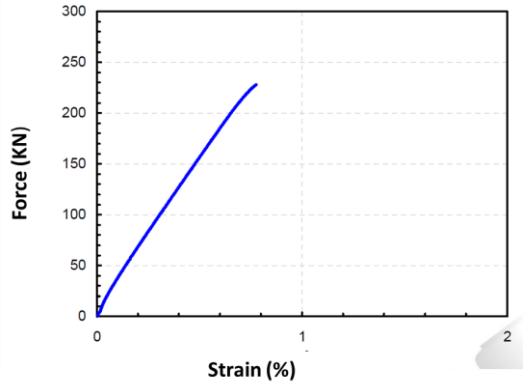


Characterization:

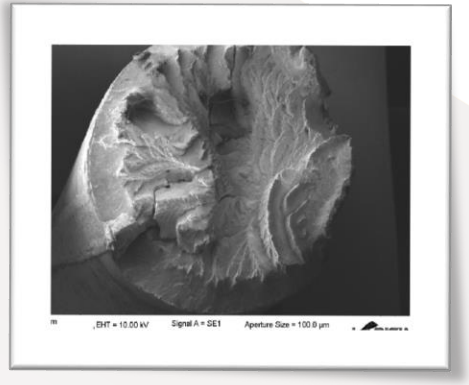
- The availability to deform is reduced
- The breakage gets more brittle and the cracking in the radial direction grows







X < 8 ppm



10 < x < 25 ppm

30ppm > X

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III. **Other Researches in progress**

- 2 Papers to be sent for publishing to impact research journals in the upcoming months.

  ‘‘*Analysis of cleaning methods in hydrogen content determination tests. Application to a failure in a suspenders bridge”*

  “Measurement of Hydrogen Embrittlement Threshold in X80 and S420 Steels by the Incremental Step Loading Technique”

- Paper presented 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.

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