ICHS 2013

5th International Conference on Hydrogen Safety

Brussels, 11th Semptember

HELIOS: A new method for Hydrogen permeation test

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Concerning Hydrogen Induced Cracking (HIC) risk

Hydrogen Embrittlement (HE) and Stress Corrosion Cracking (SCC) are still **severe and current threats**.

The mitigation of HIC risk is fundamental for the safety of the **hydrogen storage** and **transportation**.

Hydrogen sensing and detection

Current techniques (electrochemical & amperometric sensors)

- Parts fragility
- Apparatus complexity
- Poor sensitivity
- Expensive

→ Not suitable for Non-Destructive Testing

Innovative technology (metal oxide solid state sensors)

- Resistant hard-ware
- Easy to handle
- High sensitivity
- Cost saving

→ A new method for in-situ HIC risk assessment

Sensors for hydrogen flux monitoring

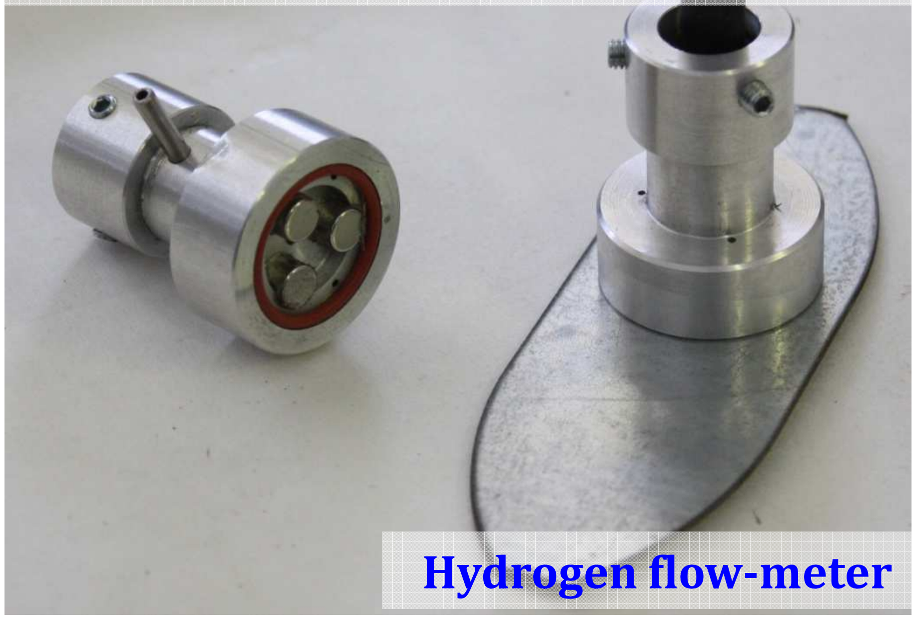
No surface preparation is demanded for hydrogen detection

Permeation cell

- *Response time (T90):*
  <15 s at 100 ppm

- *Sensor sensitivity:*
  0.5 pl/cm^2/s for hydrogen in air

Versatile solutions

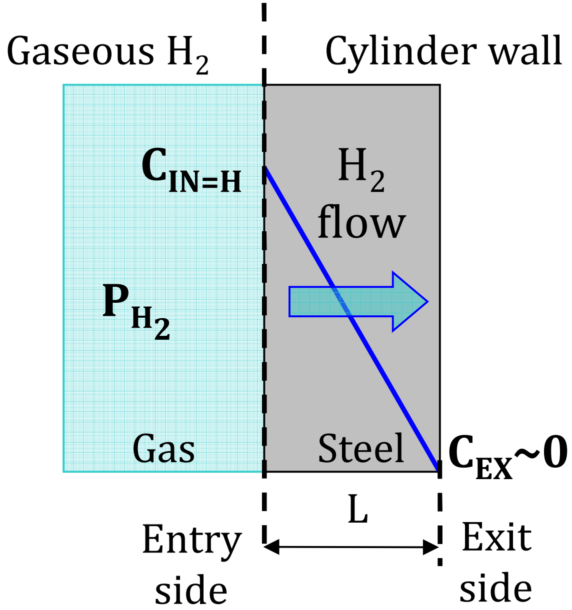


Hydrogen flow-meter

Letomec s.r.l.
Via Fiorentina 47, Pisa, Italy
+39 3930673665
www.letomec.com

Research programme

Step 1: HELIOS measurements

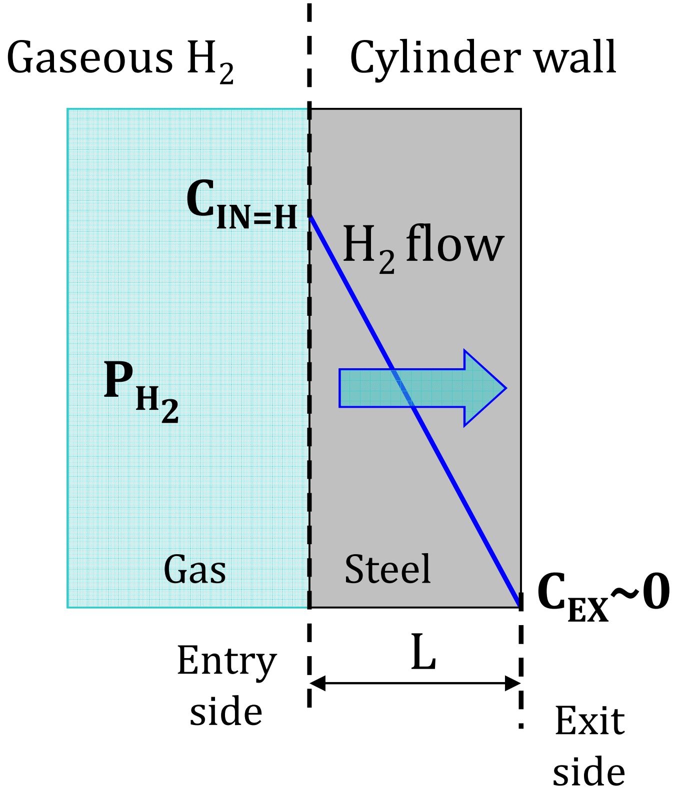


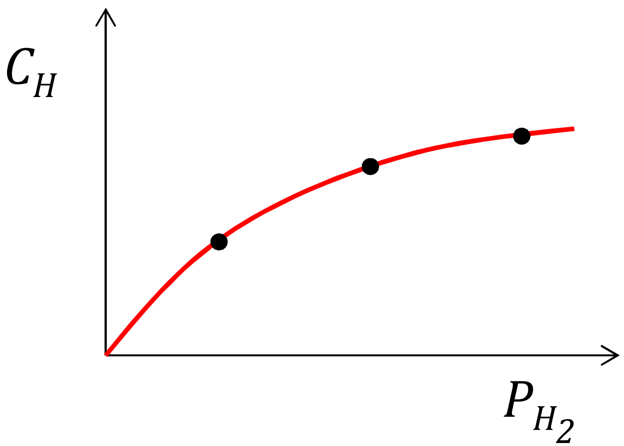
Hydrogen flow through the steel wall of a gas cylinder

Step 2: validation of HELIOS results

Traditional tecniques for hydrogenation and hydrogen content measurement

Validation procedure



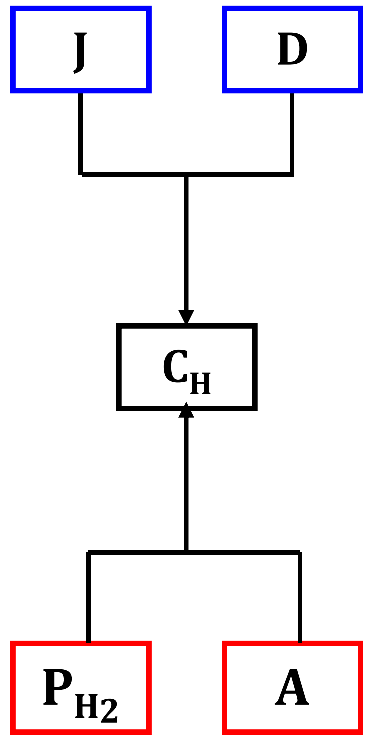


Fick's Law

$$J = D\frac{C_{IN} - C_{EX}}{L}$$

Sievert's Law

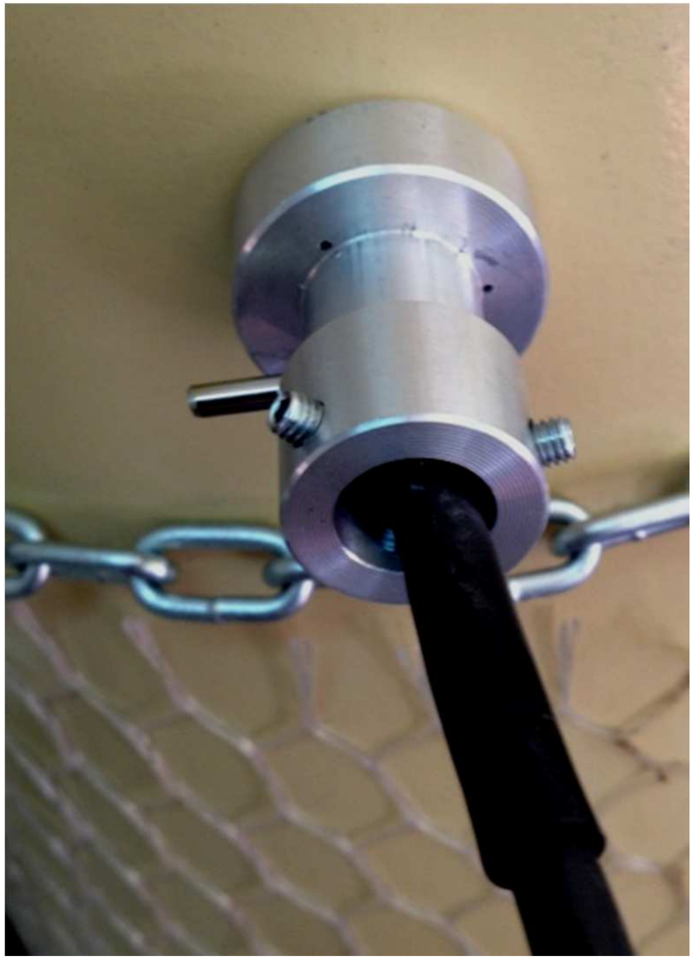
$$C_H = A \cdot \sqrt{P_{H_2}}$$



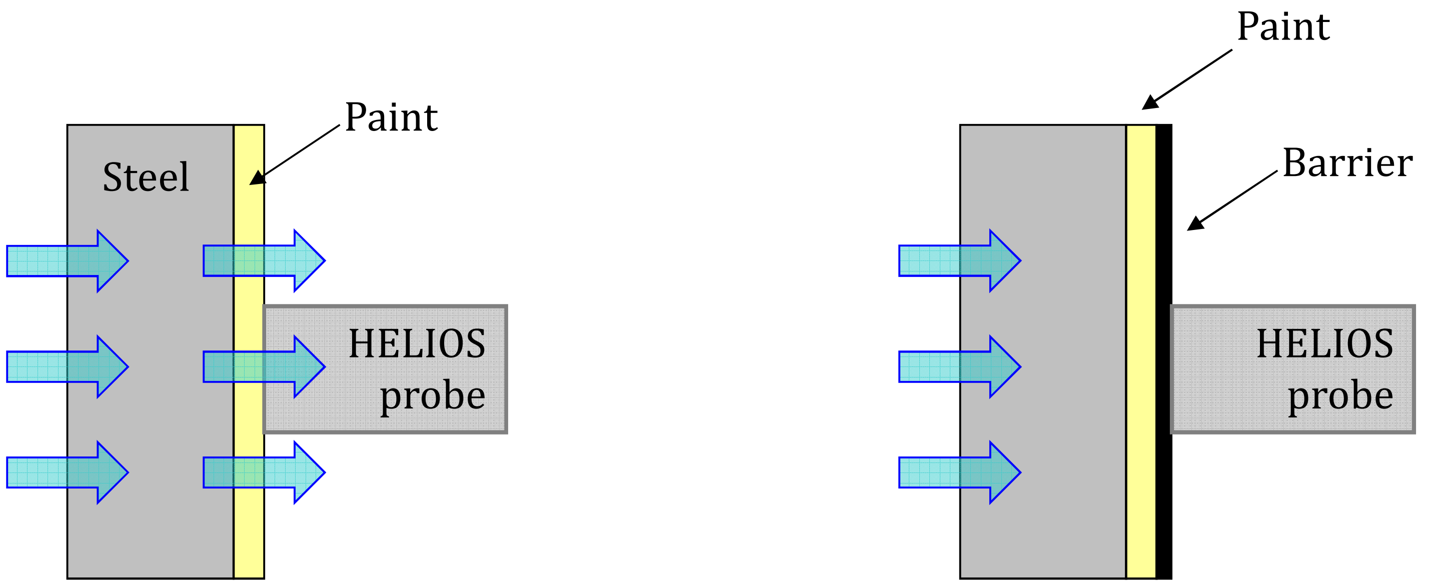
Step 1: HELIOS measurement

- Sample: gas cylinder wall
- Surface condition: **painted**
- Cylinder internal pressure: **60 bar**

X52 low strength steel ($D=2.50 \pm 0.5E\text{-}6\ cm^2/s$).



Hydrogen flux through a painted cylinder wall

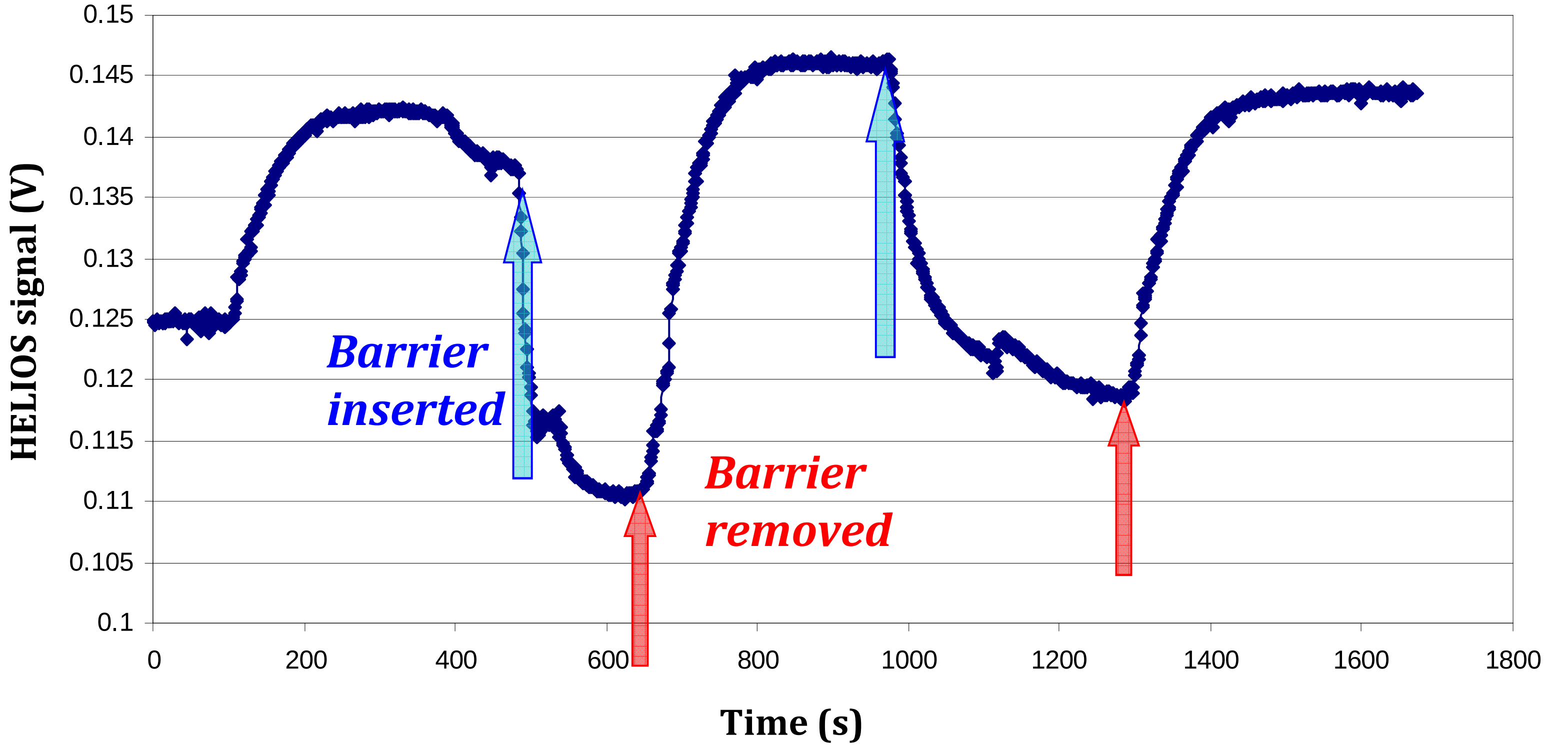


No barrier: hydrogen flux

With barrier: no hydrogen flux

A stainless steel thin sheet was inserted between the probe and the cylinder surface in order **to prevent hydrogen detection** thanks to stainless steel very low hydrogen diffusivity.

Hydrogen flux through cylinder wall: results from HELIOS



Sequence of measurements performed with or without the stainless steel barrier.

Hydrogen flux range: **135 ÷150 pl/cm²/s.**

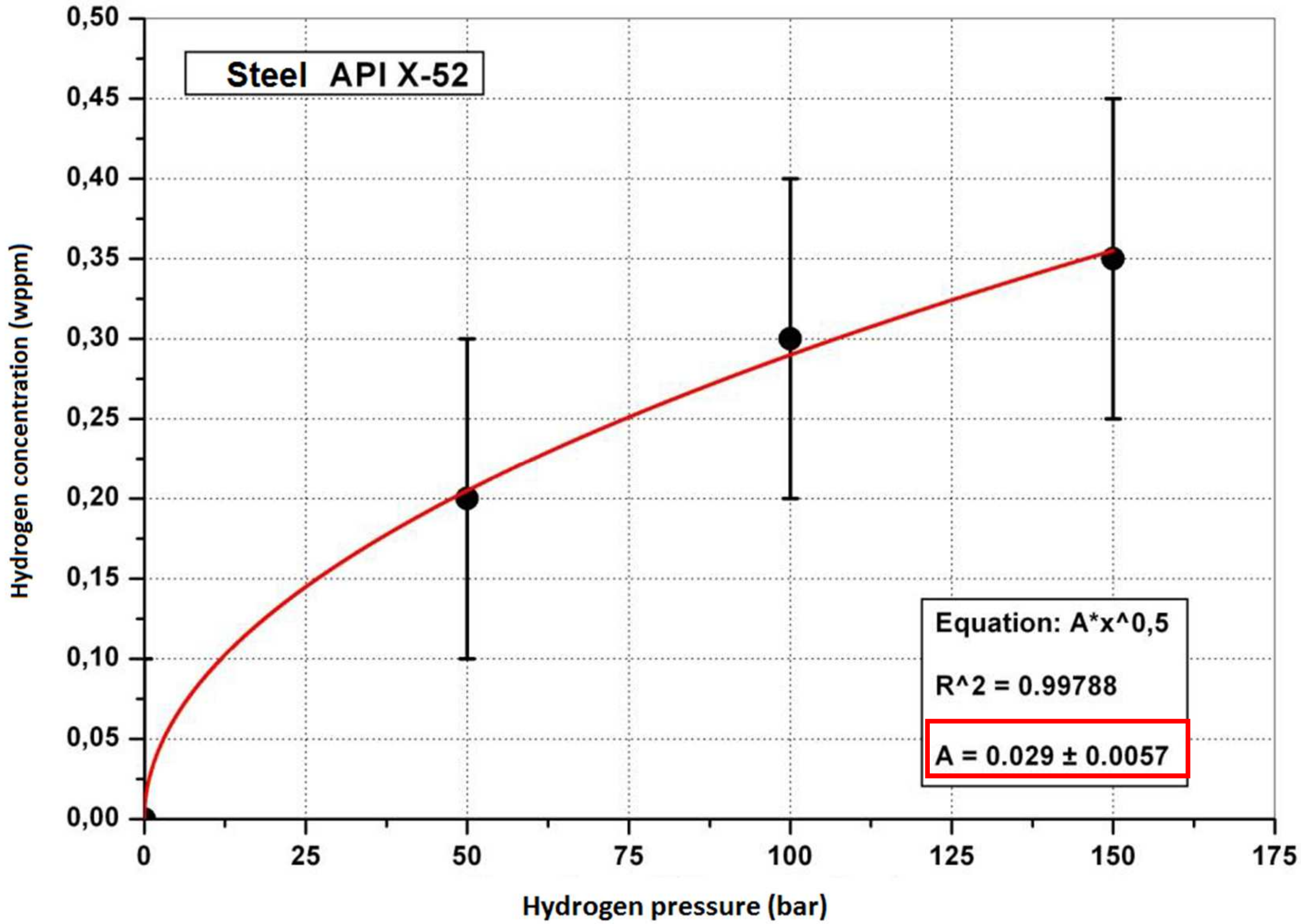
Step 2: validation of HELIOS results

a) Gaseous hydrogenation of X52 steel plane specimens in autoclave was performed at different hydrogen partial pressures (**50, 100** and **150 bar**) for a charging time of **1 week** each.

b) Thermal desorption to measure hydrogen content by means of LECO DH603.

c) Correlation between $\mathbf{P_{H_2}}$ and $\mathbf{C_H}$ gives the experimental relation to verify results obtained from HELIOS analysis.

Hydrogen concentration vs hydrogen pressure



Discussion

From **HELIOS** experimental data, C_H can be calculated by Fick's law at the steady state:

$$C_H = \frac{J \cdot L}{D}$$ **= 0.22÷0.24 wppm**

Substituing gas cylinder pressure and the constant A values in **Sievert's law** expression, it results:

$$C_H = A \cdot \sqrt{P_{H_2}}$$ **= 0.22 wppm**

Conclusions

1) Hydrogen flow measurements, and accordingly in-situ HIC risk assessment, can be successfully faced using HELIOS. In particular, hydrogen flux through a **gas cylinder painted wall** was detected with no preliminary surface preparation.

2) Hydrogen concentration values, obtained by a proper elaboration of HELIOS data, are **coherent** with the results of the analysis carried out according to Sievert's theory.

Future works and potential applications

1) The extreme sensitivity of HELIOS sensor can be usefully employed to measure very **low hydrogen flux in operative conditions** related to hydrogen or hydrogenating fluids (Oil&Gas applications) storage and transportation.

2) Being the detection independent from the tank material, HELIOS can be usefully used to conduct tests on **polymers.** A future campaign on composite materials and multilayer walls is planned.

Thank you for attention.