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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

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

- Sensor sensitivity:
  0.5 pl/cm²/s for hydrogen in air

Versatile solutions

Letomec s.r.l.
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Research programme

Step 1: HELIOS measurements

Step 2: validation of HELIOS results

Traditional techniques for hydrogenation and hydrogen content measurement
Validation procedure

Gaseous $H_2$ | Cylinder wall
---|---
$C_{IN=H}$ | $H_2$ flow
$P_{H_2}$ | Gas
Steel

$C_{EX} \approx 0$

**Fick’s Law**

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

**Sievert’s Law**

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

Diagram:

- $J$ and $D$
- $C_H$
- $P_{H_2}$ and $A$
Step 1: HELIOS measurement

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

X52 low strength steel
(D=2.50 ± 0.5E-6 cm²/s).
Hydrogen flux through a painted cylinder wall

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: $135 \div 150 \text{ pl/cm}^2/\text{s}$. 
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 $P_{H_2}$ and $C_H$ gives the experimental relation to verify results obtained from HELIOS analysis.
Hydrogen concentration vs hydrogen pressure

Equation: $A^x^{0.5}$

$R^2 = 0.99788$

$A = 0.029 \pm 0.0057$
Discussion

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

$$C_H = \frac{J \cdot L}{D} = 0.22 \div 0.24 \text{ wppm}$$

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

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