

Permeation and Leak Testing of Composites for Tank Systems under Tensile Stress

tank.tech 2009, Tüv Süd Automotive GmbH, Bad Gögging bei München

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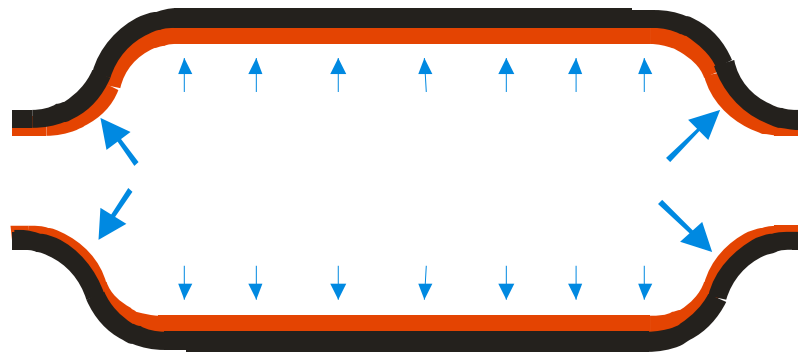
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Contents

- Objectives
- Measurement method and apparatus
- Results
- Integration of method in tank development process

High Pressure Hydrogen Storage Tank



Conditions:

- -40 °C ... +85 °C
- Up to 100 MPa
- Resulting tensile stress estimated 2 % ... ≤ 10 %

Concept: **Barrier liner** inside for permeation reduction and carbon fiber reinforced polymer outside.

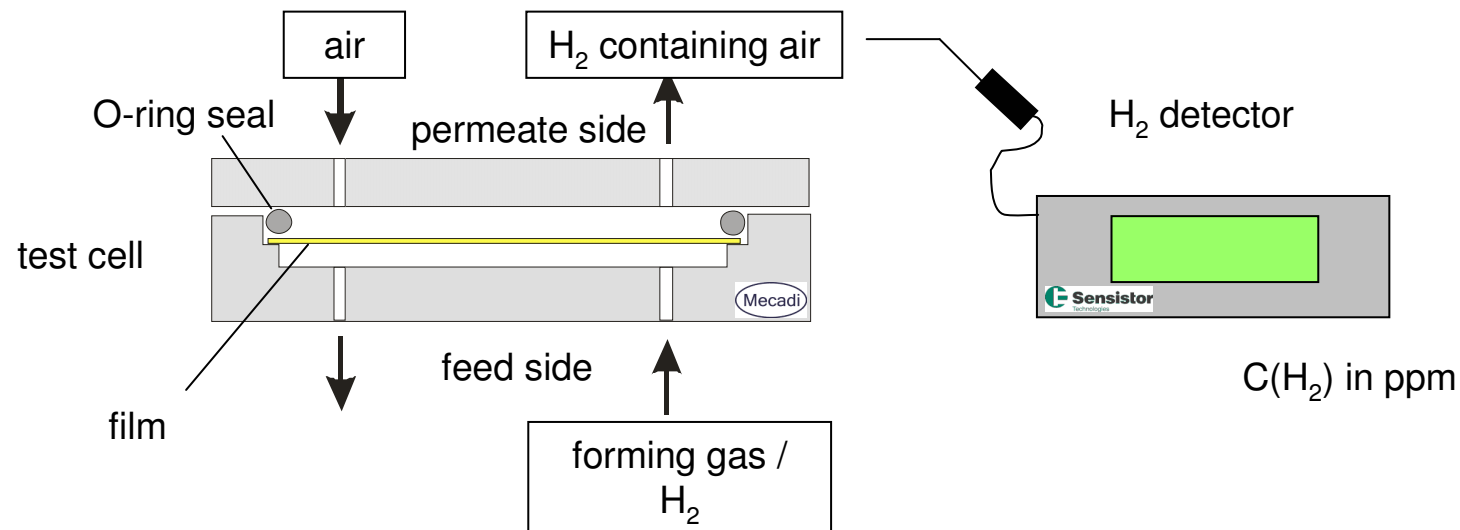
→ Tensile stress for barrier layer

Materials for Barrier Layers

- Selection of barrier materials with **permeation** specification to be fulfilled at:
 - high temperatures and pressures
 - low temperatures (no cracks)
 - **under tensile stress**
 - Opposite direction of changes in **mechanical** and **barrier** properties
 - Material candidates like polymers with a high degree of crystallinity (EVOH, LCP), inorganic coating (SiO_x) or metal (Al) show low tolerance to tensile stress (forming of defects, cracks)
- ⇒ Permeation as a function of tensile stress has to be examined!



Measurement Method for Hydrogen Permeation



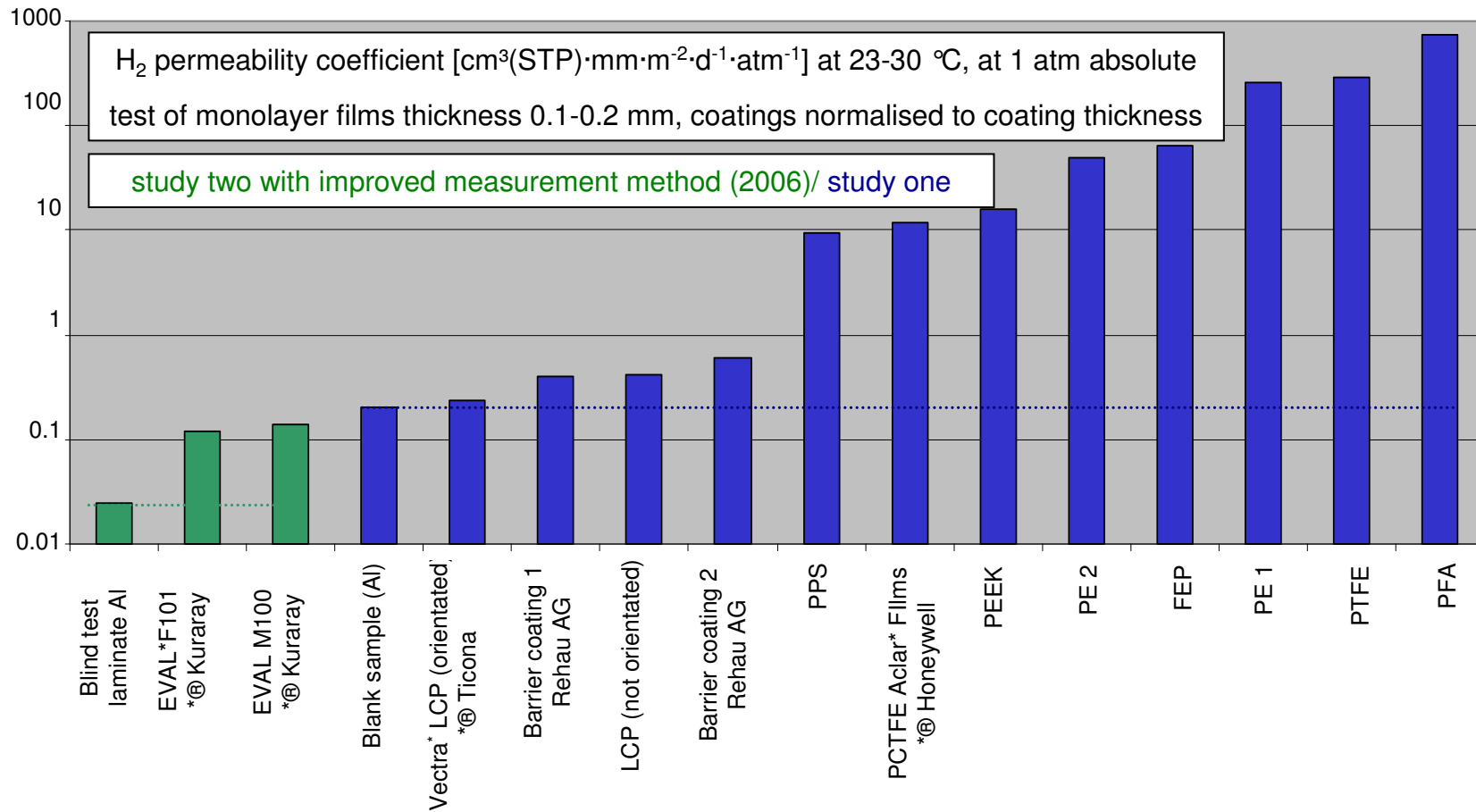
- Forming gas or H₂ flows across the film on the feed side
- H₂ permeates through the leak-free film
- Airflow transports the permeated H₂ to the detector
- Permeation rate is calculated from flow and concentration

Hydrogen Trace Analysis

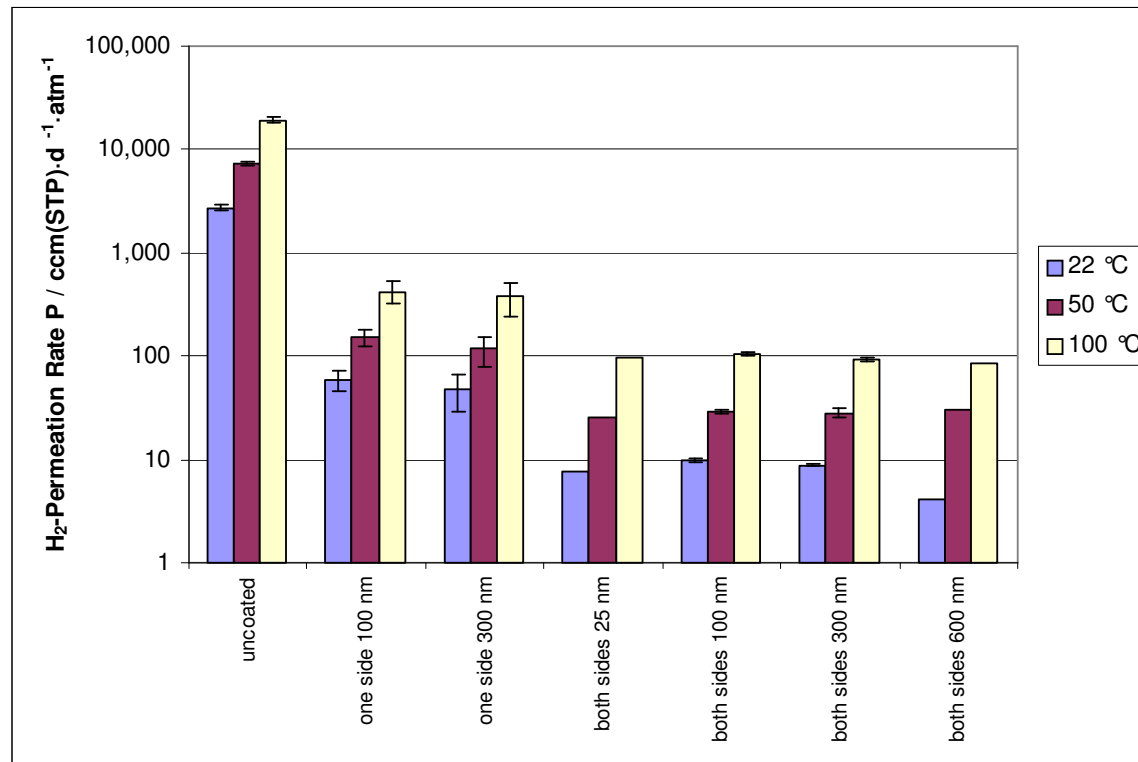


- Used for hydrogen detection:
Adixen Hydrogen Leak Detector H2000 Plus
- Detection range: 0.5 ppm - 100 ppm H₂

Material Screening for Low H₂ Permeation



Permeation of Coated Polyimide Films (2009)



Calculated
H₂ permeability
coefficient
barrier layer of
double-sided
25 nm coating

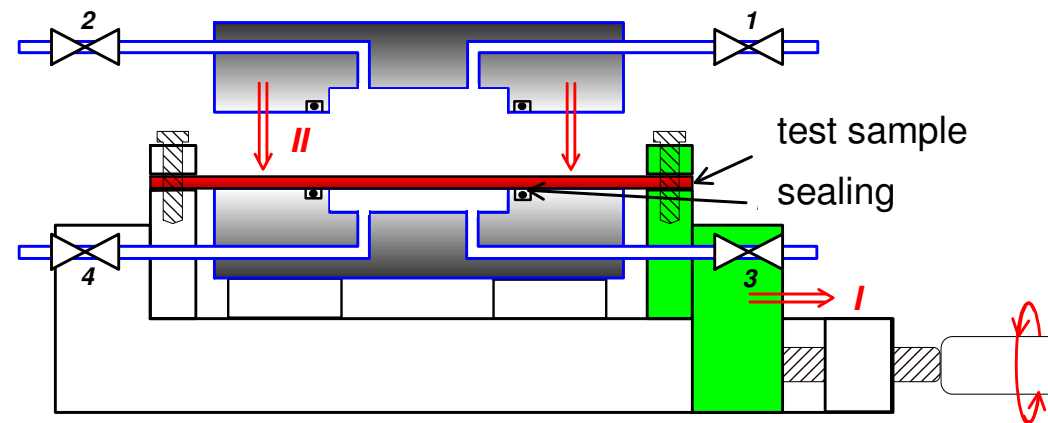
23 °C

4·10⁻⁴

cm³·mm·m⁻²·d⁻¹·atm⁻¹
(STP)

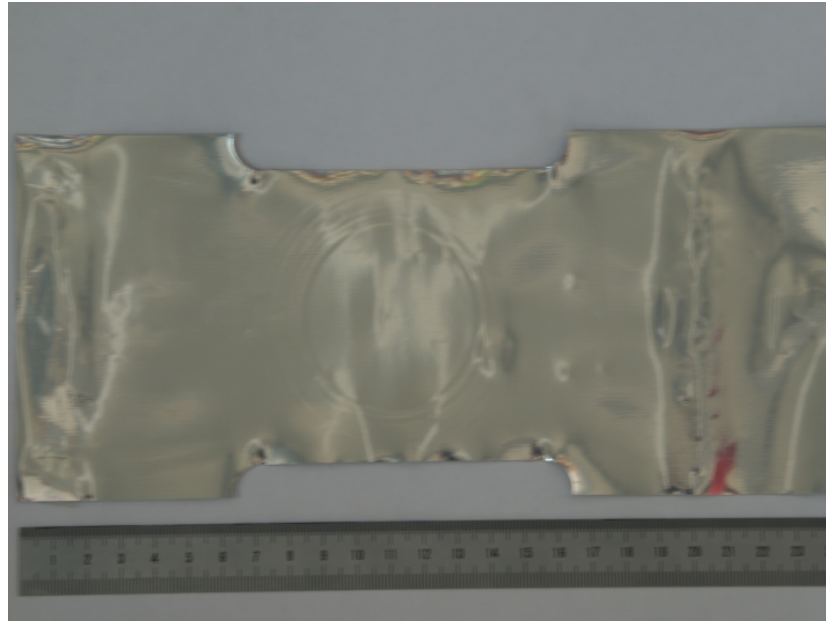
Films supplied by *JOANNEUM RESEARCH*

Permeation Measurement under Tensile Stress



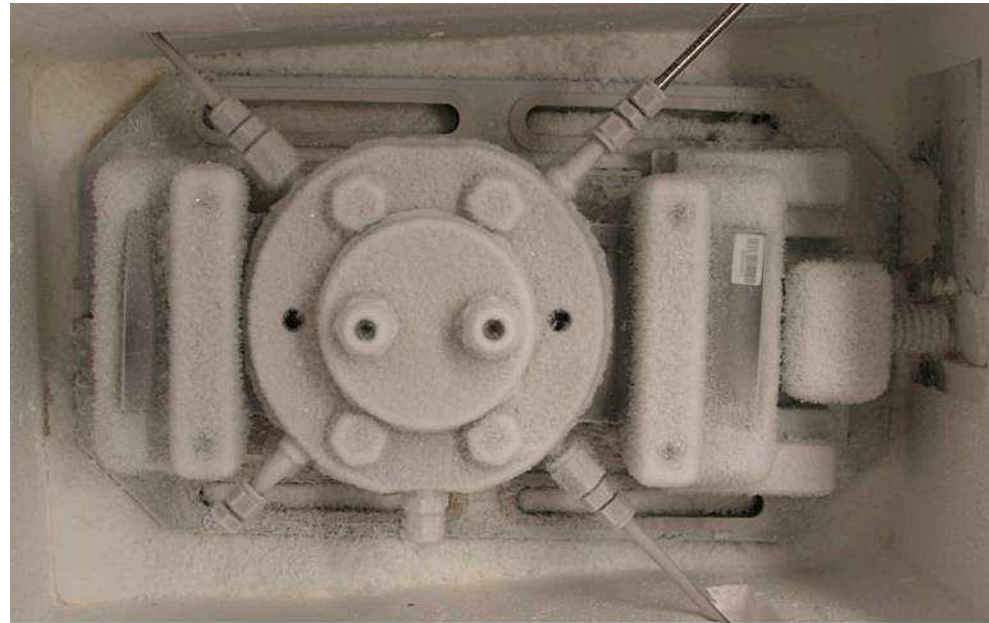
- Polymer film/ composite fixed with clamps (no thickness limit)
- Stepless elongation (0 - 25 % possible, tensile force max. 1000 kg)
- Permeation measurement cell is closed after tempering (-80 °C ... +200 °C range possible) and elongation
- Test media can be gas or liquid, pressure up to approx. 10 MPa
- Development in cooperation with University of Saarland

Specimen



- Flat samples 300 x 100 mm
- Broadness reduced in permeation measurement area
- Measurement area 50 cm²

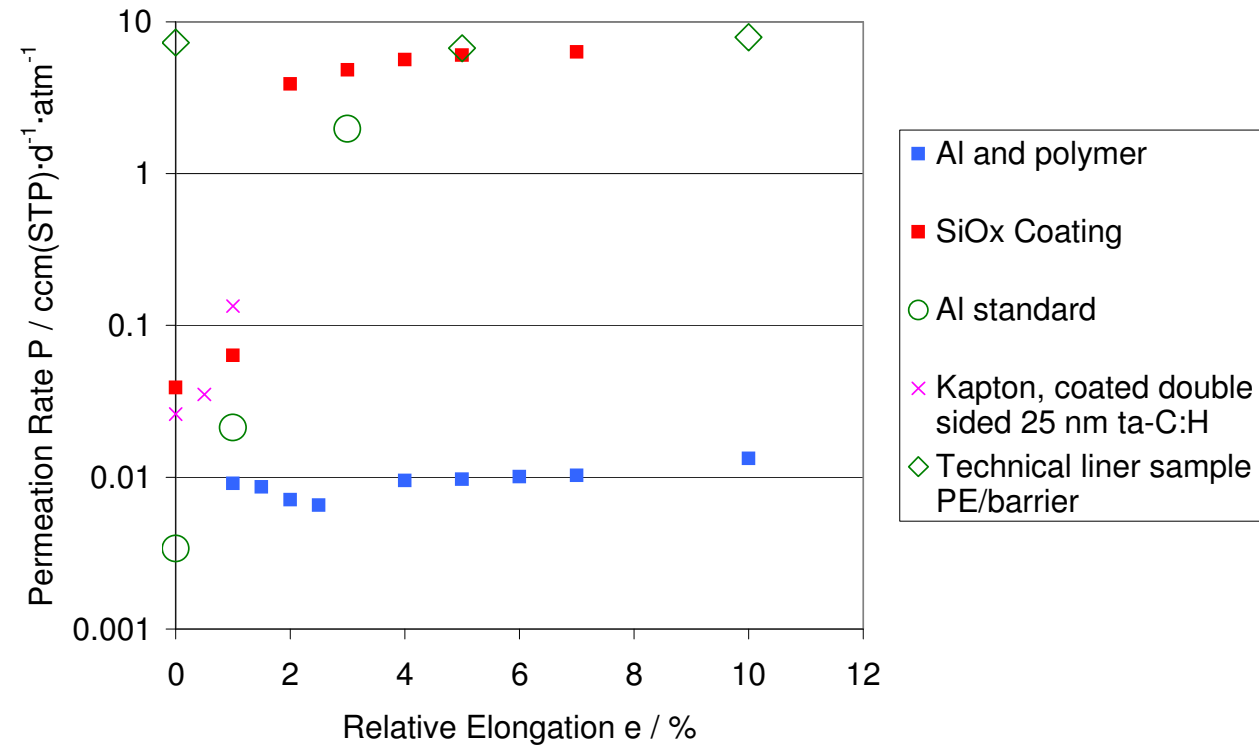
Low Temperature (-80 °C) under Tensile Stress



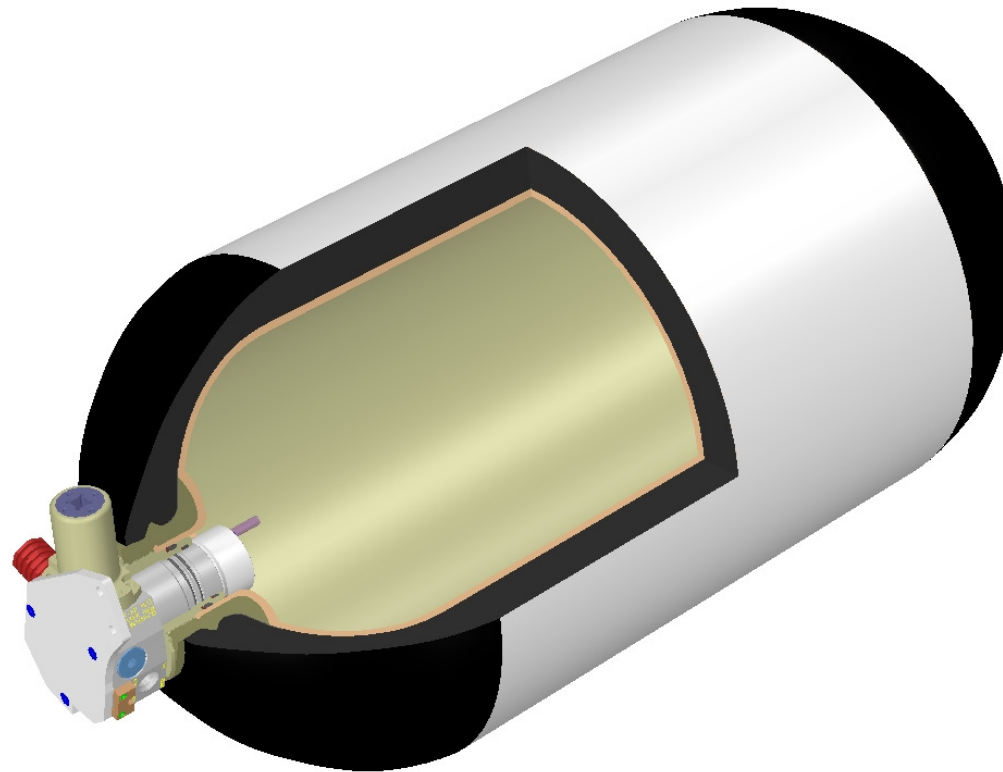
- Permeation/ leakage measurement detects cracks/ failure at low temperature
- Polymer films can be cooled down to or below glass transition point

Results of Screening of Barrier Films

Permeation vs. Relative Elongation (Internal Study)

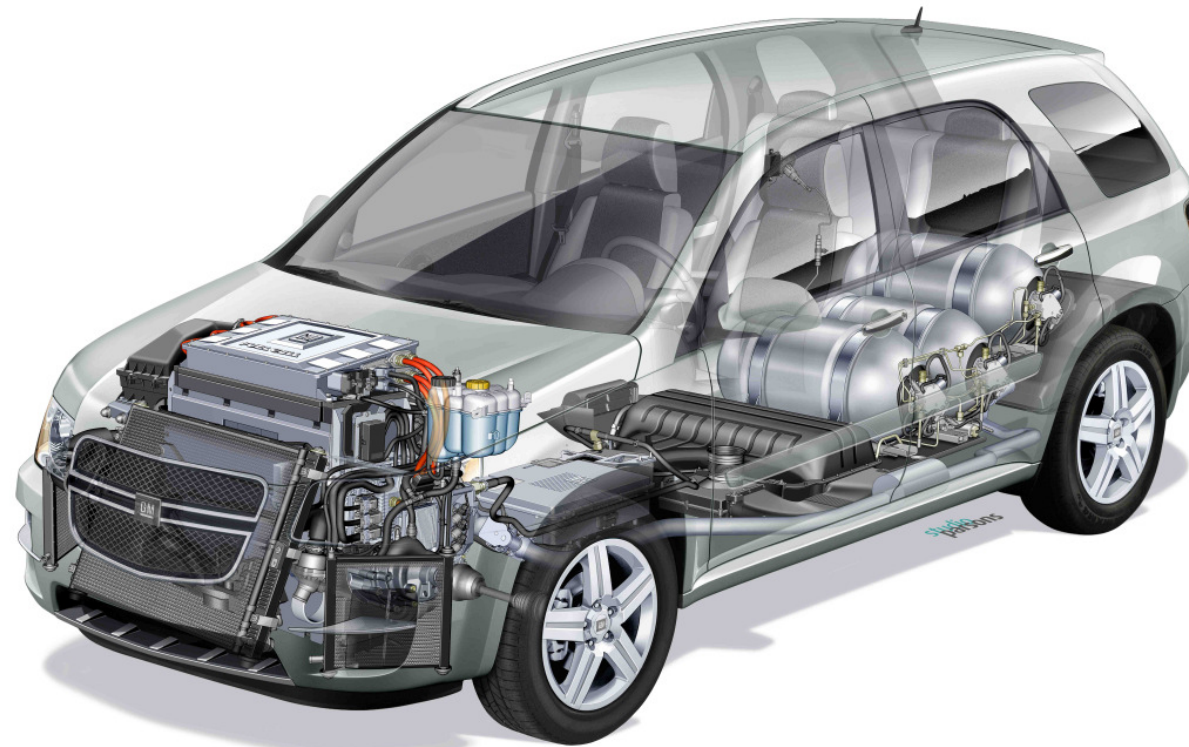


Tank design with barrier liner inside



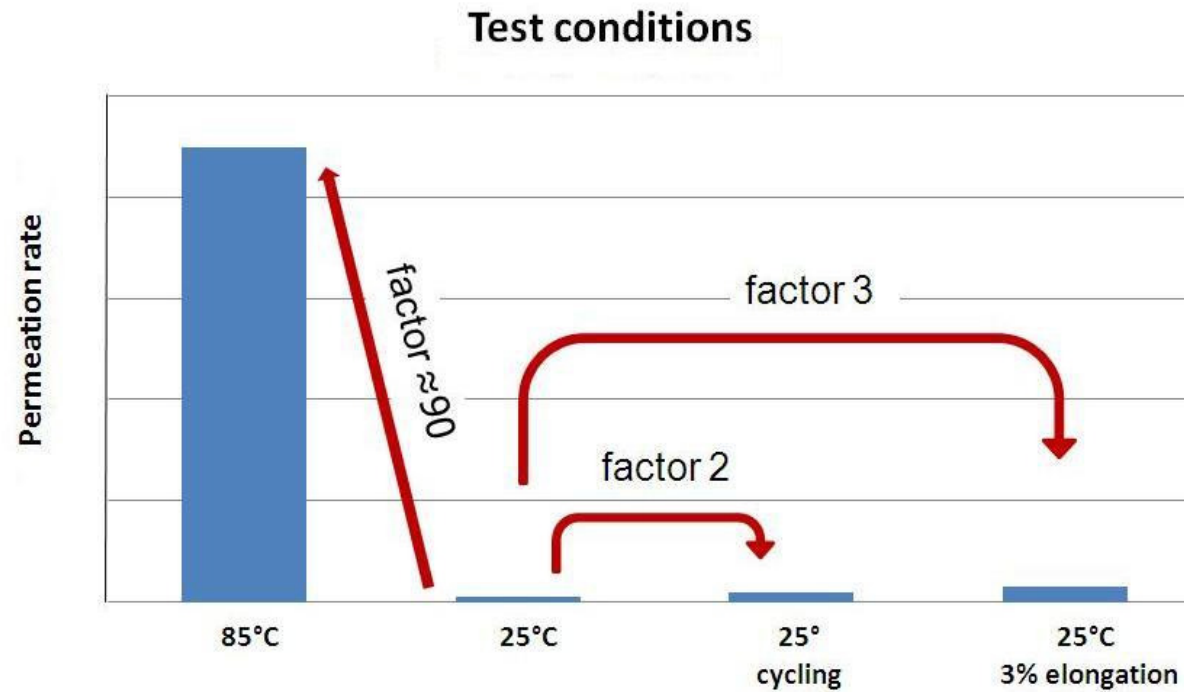
High pressure hydrogen storage tank (GM)

GM HydroGen4



GM HydroGen4 Equinox Hydrogen Fuel-Cell Vehicle

Testing of Hydrogen Tank Liner Candidate



Influence of temperature, cycling and elongation to permeation of liner

Testing of Hydrogen Tank Liner Candidates

Study Summary:

For the examined material composition:

- tensile stress enlarges permeation,
- total permeation is mainly governed by temperature effects,
- materials/ laminate combinations can be preselected by combined permeation/ tensile strength measurements,
- low temperature behaviour/ cycles can be implemented,
- total permeation can be calculated with design/ temperature data.

Summary

Combined measurement of permeation and tensile stress leads to preselection of materials before prototyping:

- Implementation of permeation as a method to detect defects, cracks under practical conditions.
- Definition of tolerable mechanical stress without destruction as a basis for construction and design.

Future tasks:

- Transfer of existing technologies in packaging applications to barrier systems of tanks: combination of barrier layers with intermediate layers to improve mechanical properties and for defect decoupling.

Thank you for your interest !

The Potential

Corresponding author (lecturer):	Energy density [kWh·kg ⁻¹]
Dr. Andreas Konrad	Gasoline 11,0
Mecadi GmbH Chemicals/Processing	Diesel 11,7
Industriegebiet In der Kolling	Methanol 5,5
In der Kolling 9	Ethanol 7,5
66450 Bexbach	Hydrogen 33,3
Germany	Li-battery (Co) 0,55
mail: info@mecadi.com	Li-battery (Mn) 0,43
phone: +49 6826 93383 0	

Energy storage capacity of pure substances
(without containment and enclosed components)

Mecadi - your service partner for customer specific permeation measurement