DE LA RECHERCHE À L'INDUSTRIE

MATERIAL TESTING IN H_2 GAS

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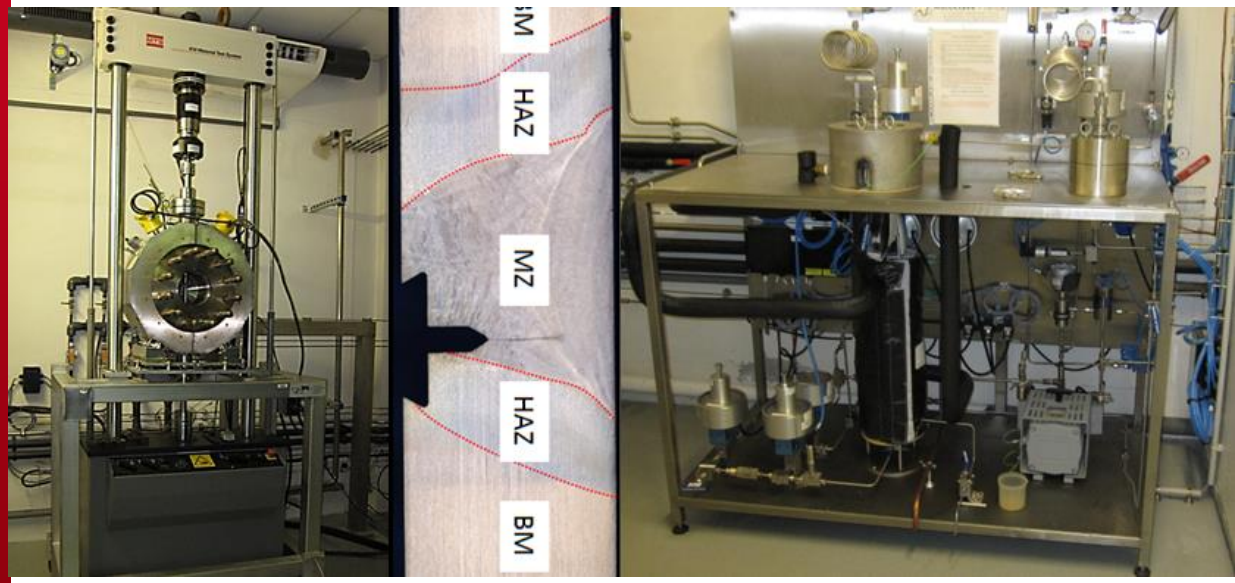
SANDIA NATIONAL LAB



I. Moro, L. Briottet, P. Lemoine

APRIL 9-10, 2013

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MATERIAL TESTING UNDER HYDROGEN GAS AT CEA/LITEN

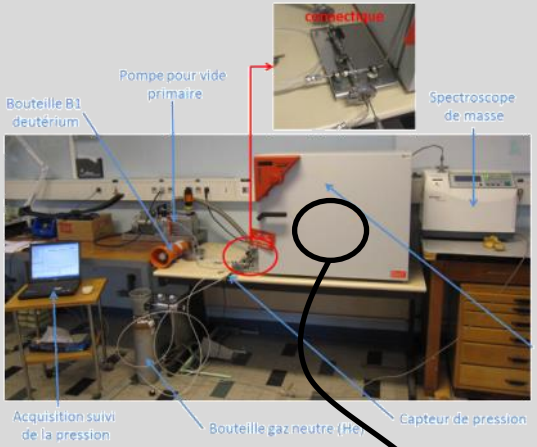
OVERVIEW

MATERIAL TESTING IN GASEOUS H_2

Uniaxial loading under H_2 < 350 bar and < 300°C

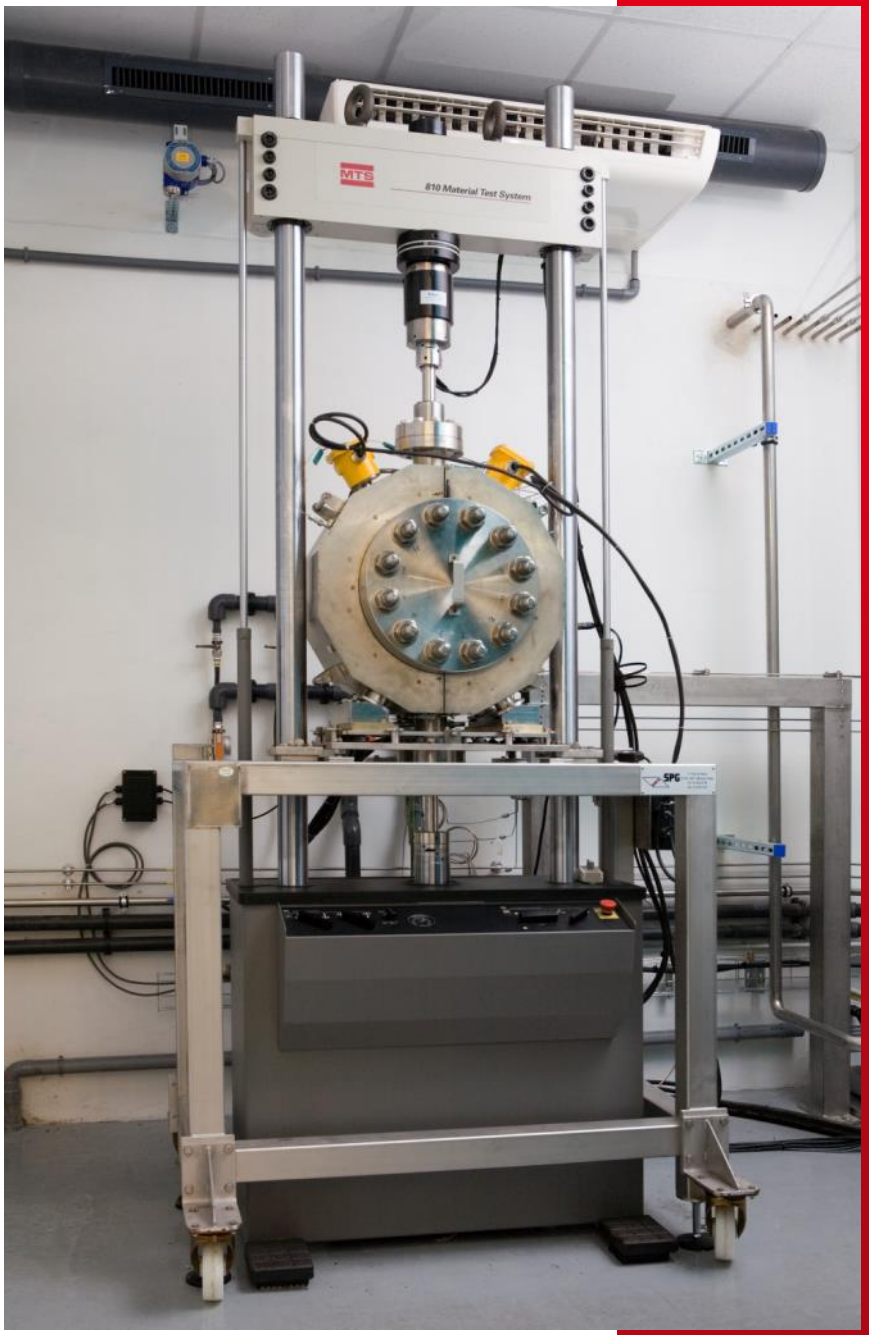
Disc/tube test under H_2/He < 1000 bar - Impurities

H_2/D_2 gas permeation tests < 5 bar and < 300°C



Permeation cell

liten



PRESSURE VESSEL
+
SERVO-HYDRAULIC TEST FRAME

- MTS servo-hydraulic test frame (250 kN)
- Custom vessel
- Frontal opening

PRESSURE VESSEL : OVERVIEW

PRESSURE VESSEL : OVERVIEW

Specific design to allow thermal expansion while remaining aligned

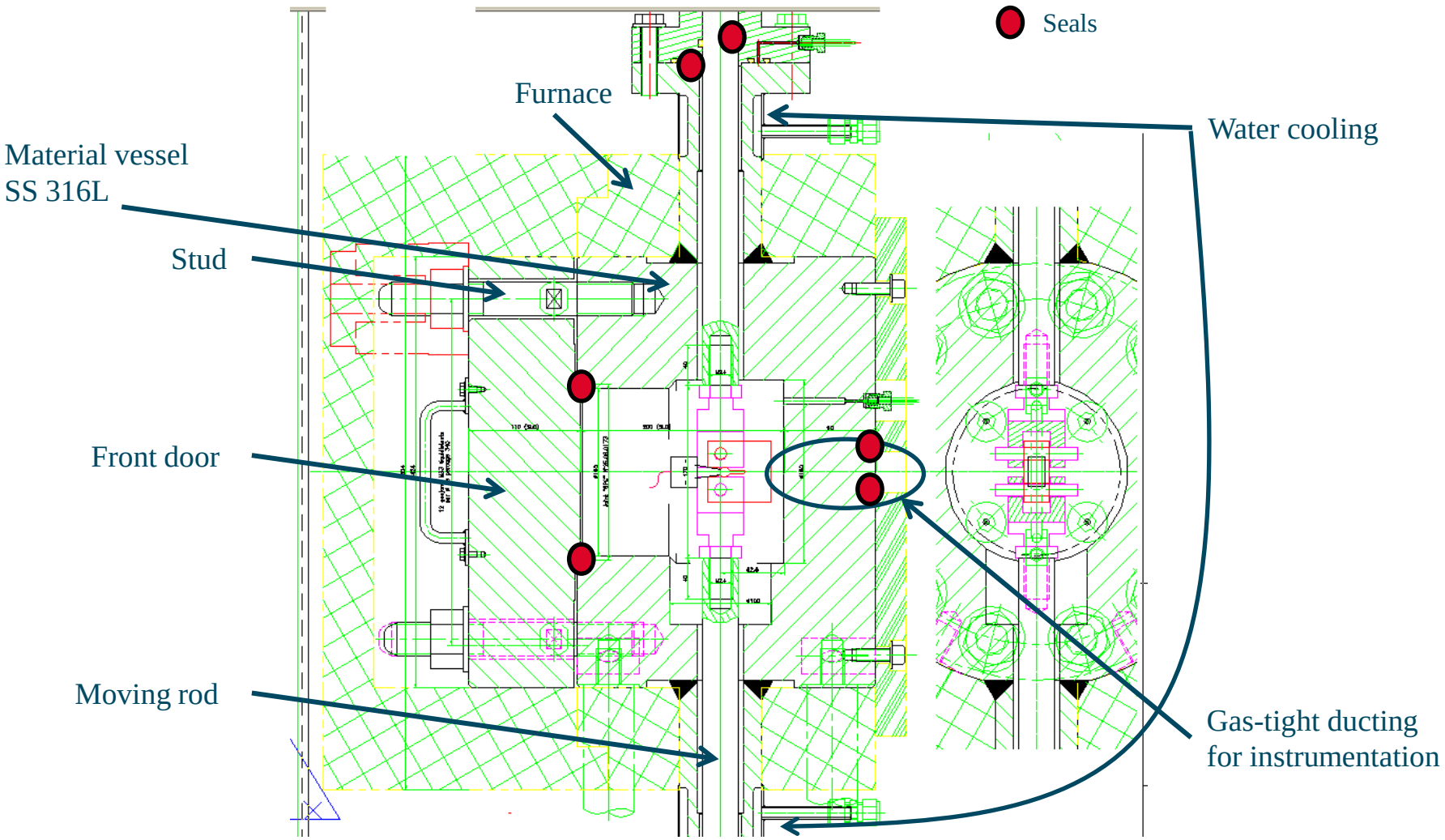
Specific device to screw the door at nominal torque without damaging the load line

Device for pressure vessel alignment

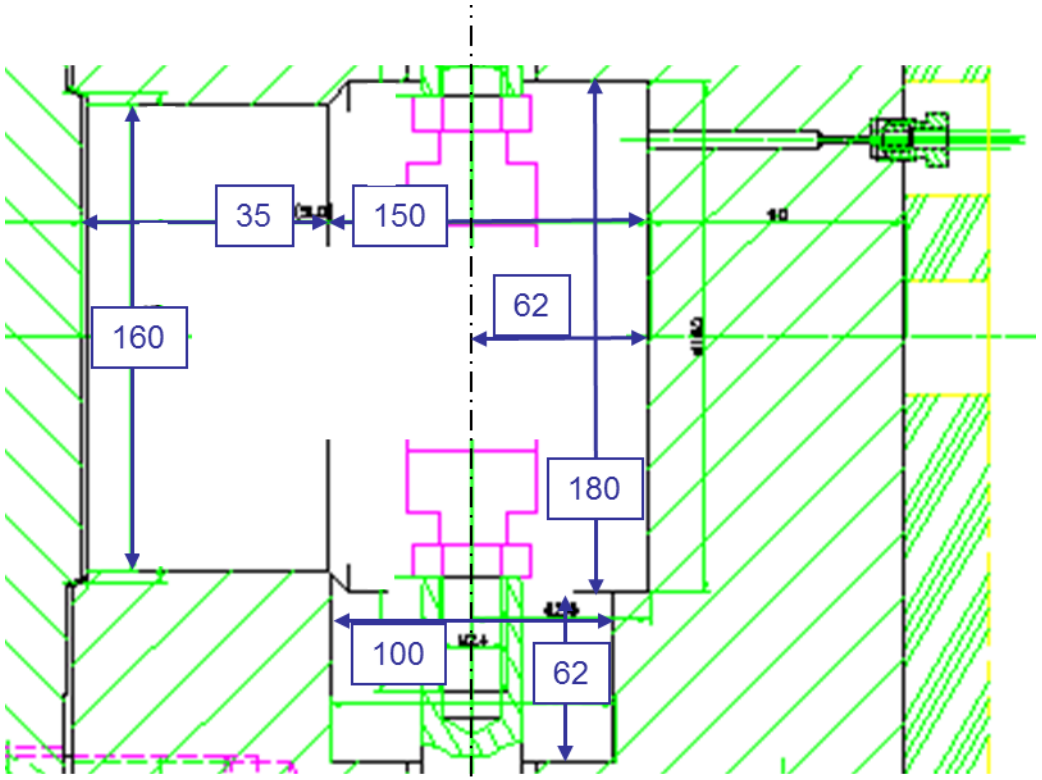
PRESSURE VESSEL : TESTS UNDER HIGH PRESSURE H_2

Pressure vessel	
Maximum volume available	5,5L + use of dedicated tool to reduce the gas volume
Max pressure (MPa)	35
T (°C)	RT - 300
Test frame	
Device maximum load	50 kN
Displacement	~100 mm
Min displacement rate µm/s	~1
Fatigue test	R > 0
Frequency	Tested < 10Hz under H_2

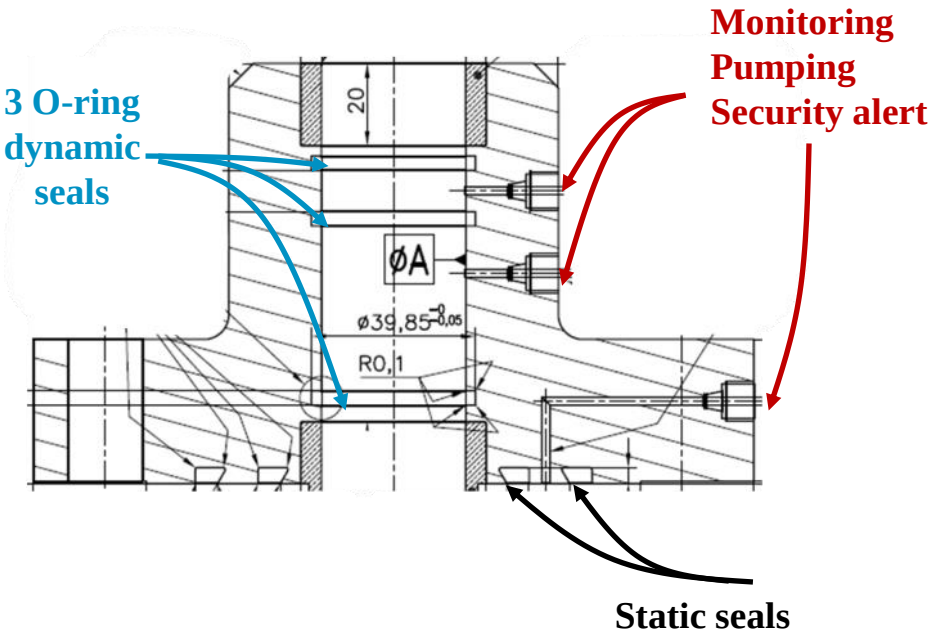
PRESSURE VESSEL : CROSS SECTION



PRESSURE VESSEL : CROSS SECTION



PRESSURE VESSEL : SEALS

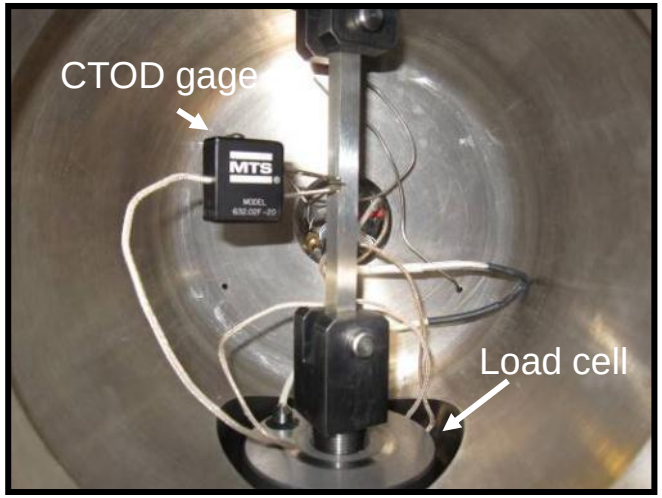


Kalrez seals
(perfluoroelastomer)

SS S-type seals (temperature)

PRESSURE VESSEL : TRANSDUCERS

Strain gages COD or extensometers

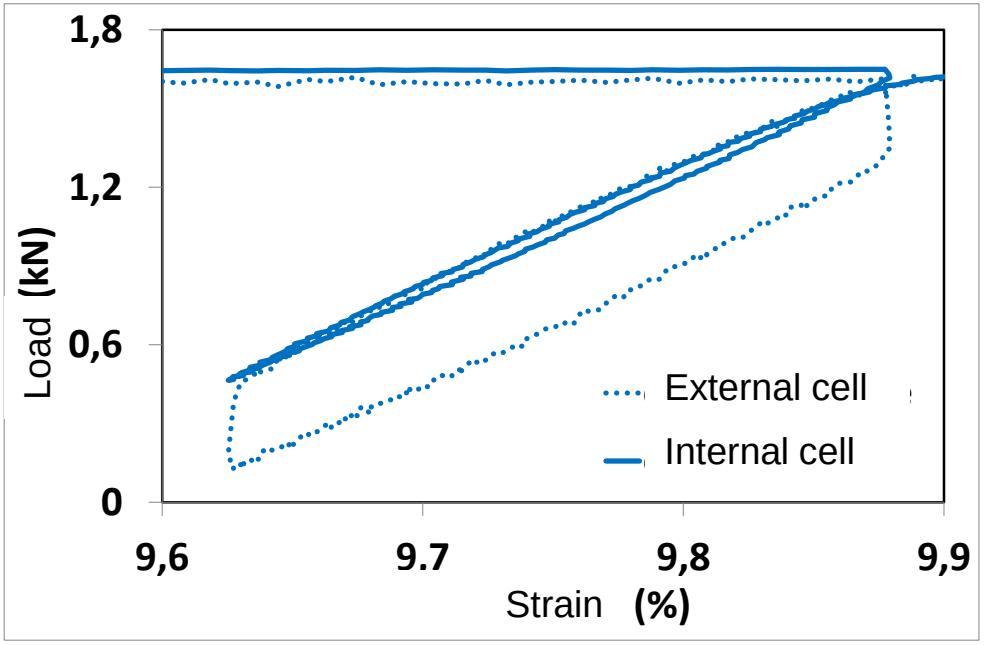


- Signal drift during long term tests under hydrogen pressure (> few hours)
- Not stabilised after 2 days under 30 MPa H_2
- Slow drift : not critical for cyclic loadings
- WOL test (with in-situ precracking) not possible

PRESSURE VESSEL : LOAD CELL

- External load cell (100kN) : friction between rod and seals estimated < 300 N
  => detrimental for tests below ~10 kN

- Development of an internal 50kN load cell (strain gages protected from H_2 gas)
  no drift
  very sensitive to alignment and clamping



PRESSURE VESSEL : GAS MANAGEMENT

- H_2, N_2
- Remote control and monitoring
- Pneumatic valves
- H_20 et 0_2 sensors : not effective for low ppm (< 10 ppm)

  Hygrometer : capacitance principle using a gold/alumina oxide probe

  Trace Oxygen Analyser : Micro fuel cell (electrochemical galvanic device)

⇒ Tests under H_2 : N60 purity gas

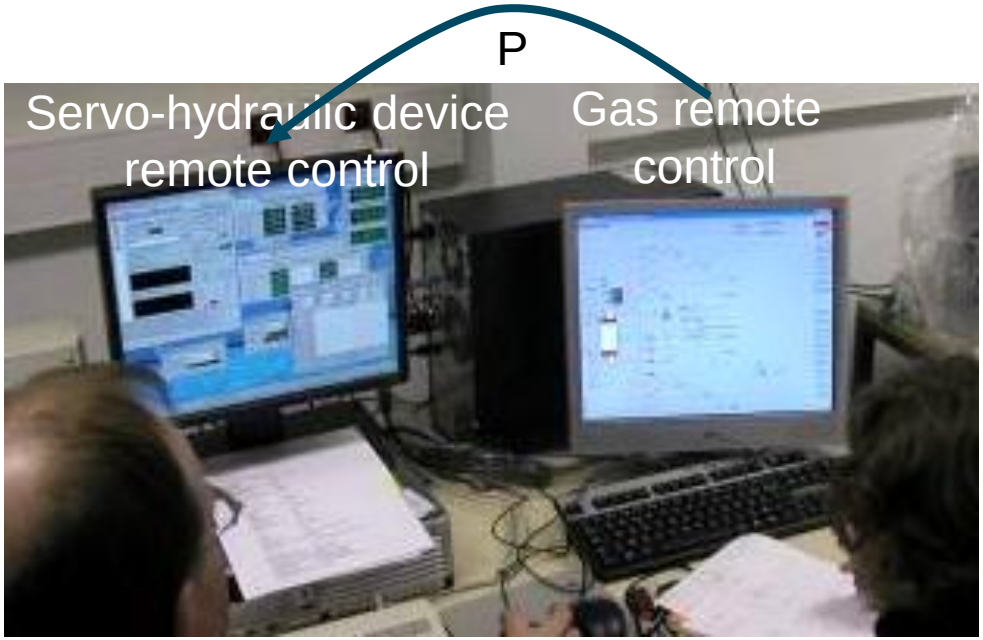
Always the same procedure including :[Vacuum / N_2] (x 3) before H_2 inlet

liten

PRESSURE VESSEL : TEST PROCEDURE

- Sample preparation :
  - traction / LCF : surface roughness
  - crack growth / toughness : precrack under air

- Gas management procedure to ensure gas purity
- N_2 pressure at testing pressure to check tightness
- H_2 pressure
- Tests using MTS (MPT or specific procedure)
- After testing : H_2 sweeping out, N_2 inlet, vacuum, 1 bar N_2



External load = f(P)

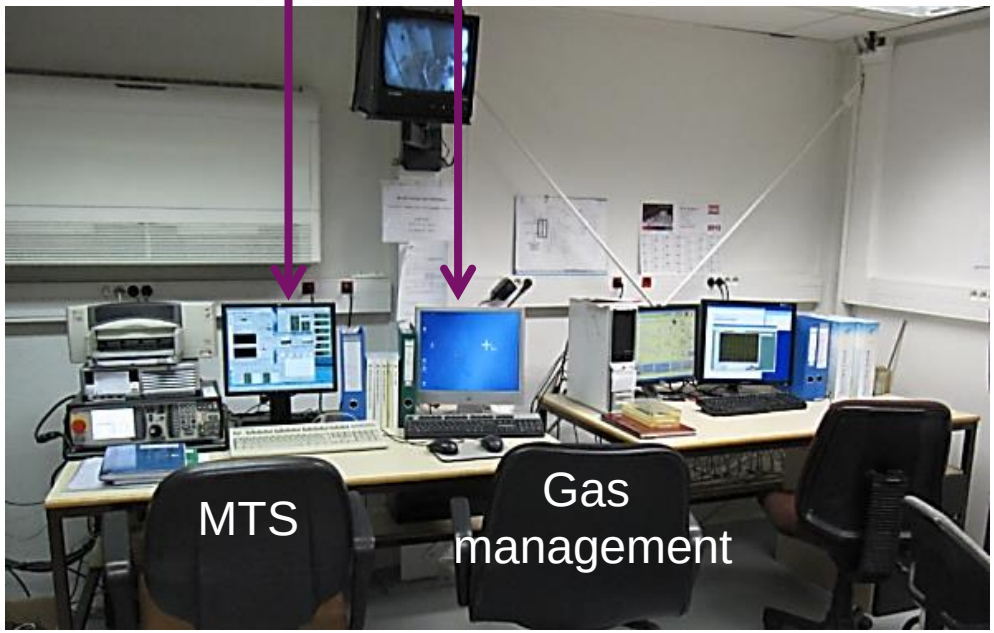
PRESSURE VESSEL : SAFETY ISSUES

- Remote control – No access under H_2
- Automatised gas management procedure
- Security camera
- Min venting flow rate

- Seal tightness control
- H_2 and O_2 sensors
- 2 H_2 thresholds
- 10 % Inferior flammability limit in air
- 20 % Inferior flammability limit in air

Automated safety procedure

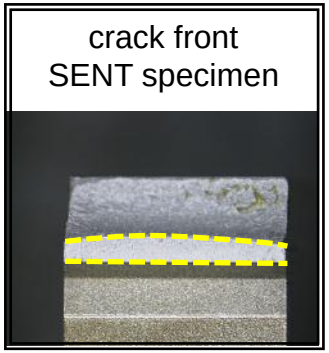
Shut down all electrical power in test room
→
H_2 sweeping out
N_2 inlet
Max venting flow rate in the room



PRESSURE VESSEL : SUMMARY

MAIN ADVANTAGES

- Available volume (+ use of tools to reduce gas volume) / instrumentation
- Remote gas management
  → "Fast" atmosphere switches
- Reliable : 6 years old – No major trouble
- Precise measurement from low to high loads
- Load cell : no drift under H_2
- Alignment : ~ straight crack fronts



MAIN DISADVANTAGES

- Test in temperature : thermal inertia
- Test preparation : ~3 hours, alignment
- Gas purity analysis
- Pressure below 35 MPa / Low P accuracy below 2 bar

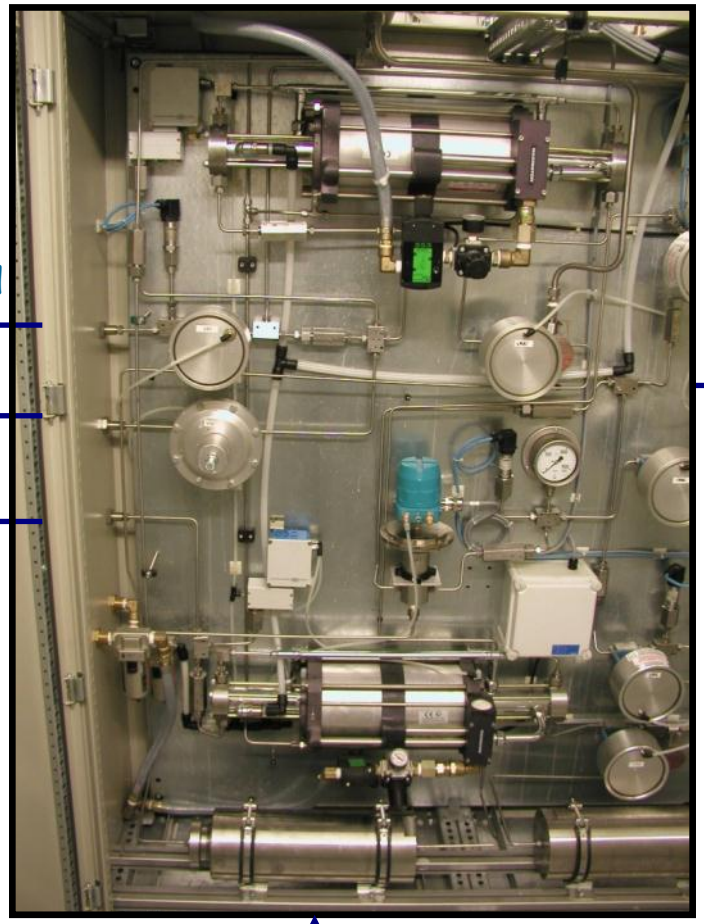
FUTURE IMPROVEMENTS

- Gas purity analysis
- Extensometer with strain gages : signal drift → LVDT ?
- Crack initiation detection from small defect (calibrated hole)

- Towards 100 MPa ?

DISK PRESSURE TESTS

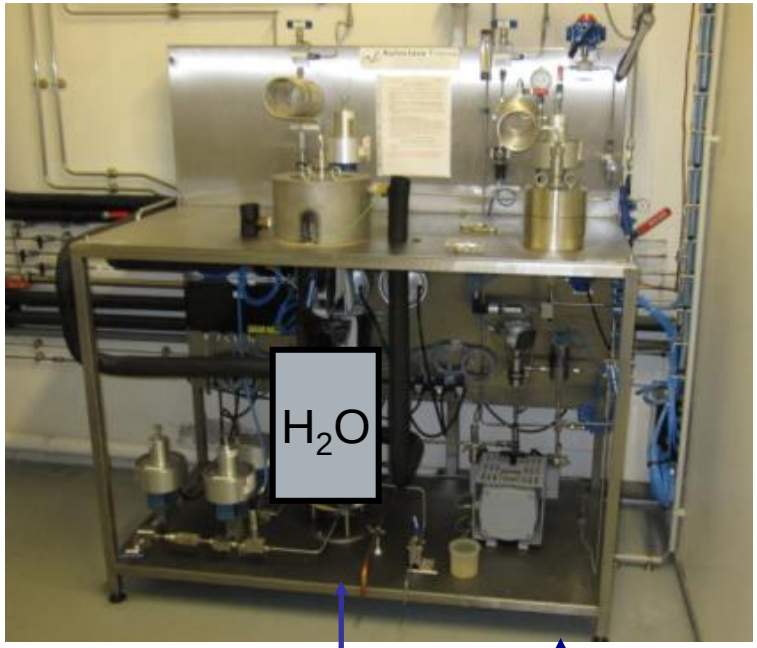
DISC PRESSURE TEST : SET-UP



Compressed air

He

H_2



Remote control

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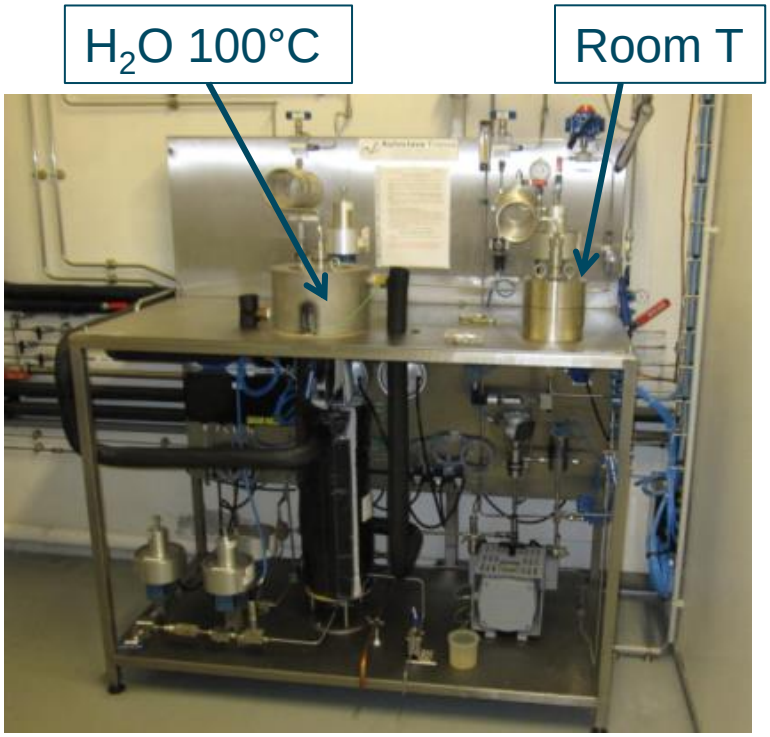
DISK PRESSURE TESTS : OVERVIEW

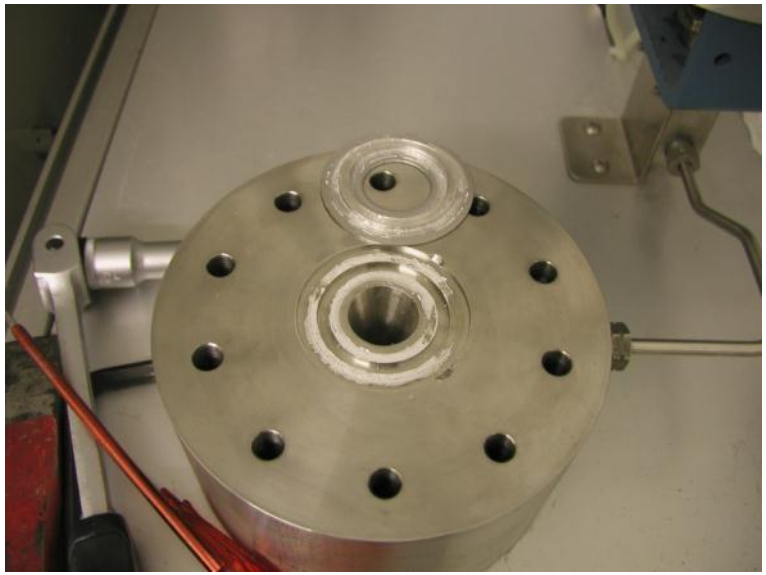
- Remote control
- H_2, He : Up to 100MPa, Tmax = 100°C
- 2 cells
- Pressure rate: up to 1000 bar/min
  above 0,1 bar/min
- Controlled pressure rate
- Working with impure gas
  (dedicated cell)
  O_2
  H_2O (bubbler up to 100°C)
  (from few ppm to 5000 ppm)
  Including long time steps



DISK PRESSURE TESTS

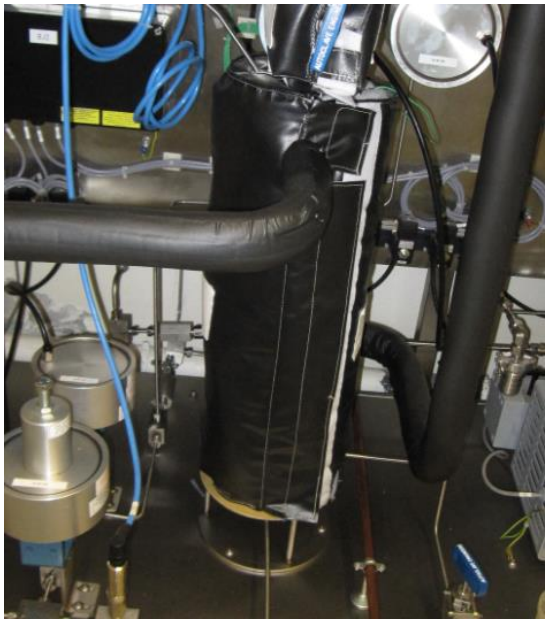
SEALS

- O-ring (elastomer) at high rates
- Indium at low rates



WATER CONTENT CONTROL

- Dedicated H_2 + H_2O gas bottle
- Using bubbler
  Measuring P and T
  Assuming saturated gas



TUBE PRESSURE TESTS

DEVICE ADAPTED TO TUBE PRESSURE TESTS

Long time range tests
under H_2 pressure (few months)

DISC PRESSURE TESTS

Advantages

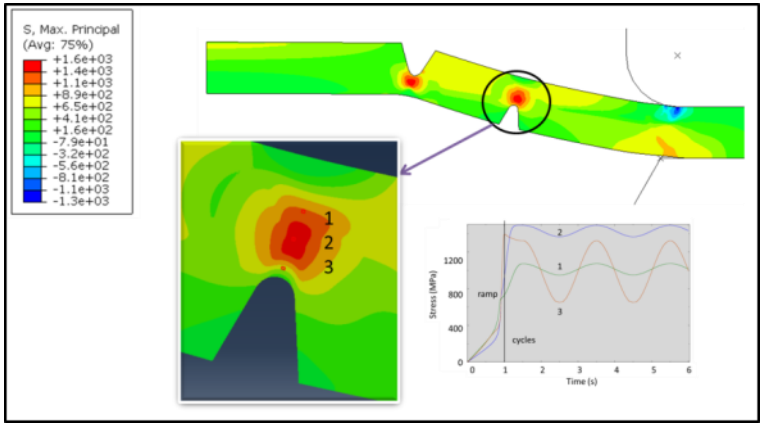
- Low volume
- Easier to proceed
- Dedicated cell for impure gas
- Fast gas switches (few seconds)
- Closer to service-life loadings

Disadvantages

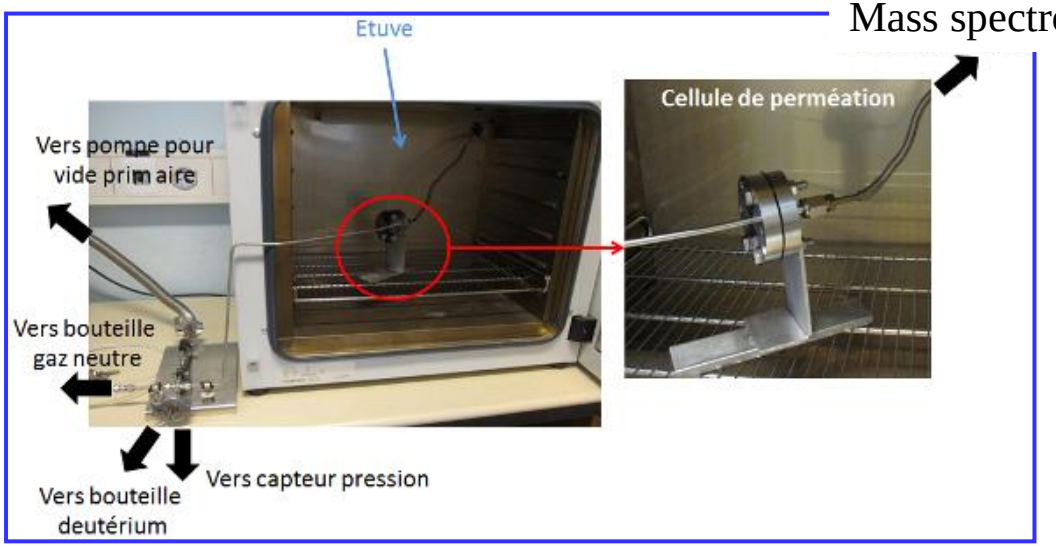
- Less instrumented
- More difficult to analyze mechanically (use of FEM)
- Boundary conditions
- Small thickness

Improvements

- Measuring in-situ deflection
- Developing tests under fatigue (cycling pressure) with / without defects

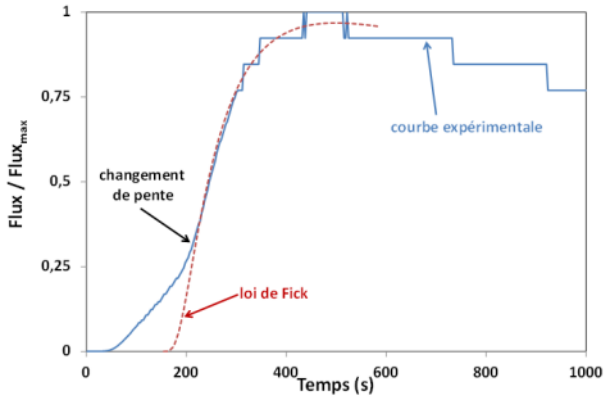


H_2/D_2 GAS PERMEATION DEVICE



20°C < T < 300°C
Pabs < 5 bar

- Diffusion coefficient
- Solubility
- Traps (binding energy)
- Oxide layer



Developments

Permeation under tensile loading
Influence of hydrostatic stress gradient

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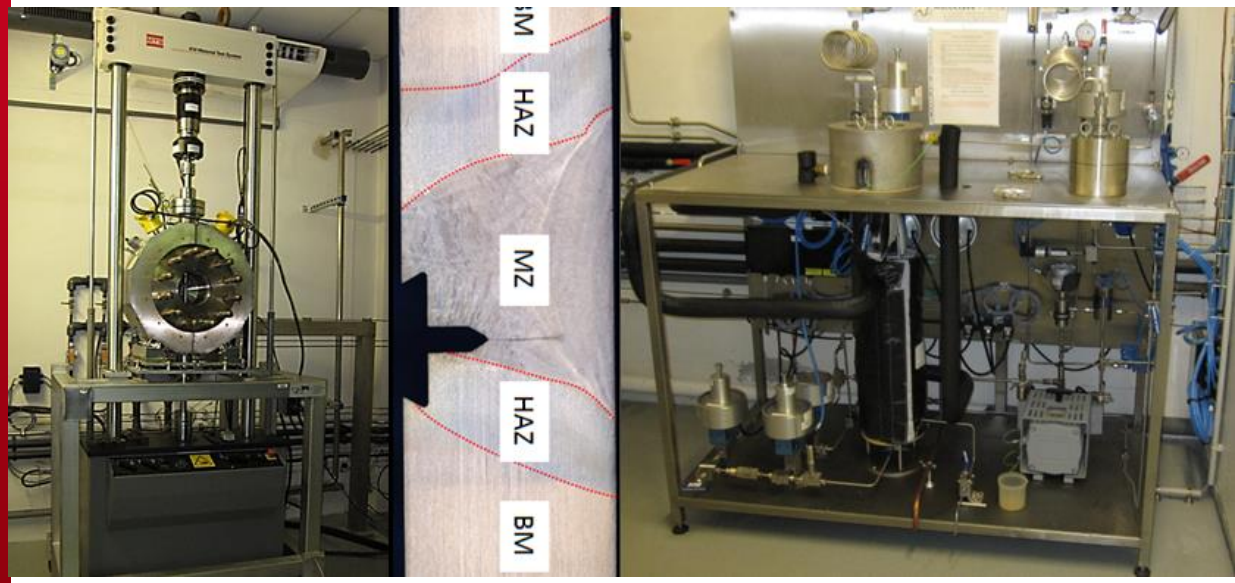
THANK YOU



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