HYCOMAT: test rig under high pressure of gaseous hydrogen at Pprime Institute, France

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Outline

- Presentation of Pprime Institute – Damage & Durability
- Design approach
- Safety
- Capabilities
- Gas management
- Test specimens
- Problems (solved or not)
- Test procedures:
  - tensile and compressive test, explosive decompression failure, dilatation (polymers)
  - Fatigue crack growth (metals)

Damage & Durability Group – Pprime Institute

- Pprime Institute: > 500 persons – 3 scientific departments (Fluid mechanics, Combustion, Energetics, Mechanical Engineering, Materials Science)

- Department of Physics & Mechanics of Materials, Damage & Durability Group
  - Academic staff: 30 persons (11 CNRS + 19 ENSMA/UP)
  - Staff: 18
  - PhD students: 45

- FUNDINGS:
  ~1 M€ per year without salaries of permanent staff
  - 50 % industrial contracts
  - 25 % european and national programs
  - 25 % institutional grants

- Relations between microstucture, mechanical behavior and durabilty

HYCOMAT design approach

- Flexibility: Suitable for mechanical testing on metals as well as on polymers, in H_2 as well as in CO_2
- Servo-hydraulic load frame
- Dedicated metrology
- Maximum pressure: 40 MPa



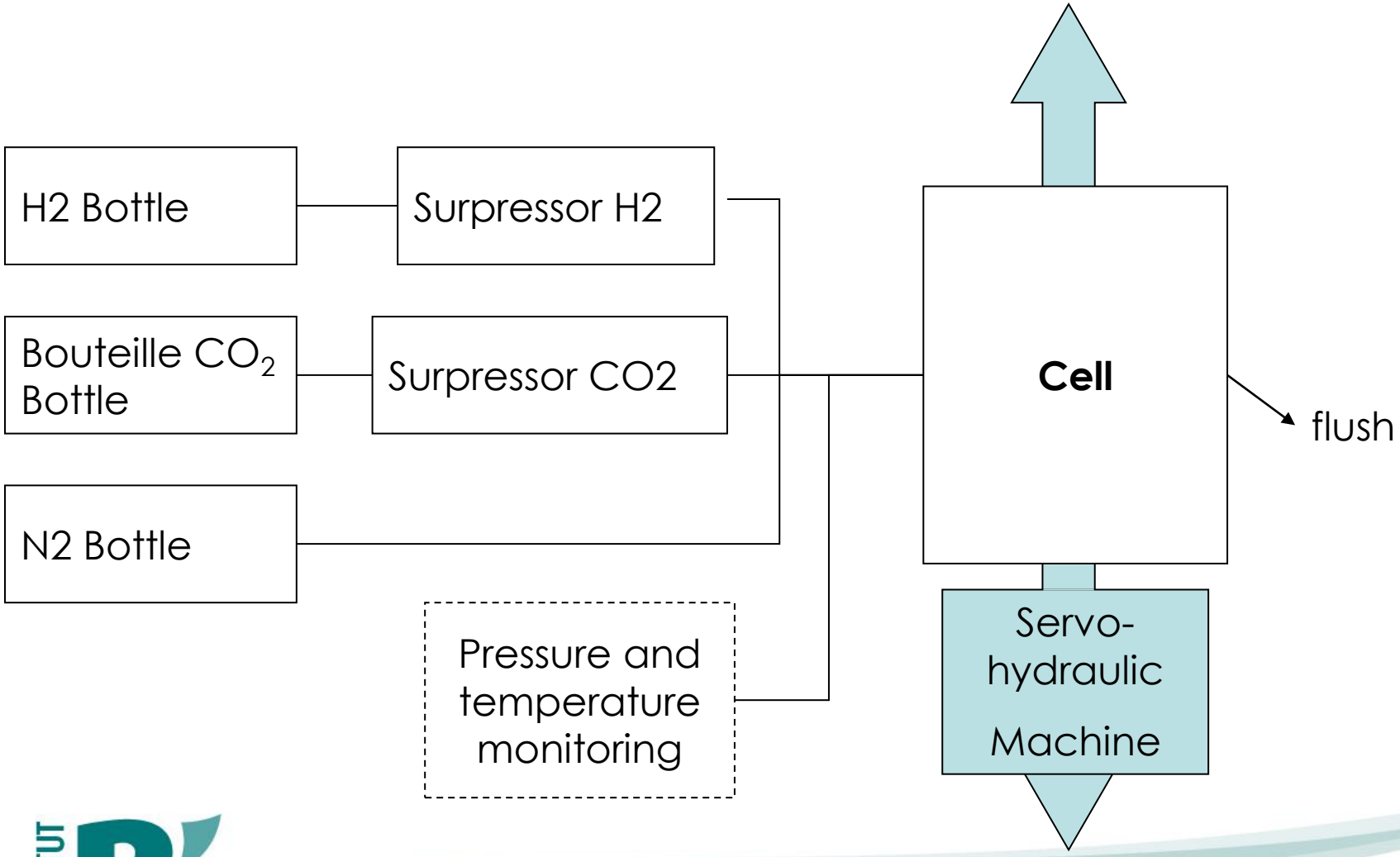
HYCOMAT design approach (*Cont'd*)

- Pressure vessel and gas supply : design by TOP INDUSTRIE®

- Specific cells for :
  - Instron Servo hydraulic test frame
  - Power unit
  - Outdoor Chiller
  - Remote control for gas and test
  - Outdoor gas storage and gas surpressors



Schematic overview



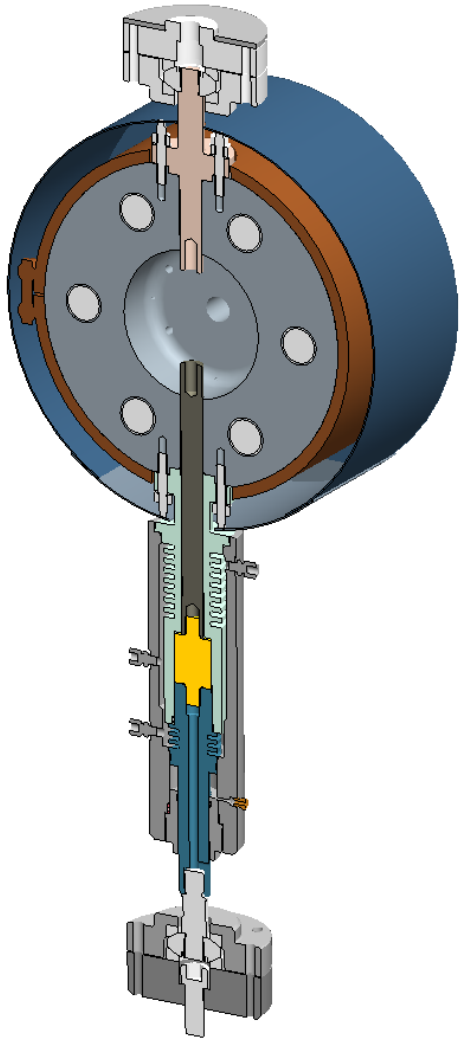
HYCOMAT : Safety

- Metallic structure for the test cell
- Separated command room / Concrete wall
- H_2 and CO_2 detectors with 2 levels of detection linked to :
  - A double speed exhaust fan
  - Interlocks (Vessel purge)
- Bursting discs
- Limited H_2 volumes
- Water cooling flow and temperature detectors



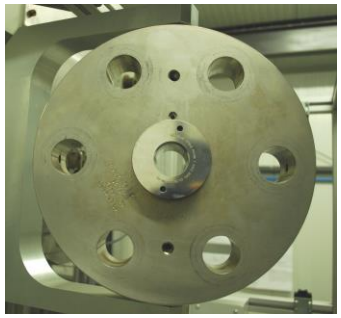
HYCOMAT capabilities

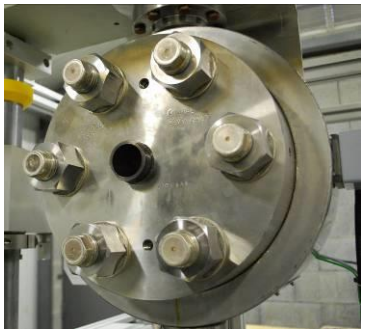
- Pressure : up to 400 Bars (40 MPa)
- Gas nature : H_2, CO_2, N_2
- Temperature : up to 150° C
- Load cells:
  - 25 kN (tension) pressure compensated internal cell Sixaxes®,
  - Lower rod pressure compensated, no piston effect due to internal pressure
  - 25 kN external cell Instron®
- Actuator displacement (LVDT) : 20 mm (7,9 in)



HYCOMAT capabilities (*Cont'd*)

- 2 front doors :
  - 40 mm (15,7in) diameter window up to 40 Bar
  - 26 mm (10,2 in) diameter window up to 400 Bar
  - 1 rear window 26mm up to 400 Bar (10,2 in)





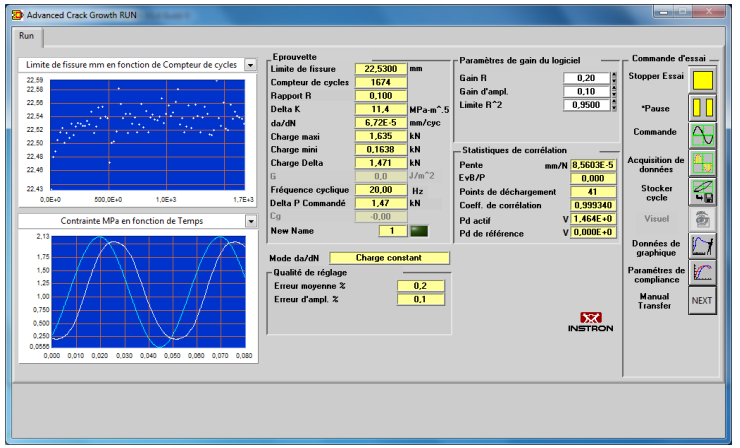


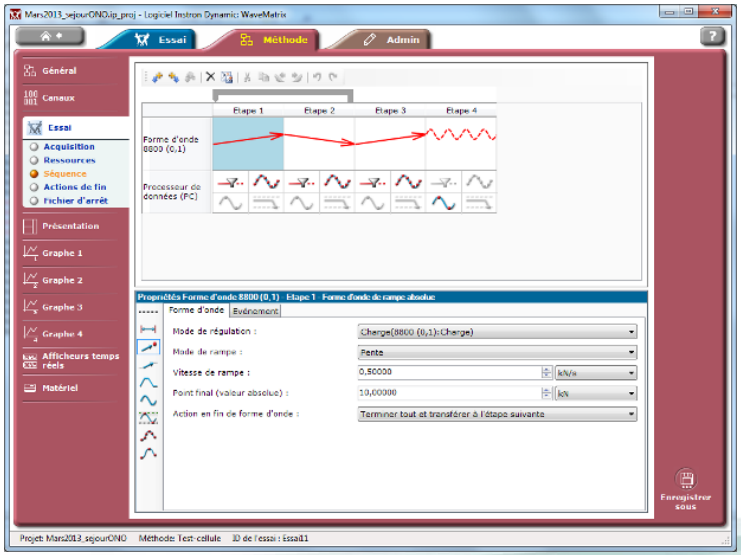
- Electrical connections for strain gages and DCP

- Gas temperature measurement (K-type TC)

HYCOMAT : capabilities (*continued*)

- Self alignment of the rods with ball joint
- Frequency : up to 20 Hz
- Test monitoring : Instron softwares :
  - Wavematrix ® (Multi purpose software)
  - Advanced Crack Growth ®





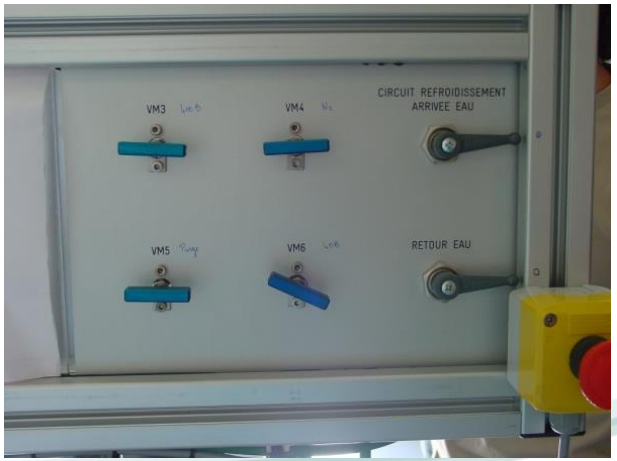
HYCOMAT : Gas Management

- Automatic regulation for temperature and pressure (PID control)
  - Max H_2 compression rate : 30 bar/minute
  - Max H_2 decompression rate : 50 bar/minute
- Manual purge of the vessel with inert gas (N_2)



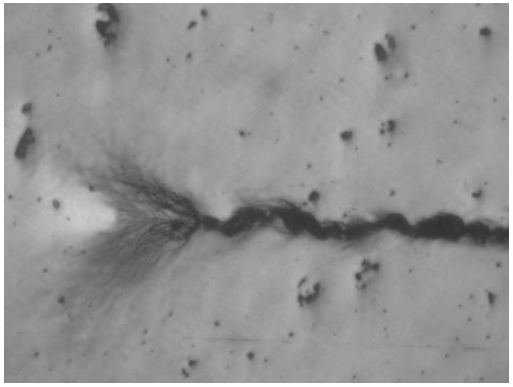






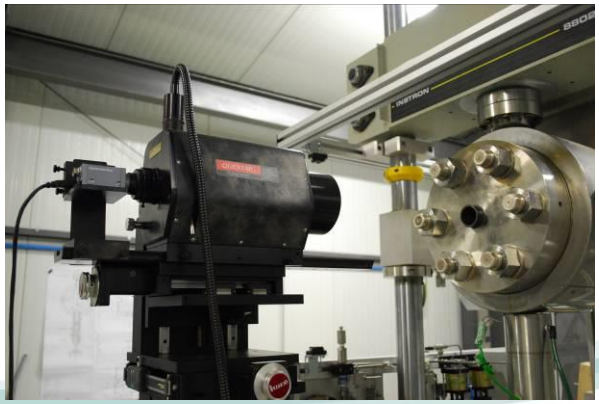
HYCOMAT: Test Specimens

- CT specimen (W= 40 mm, B=8mm)





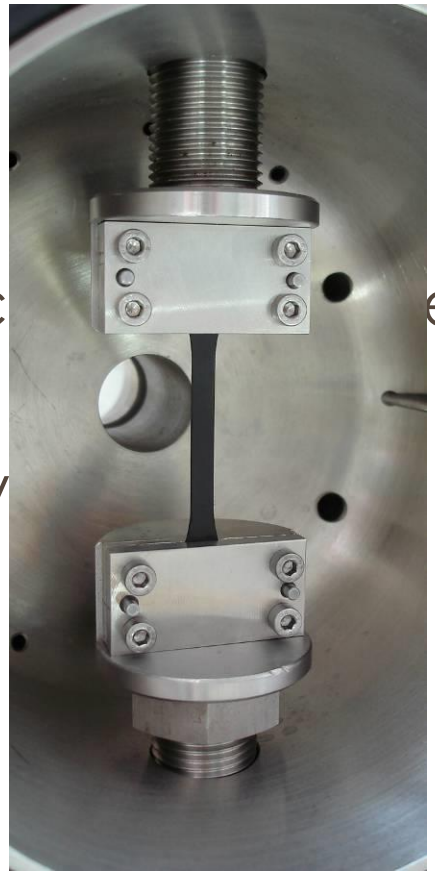
- Optical for observations and measurements - Questar®
- DCP crack measurement



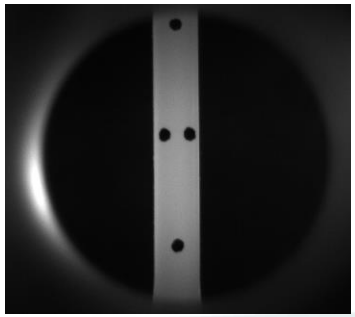
- Strain gage for Crack closure measurement

HYCOMAT: Test Specimens

- Cylindrical specimens
- Standard tension spec ers
- Strain measurement by





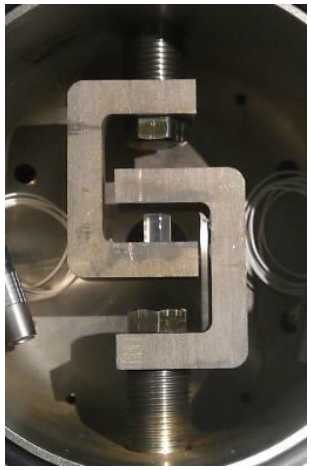


HYCOMAT: Problems (solved or not)

Hardware	Problems	Responses
Gas compressor	Valves & diaphragm failures	Frequent changes
Water jacket Seals	Explosive decompression of seals due to CO2 experiments	Anti extrusion ring
Rods	Rod galling	Anti extrusion ring
Internal load cell	Residual deformation of the compensation socket	New design

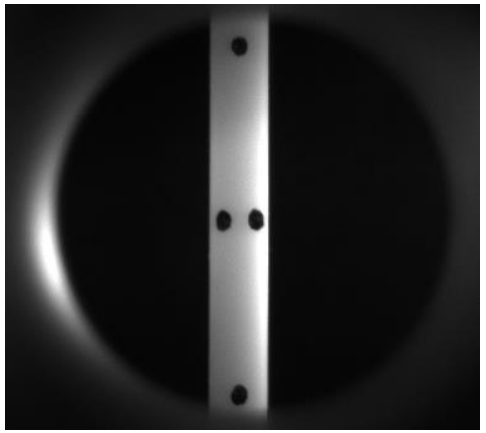
Test procedures: tension & compression test (polymers)

- Specimen design : flat dumbbell specimens (tension, cylindrical samples (compression)

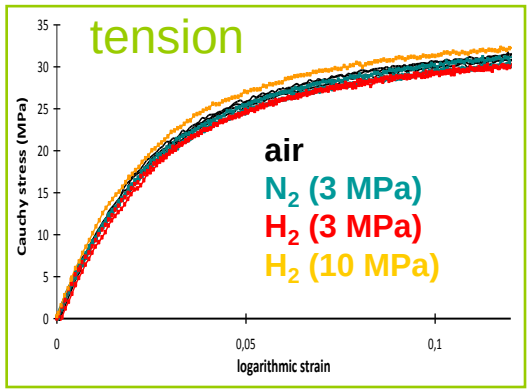


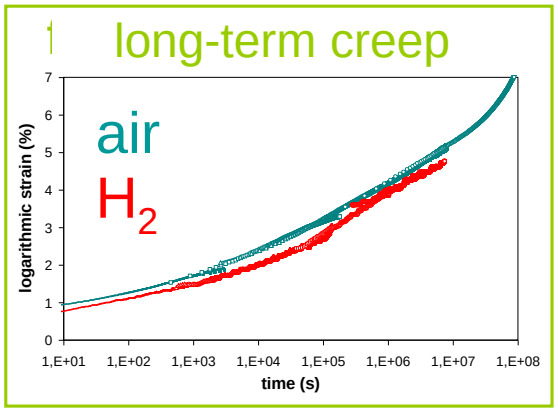
- Metrology : crosshead displacement / optical extensometer

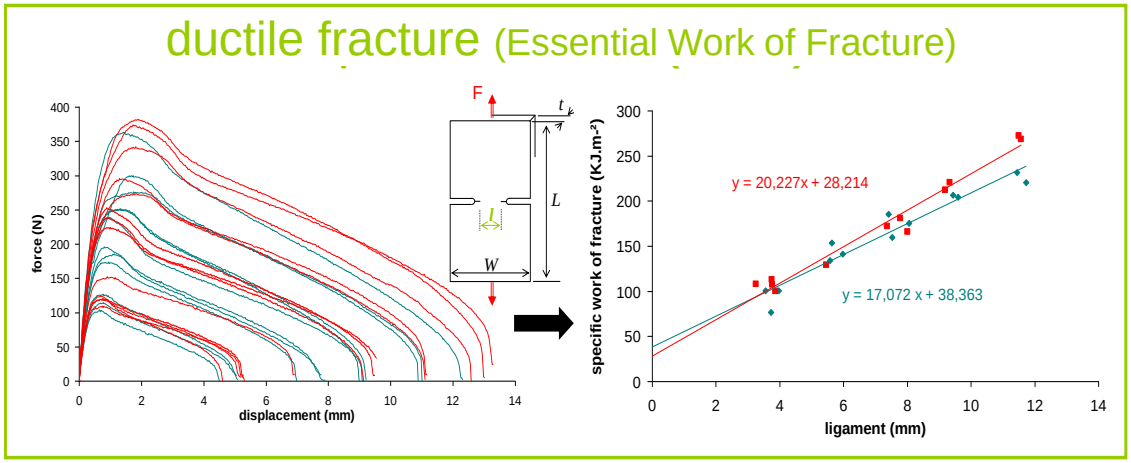
- Example of results : hydrogen influence on some key mechanical properties for gas high-density polyethylene for current gas networking (=> 3 MPa)



Test procedures: tension & compression test (polymers) *(Cont'd)*





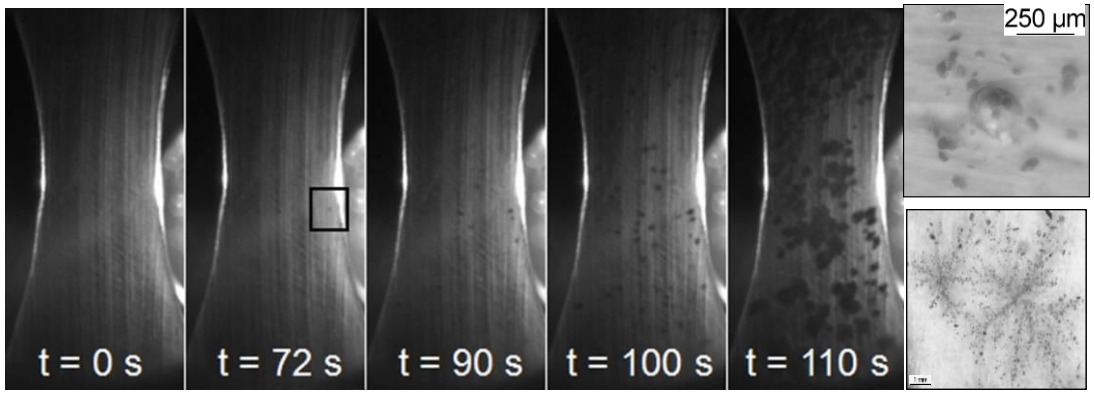


Limitations and challenges: *length / extension ratio to be compromised*

Test procedures: Explosive Decompression Failure

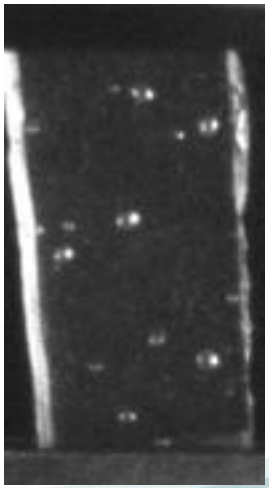
- Specimen design : flat and cylindrical samples (especially for superimposed tension or compression)
- Metrology : CCD camera => optical detection of cavitation onset (magnification of pictures)
  - ↳ current hydrostatic pressure value

Depressurization of a silicon rubber at 15 MPa/min after 1h-saturation at P_{sat} = 24 MPa



primary / satellite bubbles

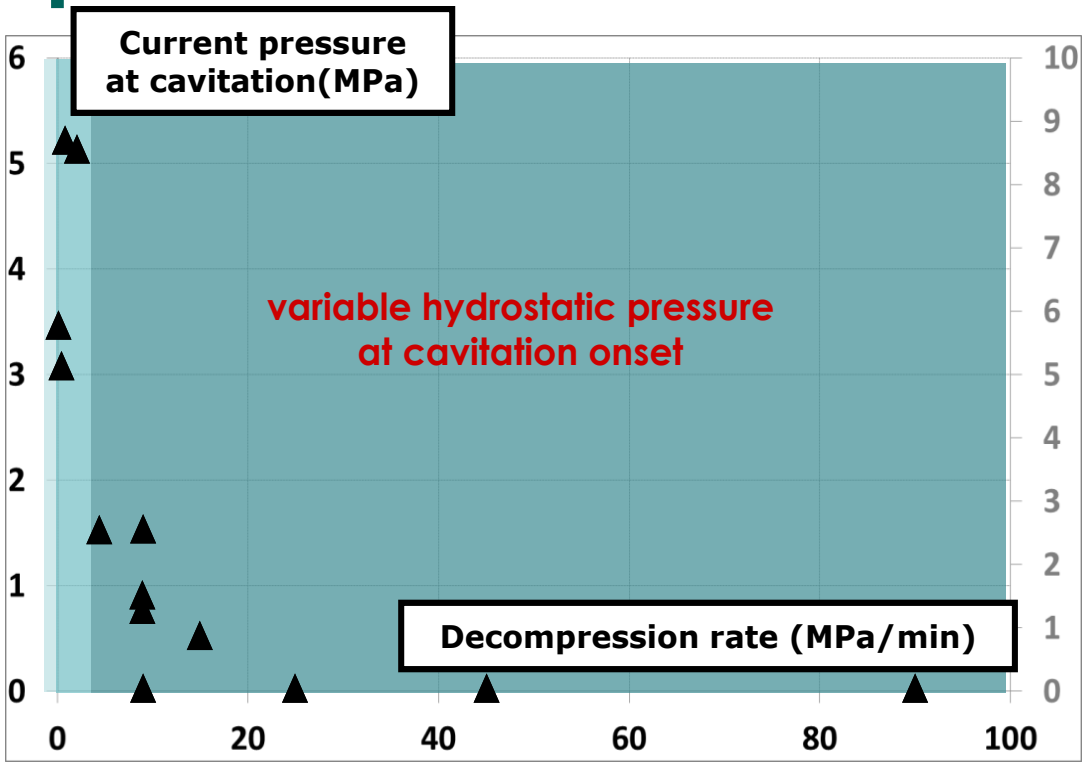
star-shape morphology



Depressurization of a polyethyl acrylate rubber at 10 MPa/min after 1h-saturation at P_{sat} = 12 MPa

Test procedures: Explosive Decompression Failure

- Example of Results



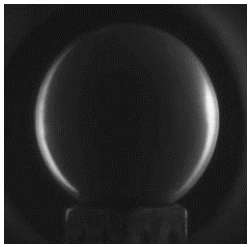
- Limitations and challenges: *experimental protocol limited to transparent rubbers, detection of early stages of cavitation (acoustic emission?)*

Test procedures: dilatation test (rubber)

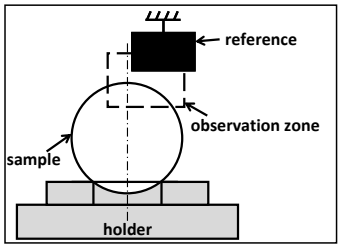
- Specimen design : cylindrical and spherical samples
- Metrology : *weak volume changes during saturation vs. large ones upon decompression*

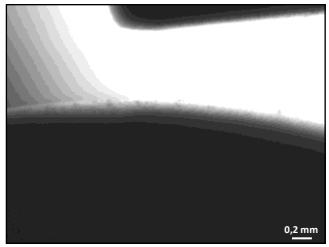
  two devices :

low resolution
(lens 12.5–75 mm f1.8)



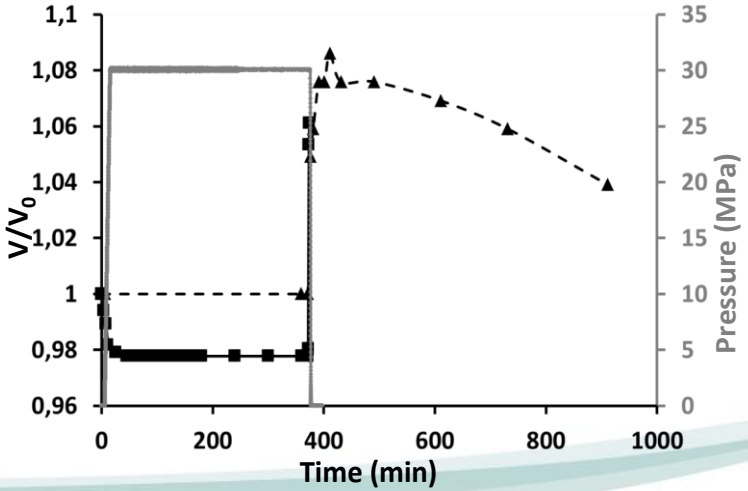
high resolution
(Questar)





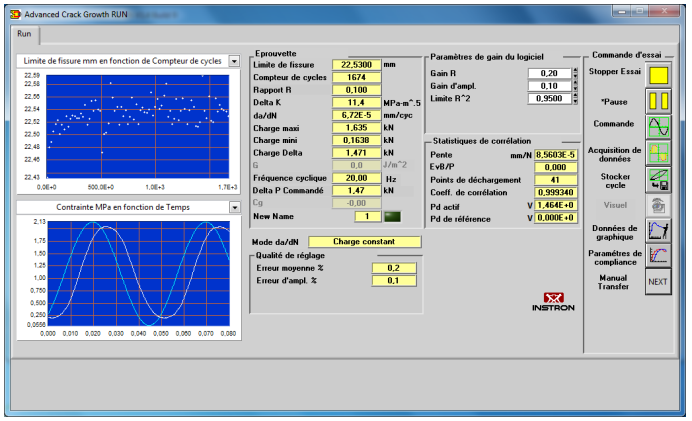
- Example of results : silica filled NBR rubber

Limitations and challenges : *large temperature transients => calculation needed to extract the volume change linked with gas diffusion*



Test procedures: fatigue crack growth in metals

- Specimen design: Compact Tension W=40mm B=8mm
- Advanced Crack Growth Instron® software
- Crack length monitoring: potential drop (DC) technique (Current intensity 1A)
- Crack growth calculation: ASTM E647
- Possibility to monitor the crack growth by means of a long-distance microscope

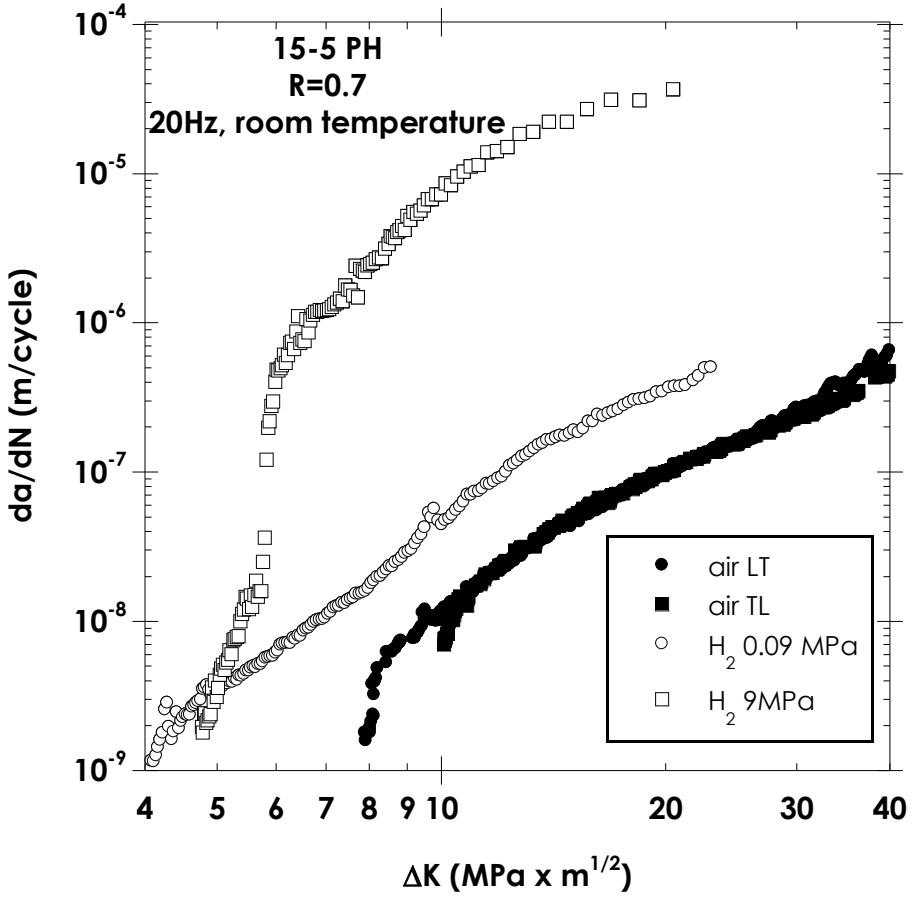






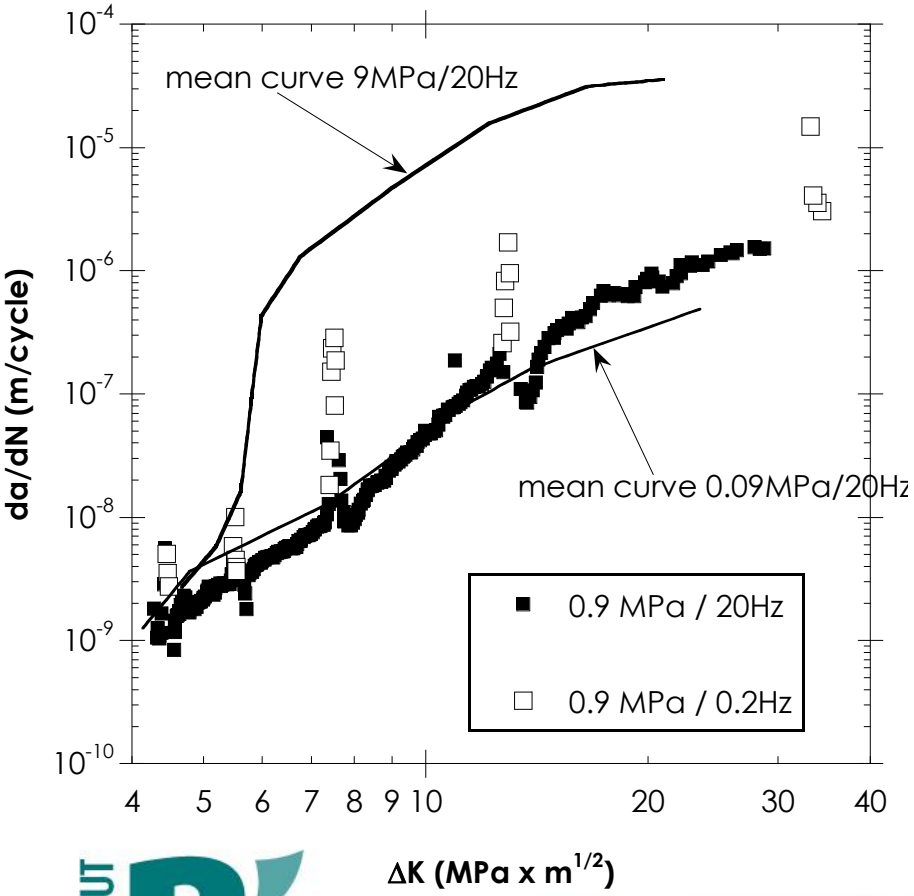
Fatigue crack propagation: Results

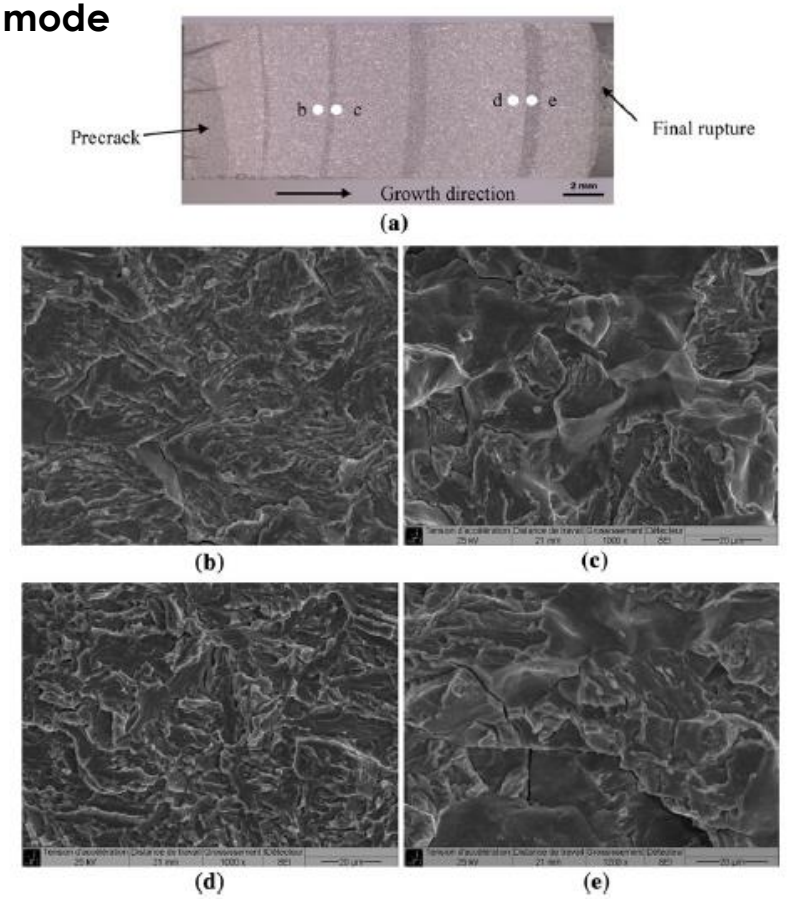
Influence of pressure in a precipitation-hardened martensitic stainless steeel
→Sharp increase in da/dN at 9 MPa H_2



Fatigue crack propagation: Results

Influence of frequency at low pressure → da/dN enhancement associated with a change in fracture mode





Acknowledgements

- Fundings: ANR, FEDER…

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