Development of Evaluation Technology for Integrity and Hydrogen Embrittlement Characteristics of Metallic Materials

KRISS Korea Research Institute of Standards and Science

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S.H.NAHM & U.B.Baek

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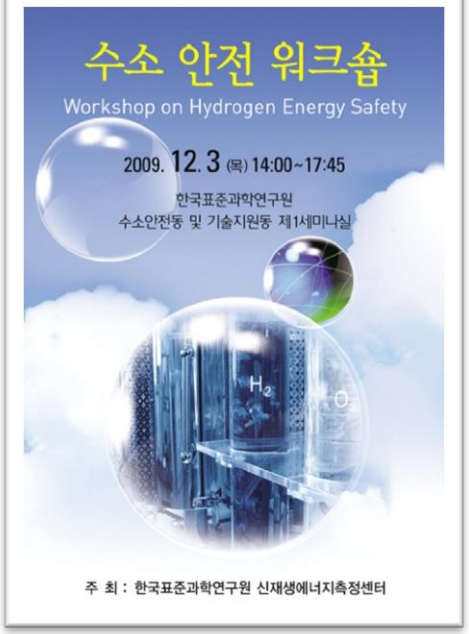
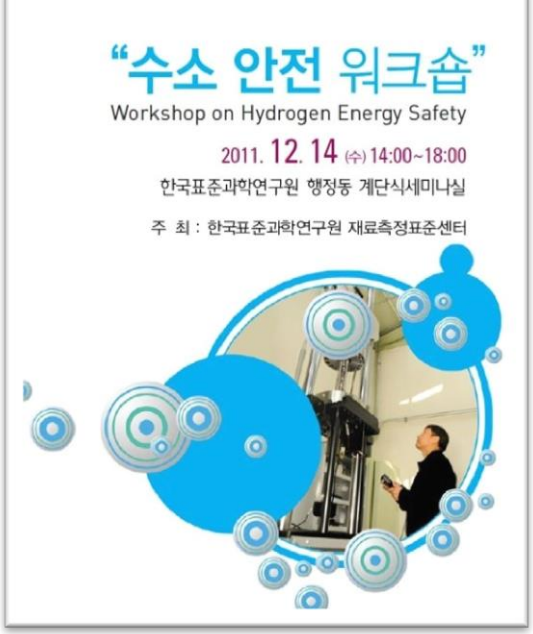
1 Status of KRISS's Hydrogen Safety R&D

2 KRISS System

Towards Global Center of Excellence in Metrology

Activities in Hydrogen Safety





- H_2 Safety Workshop: Information exchange & human network
  - The date of exhibition: 2008/12/12, 2009/12/3, 2011/12/14
- H_2 Safety Research Laboratory Open
  - Opened Date: 2009/12/3

Research Target & Contents

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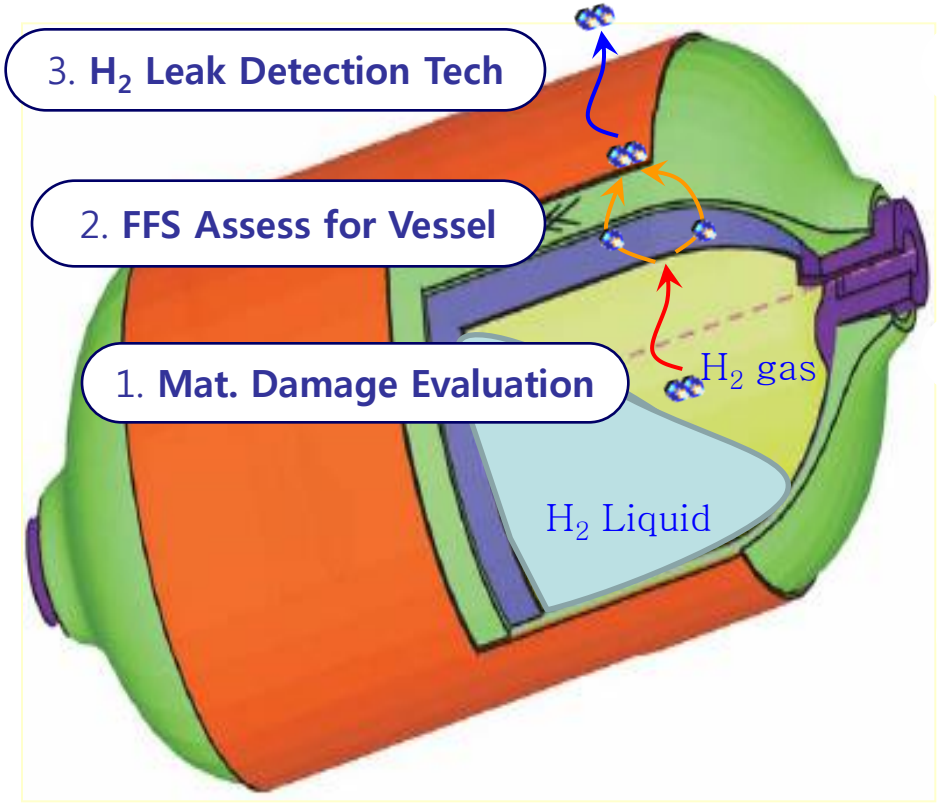
Development of integrity evaluation technology of hydrogen vessel for safe storage and transportation of hydrogen

What ?



❖ **Improvement of Storage Efficiency of H_2 & Guarantee of Storage Vessel Safety**

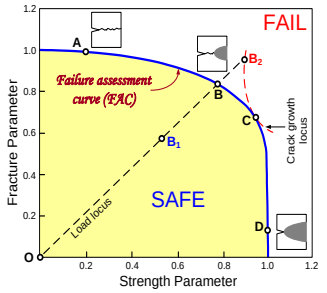
How ?

1. Mat. Damage Evaluation

- LH_2 System Design/Manuf
- LHe/LH_2 Mat. Properties
- cH_2 Mat. Properties

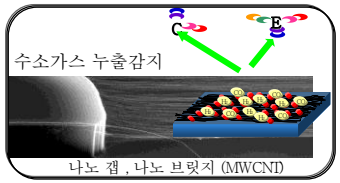
2. FFS Evaluation for Vessel

- Nondestructive H2 damage
- Defect Evaluation
- Vessel Integrity Assess



3. Leak Detection Tech

- Sensor based on CNT
- Real time Dual Monitoring System



Materials Damage Evaluation

Embrittlement msmt in Gaseous H_2

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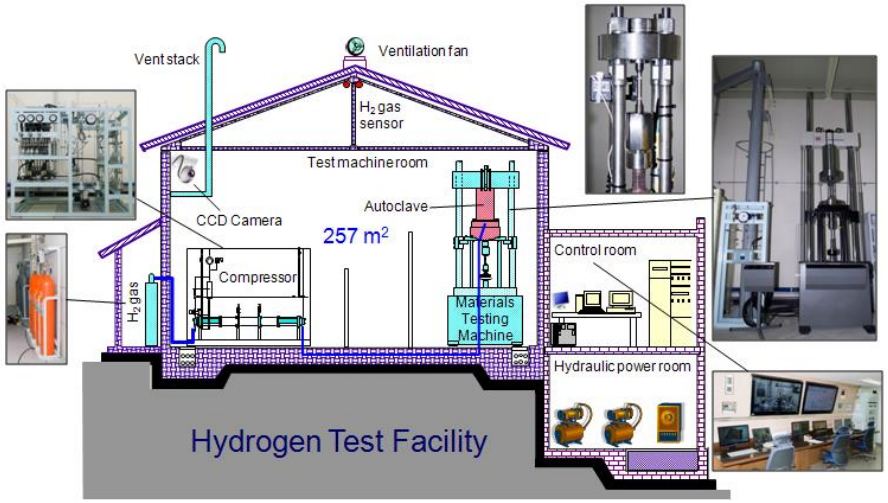
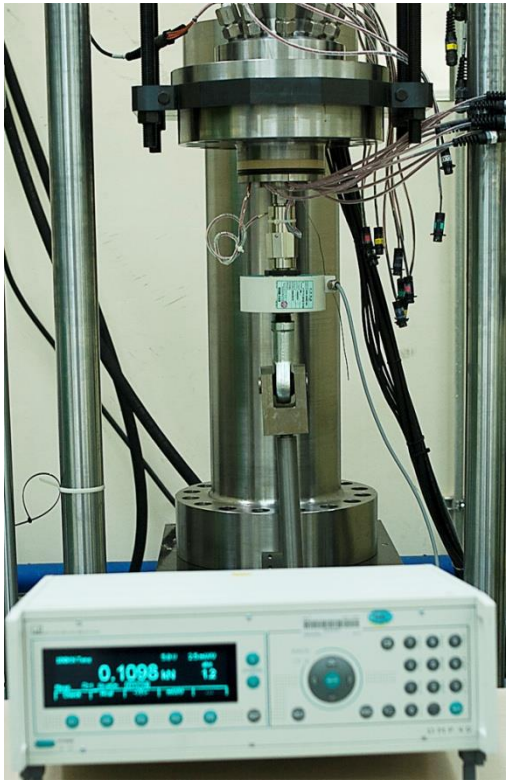
Hydrogen(cH_2) Test Facility at KRISS



Specification

- Autoclave : 80 mm ID × 400 mm
- Design Pressure : max. 120 MPa
- Temperature : Up to 80 ℃
- Force : 100 kN
- Tension, fracture toughness, LCF, FCGR, etc.

Fatigue Crack Growth Testing System Set-Up

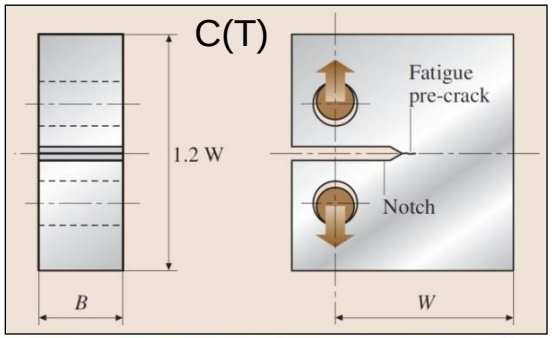


Tension (105 MPa cH_2)

Smooth tensile specimen

Notched tensile specimen

Fracture Toughness (105 MPa cH_2)



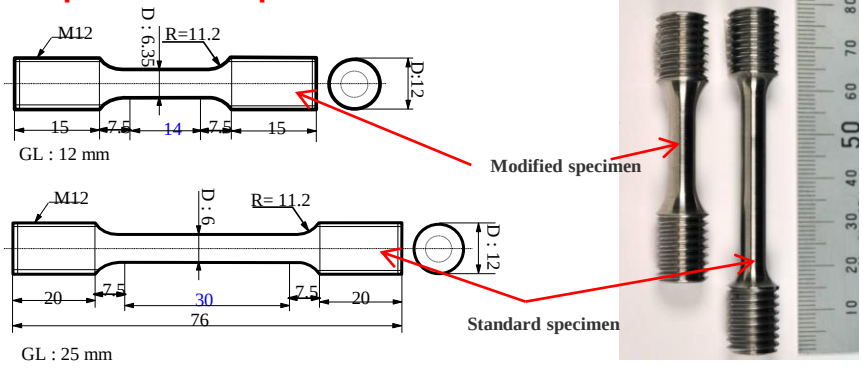
Mechanical Properties in Gaseous H_2

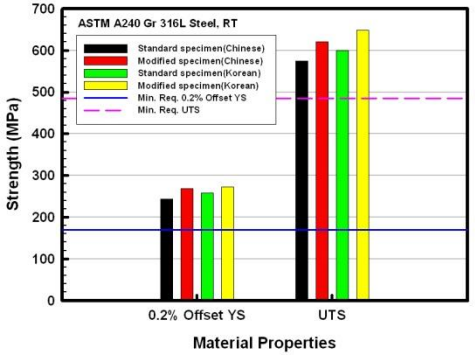
► Tensile Testing Factor in High Pressure H_2 Gas

Materials (ASTM A 240 Gr 316L)

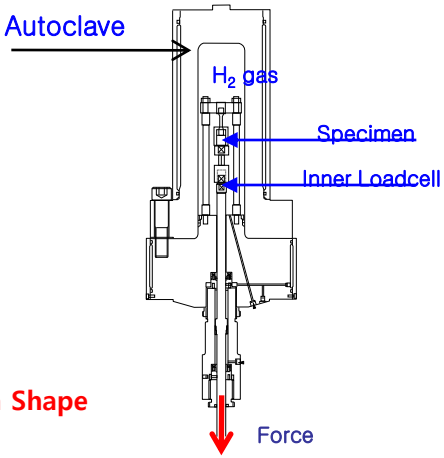
	Na	Al	Si	P	Cl	V	Cr	Mn	Fe	Co	Ni	Cu	Mo
ASTM A240Gr 316L	0.374	0.0434	0.541	0.0262	0.0658	0.0702	15.8	0.893	69.5	0.255	10.1	0.255	2.08

- **Specimen Shape**

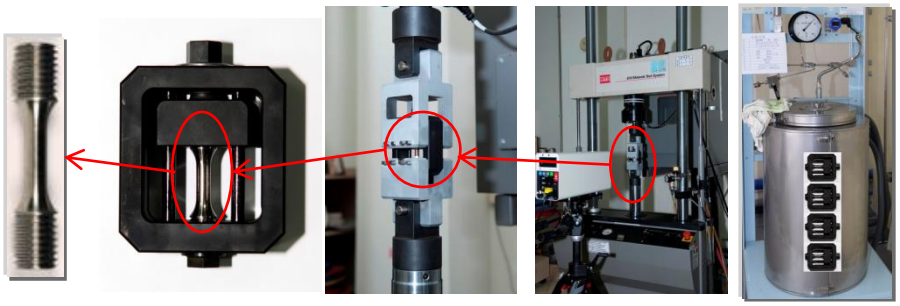




Tensile Properties depending on Specimen Shape



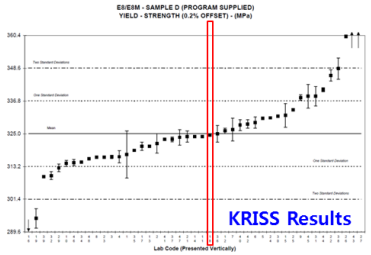
- **Experimental Equipment for Exposure time & Pre-strained specimen**



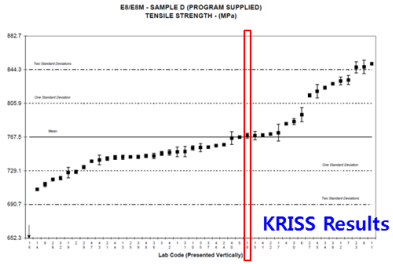
Specimen **Pre-Strained Jig** **Pre-Strain (0.1 % & 4 %)** **Long Time Exposure (1000, 2000 h)**

International Comparison on M.P.

ASTM : 48 participants



Proficiency test results for 0.2% offset **yield strength**.



Proficiency test results for **tensile strength**.

LH_2 (20 K) Testing System

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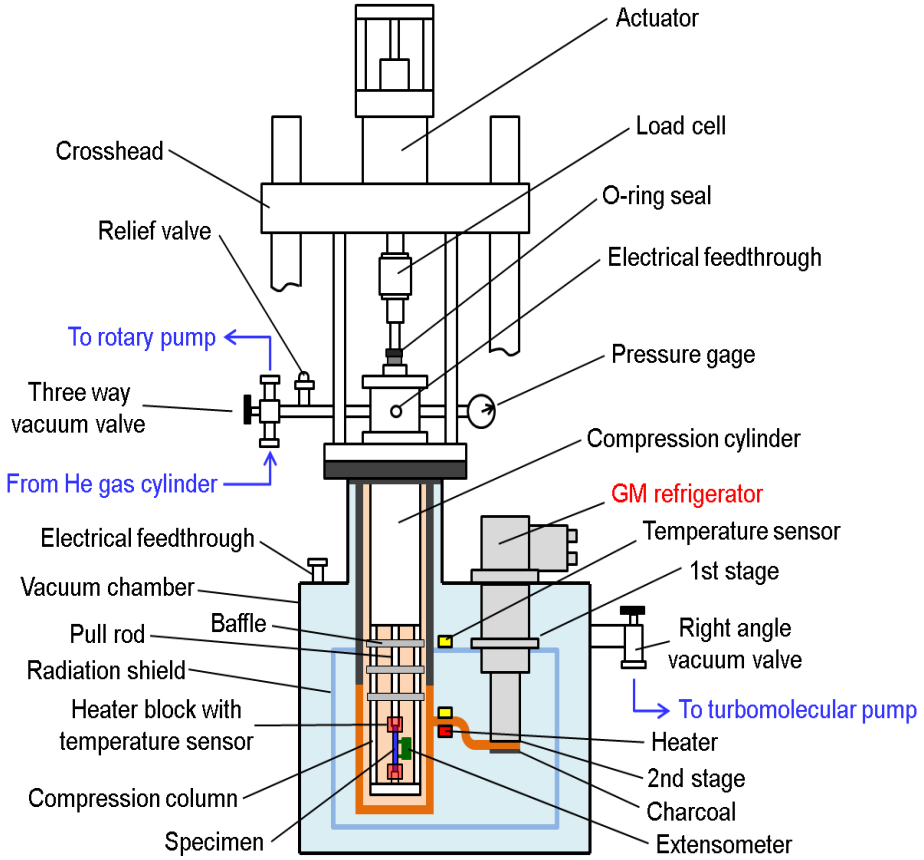
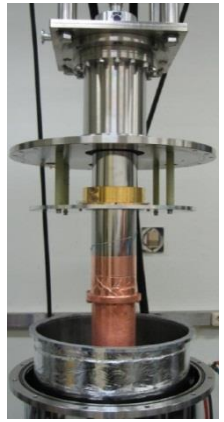
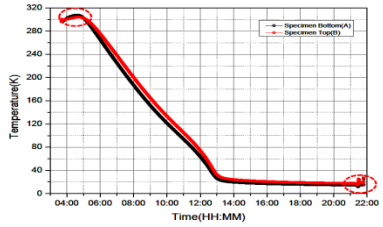
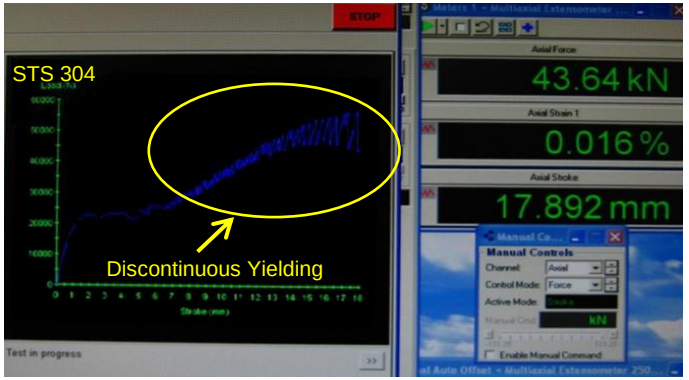
Mechanical testing cryostat with a 4 K GM refrigerator









4 K GM Refrigerator
- Manufacturer : SHI (住友重機械工業)
- Model : 415D
- 1.5 W @ 4.2 K / 45 W @ 50 K

Small Punch Test

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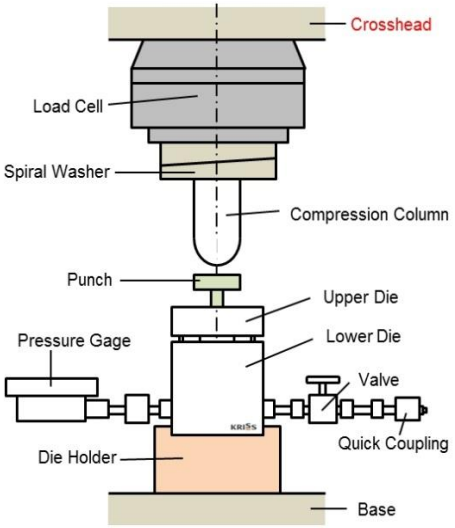
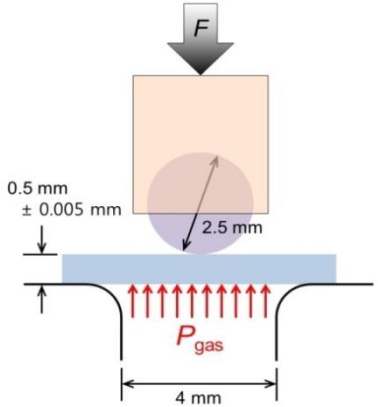
▪ SP Test in High Pressure H_2 Gas





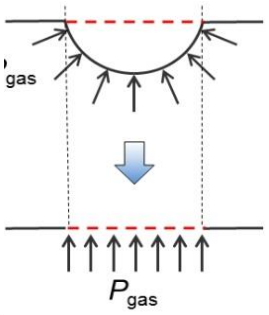
P_{gas} = 10 MPa

P_{air} = 0.1 MPa

Area = $\pi(4/2)^2$ mm^2 = 12.6 mm^2

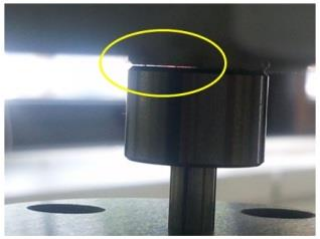
F_{gas} = 124 N

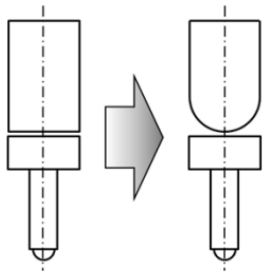
$F_{corrected} = F_{measured} - F_{gas}$



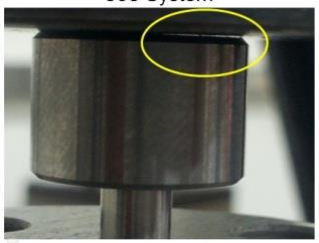
▪ SP System Improvement (Ex)

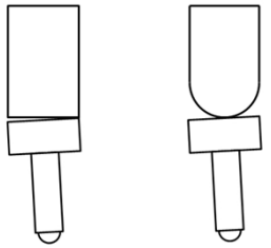
Sintech 5/G

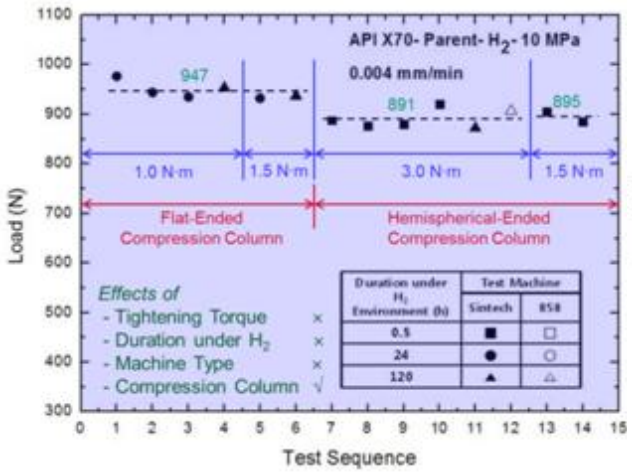


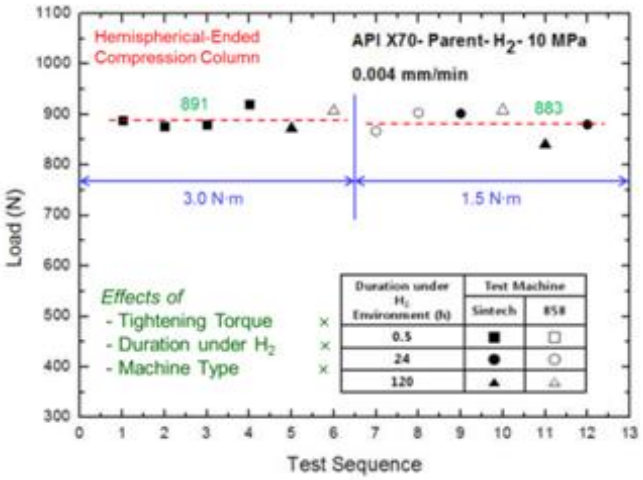


858 System









(Inside Number means after pressure correction)

Peak Force (N)	0.1 MPa Air	10 MPa Hydrogen	$F_{Hydrogen}$ / F_{Air}
X70	1,529	887 (763)	58 % (50 %)
X65	1,438	794 (670)	55 % (47 %)
X42	1,394	712 (588)	51 % (42 %)

→ **Validity Proof of SP Test as Simple Test for Hydrogen Compatibility Evaluation of Materials**

FFS Evaluation for Vessels

Damage Evaluation by Ultrasonic

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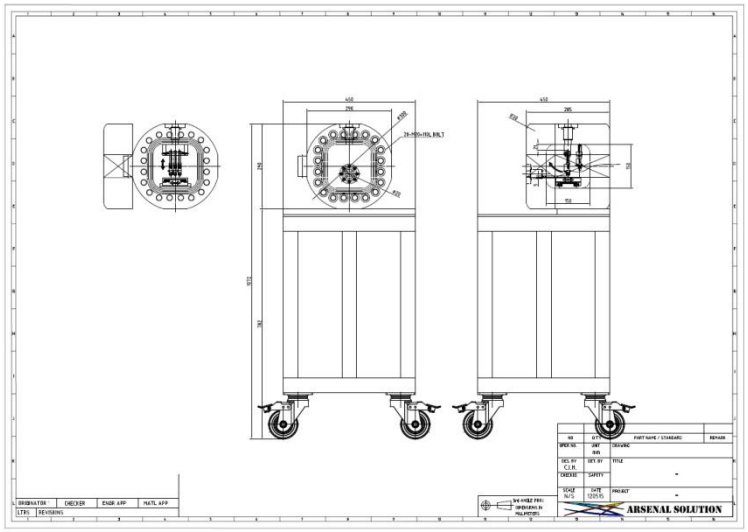
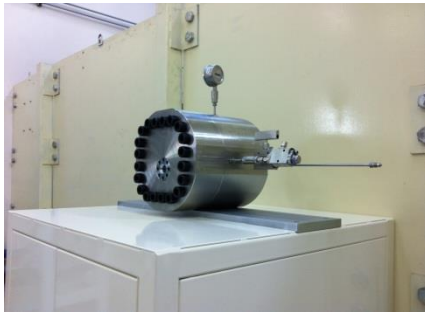
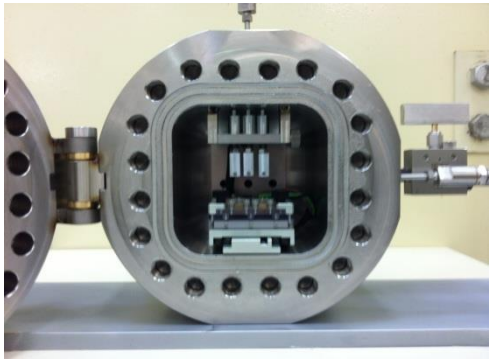
► Equipment for Measurement of Material Properties in Gaseous Hydrogen Using Ultrasonic Wave



Design Drawing for Material Properties Measurement





Nondestructive Testing Equipment

※ Main Components

Housing, Base, Sensor Insert, Ultrasonic Sensor, Gas Supply Valve, Automatic Gas Exhaust Valve, Pressure Gauge, Pressure Sensor, Ultrasonic Measurement System etc.

Fitness for Service Assessment

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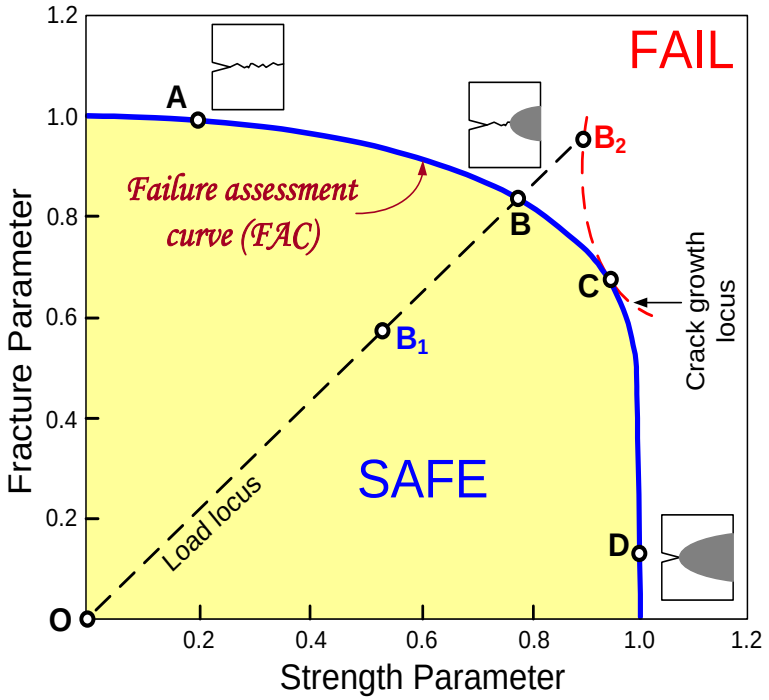
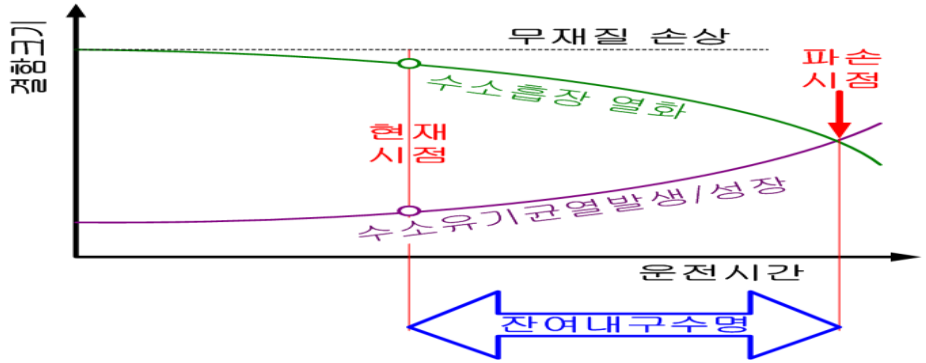
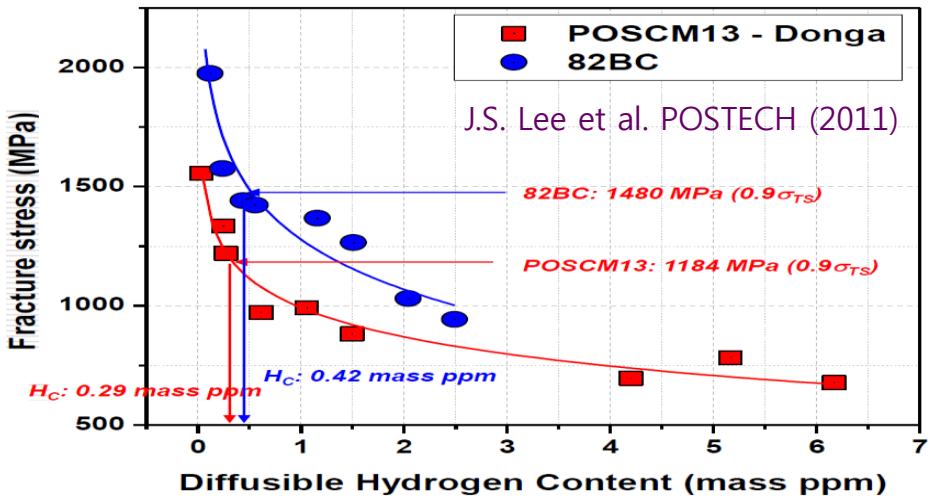
Development of FFS for Vessel



- Development of FFS Evaluation diagram for H2 Vessel Using Strength/Fracture Parameter
- Application of FFS Diagram for Evaluating the integrity of Vessel Damaged by Hydrogen

Development of Durability Life Evaluation





- Residual Life Assessment Based on Failure Modeling & Physical/Chemical Limit Life
- Establishment of Failure Criterion & Damage Change Route

Reliability msmt for Infrastructure

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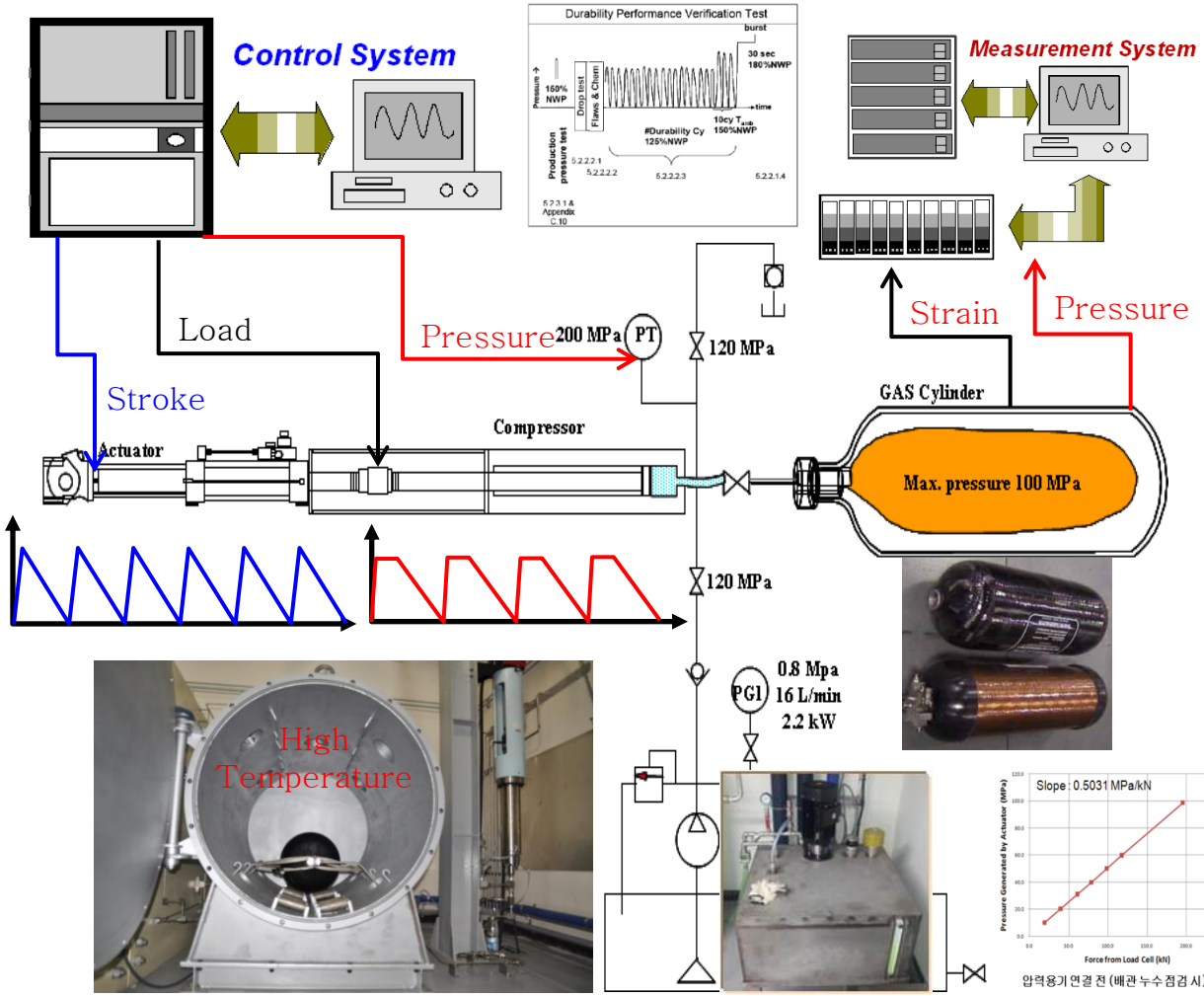
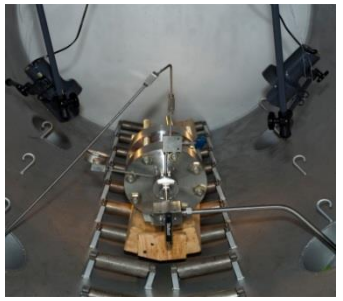
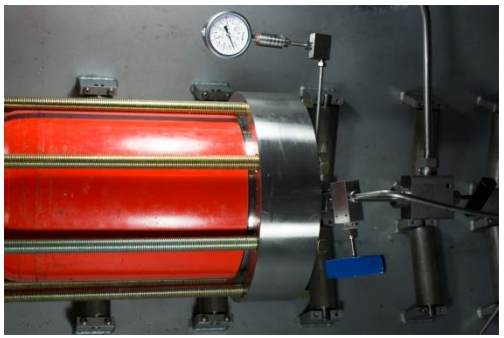
High capacity high pressure hydrogen gas infrastructure durability measurement
LPG, CNG, cH_2 gas tank, valves infrastructure durability measurement
1000 L Vessel reliability measurement (70 MPa, 0.5 Hz)

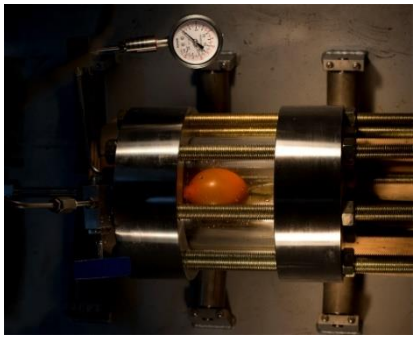




Mounting Vessel in Autoclave



Fatigue Durability Test with cH_2 Cylinder



Pre-Test for Developing Elastic Body

Hydrogen Leak Detection

Hydrogen Sensing Properies

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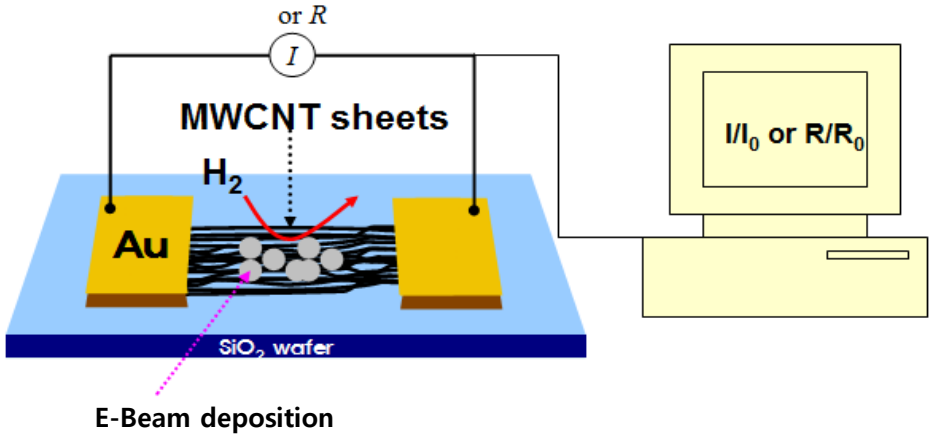
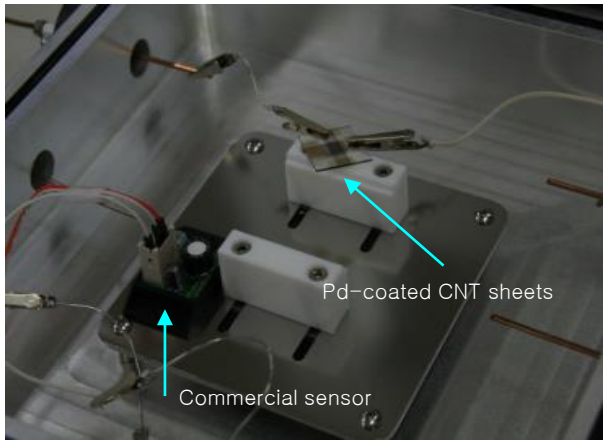
▪ **Hydrogen sensing**

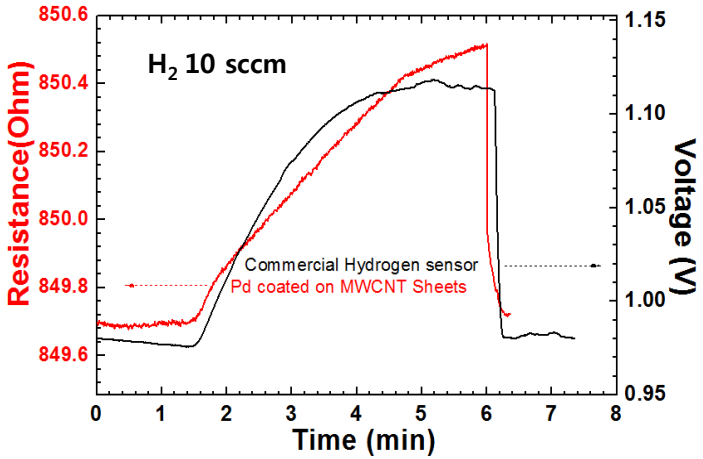


- 2 point prove measurement
- The electrical resistance of sheet was measured during hydrogen adoption/desorption process
- Nitrogen was introduced in the chamber at the desorption process.

▪ **Commercial sensor vs Pd coated CNT sheets**

-The response time of MWCNT sheets sensor is comparable with that of the commercial sensor





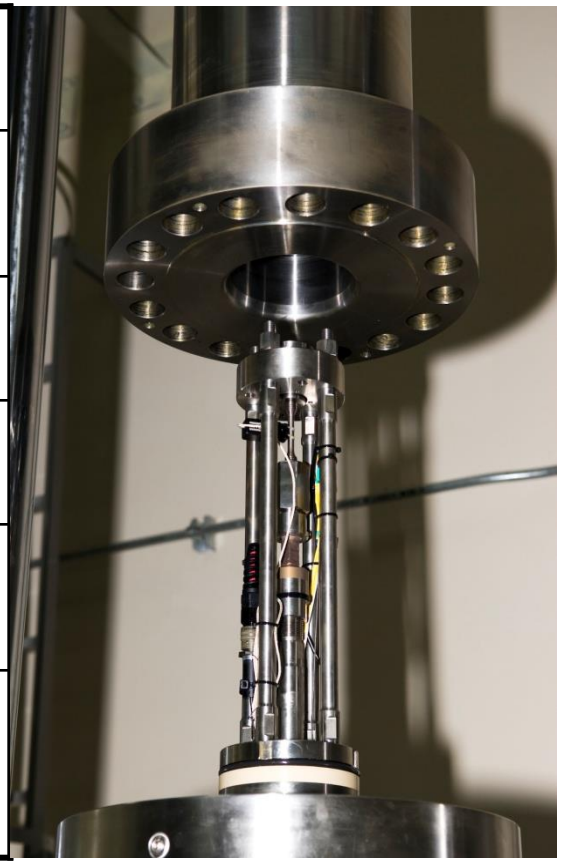
KRISS System

KRISS Design Approach

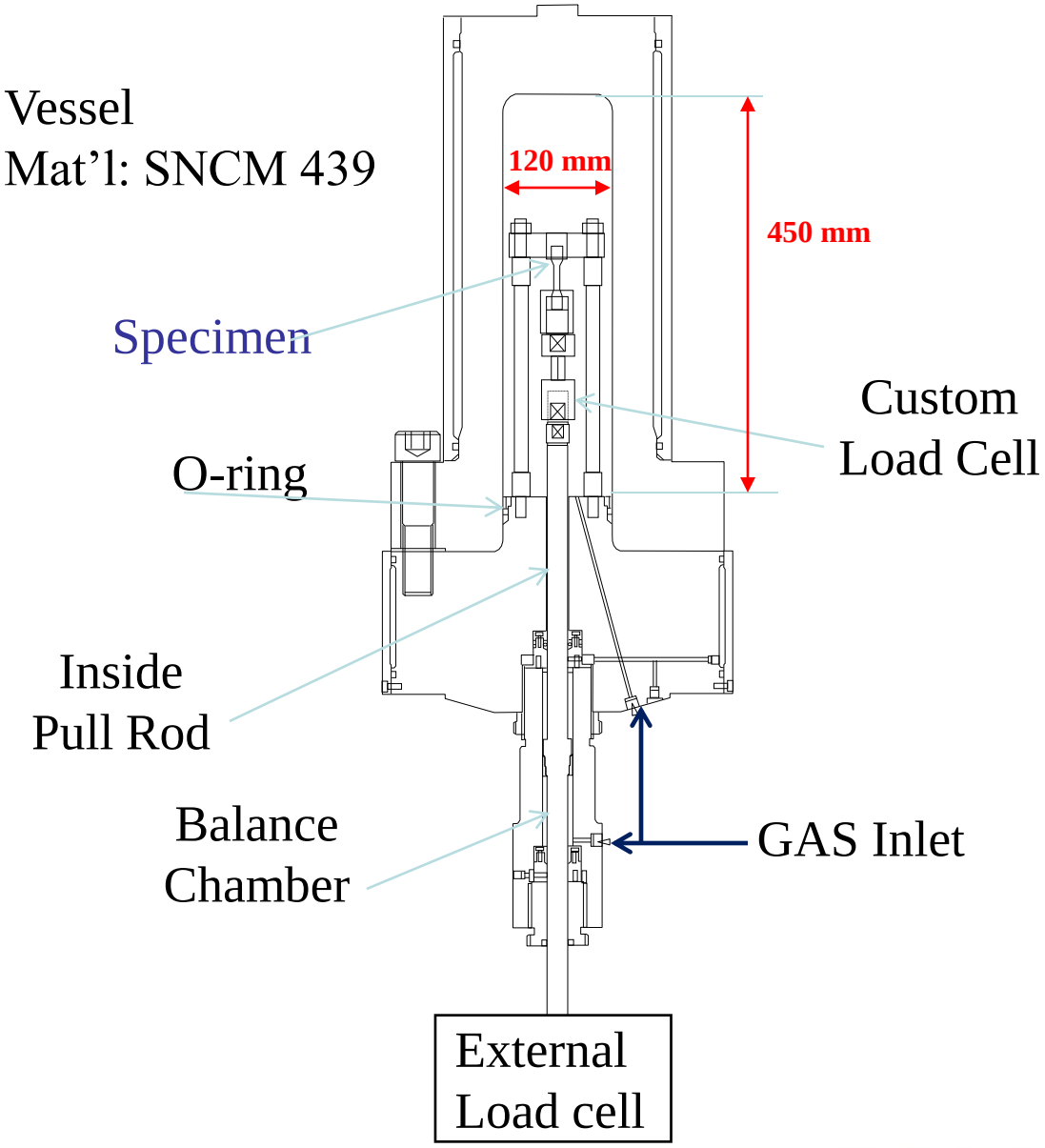
- Custom vessel
- Servo-hydraulic test frame
- Dedicated support equipment
  - hydraulic power unit
  - chiller
  - Located in a test cell

Test Capabilities

	Tensile/fracture	Fatigue
Max. Design Pressure	1200 bar (120 MPa)	1200 bar (120 MPa)
Temperature	80 deg C	80 deg C
Inner Force	50 kN	50 kN
Displacement	25 mm	Tension/Tension Tension/Compression
Test control	0.025-25 mm/s : tensile 1 minute–1 day : Fracture	0.001-10 Hz



Cross-Section of Test Vessel



Fatigue Crack Growth Test System

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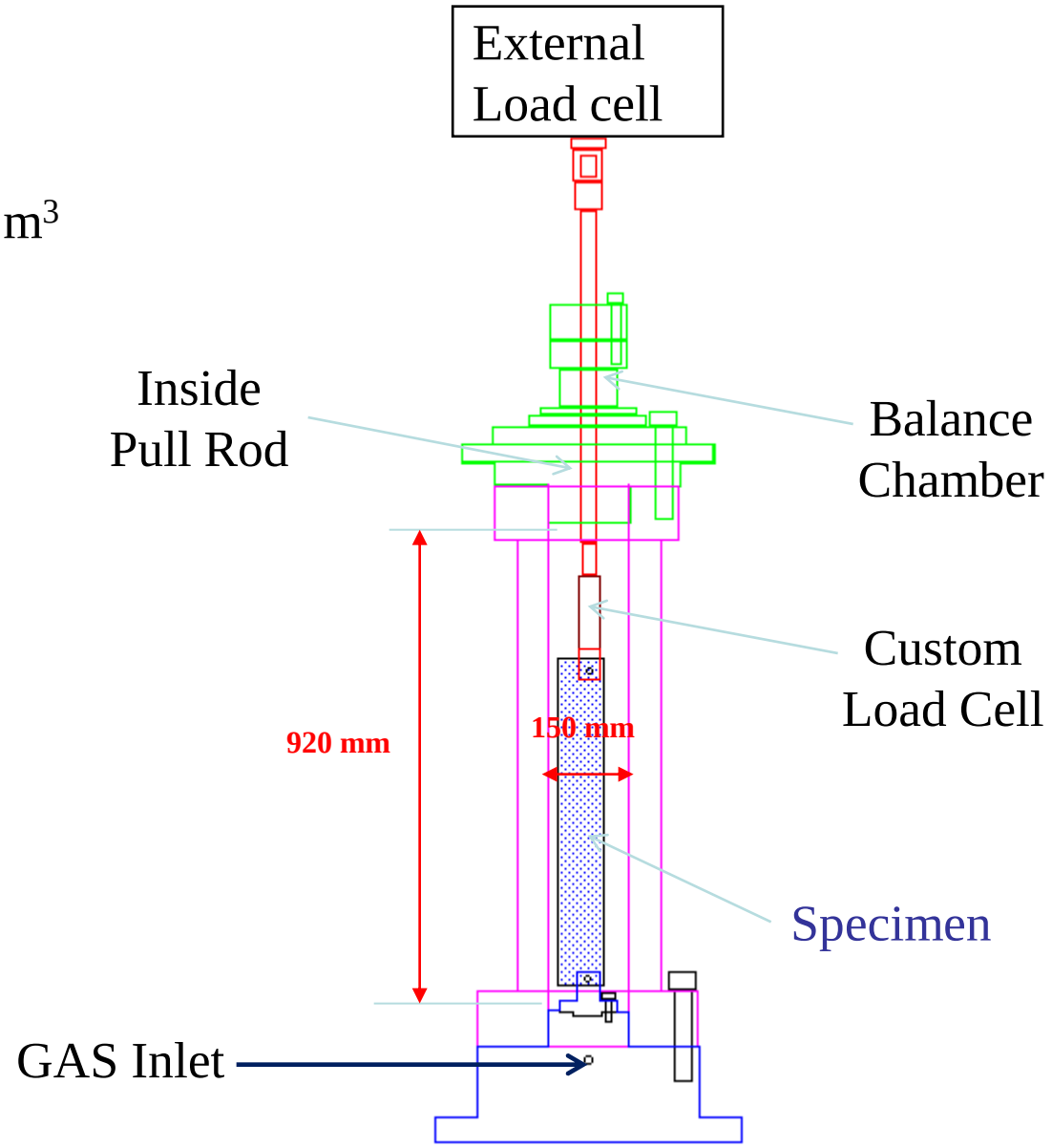
Test Capabilities

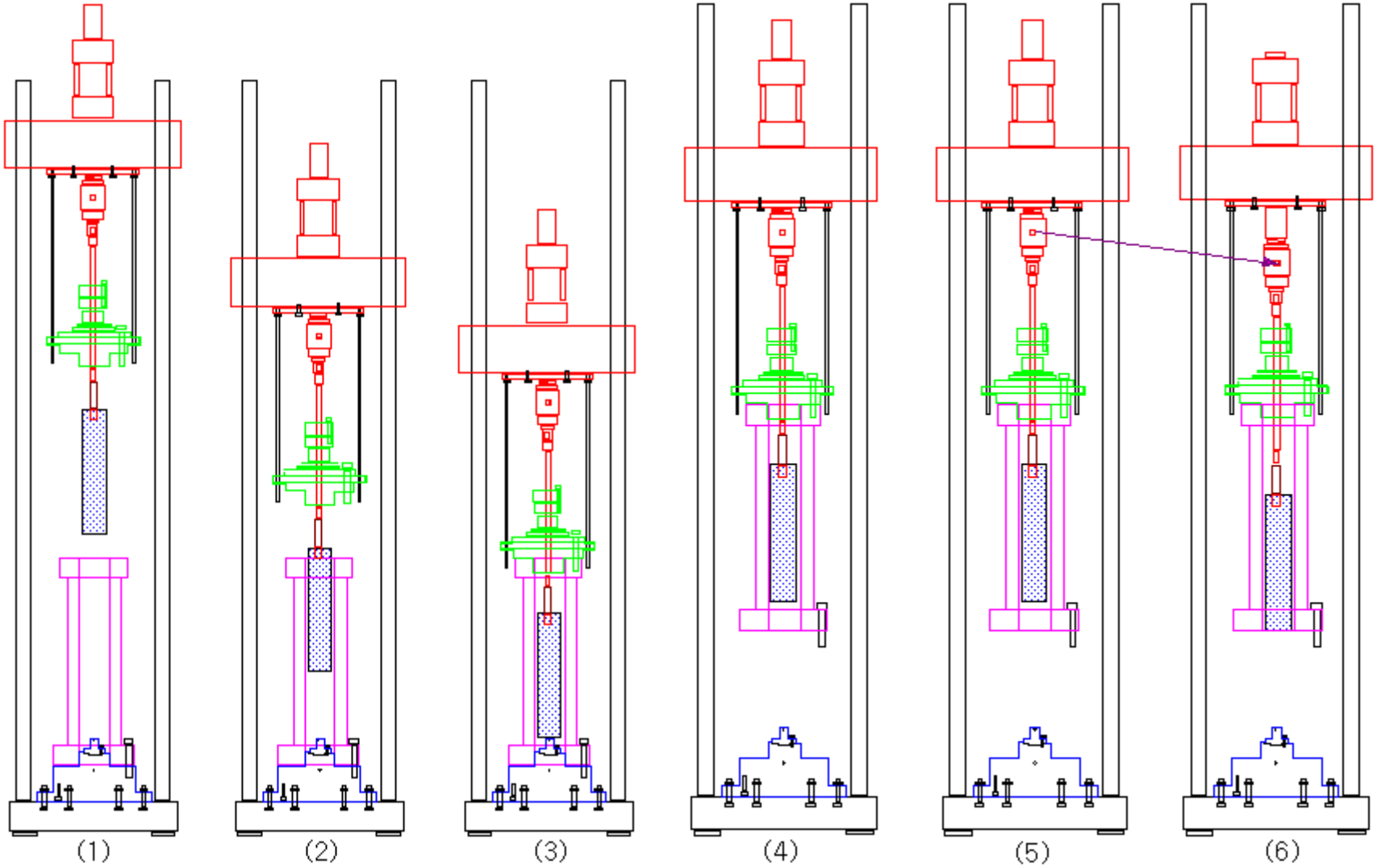
	FCG
Pressure	1200 bar (120 MPa)
Temperature	Room Temperature
Force	100 kN
Displacement	25 mm
Max. No. of Specimen	10
Test control mode	Inner Load External Load COD gage

Cross-section of Test Vessel

Vessel
Mat'l: SNCM 439
Inside Volum : 0.01626 m^3

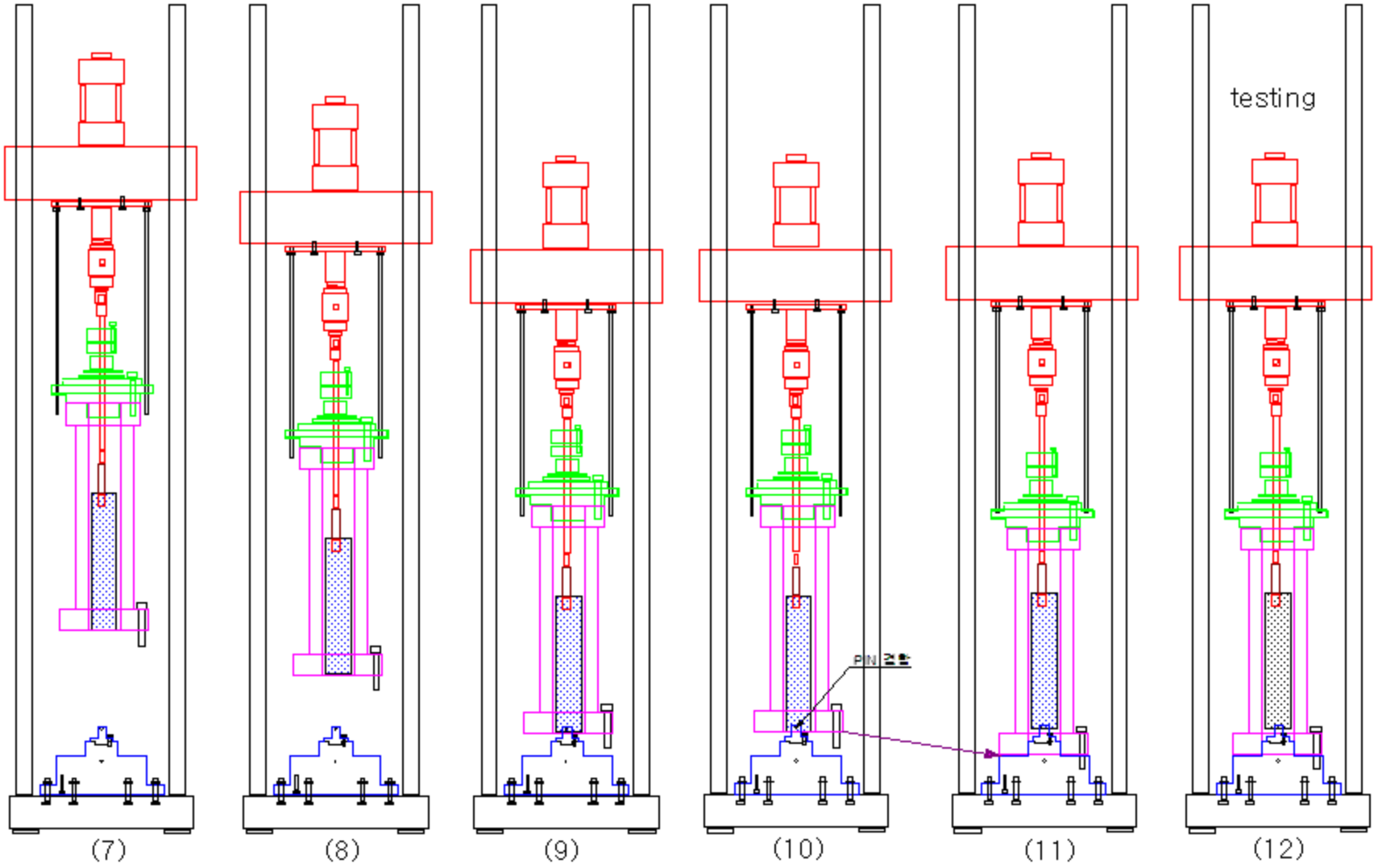


Pressure Vessel Assembly Sequence

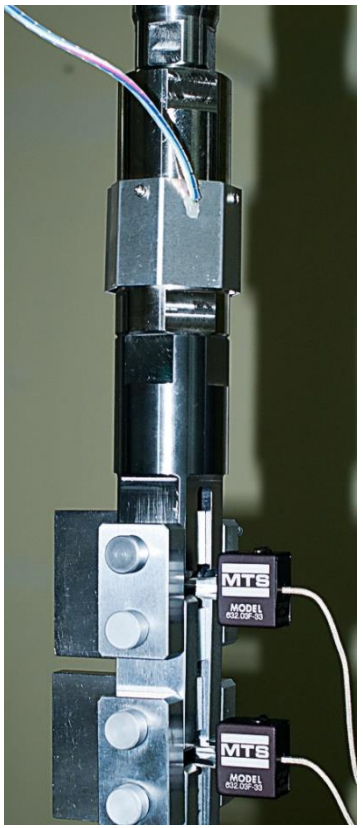


Pressure Vessel Assembly Sequence

KRISS



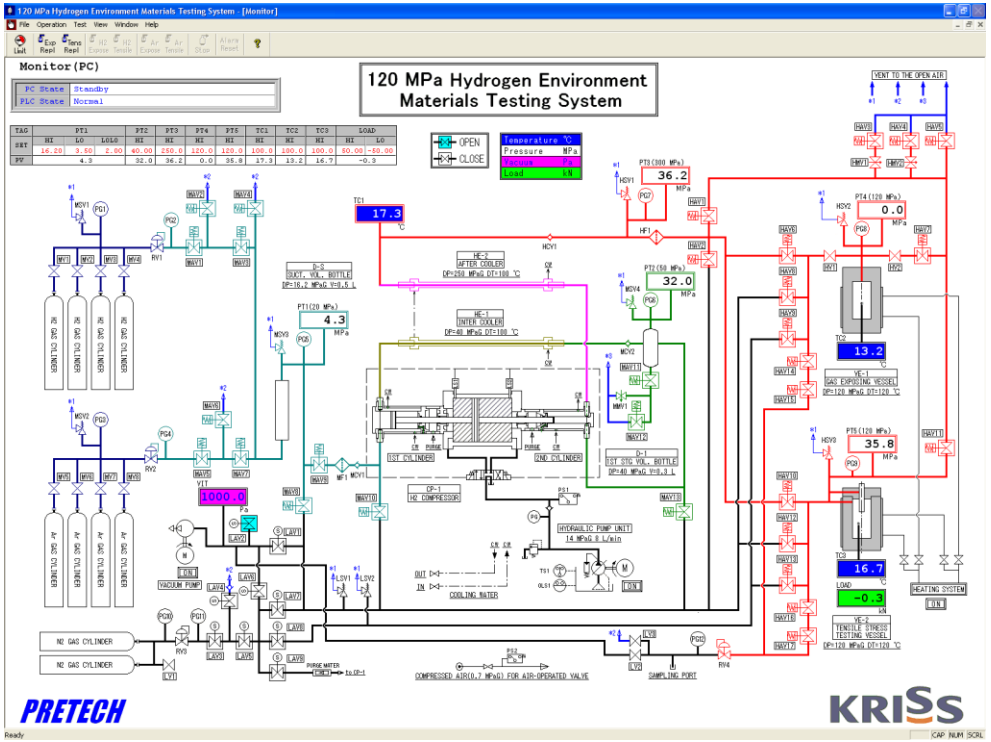
Transducers

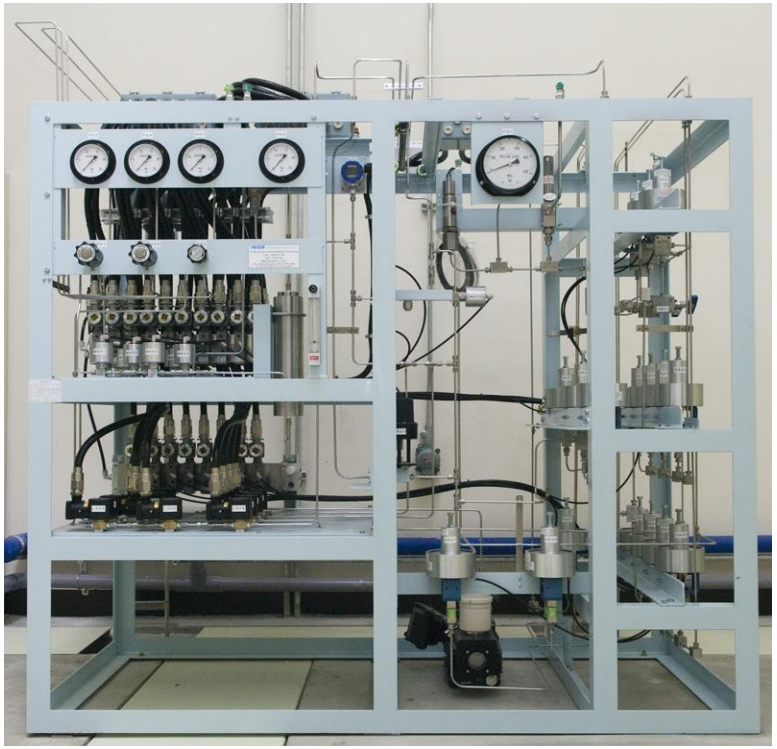


Strain gaged
Load cell(100 kN) for FCG test system

Gas Management

- Design Pressure : 120 MPa
- Computer controlled



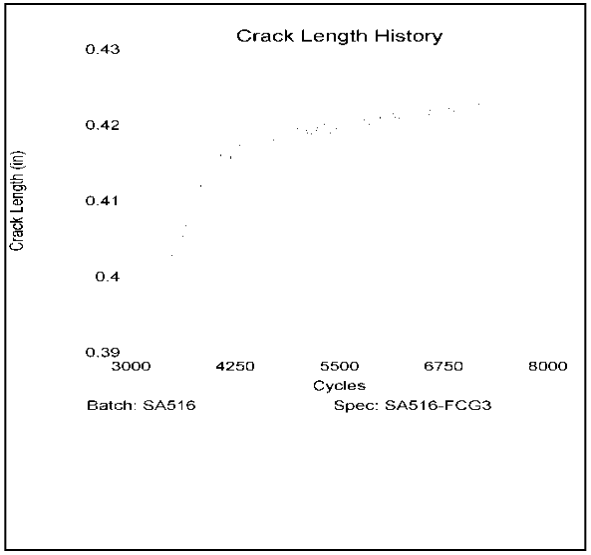


Conducting a Test

Software

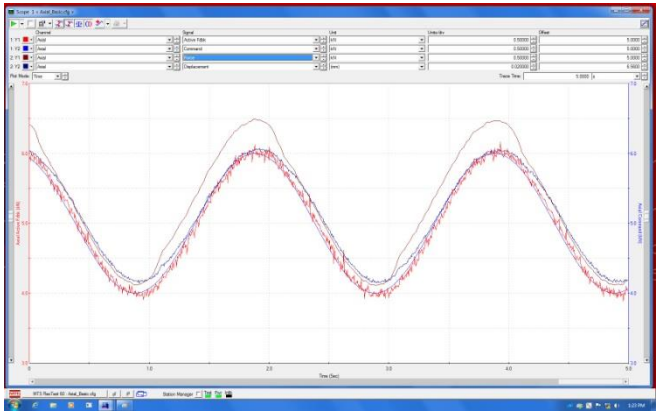
Tensile/fracture

MTS
Testworks 4
Fracture Toughness



Fatigue

MTS
Fatigue Crack Growth

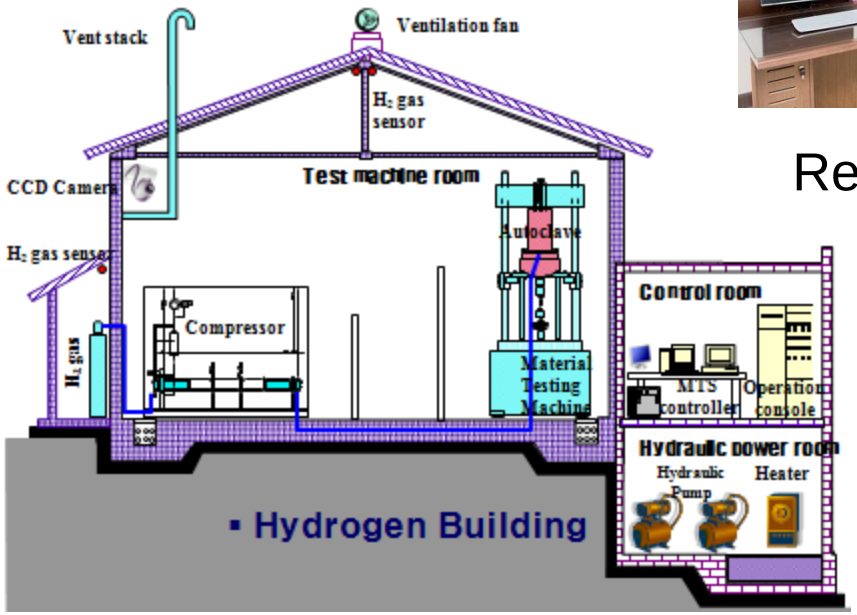


Safe Operations Using Engineering Controls

- Explosion proof test cell
- Secondary containment
- H_2 sensors
- H_2 interlocks
- Limited H_2 volumes

- ASME rated vessel
- Material: SNCM 439
- BPV Sect VIII, Div 2
- Main seal vent ports

Remote control



Hydrogen compatible material in all wetted applications.


Better Standards,
Better Life