Materials Testing Facilities in High Pressure Hydrogen Gas Circumstances in NSSMC

Hideki FUJII

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National Projects in Japan

- **International Clean Energy Network Using Hydrogen Conversion (WE-NET) Program (1993 -)**
  - **Liquid hydrogen (LH2)**
  - **Large scale vessels for LH2 transportation & storage**
- **Development for Safe Utilization and Infrastructure of Hydrogen (2003 -)**
- **Establishment of Codes & Standards for Hydrogen Economy Society (2005 -)**
- **Development of Technologies for Hydrogen Production, Delivery & Storage Systems (2010 -)**
  - **Compressed gaseous hydrogen (GH2) as well as LH2**
  - **Fuel cell vehicles --- On-board fuel tanks, Hydrogen refueling stations, etc.**

Test Facilities

Evaluation and investigation of materials used in hydrogen environments such as ;

- **hydrogen storage and transportation vessels**
- **pipes and tubes**
- **valves, pumps, etc.**

in the circumstances in which materials are actually exposed.

Total research facilities in

- **Futtsu R&E Center (Chiba)**
  - **– former Nippon Steel Corporation**
- **Amagasaki R&D Center**
  - **– former Sumitomo Metal Industries, Ltd.**

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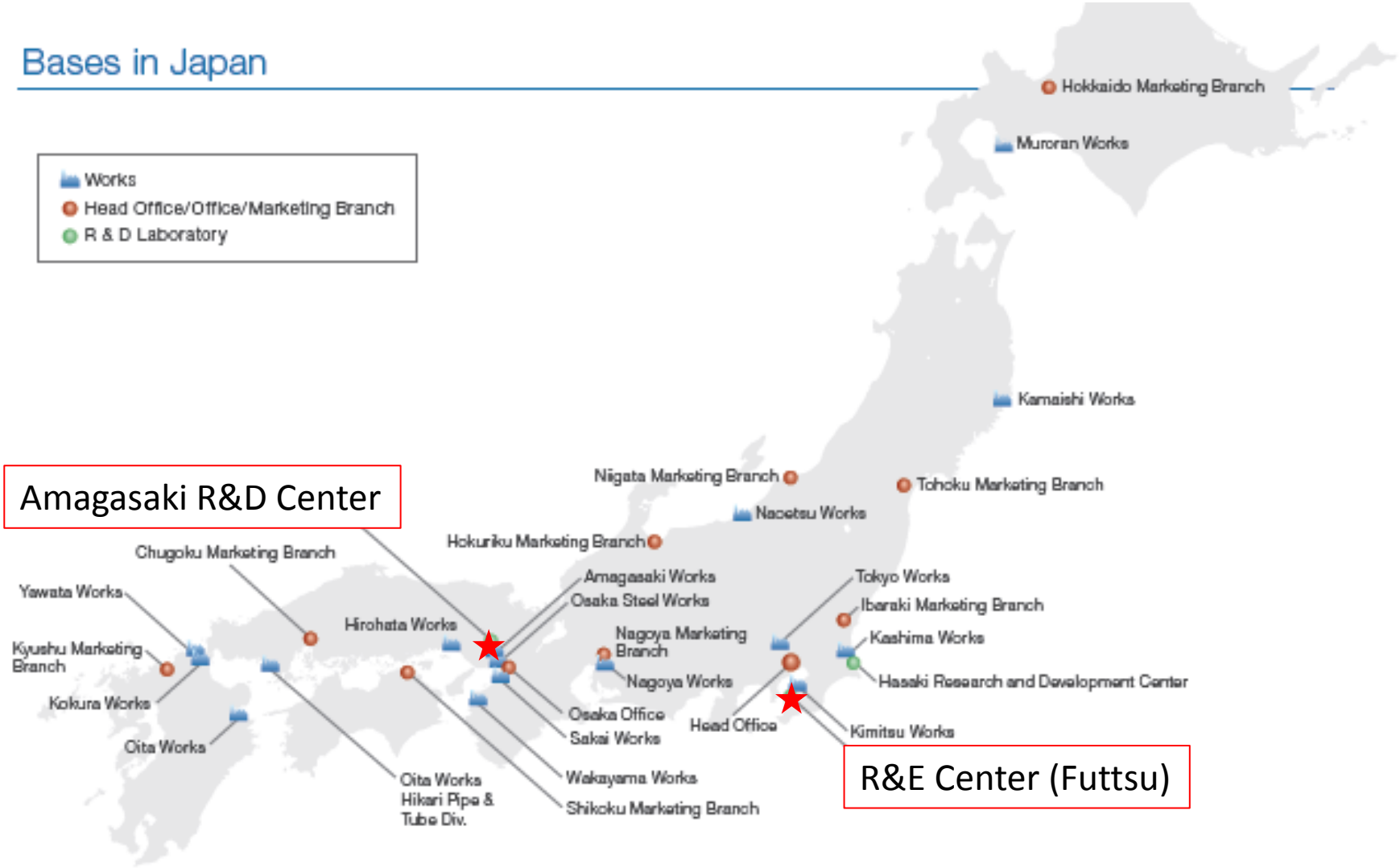
Locations



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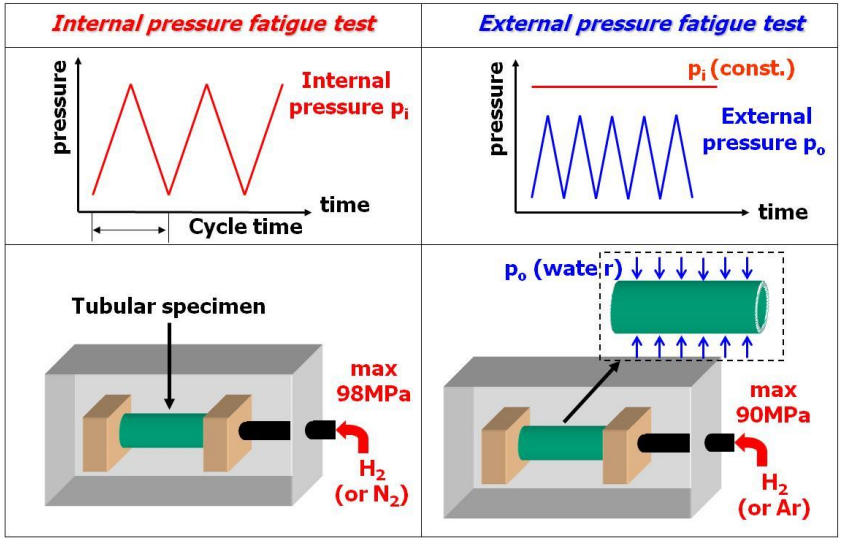
List of main test facilities

Amagasaki

- Slow Strain Rate Testing Machine in up to **45MPa** GH2 at -40 to 150ºC
- External Cyclic Pressure Fatigue Testing Apparatus (max.**90MPa**, R.T. to 100ºC)
- Internal Cyclic Pressure Fatigue Testing Apparatus (max.**98MPa**, -40ºC to R.T.)
- Compact Bending Fatigue Testing Machine in up to **120MPa** GH2 at R.T.



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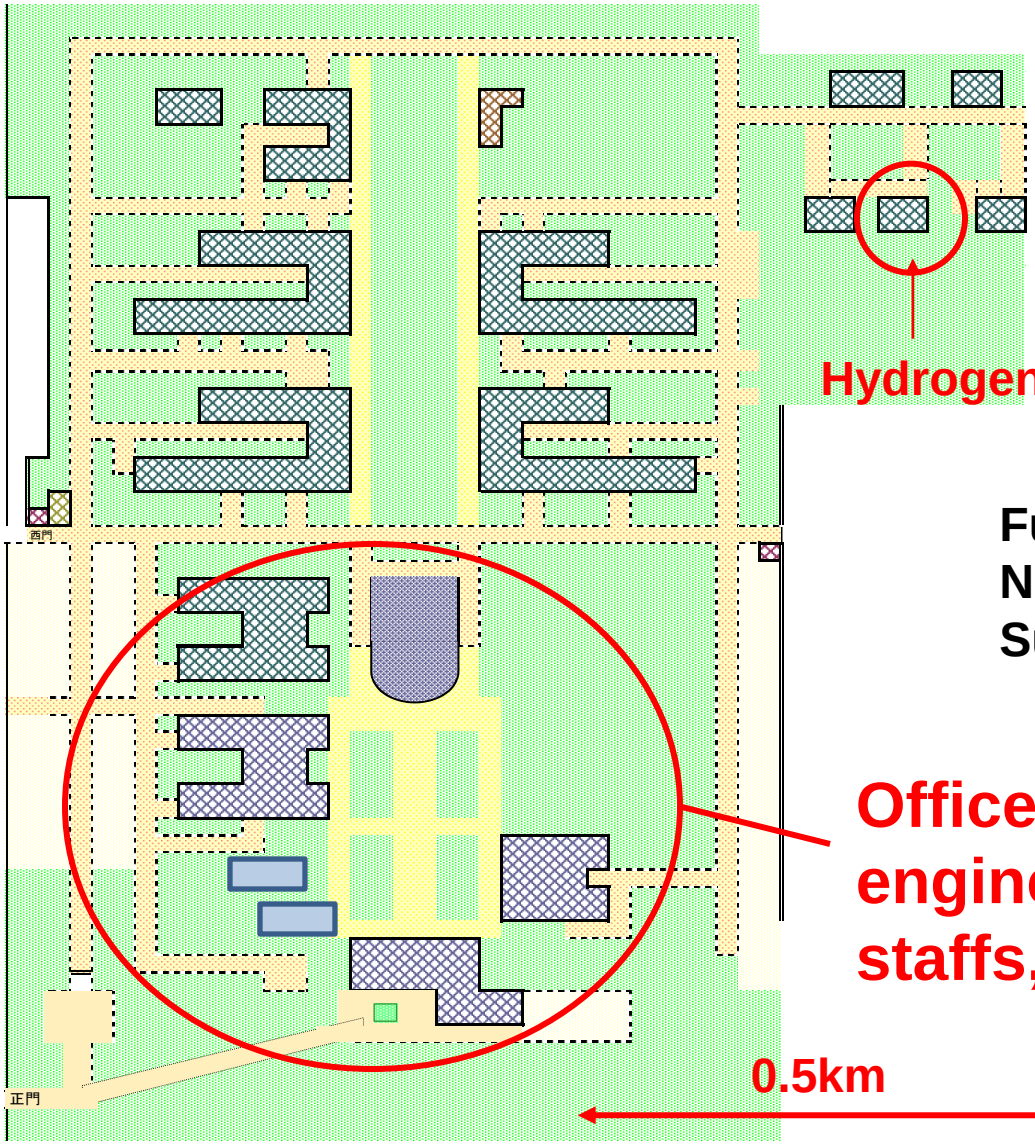
List of main test facilities

Futtsu

- Mechanical Testing Machine in Liquid Hydrogen
- Mechanical Testing Machine in up to 45MPa GH2 at -45 to 90ºC
- Mechanical Testing Machine in up to 99MPa GH2 at -45 to 90ºC
- Compact Bending Fatigue Testing Machine in up to 120MPa GH2 at R.T. (five)



Hydrogen experiment lab.

**Futtsu R&E Center,
Nippon Steel &
Sumitomo Metal Corp.**

Offices of researchers, engineers, managing staffs, etc,

0.5km

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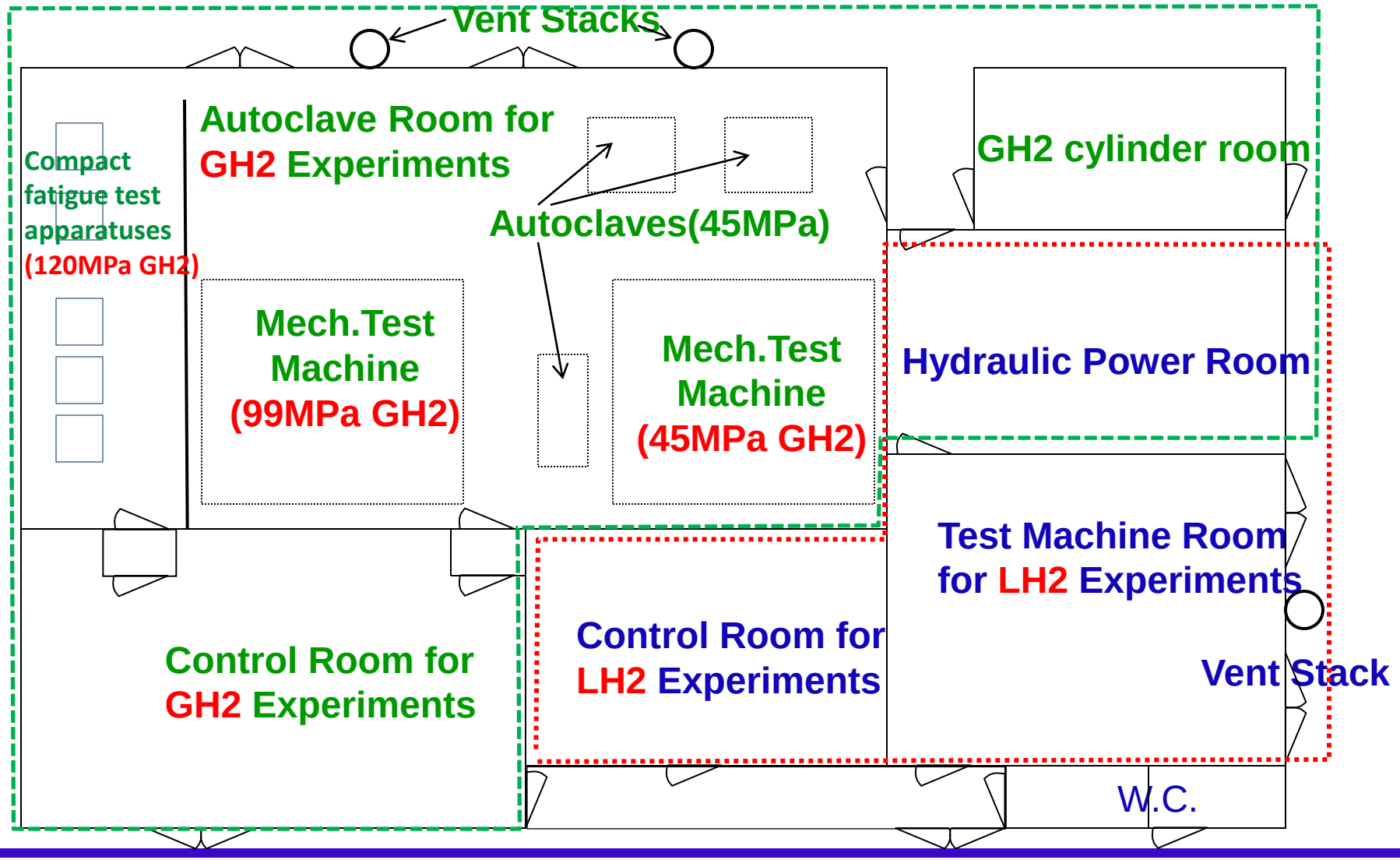
Outline of the Building



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Outline of Facilities

Combination of autoclaves and mechanical test machines

- Mechanical test machines

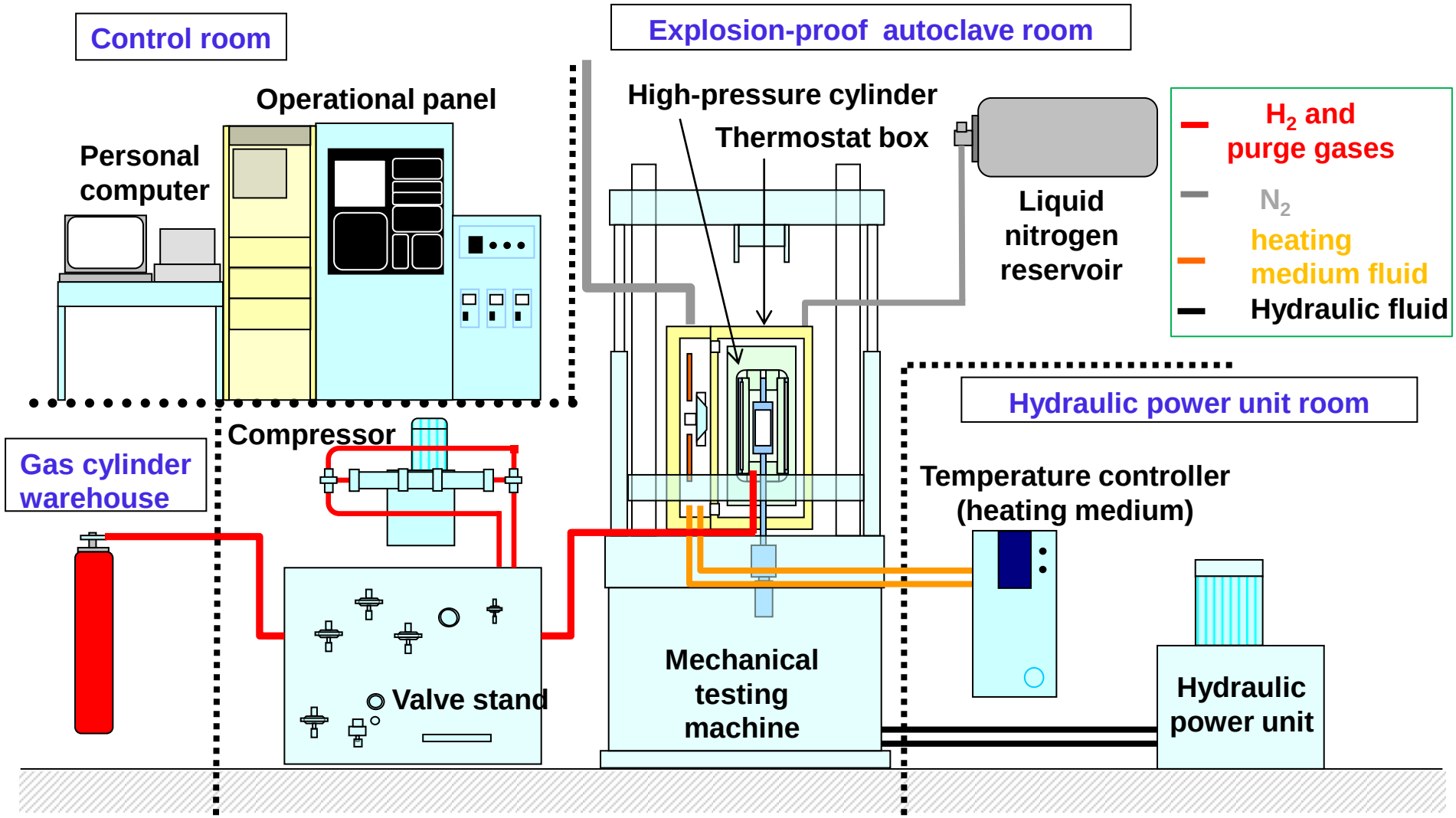
- Full digital servo hydraulic
  (45MPa-Saginomiya, 99MPa-Shimadzu)
- Max. static load ; 100kN, Piston stroke ; ±50mm
- Tensile, Fracture toughness (KIC, JIC), S-N fatigue,
- Fatigue crack growth, etc.

- Autoclaves

- Max. pressure ; 45MPa, 99MPa
- Temperature ; - 45 to 90ºC
- Internal load cell, Pressure balance system, etc.

- Safety devices

Layout Image



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Manufacturer and Sister Apparatuses

- **Designed and Manufactured by**
  KOBELCO (Kobe Steel, Ltd.)
  in strong cooperation with then Nippon Steel
  - 45MPa machine ;
  1st machine installed in 2004
  - 99MPa machine ;
  2nd machine installed in 2006
  ⇨ **Establishment of basic design concept**

- **Brothers and Sisters**
  - One machine in JSW (99MPa)
  - Three machines in Hydrogeneous (max.120MPa)

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Test Capabilities

- **Pressure ; max 45MPa / 99MPa**
- **Temperature ; - 45 to 90ºC**
- **Load ; max 100kN (- α)**
- **Piston stroke ; ±50mm (fatigue; ±1mm)**
- **CHS ; 0.001 to 60mm/s**
- **Frequency ; up to 10Hz**
- **Test duration ; up to seal deterioration**
- **Standard tests ;**

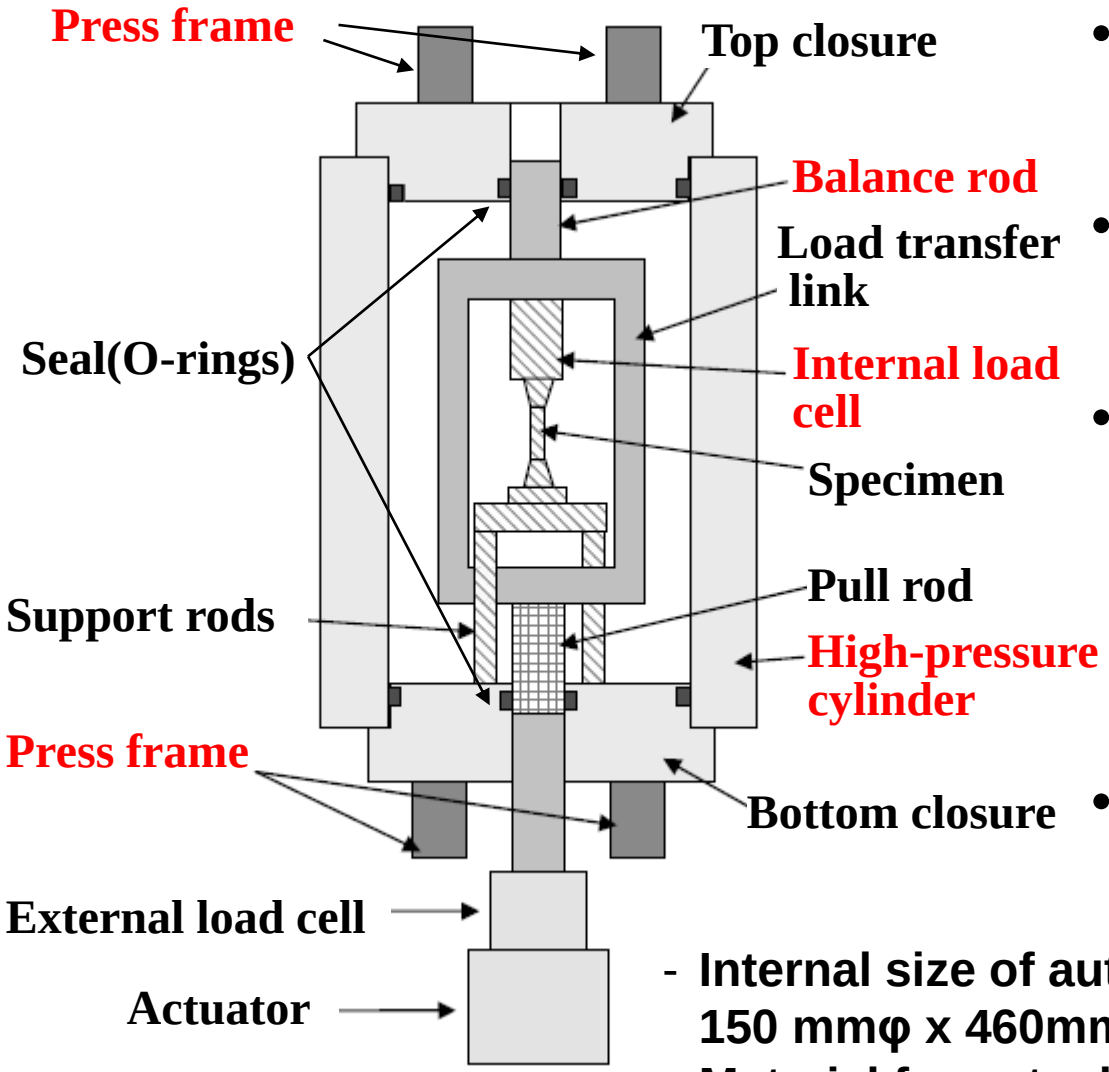
  Tensile test --- 7mm φ, 25mmGL round bar (JIS No.4),
  50mm GL plate type (JIS No.13B)

  S-N fatigue test --- 6mm φ round bar / sand glass

  Fatigue crack growth test --- 1TCT

  Fracture toughness test --- KIC, JIC with 1TCT

Structure of Autoclave and T.P. setting



- **Balance rod ;**
  - balance of high pressure
    → smaller mech. test machine
- **Twin press frame ;**
  - No screw structure
    → less opportunity of leakage
- **High-pressure cylinder;**
  - Made of A286 (99MPa machine)
    → high resistance against HGE
  - Double-layer structure
    → high resistance against crack propagation
- **Internal load cell;**

  see later explanation

- Internal size of autoclave (99MPa machine) ; 150 mmφ x 460mm H
- Material for autoclave, pull rod, T.P.holders ; A286

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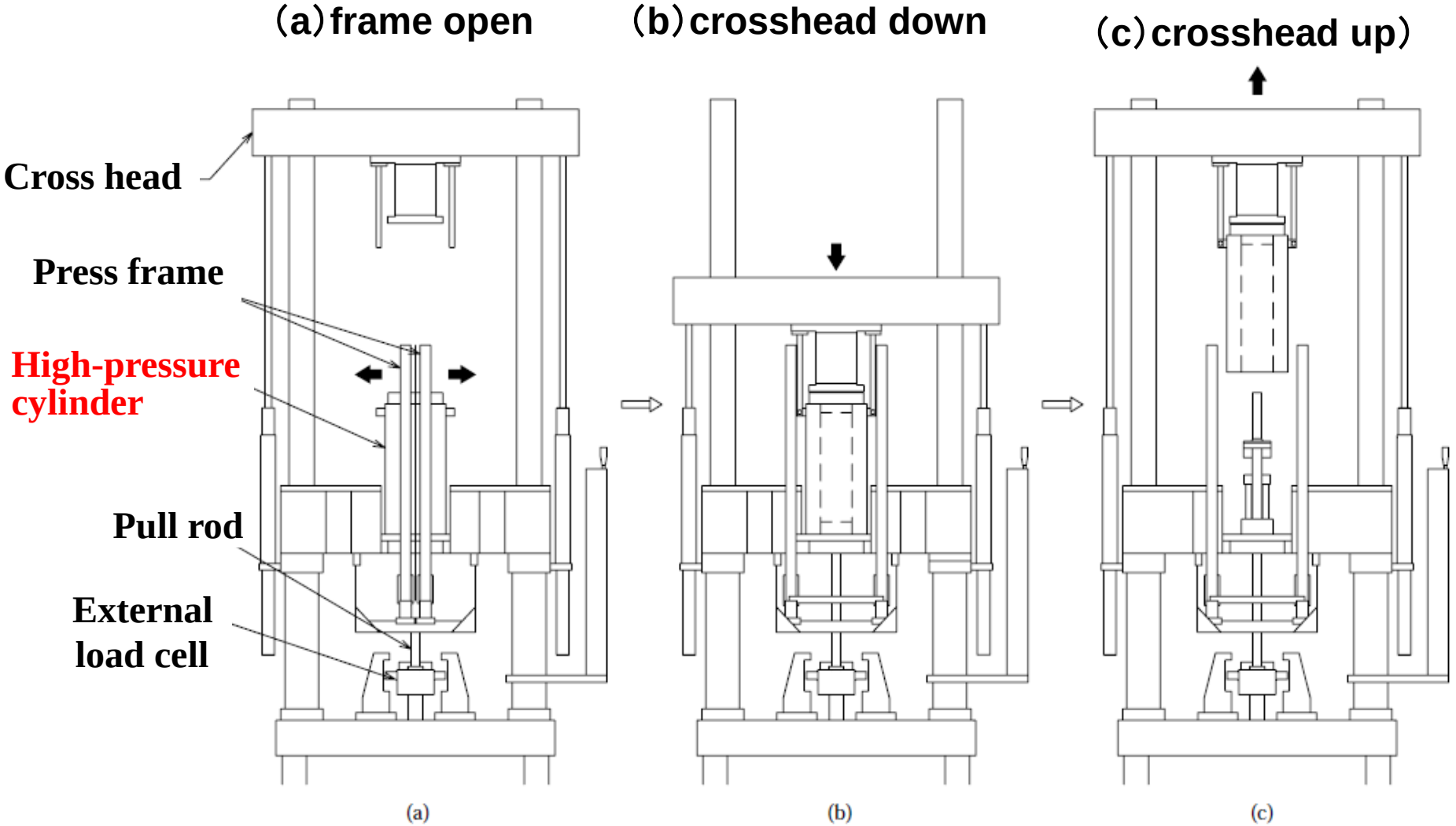
Test Procedures



Operating procedure of testing equipment (1)

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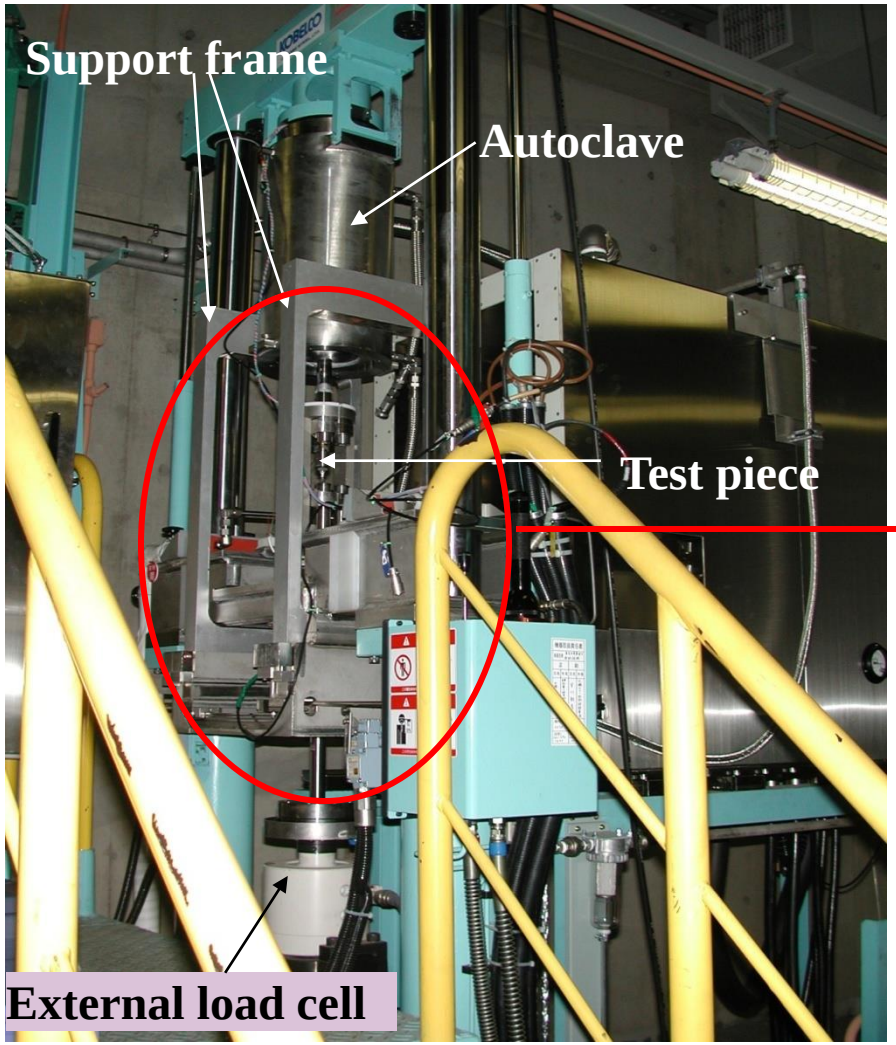
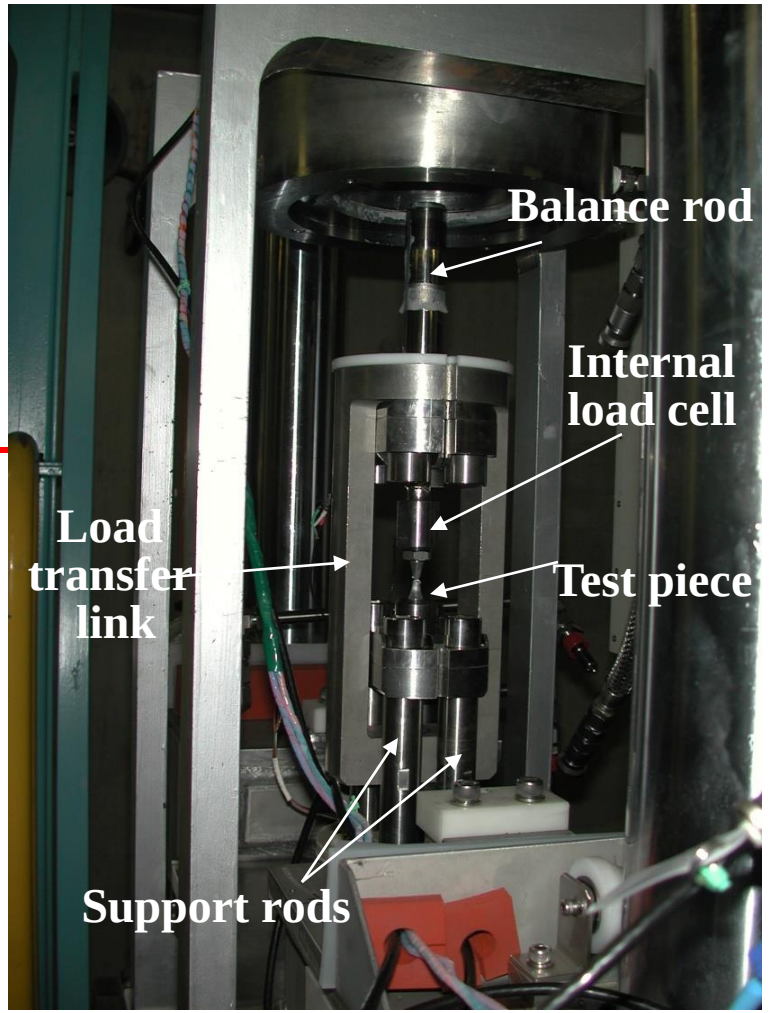
Test Procedures



Just after mounting test piece



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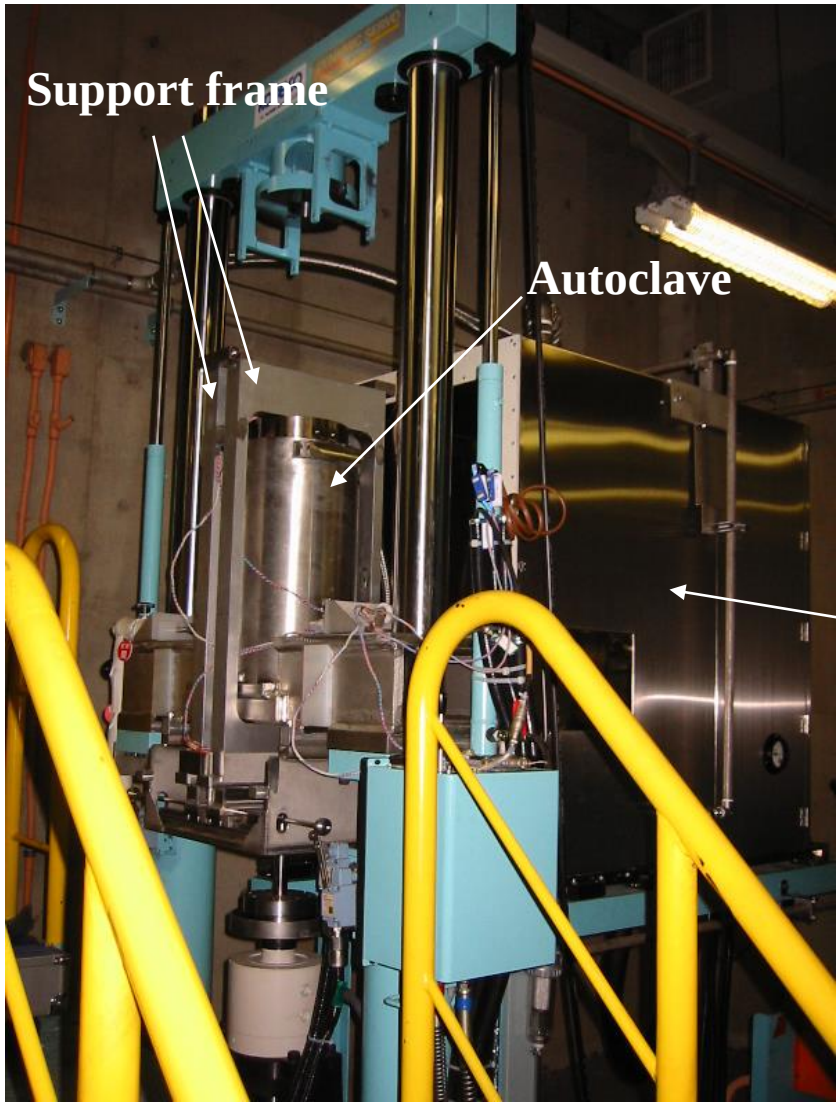
Test Procedures



Autoclave closed

Thermostat box with H_2 leakage detector

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Test Procedures

Thermostat box closed

Thermostat box

- **Heated or cooled N_2 gas at temperatures from -45 to 90°C flows**
- **H_2 detector installed**

- Less opportunity of leakage into the room

- Early gas leakage measures

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Test Procedures

Remote control after T.P. mounting

- **Open, close of valves**
- **Introduction and release of hydrogen gas**
- **Monitoring and control of temperature and pressure**
- **Operation of mechanical testing machine**

Various inter-rock systems

Valve stand

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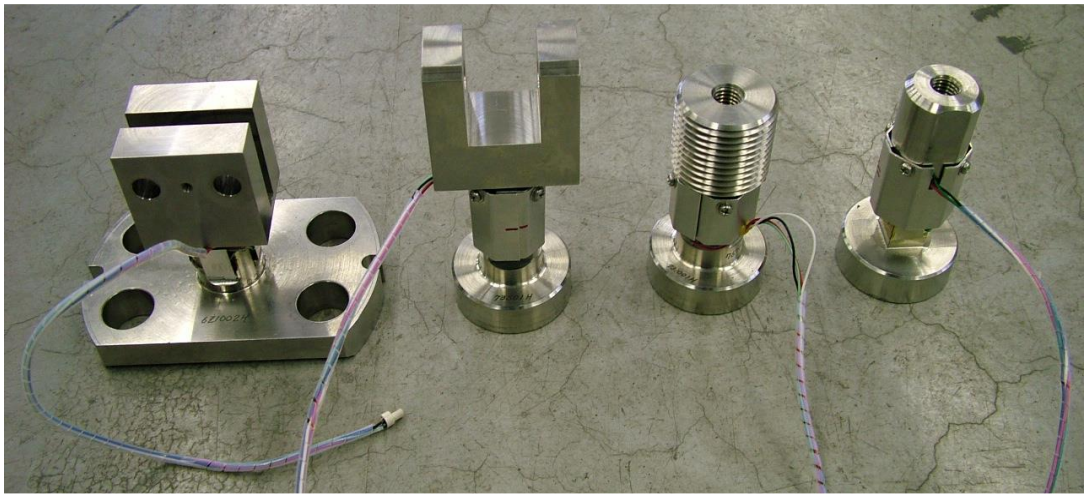
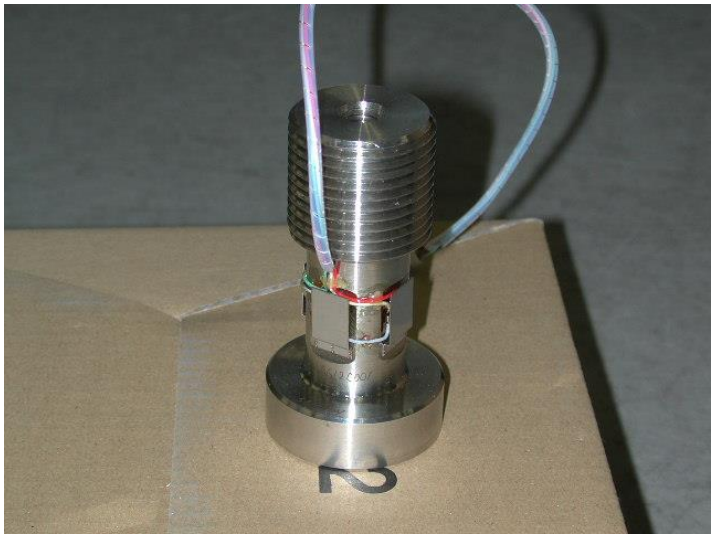
Internal Load Cell

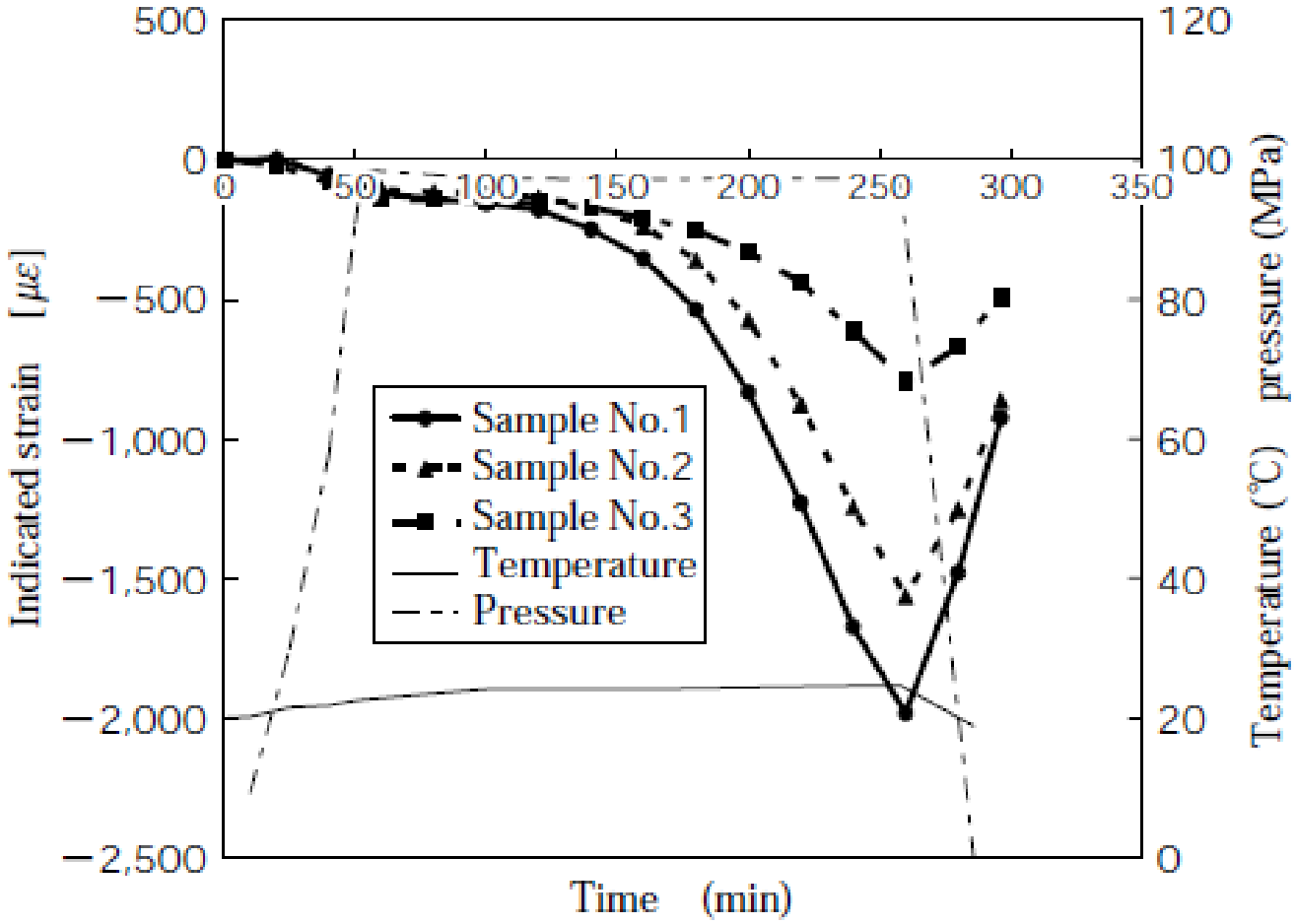
- Four strain gauges (Kyowa Electronic Instruments Co., Ltd.) with Wheatstone bridge on each upper T.P. holder
- Material for strain gauge ; Fe-Cr-Al (bcc)





- Input voltage ; less than 5V
- Zener barrier

Internal Load Cell



Output drift of commercial strain gauge (made of Ni-Cr, fcc) under high hydrogen pressure [1]

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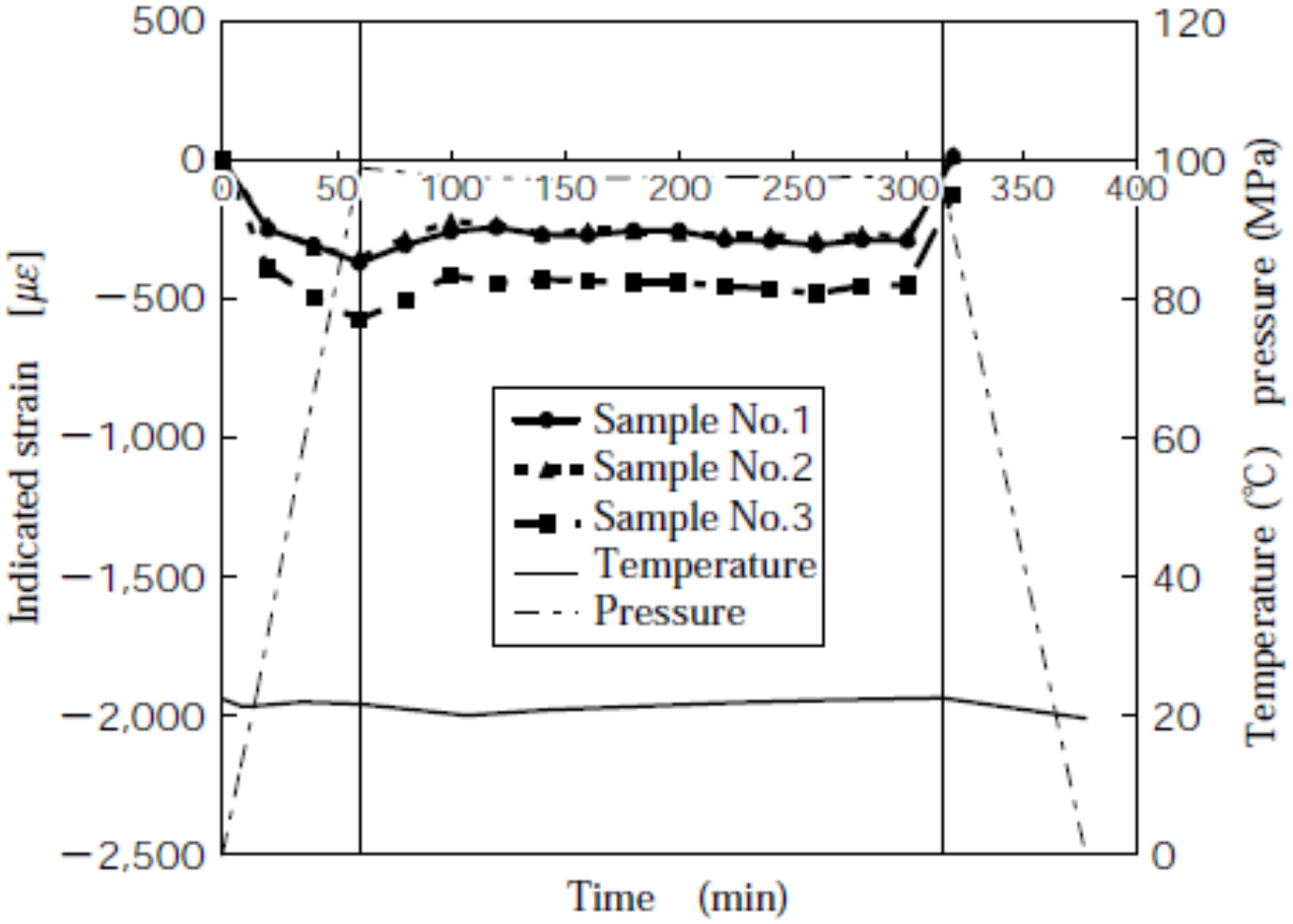
Internal Load Cell



Output drift of developed strain gauge (made of Fe-Cr-Al, bcc) under high hydrogen pressure [1]

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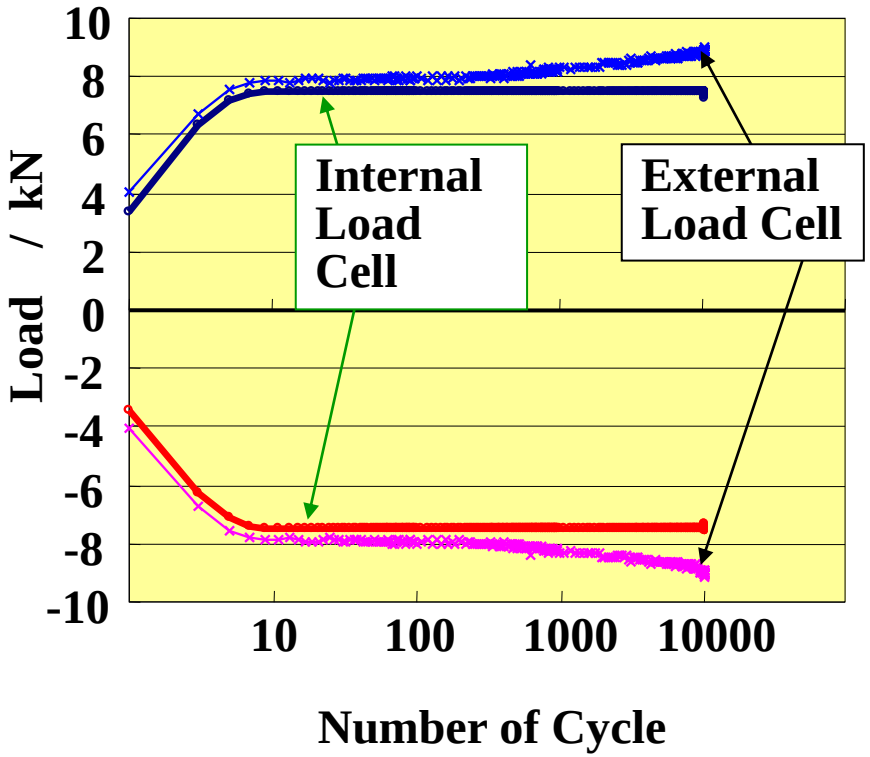
Transition of load during fatigue test (2)



Internal load cell maximum
internal load cell minimum
External load cell maximum
External load cell minimum

Controlled with internal load cell

- Load measured by external load cell is considerably deviated because of friction increase during testing.
- Internal load cell is quite effective for accurate fatigue tests.

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Other transducers

In Autoclave

- **Extensometer ; MTS Model 632.27F-20**
- **Clip gauge ; Kyowa DTR-S-6SA20B**
- **Two thermocouples ; JIS T (C-C)**

  - Input voltage ; less than 5V
  - Zener barrier

Outside of Autoclave

- **External load cell**
- **Displacement detector**

  - Explosion proof

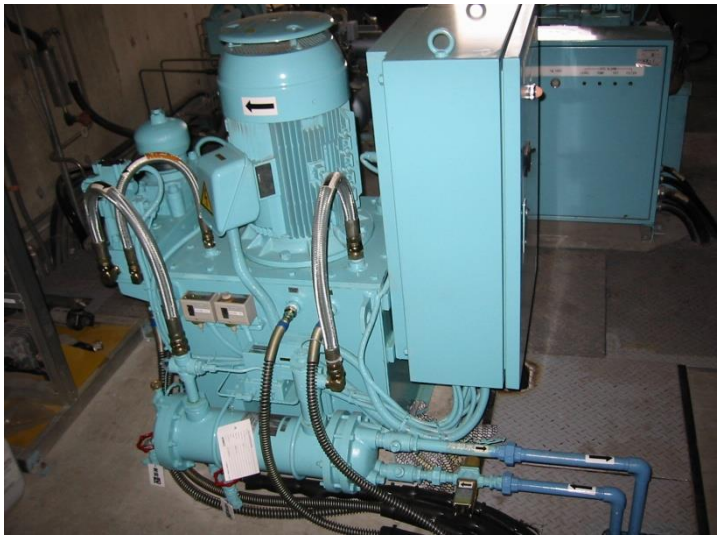
Hydrogen Supply

Direct supply from compressor

- **Compressor ;**

  Hydro-pac Inc.'s reciprocation type,

  - Inlet; 5 to 15MPa
  (from 14.7MPa gas cylinders)
  - Outlet ; max 100MPa



- **Procedure ;**

  - Evacuation by vacuum pump
  - 5MPa H_2 gas filling
  - Releasing
  3 times
  - Pressurizing to appointed pressure
  - Adjustment after temperature settled

- **Purity ; 7N (nominal)**

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Vent stack

- After a test, H2 gas is released to outside air through a vent stack
- Water sealing with heater to prevent from freezing

Temperature control

- **Temperature monitoring with thermocouples in the autoclave**

- **Heating jacket around high-pressure cylinder ;**
  - **circulation of heating medium fluid**
- **Thermostat box ;**
  - **heated or cooled N_2 gas flows**

- **Automatic temperature control from -45 to 90ºC**

Safety devices / measures

- Remote laboratory building
- 300-400mm thick explosion-proof concrete wall
- Secondary containment with steal walls
- Explosion-proof electric apparatuses
  - Electric lights, Servo valve, External load cell etc.,
- Hydrogen leakage detectors incl. in thermostat box
- Various interlock systems
- Oxygen content detector (to prevent from anoxia)
- Materials with hydrogen compatibility for pull-rod, autoclave, tubes & pipes etc.
- Annually regulated inspections incl. NDI
- Remote control
- Twin press frame without screw structure
- Others

Issues to be solved / improved -1

- **Durability of internal load cell**
  - **Currently, life time is 5-6 months**
  - **High temperature tests (at around 85°C) shorten its life time to a half**
- **Sealing by O-rings, back-up rings**
  - **No suitable materials are found for both high temperature testing and low temperature testing**
- **Dew condensation around mech. testing machine**
  - **Currently, low temperature tests are limited in dry winter time in Kanto area, Japan**

Issues to be solved / improved -2

- **Limited number of test facilities**
  - **High initial cost of test facilities**
  - **Delay of expansion of material kinds allowed to be used in hydrogen in Japan**

- **Low productivity**
  - **High running cost**
  - **Complicated testing procedures**
  - **Regulations related to high pressure gases**

- **Others**

NSSMC's future challenges -1

1. Expansion of kinds of materials
   - Various grades of Steels, Titanium and its alloys, etc. , based on requests from users and institutions in charge of standardization

2. Contribution to new codes and standards, their amendments

3. Development and application of new materials for hydrogen use (both LH2 and GH2),
   - high performance, low cost , high reliability

NSSMC's future challenges -2

To accelerate evaluations

- Development of more simplified evaluation methods
  - e.g. Compact Bending Fatigue Testing Machine in up to 120MPa
- Combination of various evaluation methods
  - e.g. SSRT, CBFT, Internal/External cyclic pressure fatigue testing, etc.
- Domestic and International cooperation
- Others

References

(1) Y.Manabe and Y.Miyashita ; “Development of 100MPa Class High Hydrogen Pressure Testing Equipment”, KOBE STEEL ENGINEERING REPORTS, Vol. 58 No. 2（2008）, p.19

(2) S. Ohmiya and H.Fujii ; “Fatigue Properties of Liner Materials used for 35MPa-class on-board Hydrogen Fuel Tanks”, Proc. of ASME Pressure Vessels and Piping Division Conf,（2005）PVP2005-71735

(3) H.Fujii, S.Ohmiya and T.Kayama ; “Comprehensive Research Facilities for Evaluation of Structural Materials Used in Hydrogen Environments”, Proc. of WHEC 16 – Lyon France, (2006) CD-ROM

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Thank you very much for your kind attention !