Research Facilities Fatigue and Fracture of Materials

For the establishment of the guidelines for the design of materials strength and the construction of a database, the basic principle of hydrogen embrittlement is being investigated by the use of various high-pressure hydrogen fatigue testing machines and vessels, high-resolution microscopes and precise measurement instruments.

120MPa,100MPa Hydrogen Fatigue Testing Machine

To clarify the effect of hydrogen on materials strength, fatigue tests are conducted under hydrogen environments at pressures up to 120MP, 100MPa

[120MPa Hydrogen Fatigue Testing Machine]

[120MPa Hydrogen Fatigue Testing Machine (3 unit)]
- Gas pressure:120MPa(max.)
- Environmental gas: hydrogen;helium;nitrogen
- Testing frequency:10Hz(max.), 20Hz(max.)
- Load capacity:±100kN(max.), ±50kN(max.)
- Available specimen shape: round bar, plate, compact-tension
- Testing temperature:-45 ~ 120℃
- Hydrogen vessel(inside): 145mm(dia.)×460mm(height)

[100MPa Hydrogen Fatigue Testing Machine (1 uniit)]
- Gas pressure: 99MPa(max.)
- Environmental gas: hydrogen;argon;helium
- Testing frequency: 10Hz(max.)
- Load capacity: ±100kN
- Available specimen shape: round bar, plate, compact-tension
- Testing temperature: -45 ~ 90℃
- Hydrogen vessel(inside):150mm(dia.)×460mm(height)

(41 machines [incl. low-pressure fatigue testing machines])

100MPa Hydrogen Gas Vessel

For charging metallic materials and rubbers with hydrogen gas

- Environmental gas: hydrogen
- Gas pressure:100MPa (max.)
- Operation temperature: 280℃ (max.)
- Volume: 0.5ml

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Blister Testing Machine for Rubber

To evaluate the durability of O-rings

- Environmental gas: hydrogen
- Pressure range: atmospheric pressure to 90 MPa
- Holding time: 0 ~ 99h
- Pressure-increasing rate: 6MPa/s
- Pressure-decreasing rate: 0.025 MPa/s ~ 20 MPa/s
- Testing temperature range: -60℃~ 100℃

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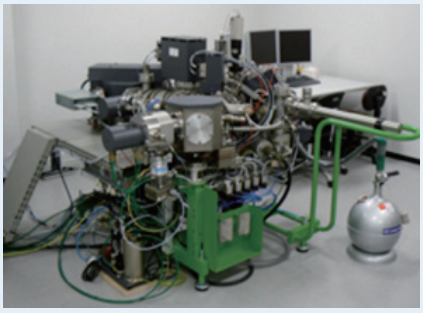
Secondary Ion Mass Spectrometry (SIMS)

To visualize the 2D/3D distribution of hydrogen in materials



- Ion source: cesium (Cs)
- Resolution: 7 × 1016 at./cm3 (for H in Si)
- XY resolution: < 1μm
- Available measurement area: 35 ~ 250μm (dia.)

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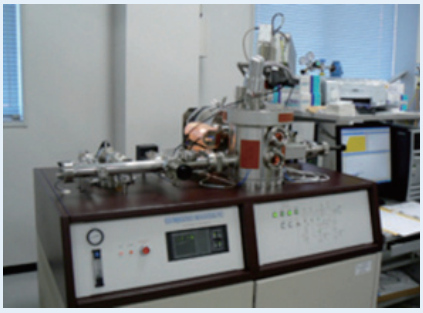
Thermal Desorption Spectrometry (TDS)

For the measurement of hydrogen content in metallic materials



- Measurement resolution: 0.01 mass ppm
- Measurement mass range: M/z:1 ~ 200
- Measurement temperature range: RT ~ 1200℃
- Vacuum pressure: < 5 × 10^{-7} Pa

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Thermal Field Emission Scanning Electron Microscope with Electron Backscatter Diffraction Analyzer

To analyze the crystallographic orientation of fracture surface originating from hydrogen embrittlement

- Electron gun: shot-key field emission type (ZrO/W cathode)
- Resolution: 1.2 nm (for 30 kV), 3.0 nm (for 1 kV)
- Accelerating voltage: 0.5 kV ~ 30 kV
- Probe current: a few pA ~ 200 nA

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Research Facilities Tribology

These test rigs and analyzers are used to study tribological properties of materials in hydrogen for establishing a proper design methodology of tribological elements used in hydrogen systems.

Hydrogen environment friction test rig

for conducting reciprocating sliding tests in hydrogen gas

- Test gases: hydrogen, argon, helium, vacuum
- Maximum gas pressure: 0.2 MPa
- Minimum pressure when evacuated: less than 1 x 10^{-5} Pa
- Maximum frequency of reciprocating motion: 20Hz
- Maximum normal load: 50 N

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Friction test rig with highly-controlled environment

for conducting sliding tests in hydrogen with controlled purity

- Test gases: hydrogen, argon, helium, vacuum
- Minimum pressure when evacuated: less than 1x10^{-5} Pa
- Concentration of water detected: down to 2 mass ppb
- Concentration of oxygen detected: down to 1 mass ppb
- Test configuration: Pin-on-disk test or reciprocating test
- Maximum rotational speed: 200 rpm
- Maximum normal load: 200 N

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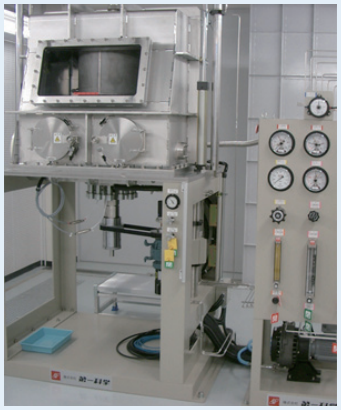
Ultra high pressure hydrogen friction test rig

for conducting pin-on-disk sliding tests in ultra-high pressure hydrogen



- Test gases: hydrogen, argon, helium
- Maximum gas pressure: 45 MPa
- Temperature: -50 to 120 C
- Minimum pressure when evacuated: 0.1 Pa
- Test configuration: Pin-on-disk test
- Diameter of disk specimen: between 15 and 60 mm
- Maximum rotational speed: 100 rpm
- Maximum normal load: 50 N

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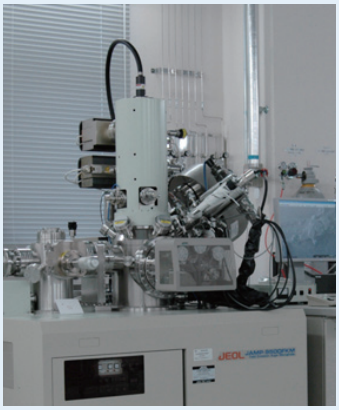
Auger Electron Spectroscope for hydrogen tests (AES)

for analyzing surfaces before and after sliding tests in hydrogen



- Ion guns: Shot key field emission ion gun
- Resolution of secondary electron images: 3 nm
- Resolution of Auger images: 8 nm
- Minimum pressure when evacuated: less than 5 x 10^{-8} Pa

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X-ray Photoelectron Spectroscope for hydrogen tests (XPS)

for analyzing surfaces before and after sliding tests in hydrogen

- X-ray source: Al/Mg twin target and Al target
- Minimum diameter of analysis: 30 μm
- Minimum pressure when evacuated: less than 7 x 10^{-8} Pa

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Sealed transfer vessel for carrying tribology specimens

for carrying specimens before or after friction tests in hydrogen to the analyzers in high vacuum or inert gas

- Test rigs to which the vessel can be attached: The friction test rig with highly-controlled environment and the ultra high pressure hydrogen friction test rig
- Analyzers to which the vessel can be attached:The X-ray Photoelectron Spectroscope (XPS) and the Auger Electron Spectroscope (AES)
- Minimum pressure when evacuated:less than 1 x 10^{-4} Pa

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Research Facilities Thermal engineering, Thermal properties

PVT property (Pressure-Volume-Temperature relationship), viscosity, and thermal conductivity of hydrogen are measured at high pressures up to 100 MPa accurately by self-developed apparatuses. Permeability of hydrogen at high temperatures up to 500 ℃ is also measured.

Magnetic Suspension Densimeter

Density is measured based on the Archimedes buoyancy principle.
The sinker in the pressure vessel is levitated by the magnetic coupling of a permanent magnet and electromagnet, and the buoyancy force working on the sinker is measured by an electronic balance. The density in the vessel is determined from the buoyancy force and the volume of the sinker.

- Temperature range : from room temperature to 250 ℃
- Pressure range : up to 100 MPa
- Sinker : mass 28 g, volume 3.3 cc

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PVT Measurement Apparatus by the Isochoric Method

PVT is measured using the isochoric method. In this method, the pressure vessel is filled with sample fluid, and the temperature and pressure are measured along the isochores.

- Temperature range : from 200 ℃ to 500 ℃
- Pressure range : up to 100 MPa
- Volume of the pressure vessel : 250 cc

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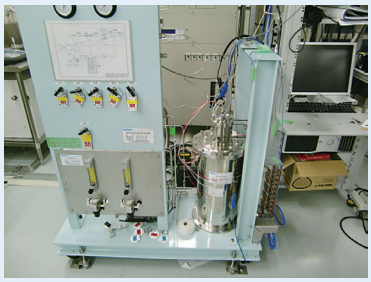
Viscosity Measurement Apparatus

Viscosity is measured using the vibrating wire method. AC voltage with different frequencies is supplied to the wire set in a magnetic field, and viscosity is determined from the resonant curve obtained by the measurements of induced voltages.



- Temperature range : from -40 ℃ to 500 ℃
- Pressure range : up to 1 MPa
- Wire dimensions : diameter 50 μm, length 24 mm

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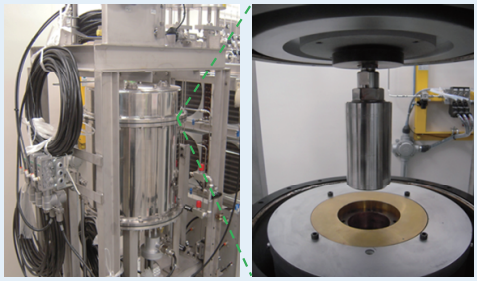
Thermal Conductivity Measurement Apparatus

Thermal conductivity is measured using the transient short hot-wire method. A small electrical current is supplied to a fine platinum wire inside the pressure vessel, and the thermal conductivity is determined from the transient temperature change of the wire.



- Temperature range : from room temperature to 500 ℃
- Pressure range : up to 100 MPa
- Wire dimensions : diameter 10 μm, length 15 mm

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Hydrogen Permeability Measurement Apparatus

Hydrogen is filled in a seamless coiled tube, and permeability of hydrogen through metals is measured from the pressure drop. The permeability is also measured using a gas chromatograph.

- Temperature range : from 300 ℃ to 500 ℃
- Pressure range : up to 1 MPa
- Volume of the vessel : 50 cc

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Flow Rate Through Orifice Measurement Apparatus

High-pressure hydrogen is rapidly expanded from a pressure vessel through an orifice imitating a crack of container, and the temperature and pressure changes of the sample during the expansion are measured.

- Temperature range : room temperature
- Pressure range : up to 100 MPa
- Diameter of the orifice : 0.2 mm

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Research Facilities Fuel Cells

We have been conducting a wide range of fuel cell R&D, from materials to systems using advanced facilities such as highest-resolution FESEM-EDX and STEM-FIB systems and fuel-cell evaluation systems as shown below.

Fuel Cell (SOFC) Evaluation Systems

Automatic electrochemical evaluation system for SOFCs which can continuously monitor the overvoltages caused by electrode reactions and ohmic resistance of the component materials separately as well as current-voltage characteristics of SOFCs.

- Anode gas: H_2, CO_2, CO, CH_4, N_2 (< 200 ml min-1)
- Cathode gas: Air (< 200 ml min-1)
- Current resolution: 1 mA
- Voltage resolution: 1 mV

(Hydrogen Utilization Processes Lab., Kyushu University: 50/Fuel Cell System Lab., Kyushu University: 3)

Fuel Cell (PEFC) Evaluation Systems

Automatic electrochemical evaluation system for PEFCs which can measure the overvoltages caused by electrode reactions and ohmic resistance of the component materials separately as well as current-voltage characteristics of PEFCs.

- Anode gas: H_2 (< 200 ml min-1)
- Cathode gas: Air (< 200 ml min-1)
- Current resolution: 1 mA
- Voltage resolution: 1 mV

(Hydrogen Utilization Processes Lab., Kyushu University: 10/Fuel Cell System Lab., Kyushu University: 5)

Scanning Transmission Electron Microscope

Ultra high-resolution scanning transmission electron microscope (STEM) equipped with highly-sensitive EDX and EELS systems.

- Magnification: x 4,000,000
- Resolution: 0.204 nm
- Accelerating voltage: 200 kV
- Electron gun: cold FE attachment:highly-sensitive EDX and EELS system

(Hydrogen Utilization Processes Lab., Kyushu University)

Focused Ion Beam System

Processing machine for micro-sampling of a specific part of materials under investigation with a thickness of less than 100 nm for STEM observation.

- Resolution: 6 nm at 40 kV
- Accelerating voltage: 10 ~ 40 kV
- Ion source: Ga
- Specimen holder: compatible holder available for STEM

(Hydrogen Utilization Processes Lab., Kyushu University)

Automatic Gas Chromatograph

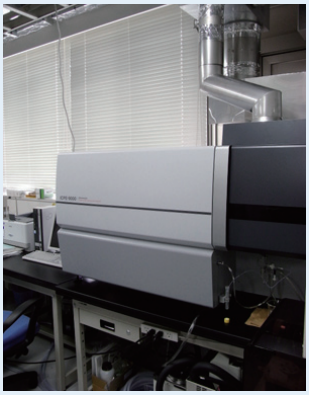
Gas chromatograph for long-term consecutive measurement of fuel gas composition equipped with automatic data output system.

- Gas species detectable:H_2, O_2, N_2, CH_4, CO, CO_2, C_2-C_6
- Carrier gas: He, N_2
- Detector: TCD
- Minimum analyzing time: 20 min
- Accuracy: 0.01%

(Hydrogen Utilization Processes Lab., Kyushu University: 3/Next-FC: 1)

Inductively Coupled Plasma (ICP) Emission Spectrometer

ICP spectrometer features high ppb level detection ability, broad 5-6 digit analysis concentration ranges, and batch analysis of multiple elements. Measurements are completed in only a few minutes.



- Crystal oscillator: 1.6 kW
- Output stability: within 0.3%

(Hydrogen Utilization Processes Lab., Kyushu University)