Material Testing and Analysis of Metals in Hydrogen Gas Environments

In response to global climate change, efforts toward decarbonization and carbon neutrality have significantly accelerated. Many countries have set their goals to achieve carbon neutrality, such as by the year 2050. Hydrogen energy is considered a promising solution due to minimal environmental impact and zero carbon dioxide emissions during combustion.

Hydrogen has exceptional capabilities in energy conversion, making it a valuable resource for electricity generation. Kobe Material Testing Laboratory Co., Ltd. has actively developed various material testing technologies in hydrogen environments. This document highlights our testing achievements and prospects for further technological advancements.

Aiming to realize hydrogen energy society

Hydrogen energy: Produce → Transport / Store → Use

The demand for using structural materials in hydrogen environments is increasing, but challenges remain.

Challenge 1

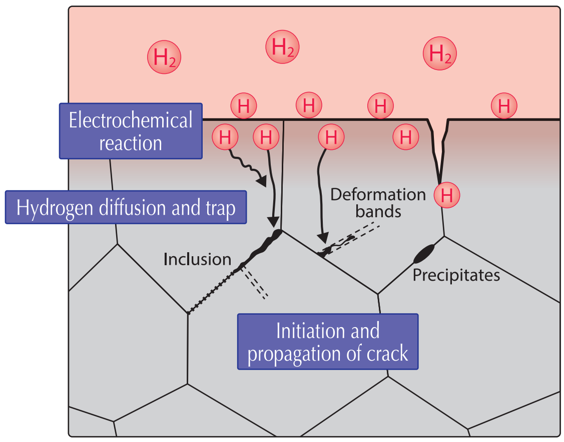
- Can existing designs be used as they are?
- Is fundamental improvement necessary?

Concerns in Strength Design

Hydrogen Embrittlement

Hydrogen atoms infiltrate and diffuse within metallic materials, reducing their strength.

- Delayed Fracture
- Decrease in tensile properties such as elongation at break and reduction of area
- Reduction in fatigue life and fatigue limit
- Acceleration of fatigue crack growth rate
- Decrease in fracture toughness



Material testing in hydrogen environments is essential to evaluate the hydrogen compatibility of intended metallic materials.

Challenge 2 | Current situation

- Handling hydrogen gas requires specialized knowledge and rigorous safety measures due to its flammability.
- Testing in hydrogen environments requires expensive, explosion-proof equipment.
- The high cost of testing services in hydrogen environments hinders the development of a hydrogen energy society.

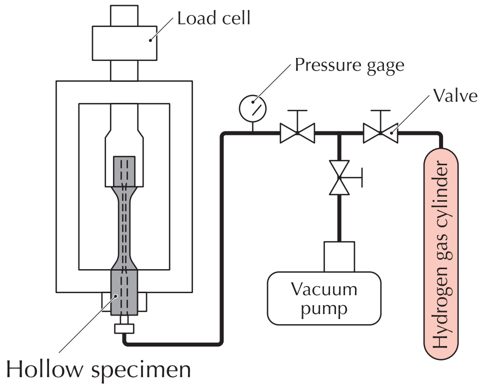
Please refer to the next pages for our proposed solutions.

Solution Various strength tests using hydrogen gas-sealed hollow specimens

Benefit 1 Realizes strength testing while exposing the material's surface to hydrogen gas

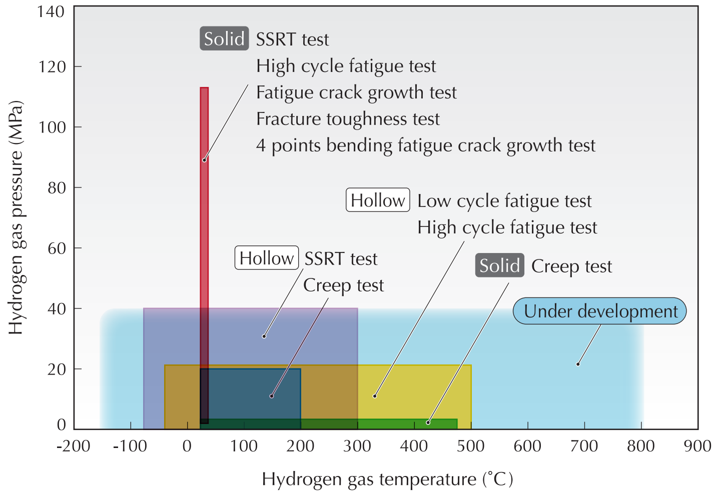
Benefit 2 Minimal amount of hydrogen gas
Ensures safety even if hydrogen gas diffuses outside the specimen

Benefit 3 Integration with KMTL's core testing technology
➡ Responds to diverse testing needs at an appropriate cost



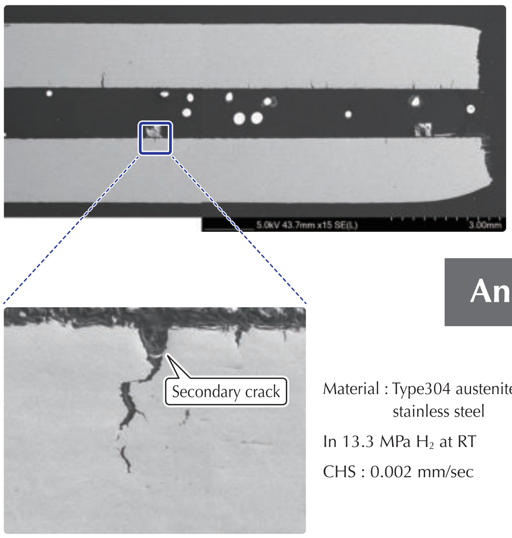
Our hydrogen material testing lineup

Specimen	Material Strength Tests	Pressure (MPa)		Temperature (°C)	
		Lower	Upper	Lower	Upper
Hollow	SSRT (Slow strain rate tensile) test	0.1	40	−80	300
	Creep test	0.1	20	23	200
	Low cycle fatigue test	0.1	20	−40	500
	High cycle fatigue test				
Solid	Creep test	0.1	0.1	23	475
	SSRT test	1	113	23	23
	High cycle fatigue test				
	Fatigue crack growth test				
	Fracture toughness test				
	4 points bending fatigue crack growth test				



EBSD analysis of fractured hollow specimens

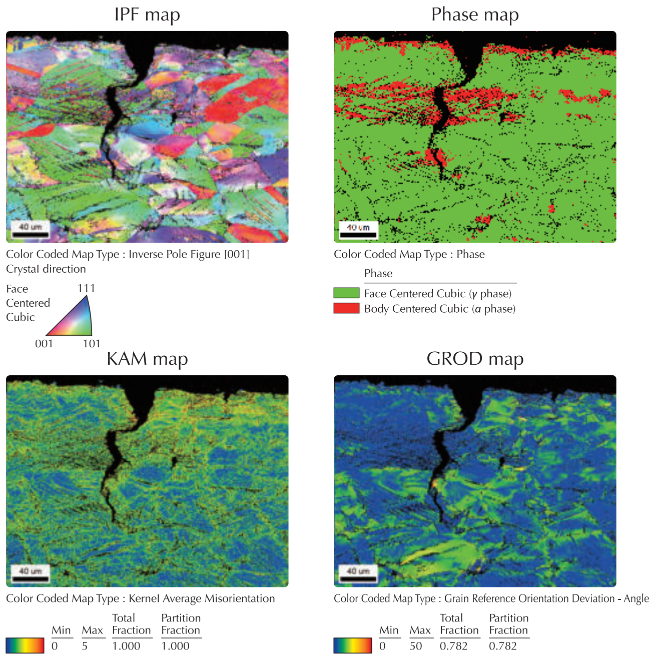
Cross-sectional SEM image near fracture



Magnified view of secondary crack

Material : Type304 austenite stainless steel
In 13.3 MPa H_2 at RT
CHS : 0.002 mm/sec

Analysis



Scope

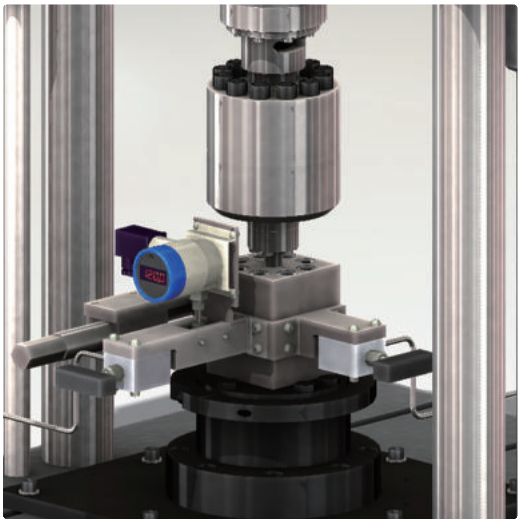
- **Fracture surface observation of a hollow specimen confirmed crack initiation from the inner surface exposed to hydrogen.**
- **Phase transformation from austenite to martensite was observed along the crack path.**
- **Type304, with relatively low austenite stability, exhibited lower ductility due to hydrogen exposure.**

SSRT, fatigue life and fatigue crack growth tests under 100 MPa class high pressure hydrogen gas environment

High pressure hydrogen gas vessels and piping are essential for hydrogen-energy related products and infrastructure, including FCV fuel tanks, hydrogen station refueling equipment, and large-scale hydrogen transportation and storage facilities. While these components enhance energy density and efficiency, they require specialized technologies and materials to handle the pressure safety. Strict safety standards and regulations necessitate extensive material testing in an high pressure hydrogen gas environment.

Kobe Material Testing Laboratory Co., Ltd. has established SSRT, fatigue life, and fatigue crack growth tests under high pressure hydrogen gas environments. This was achieved by mounting a specially structured small pressure vessel on a general-purpose hydraulic servo-type fatigue testing machine. This document introduces the technology.

New test method for small-sized specimens under high-pressure gaseous hydrogen

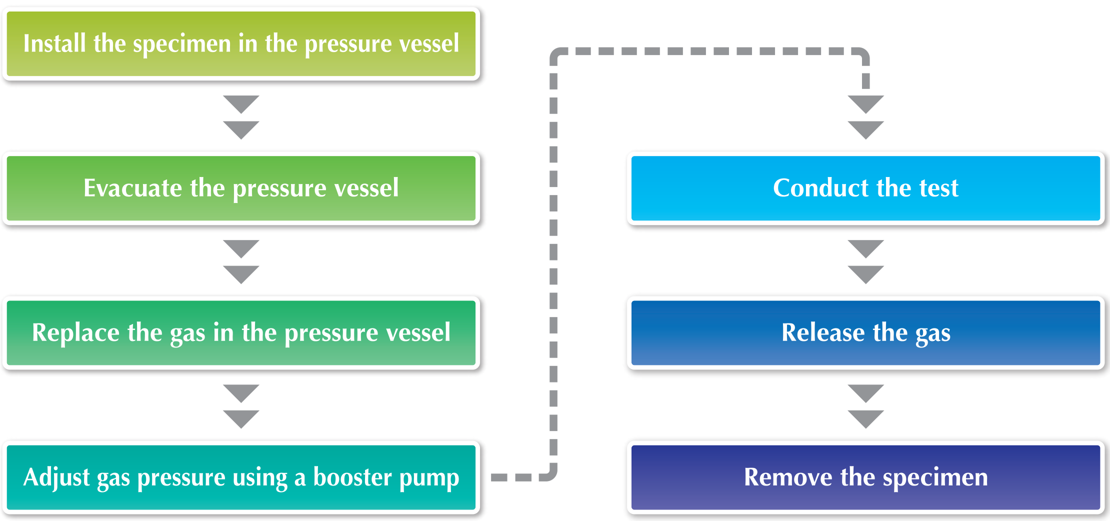


System for SSRT and fatigue life tests

System for fatigue crack growth and fracture toughness tests

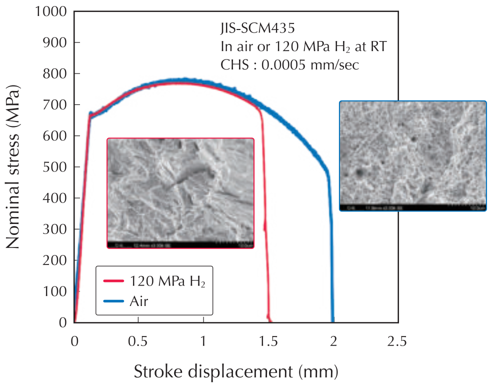
Our dedicated explosion-proof test room has been registered as a High-Pressure Gas Production Facility in Hyogo Prefecture.
Tests are conducted in full compliance with safety regulation by Japanese law, including gas detectors, forced exhaust fans, explosion-proof fluorescent lights, and regular voluntary inspections.

Test procedures



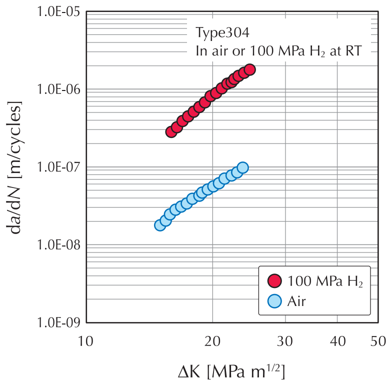
Testing performance in high pressure hydrogen gas environment

SSRT test result



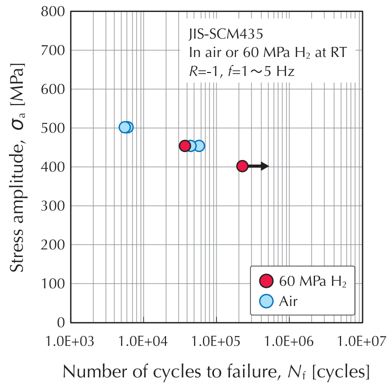
Ductility loss
Relative reduction of area, RRA : 0.6
Relative elongation, REL : 0.6

Fatigue crack growth test result



Accelerated crack growth rate
10 times faster
than that in air!

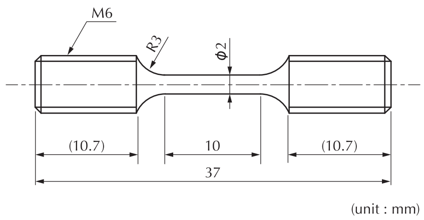
Fatigue life test result



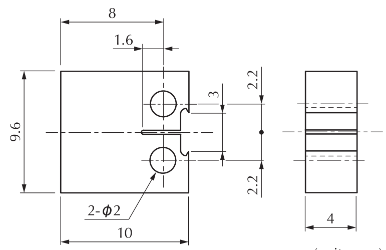
Impact in low cycle life regime
Minimal impact in high cycle life regime

Our standard test specimens in KMTL

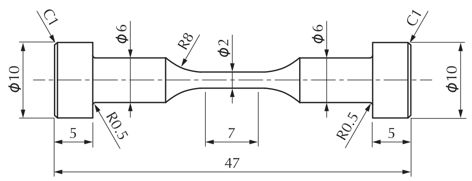
SSRT test specimen



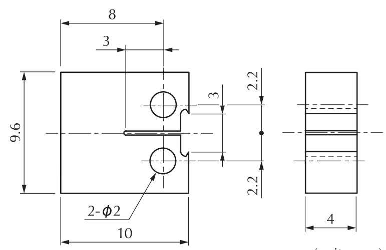
CT specimen for
fatigue crack growth test



Fatigue life test specimen

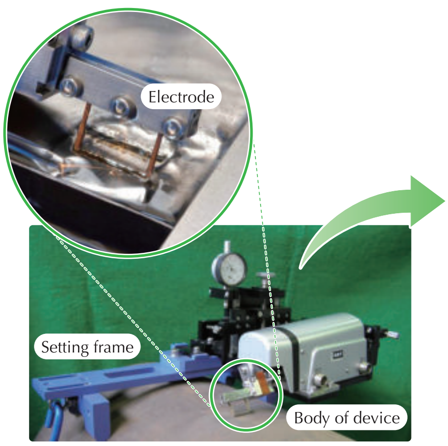


CT specimen for
fracture toughness test

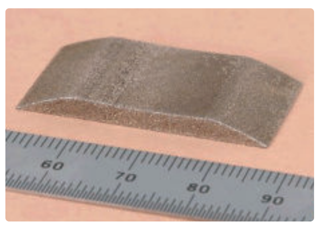


Core technology of KMTL miniature testing method

In all industrial fields, there is a need to take samples from actual products or components and conduct various material tests while simulating similar environments. However, limitations on the dimensions of materials that can be taken from actual machines often result in specimens that do not meet the size recommended by general test standards.
We specialize in miniature testing technology, where small samples taken from actual components are machined into specimens and subjected to a range of testing methods. This technology has been adapted for high-pressure hydrogen gas environment, resulting in our original test method described in this document. Please view our miniature testing services using the QR code.



Discharge sampling device for
taking miniature specimens



For more information

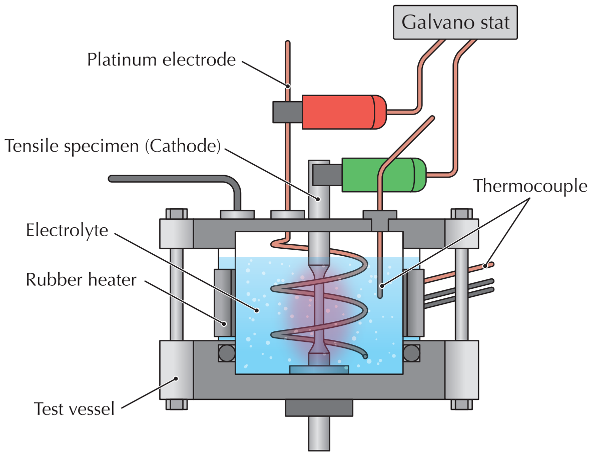
Continuous electrolytic H-charging for SSRT test

Electrolytic hydrogen charging is a method used to add hydrogen to metallic materials. The specimen is polarized in an aqueous solution, allowing hydrogen to penetrate the material from its surface and diffuse. It has been primarily used to study hydrogen embrittlement and delayed fracture of steels under corrosive environments. Hydrogen embrittlement tends to increase with the hydrogen concentration in the steel. Electrolytic hydrogen charging allows us to simulate hydrogen effects in actual environments to evaluate the hydrogen resistance of steel.

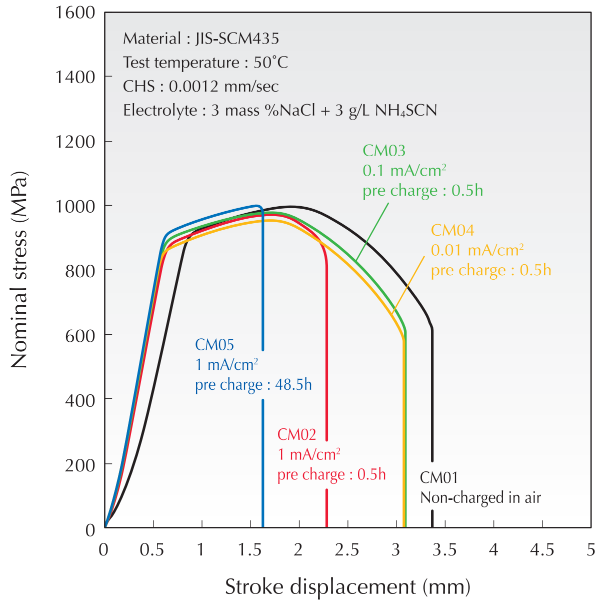
Kobe Material Testing Laboratory Co., Ltd. faced the challenge of hydrogen desorption during tensile tests in the atmosphere conducted after hydrogen charging. This document introduces our developed method for maintaining hydrogen charging during tensile tests, effectively solving this issue.

Experimental setup of the continuous electrolytic charging SSRT testing method

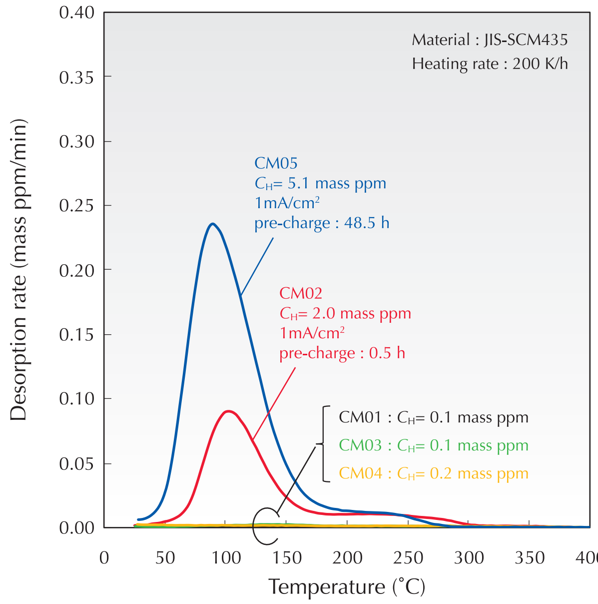
- SSRT testing is performed with a specimen hydrogen-charged using electrochemical reactions during the process.
- Optimal for evaluating hydrogen embrittlement, including delayed fracture in corrosive environments, with a wide range of parameter settings such as electrolyte composition, current value, and test speed.
- Hydrogen concentration analysis is conducted by immersing the fractured specimen in liquid nitrogen and store it without hydrogen desorption.
- Custom design and fabrication of experimental equipment are available.



Stress-stroke curves



Thermal Desorption Analysis (TDA) of hydrogen



~ Key discussions in this experiment ~

- Significant ductility reduction occurred due to hydrogen under high current density conditions.
- Longer pre-charging time caused more pronounced hydrogen embrittlement.
- Diffusible hydrogen was detected from the fractured specimen.
- Correlation exists between ductility reduction and hydrogen concentration.
- Evaluating concentration distribution in the test section requires the hydrogen diffusion coefficient.

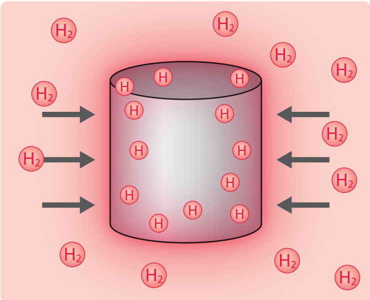
Measurement and analysis of hydrogen diffusivity, D

Hydrogen atoms in metals diffuse significantly faster than other solute atoms such as carbon. Many researchers identify hydrogen diffusion as a primary cause of hydrogen embrittlement.

Kobe Material Testing Laboratory Co., Ltd. studied for a method of experimentally obtaining the hydrogen diffusivity, D. Hydrogen concentration distribution analysis using FEM, based on this hydrogen diffusivity is shown as follows.

Procedure for hydrogen diffusivity by thermal desorption analysis (TDA)

Step I Electrolytic hydrogen charging



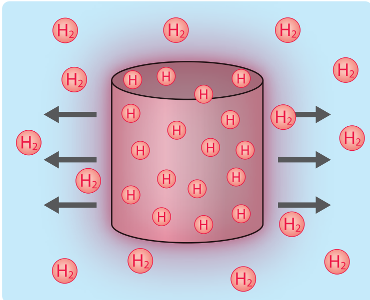
Electrolyte

- 3% NaCl + 3g/L NH_4SCN

Charging conditions

- Solution temperature : $T = 50$˚C
- Charging time : $t = 48$ h

Step II Hydrogen desorption curve acquisition by TDA



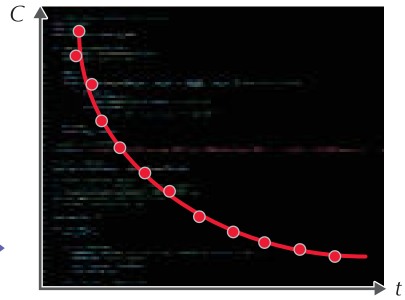
Pre-measurement tasks

- Mass measurement after sample thawing

Measurement conditions

- Equipment : JEOL GC-MS
- Holding temperature : $T = 24$, 50, 80 ˚C

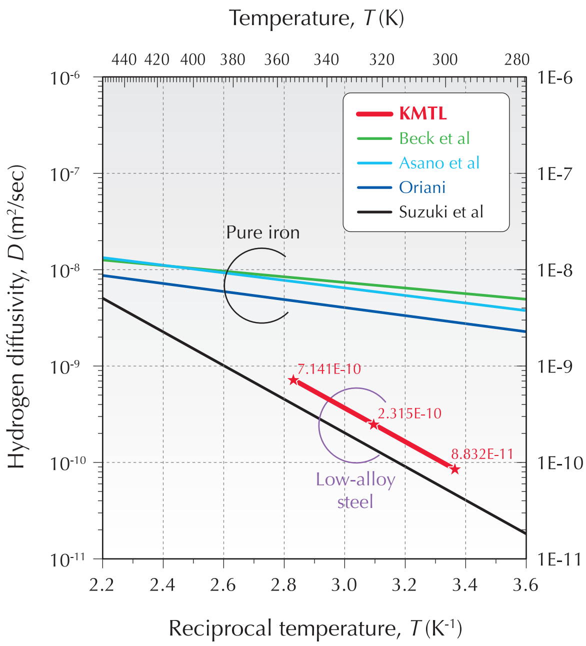
Step III Fitting analysis



Diffusion equation

- Reference : Acta Metallurgica, Vol.2, 2, 1954, 214-223
- Fitting : Least squares method
- Numerical calculation code : Python

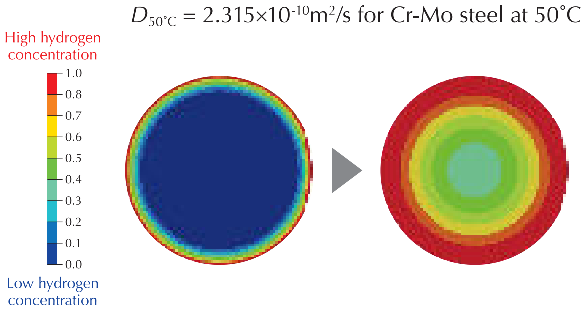
Fitting analysis results



Diffusion simulation by FEM analysis

Time history change of hydrogen concentration distribution in the cross-section of round bar specimens

Analysis conducted using the hydrogen diffusion coefficient $D_{50^\circ C} = 2.315\times10^{-10}$ m²/s for Cr-Mo steel at 50˚C



For more information

* This is an animation of the analysis results using FEM analysis software Abaqus 2021.

- **Hydrogen concentration saturation is reached in approximately 10 hours.**
- **Hydrogen charging of 0.5 hour results in a concentration gradient.**

Kobe Material Testing Laboratory Co., Ltd.

HP https://en.kmtl.co.jp/

TEL +81-79-435-5010

Contact