

Current status of round-robin tests for hydrogen material compatibility

Transmitted by Japan

6th Meeting of the informal working group on GTR No.13 (Phase 2)
18-20 June 2019 @ CATARC, Tianjin, China

Participant institutes, material, and remaining bars

Started in 2015

Japan



Sandia
National
Laboratories

USA



Materialprüfungsanstalt
Universität Stuttgart

Germany



Participation

KRISS

Korea



浙江大学

ZHEJIANG UNIVERSITY

China

in 2019

$\phi 25 \text{ mm} \times \text{L}500 \text{ mm} \times 12 \text{ bars}$



SUS316L (MPA)

Supplied numbers of bars

SNL	3 bars
MPA	2 bars
HYDROGENIUS	7 bars

Chemical composition and mechanical properties

Chemical composition (mass%)

Heat	C	Si	Mn	P	S	Ni	Cr	Mo	N
MPA	0.019	0.49	1.41	0.029	0.024	12.19	17.13	2.05	—
Requirement ¹⁾	≤0.03	≤1.00	≤2.00	≤0.045	≤0.030	12.00 ~ 15.00	16.00 ~ 18.00	2.00~ 3.00	—

1) JIS G 4303 (1981), “Stainless steel bars”

$$Ni_{eq} = Ni + 12.6C + 0.35Si + 1.05Mn + 0.65Cr + 0.98Mo = 27.2 \text{ mass\%}$$

Mechanical properties

Heat	$\sigma_{0.2}$ [MPa]	σ_B [MPa]	Elongation [%]	RA [%]	<i>HBW</i>
MPA	245	551	60	78	141
Requirement ¹⁾	≥175	≥480	≥40	≥60	≤167

1) JIS G 4303 (1981), “Stainless steel bars”

Test procedures of round-robin test

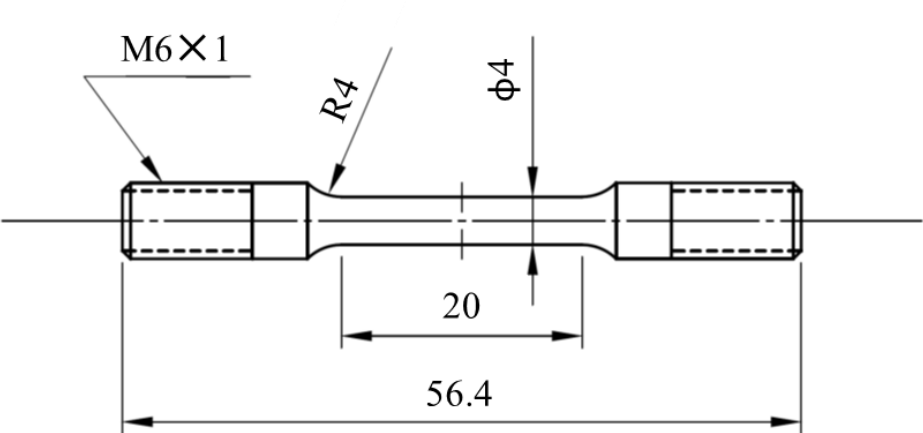
Test condition and required numbers of specimens for each test

Test	Environment	Condition	Number of specimens
SSRT at -40°C	0.1-MPa N ₂ gas	5×10^{-5} /s	3
	90-MPa H ₂ gas		3
Notch fatigue at $R = 0.1$ at -40°C	0.1-MPa N ₂ gas	1Hz, $\sigma_a = 200$ MPa $\sigma_{\max} = 444$ MPa, $\sigma_{\min} = 44$ MPa	3
	90-MPa H ₂ gas		3
Smooth fatigue at $R = -1$ at -40°C	0.1-MPa N ₂ gas	1Hz, $\sigma_a = 320$ MPa $\sigma_{\max} = 320$ MPa, $\sigma_{\min} = -320$ MPa	3
	90-MPa H ₂ gas		3

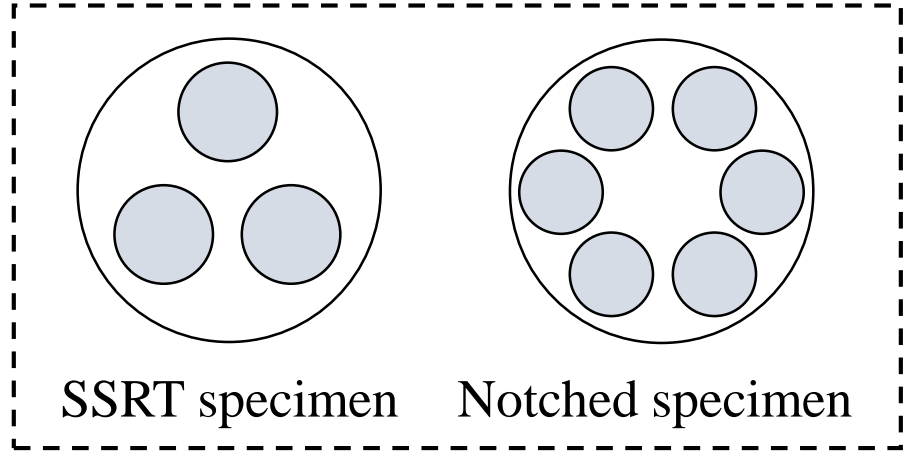
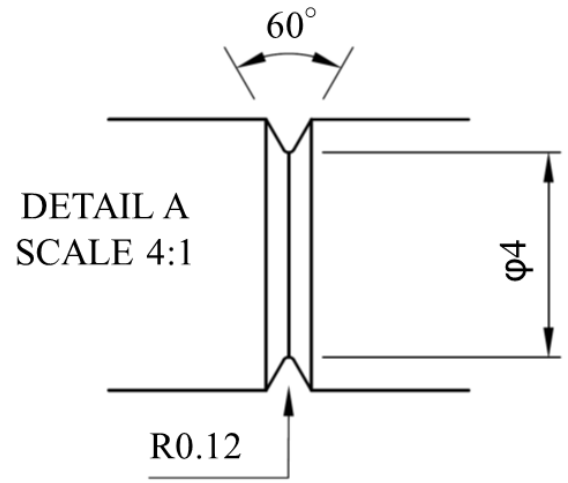
Manufacture of specimens

- ◆ The **SSRT** specimen was manufactured by **MPA**.
- ◆ The **circumferentially-notched** specimen was manufactured by **SNL**.
- ◆ The **smooth, round-bar specimen** was manufacture by **HYDROGENIUS**.

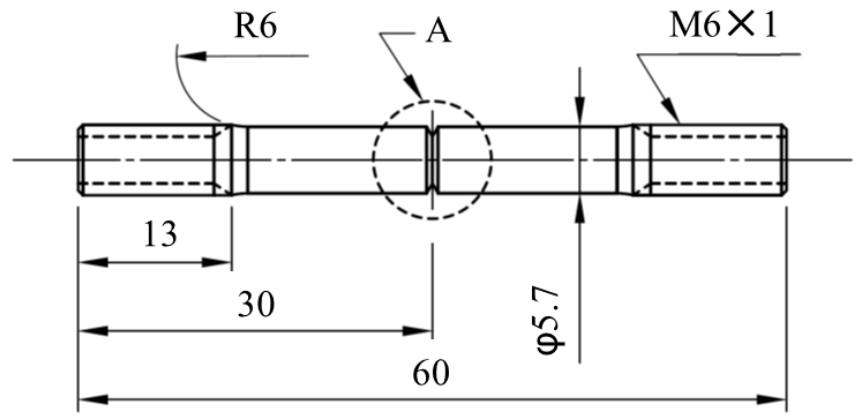
Specimen geometries (SSRT and notched specimens)



SSRT specimen
(MPA, ASTM E8 Specimen 4)



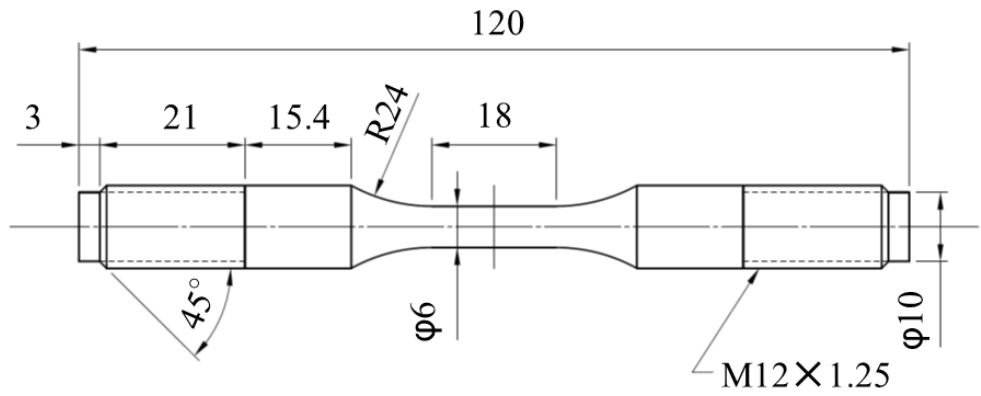
SSRT specimen Notched specimen



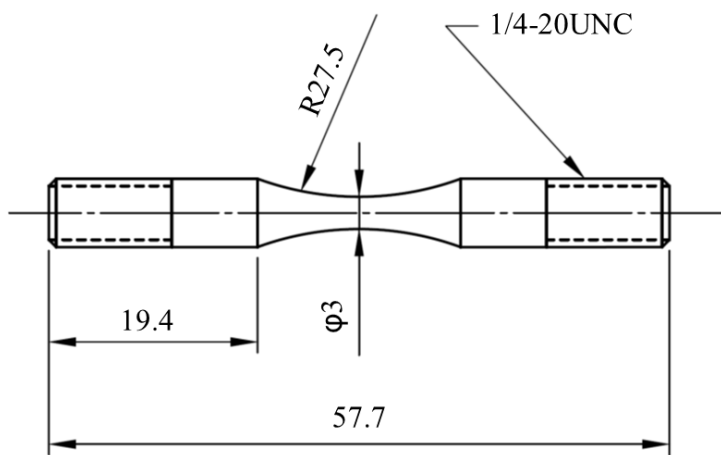
Circumferentially-notched specimen
(SNL, $K_t=4.1$)

Specimen geometries (smooth, round-bar specimens)

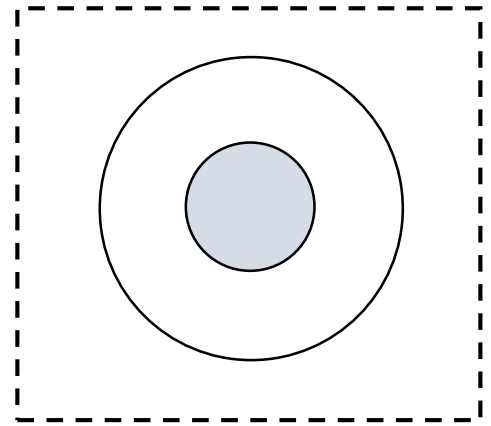
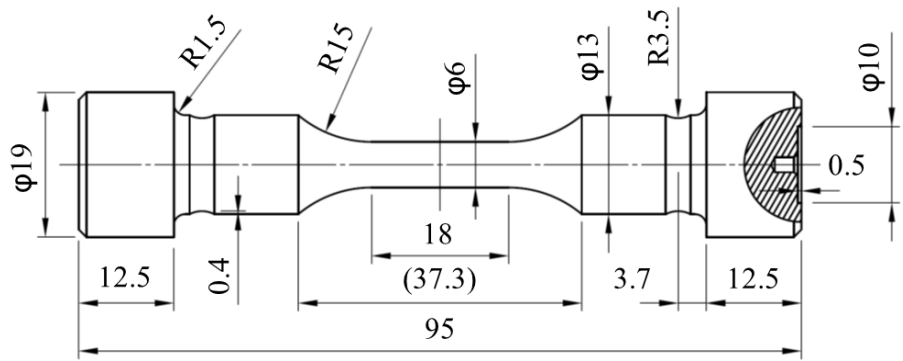
MPA



SNL

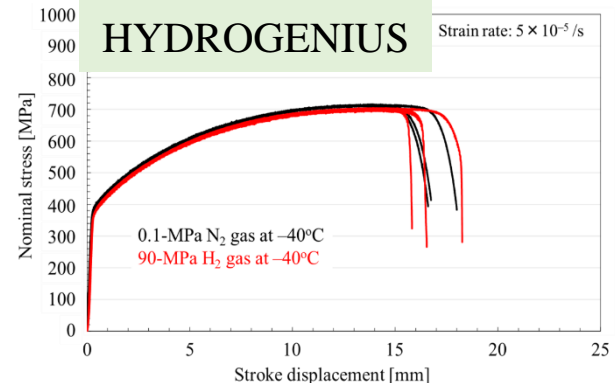
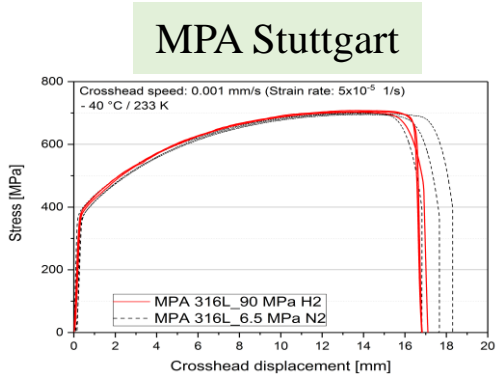
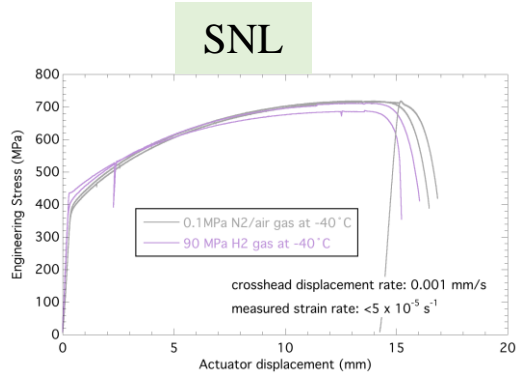


HYDROGENIUS



Smooth, round-bar specimens (manufactured by HYDROGENIUS)

Results of SSRT test (comparison SNL, MPA Stuttgart and HYDROGENIUS)

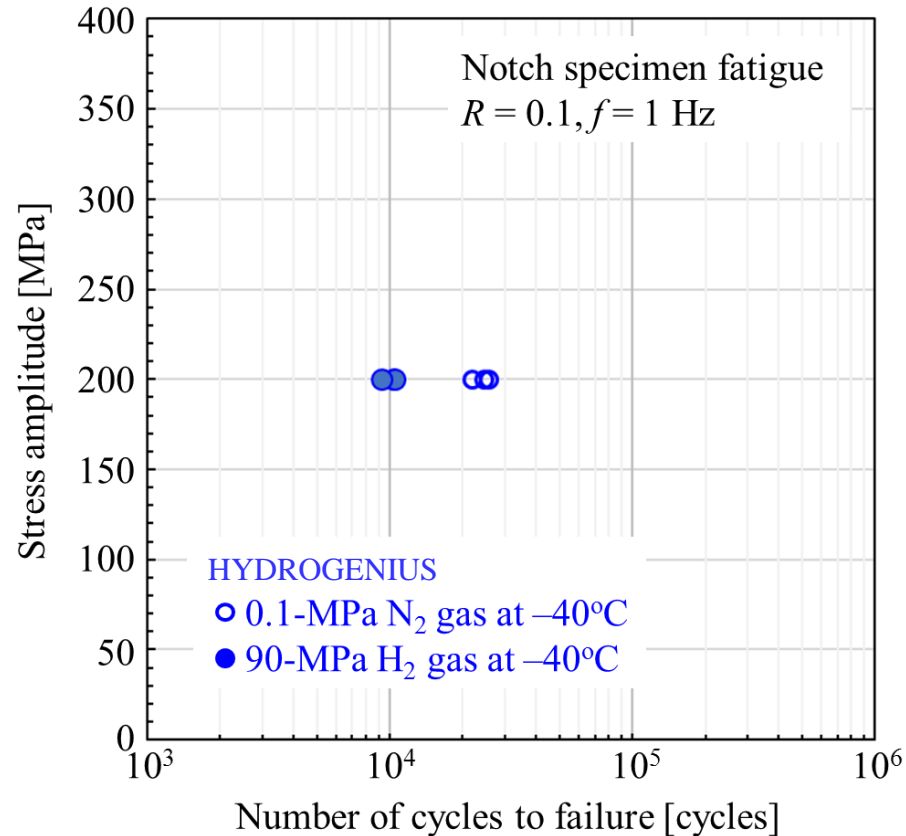


Test environment	Tensile strength [MPa]			Elongation [%]			Reduction in area [%]		
	SNL	MPA Stuttgart	HYDRO GENIUS	SNL	MPA Stuttgart	HYDRO GENIUS	SNL	MPA Stuttgart	HYDRO GENIUS
0.1 MPa N ₂ at -40° C (MPA:6.5MPa)	717	699	716	77	81.99	81	82	83.52	84
	719	696	716	77	87.95	84	83	85.6	83
	–	698	716	–	88.98	90	–	83.46	85
	Average	718	698	716	77	86.3	85	83	84.19
90 MPa H ₂ at -40° C	712* ¹	727	704	80* ¹	82.84	94	84* ¹	62.52	76
	689* ²	729	703	76* ²	79.95	79	69* ²	60.77	66
	–	–	699	–	–	75	–	–	58
	Average	701	728	702	78	81.40	83	77	61.65

(SNL) *¹ Pressure > 90 MPa
 *² Slightly overloaded prior to testing

◆ The experimental results from SNL, MPA Stuttgart and HYDROGENIUS were nicely consistent.

Results of notched specimen fatigue test (HYDROGENIUS)



Test data (HYDROGENIUS)

Test environment	Number of cycles to failure [cycles]
	Notch specimen fatigue : $\sigma_a = 200 \text{ MPa}, R = 0.1, f = 1 \text{ Hz}$
0.1-MPa N ₂ gas at -40°C	2.20×10^4
	2.57×10^4
	2.44×10^4
90-MPa H ₂ gas at -40°C	1.03×10^4
	1.04×10^4
	9.25×10^3

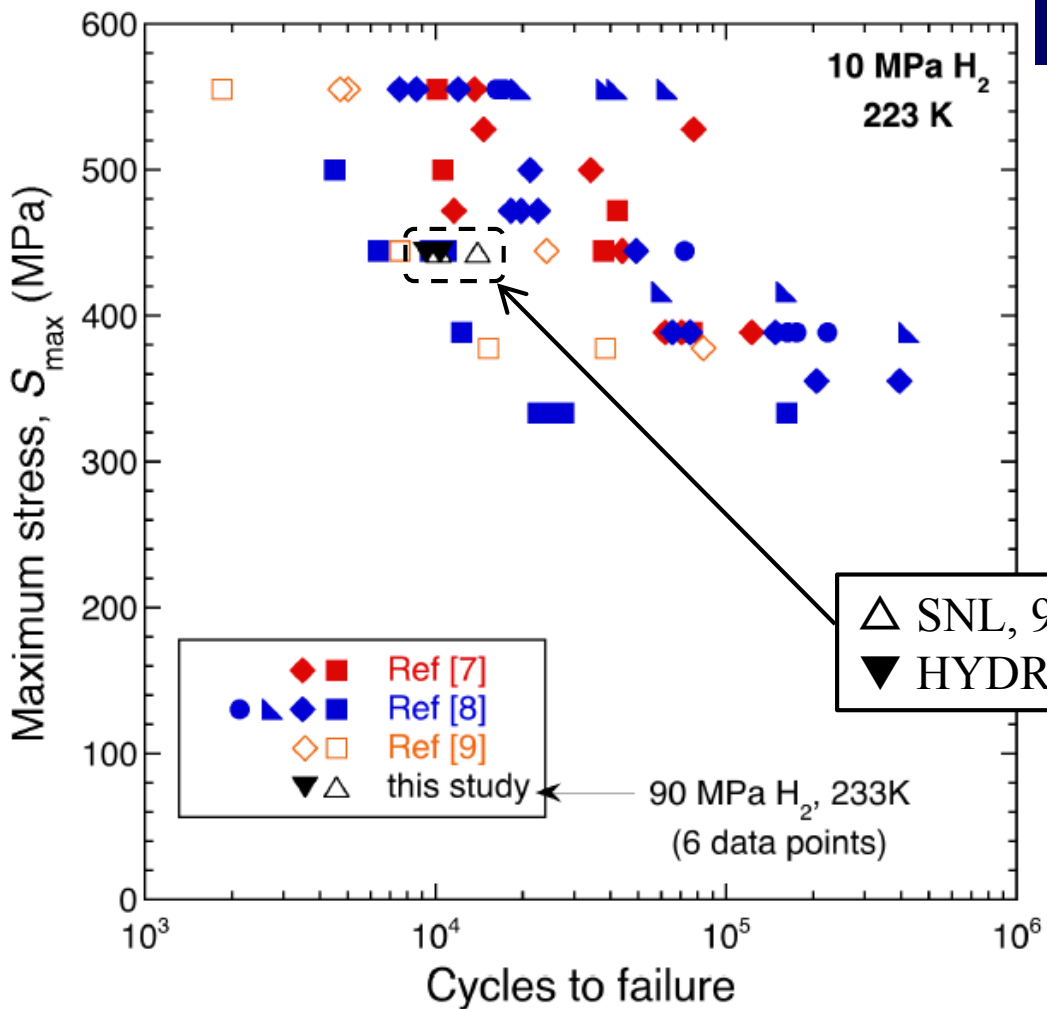
Notched specimen fatigue properties (HYDROGENIUS)

- ◆ In HYDROGENIUS, all the tests (3 tests at each environment) were finished.
- ◆ At -40°C, the notch fatigue life was shorter in 90-MPa hydrogen gas than 0.1-MPa nitrogen gas.

Results of notched specimen fatigue test

(comparison SNL and HYDROGENIUS)

Chris San Marchi et al.,
ASME PVP2018-84898

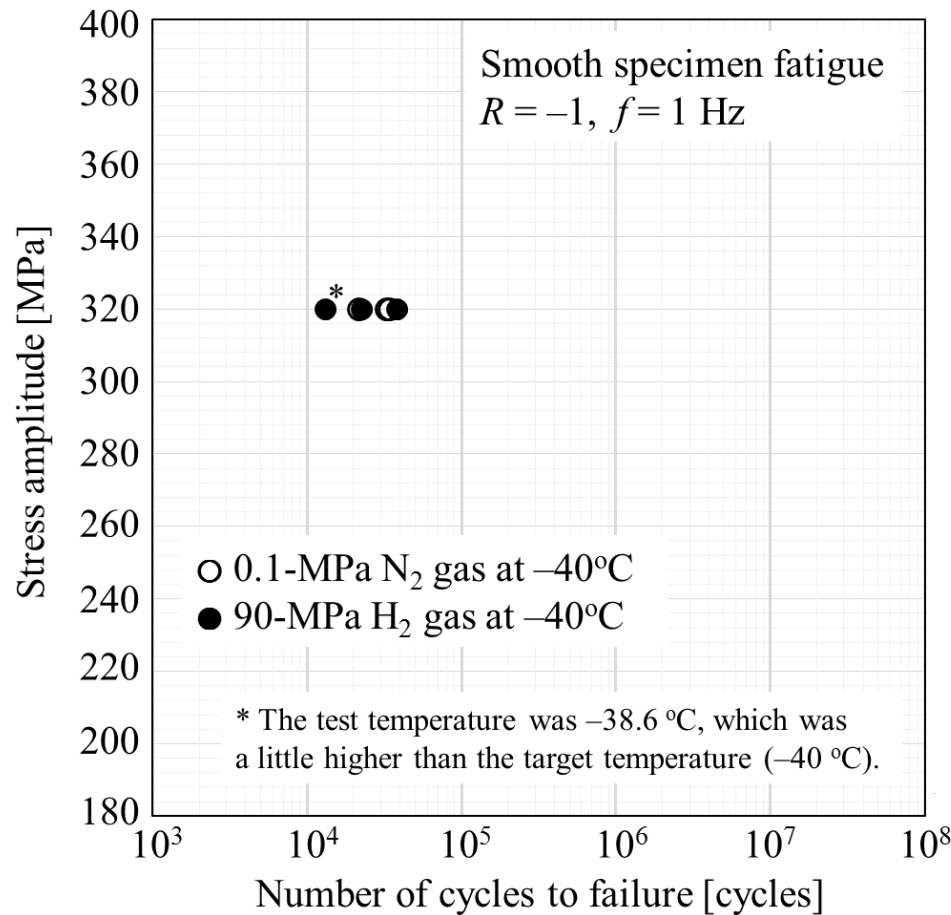


△ SNL, 90-MPa H_2 at $-40^\circ C$
▼ HYDROGENIUS

Notched specimen fatigue properties (SNL and HYDROGENIUS)

- ◆ In SNL, 3 tests in 90-MPa hydrogen gas were finished.
- ◆ The experimental results from SNL and HYDROGENIUS were nicely consistent.

Results of smooth specimen fatigue test (HYDROGENIUS)



Test data (HYDROGENIUS)

Test environment	Number of cycles to failure [cycles]
	Smooth specimen fatigue : $\sigma_a = 320 \text{ MPa}, R = -1, f = 1 \text{ Hz}$
0.1-MPa N ₂ gas at -40°C	3.29×10^4
	2.17×10^4
	3.41×10^4
90-MPa H ₂ gas at -40°C	2.26×10^4
	1.31×10^4 *
	3.83×10^4

Smooth specimen fatigue properties (HYDROEGNIUS)

◆ In HYDROGENIS, all the tests (3 tests at each environment) were finished.

Summary of current status of round-robin test

Current status of round-robin test

Institute	Environment	SSRT	Notched specimen fatigue	Smooth specimen fatigue
SNL	H ₂	2	3 (finished)	(declined)
	Inert	2	0	(declined)
MPA Stuttgart	H ₂	2	0	0
	Inert	3 (finished)	0	0
HYDRO GENIUS	H ₂	3 (finished)	3 (finished)	3 (finished)
	Inert	3 (finished)	3 (finished)	3 (finished)

Plan of round-robin tests

- ◆ At China and Korea, **notched specimen fatigue in H₂ gas (3 specimens)** and **smooth specimen fatigue in H₂ gas (at maximum, 3 specimens)** will be expected.
- ◆ Supply the specimen for SSRT from MPA Stuttgart ✘ Preferentially
- ◆ Supply the notched specimen from SNL
- ◆ Supply the raw material for the smooth specimen ($\phi 25 \times L275\text{mm}$) from HYDROGENIUS. The specimen geometry of the smooth specimen can be determined at each institute.

Notch fatigue at $R = 0.1$ at -40°C	0.1-MPa N ₂ gas	1Hz, $\sigma_a=200$ MPa	3
	90-MPa H ₂ gas	$\sigma_{\max}=444$ MPa, $\sigma_{\min}= 44$ MPa	3
Smooth fatigue at $R = -1$ at -40°C	0.1-MPa N ₂ gas	1Hz, $\sigma_a=320$ MPa	3
	90-MPa H ₂ gas	$\sigma_{\max}=320$ MPa, $\sigma_{\min}= -320$ MPa	3

Schedule to be expected

1. Supply the specimen and raw material to China and Korea
: will be Completed soon
2. Finished of Round Robin tests in H₂ gas : September, 2019 (hopefully)

**Thank you for
your kind attention**