

# **Introduction to GTR#13 Phase2 IWG - Discussed items in SAE -**

**(c) Requirements for material compatibility**

**Prepared by Technical Secretary**

GTR#13 Phase2 Informal WG : 17-19/Oct/2017@ Brussels

United Nations




**Economic and Social Council**

ECE/TRANS/WP.29/AC.3/49

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**5. Scope of work in Phase 2 should cover:**

- (a) Original items described in ECE/TRANS/WP.29/AC.3/17 shall be kept;
- (b) Potential scope revision to address additional vehicle classes;
- (c) Requirements for material compatibility and hydrogen embrittlement;** 
- (d) Requirements for the fuelling receptacle;
- (e) Evaluation of performance-based test for long-term stress rupture proposed in Phase 1;
- (f) Consideration of research results reported after completion of Phase 1 – specifically research related to electrical safety, hydrogen storage systems, and post-crash safety;
- (g) Consideration of 200 per cent Nominal Working Pressure (NWP) or lower as the minimum burst requirement;
- (h) Consider Safety guard system for the case of isolation resistance breakdown.

**The following two items are discussed in SAE FC Safety Task Force**

**【1】 Material compatibility test method** ( for austenitic stainless steels )

**【2】 Additional metal material test method** ( HG-SCC test method for aluminum alloy )

# 【1】 Material compatibility test method ( for austenitic stainless steels )

“Material compatibility ” issue has already been discussed  
at “SAE H2 Compatibility Expert meeting ” .

 **It is efficient if we can start GTR-Phase2 based on this discussion.**

Today ( GTR Phase2 Informal WG#1 @ Bruxelles ), I will present...

- **What is “SAE H2 Compatibility Expert meeting ”?**
- **Outline of “Material compatibility test method” at SAE**

# What is “SAE H2 Compatibility Expert meeting ” ?

## ■ Object

To Investigate H2 compatibility test method for metal material under high pressure H2 environment.

## ■ Framework

As an activity affiliated with SAE, metal material expert of the United States, Germany, and Japan has participated.

( Current target : To reflect H2 compatibility test method to SAE J2579 )

## ■ History

This meeting has been held  
10 times since Mar/2015.

(1) Mar2015

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(6) Feb2017@Kyushu,JPN

(7) Feb2017@Sandia,US

(8) May2017@Web meeting

(9) Jun2017 @Detroit, US

(10) Oct2017@Stuttgart@GER

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# What is “SAE H2 Compatibility Expert meeting” ?

## ■ Participant

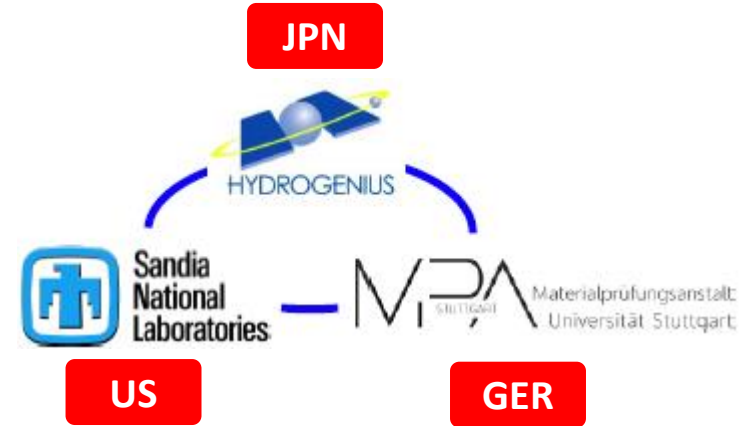
	<i>Material Expert</i>	<i>Automotive, etc</i>
<i>U.S. / Canada</i>	<p><b>Sandia National Lab.</b>                      ○○○○ △△△△△ (*Leader)                      ○○○○ △△△△△</p>	<p><b>SAE</b>                      ○○○○ △△△△△</p> <p><b>CSA</b>                      ○○○○ △△△△△</p> <p><b>DOE</b>                      ○○○○ △△△△△</p> <p><b>US Toyota</b>                      ○○○○ △△△△△</p>
<i>Germany</i>	<p><b>MPA Stuttgart</b>                      ○○○○ △△△△△</p>	<p><b>BMW</b>                      ○○○○ △△△△△</p> <p><b>Daimler</b>                      ○○○○ △△△△△</p>
<i>Japan</i>	<p><b>Kyushu Univ.</b>                      ○○○○ △△△△△, ○○○○ △△△△△</p> <p><b>AIST</b>                      ○○○○ △△△△△</p> <p><b>The University of Tokyo</b>                      ○○○○ △△△△△, ○○○○ △△△△△</p>	<p><b>JAMA</b>                      ○○○ △△△△, ○○○ △△△△ (Toyota)</p> <p>○○○○ △△△△△ (Honda)</p> <p>○○○○ △△△△△ (Suzuki)</p> <p><b>JARI</b>                      ○○○○ △△△△△, ○○○○ △△△△△</p>

■ Describe the details in the presentation slide

# Outline of “Material compatibility test method” at SAE

## ■ Current discussion item 1 : Round Robin

Phase1 2016 - 2017	Phase2 2018 - 2019	Phase3 2019 - 2020
<b>Round Robin</b>	Sensitivity Study of Test Parameters	“Testing Campaign” Material Validation
Timeline: ca. 12 months	Timeline: ca. 1.5 year	Timeline: ca. 2 years
<b>One Material:</b> 316 L SUS	> 1 Stainless Steel Sample (e.g. 3)	Multiple Materials (see B.2)
-Sample Probe Specification -SAE Round Robin Test Plan. -SAE to work to harmonize IPHE round robin.	-Parameters Sensitivity Test Study of each (e.g. stress, T,P) governing Mechanisms. -Different Samples Specification (R-1 smooth vs. R0.1 notched), etc.	-Use Modified SAE Testing Plan from Sensitivity Study as Basis
Goal: Qualification of Lab Testing Capability with Parameters and Material (Temperature, Pressure, Stress, etc.)	Goal: Determine “worst case” accelerated Test Plan and Acceptance Criteria based on Damage Analysis (with a realistic range of vehicle applications)	Goal: Share Testing Burden with multiple laboratories to validate a “complete set” of automotive Steels in H2.



### Participating institute:

- ✓ Sandia National Laboratories (SNL)
- ✓ MPA University Stuttgart (MPA)
- ✓ Kyushu University (KU)

### Round-robin tests in hydrogen-gas and inert environments:

- ✓ SSRT test of smooth, round-bar specimen
- ✓ Fatigue-life test of circumferentially notched specimen at  $R = 0.1$
- ✓ Fatigue-life test of smooth, round-bar specimen at  $R = -1$

### Material:

- ✓ Type 316L

### SSRT test:

All the institutes use the **same** specimen recommended from SNL.  
=> The specimens have been provided from **MPA**

### Fatigue-life test of circumferentially notched specimen at $R = 0.1$ :

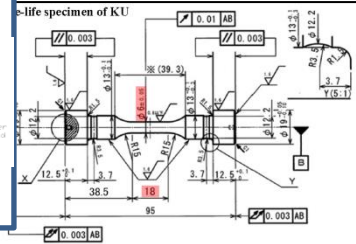
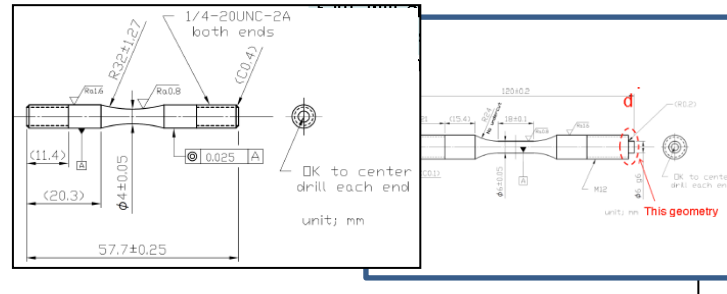
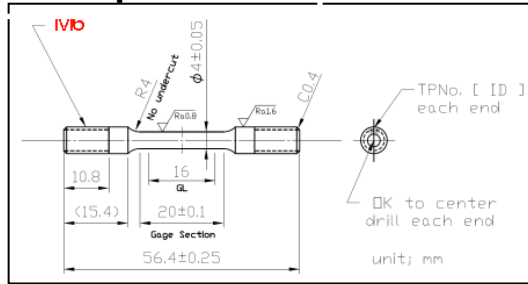
All the institutes use the **same** specimen recommended from SNL.  
=> The specimens have been provided from **SNL**

### Fatigue-life test of smooth, round-bar specimen at $R = -1$ :

Each institute uses **different** specimens.  
=> The specimens have been provided from **KU**

# Outline of "Material compatibility test method" at SAE

## SSRT specimen



**Table** Estimated schedule of round-robin tests @HYDROGENUS,JPN

			2017											
			5	6	7	11	9	10	11	12				
SSRT -40°C	Jigs	Machining												
	Specimen (MPA)	Agreement/Machining		★		●								
	'Test 3-1'	Inert												
	'Test 3-1'	90MPa H <sub>2</sub>												
R - 0.1 -40°C	Jigs	Machining												
	Specimen (SNL)	Agreement/Machining		★		●								
	'Test 3-1'	Inert												
	'Test 3-1'	90MPa H <sub>2</sub>												
R - 0.1 Smooth -40°C	Jigs	Agreement/Machining												
	Specimen (KU)	Machining												
	'Test 3-1'	Inert												
	'Test 3-1'	90MPa H <sub>2</sub>												
	Specimens for MPA and SNL	Drawing												
	Agreement		★											
	Machining													

**Round Robin (Cross-check of facility capability ) is due to be completed within 2017.**



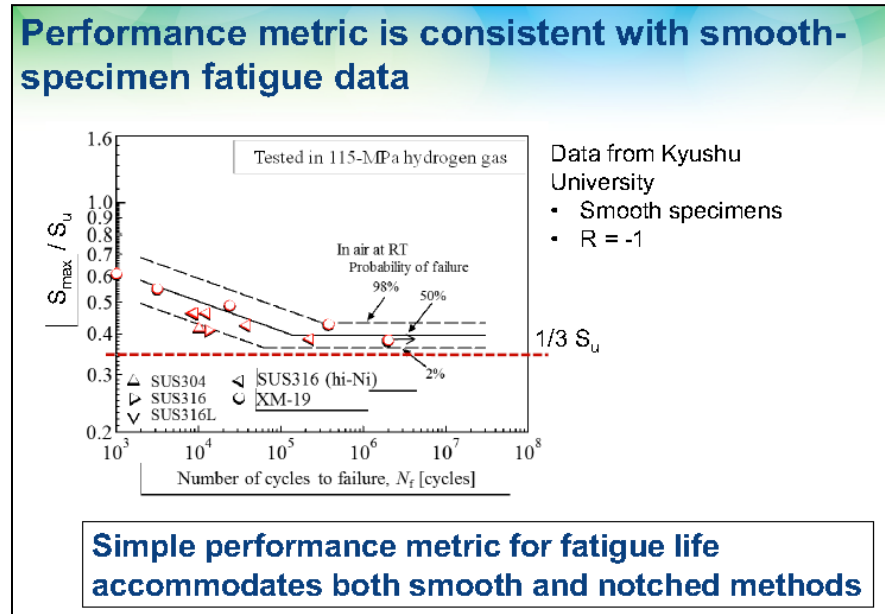
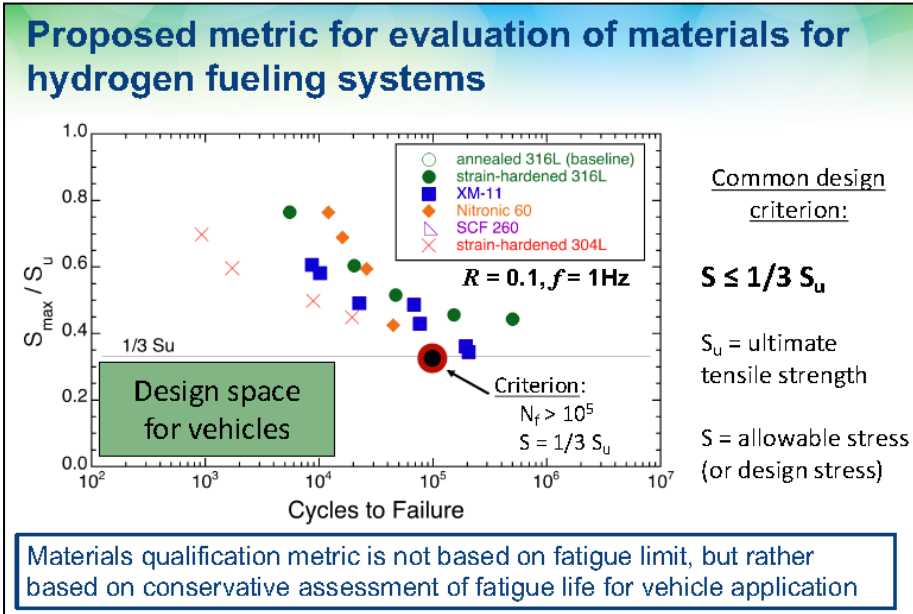
# Outline of "Material compatibility test method" at SAE

## Current discussion item 2 : Metric for evaluation

Phase1 2016 - 2017	Phase2 2018 - 2019	Phase3 2019 - 2020
Round Test	Test	"Testing Campaign" Material Validation
Timeline: ca. 1 year	Timeline: ca. 2 years	Timeline: ca. 2 years
One Material	Multiple Samples (e.g. 3)	Multiple Materials (see B.2)
-Sample Probe Specification -SAE Round Robin Test Plan. -SAE to work to harmonize IPHE round robin.	-Parameters Sensitivity Test Study of each (e.g. stress, T,P) governing Mechanisms. -Different Samples Specification (R-1 smooth vs. R0.1 notched), etc.	-Use Modified SAE Testing Plan from Sensitivity Study as Basis

**Metric for materials evaluation**

## Metric for materials evaluation ( under discussion )



Fundamental evaluation routing of CHSS material is under discussion.

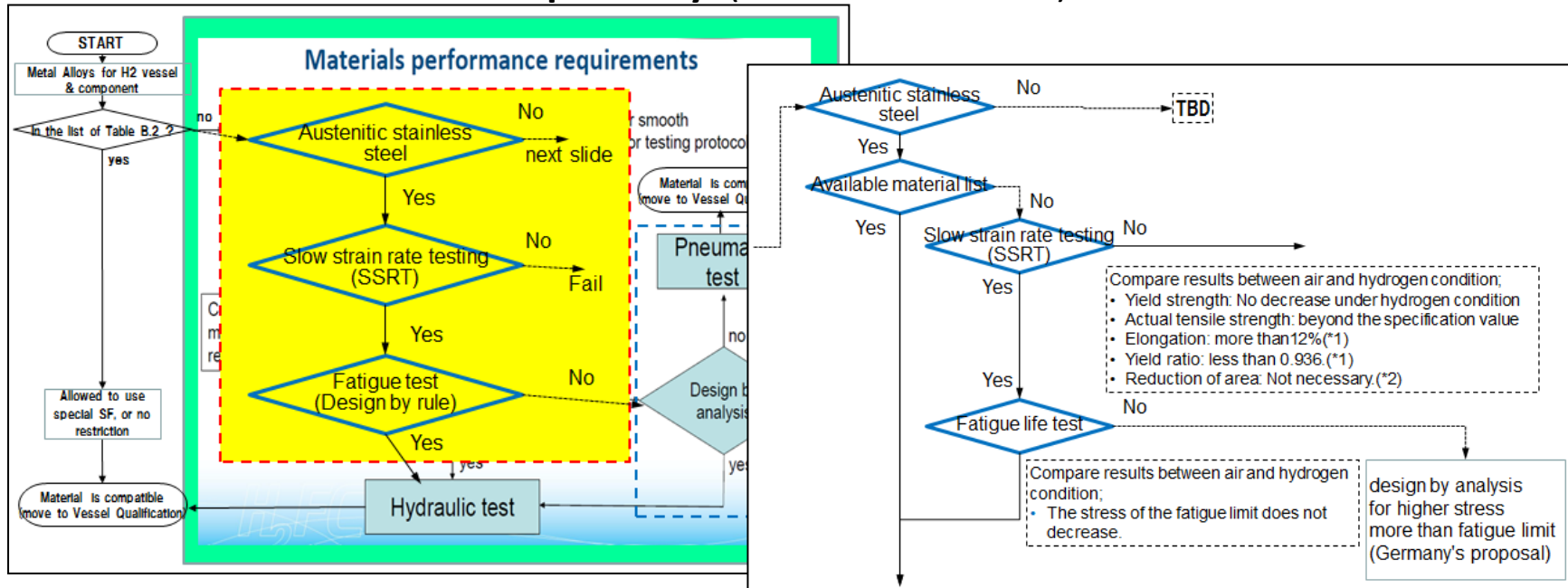
# Outline of “Material compatibility test method” at SAE

## ■ Current discussion item 3 : Verification flow

Phase1 2016 - 2017	Phase2 2018 - 2019	Phase3 2019 - 2020
Round Robin Test	“Testing Campaign” Material Validation	
Timeline	Timeline: ca. 2 years	
One Material	Multiple Materials (see B.2)	
-Sample Probe Specification -SAE Round Robin Test Plan. -SAE to work to harmonize IPHE round robin.	-Parameters Sensitivity Test Study of each (e.g. stress, T,P) governing Mechanisms. -Different Samples Specification (R-1 smooth vs. R0.1 notched) etc.	-Use Modified SAE Testing Plan from Sensitivity Study as Basis

Discussion about Verification flow

## ■ Verification flow of H2 compatibility ( under discussion )



Specific verification flow of H2 compatibility are also under discussion.

## 【2】 Additional metal material test method ( HG-SCC\* test method for aluminum alloy )

When using aluminum alloy for CHSS, it is necessary to consider corrosion under humid gas environment in addition to material compatibility .

- ➔ **The HG-SCC test method is scheduled to be published as an HPI\*\* standard and this test method is quoted in SAE J2579.**
- ➔ **Adding HG-SCC test method as one of the material requirements should be discussed in GTR-Phase2.**

**HG-SCC\*** : Humid Gas Stress Corrosion Cracking  
**HPI\*\*** : High Pressure Institute of Japan

## **【2】 Additional material compatibility test method**

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Today ( GTR Phase2 Informal WG#1 @ Bruxelles ), I will present...

- Why HG-SCC test method is necessary?
- Introduction: outline of HG-SCC test method for aluminum alloy

# Why HG-SCC test method is necessary?

## Does "Stress Corrosion Cracks" accelerate under humid gas environment?

➔ YES!

It has been found that stress corrosion cracks are greatly accelerated in some aluminum alloys under humid gas environment.  
( for example A7075)

North Atlantic Treaty Organization.  
Scientific Affairs Division 1971. J.C. Scully  
**The theory of stress corrosion cracking in alloys**

## Does humid gas environment exist in CHSS?

➔ YES!

If the hydrogen gas has a impurity water content of **5 ppm**, it corresponds to **RH 85% @ -7°C**.  
( 5ppm : regulated in ISO14687-2 and SAE J2719 as H2 fuel specification )

Zairyo-to-Kankyo,  
Vol.65, (2016), 432-437

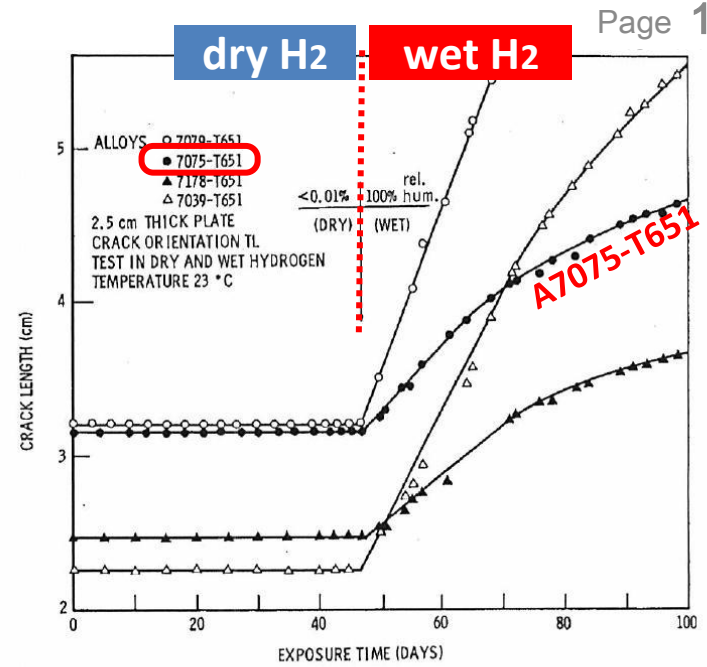
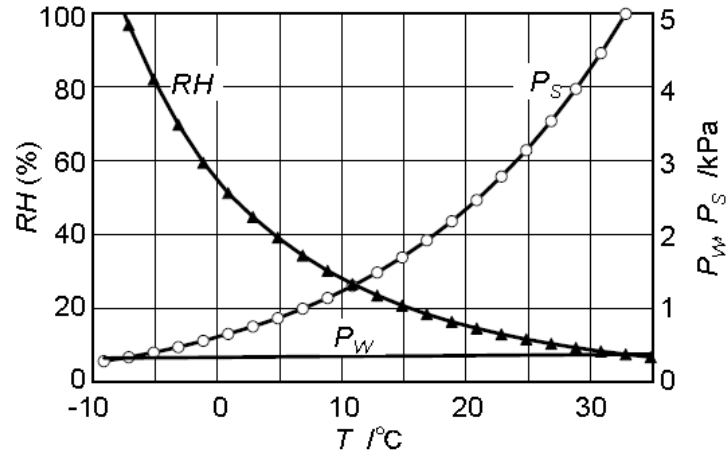


Fig. 9. Effect of humidity on SC crack growth of different aluminum alloys in hydrogen



**RH** : Relative humidity  
**Ps** : Saturation water vapor pressure  
**Pw** : Partial water vapor pressure

# Why HG-SCC test method is necessary?

## ■ Did accidents caused by stress corrosion cracking in humid gas environment exist in the past?

➔ **YES!**

Although not an example of CHSS, there are cases of accidents in containers for scuba diving in the past, according to the table below, there are many cases of accidents with A6351 material.

Accident Investigation Committee  
In KHK,JPN ( Nov.2001 )

**Accident cases of cylinder made of aluminum alloy for SCUBA**

From accident Investigation Committee in KHK (11.2001)

No.	Date (M/D/Y)	Place of occurrence	Human damage	Material	Manufacturing date	Duration of use	Damage condition	Handling condition
1	6.4.1994	Miami, Florida, USA	Severe:1	<b>A6351</b>	11.1982	11.5 years	Rupture	During refilling
2	7.1996	Alabama, USA	Unknown	<b>A6351</b>	Unknown	Unknown	Rupture	During refilling
3	5.30.1997	Vestfold, Norway	None	A5283	1973	24 years	Rupture	During storage
4	1.1998	New South Wales, Australia	Unknown	<b>A6351</b>	10.1982	15.3 years	Rupture	During storage
5	2.1.1998	Riviera Beach, Florida, USA	Severe:1 Slight :2	<b>A6351</b>	Unknown	Over 10 years	Rupture	During refilling
6	8.1998	Tairua, New Zealand	Injury	<b>A6351</b>	10.1980	18 years	Rupture	During refilling
7	12.1998	Tampa, Florida, USA	None	<b>A6351</b>	Unknown	Unknown	Rupture	During refilling
8	3.2000	Key Largo, Florida, USA	Severe:1	<b>A6351</b>	1987	13 years	Rupture	During refilling

# Why HG-SCC test method is necessary?

- Therefore, when applying aluminum alloys to CHSS, As one of the material requirements, stress corrosion cracking under humid gas environment should be considered.
- The HG-SCC test method was discussed in Japan and will be published in February 2018 as HPI standard.
- Also, this HG-SCC test method is quoted in SAE J 2579.



**Today, I will present outline of  
“HG-SCC test methods for aluminum alloys “**

Unfortunately, until February 2018, it is difficult to disclose the full text of the HG-SCC test method. I would like to focus on the summary today.

## Standard Test Method for Humid Gas Stress Corrosion Cracking of Aluminum Alloys for Compressed Hydrogen Containers

### ■ Section 1 : Scope

This standard specifies the test method for humid gas stress corrosion cracking (HG-SCC) and the applicability criterion of aluminum alloys for compressed hydrogen containers for automotive use.

### ■ Section 4 : Principle

A fatigue pre-cracked specimen is loaded by a constant-load or constant-displacement method to a  $K_{IAPP}$  equal to a defined value. Then, the specimen is maintained in the loaded state at prescribed temperature for a prescribed duration. After the test duration, the specimen is examined as to whether or not the cracking has extended from the initial fatigue pre-crack. If the crack extension length does not exceed a prescribed value, the material of the specimen is considered suitable for compressed hydrogen containers as far as the required resistance to crack extension under loading is concerned.



## ■ Specimen configurations

■ Describe the details  
in the presentation slide

Proportional dimensions and tolerances of compact specimen

Knife edge for location of displacement gauges

## ■ Test Method (Loading)

■ Describe the details  
in the presentation slide

## ■ Test environment and period

■ Describe the details  
in the presentation slide

## ■ Qualification of materials

Thank you !!