Proposal HG-SCC test method for aluminum alloys

HG-SCC:Humid Gas Stress Corrosion Cracking

Transmitted by Japan

10th Meeting of the informal working group on GTR No.13 (Phase 2)
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Background

- Some kinds of aluminum alloy show stress corrosion cracking (SCC) in humid gas conditions.
- This type of SCC occurs both outside and inside of containers under the presence of H_2O.
- Materials with higher SCC susceptibility will be selected by using HG-SCC test.
- In the case of the expansion of available material in the future, the safety material will be identified by applying HG-SCC test to new alloy.

Testing Protocol (Material)

1.1 The materials are wrought aluminum alloy products.

1.2 The material under consideration shall be defined by a materials specification The materials specification shall include requirements for the following:

1. allowable compositional ranges;
2. specified minimum yield strength, Sy
3. specified minimum tensile strength, Su
4. specified minimum tensile elongation, El

1.3 Either the materials manufacturer’s certification or equivalent testing performed in air at room temperature may be used to verify that the material meets the specification.

Testing Protocol (Environment)

2.1 Temperature: 298 K $\pm$ 5 K

(at room temperature)

2.2 Atmosphere and humidity: 85 % or higher in relative humidity and no generation of dew in air

2.3 Test period: 90 days

(in accordance with B6.6 of ISO7866:2012)

Testing Protocol (Test specimen)

3.1 Test specimen : One of the specimen geometries, or a combination of them, shall be used for test.

1) Compact specimen of ISO 7539-6:2011

2) Single edge bend specimen (SE specimen or cantilever bend specimen of ISO 7539-6:2011)

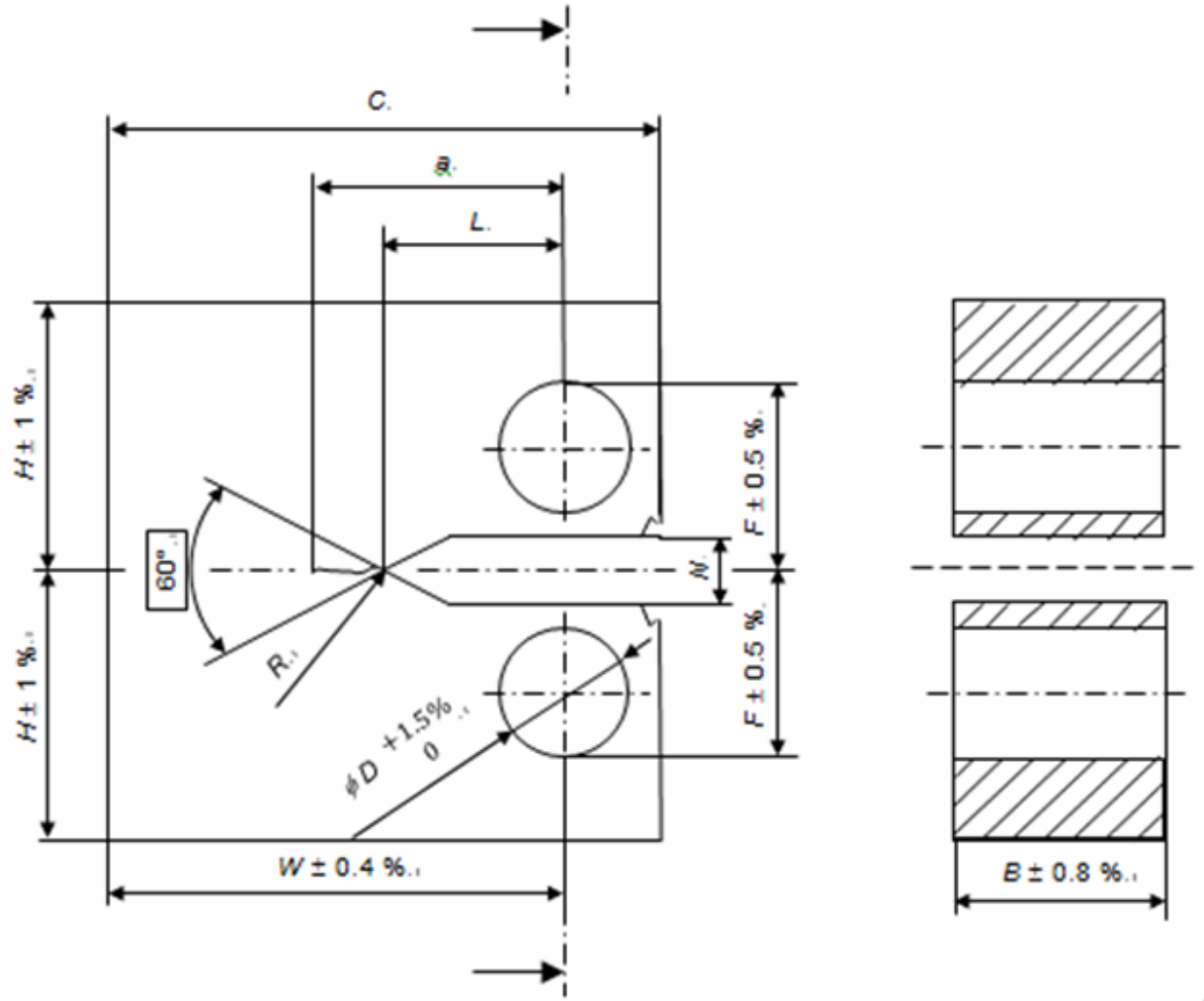
3) Double-cantilever-beam specimen (DCB specimen) of ISO 7539-6:2011

4) Modified wedge-opening-load-specimen (modified WOL specimen) of ISO 7539-6:2011

5) C-shaped specimen of ISO 7539-6:2011

3.2 Specimen orientation: the orientation of specimen sampling shall be Y-X orientation. Other orientation may be added when necessary.

Testing Protocol (Test specimen)



Example of specimen (CT)

Testing Protocol (Pre-crack and Load)

3.3 Fatigue pre-crack shall be introduced in accordance with class 6 of ISO 7539-6

3.4 A load is applied under constant load or constant displacement conditions.

The value of K_{IAPP} obtained by the following equation given in B.6.2 of ISO 7866:2012 is loaded.

$$K_{IAPP} = 0.056\sigma_{0.2}$$

For constant displacement condition, the load shall be measured after the 90-day test period.

Testing Protocol (Measurement crack length)

3.7 Measurement of crack length: After breaking of the specimen, the crack length shall be measured with an accuracy within ±0.01 mm.

1) effective crack length including the fatigue pre-crack a_{pre}

2) effective crack length up to the tip of the HG-SCC crack a_{scc}

Validity and criteria of test

Table 1 Qualification of materials

Case	Crack extension	K_{IA} versus K_{IAPP}	Judgment *
I	$(a_{scc} - a_{pre}) \leqq 0.16$ mm	$K_{IA} < K_{IAPP}$	invalid
II		$K_{IA} \geqq K_{IAPP}$	pass
III	$(a_{scc} - a_{pr}) > 0.16$ mm	$K_{IA} \leqq K_{IAPP}$	fail
IV		$K_{IA} > K_{IAPP}$	invalid

A minimum of three valid specimens must pass this test.
(Crack extents less than 0.16mm)

Plan of verification test (in humid hydrogen gas environment)

Table 2 Summary of test conditions and Requirement

Load	Constant load or Constant displacement
Temperature	298 K±5 K
Atmosphere and humidity:	Air (85 % of higher in relative humidity)
Number of specimens	3 (valid)
Test period	90 days
Criteria	$(a_{scc} - a_{pre}) \leqq 0.16$ mm $K_{IA} \geqq K_{IAPP}$

Rationale (Environment)

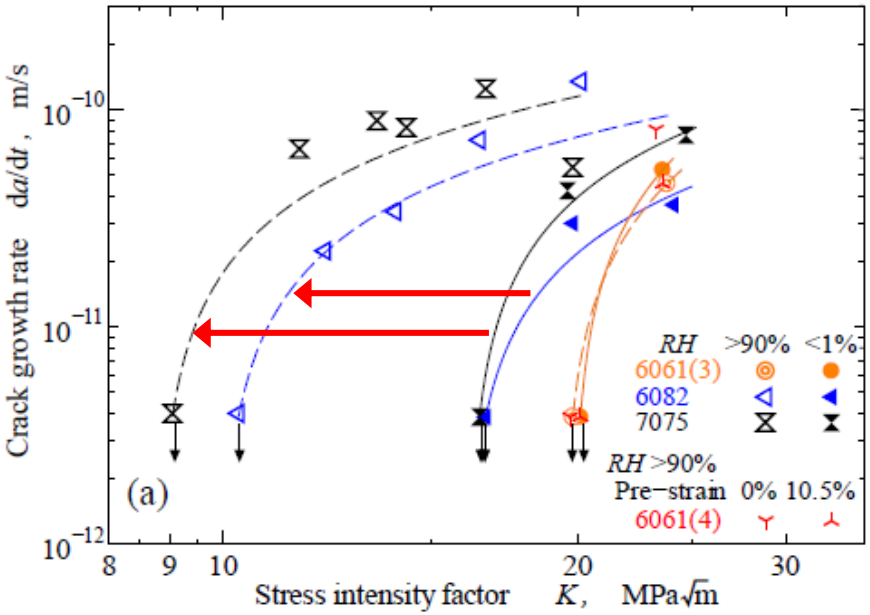
Test temperature

- The environmental temperature range for the vehicle is generally considered to be 233 K to 358 K (-40˚C to +85˚C).
- Susceptibility for SCC at cold temperature is low, the test temperature for this test shall be at room temperature (293±5K).

Rationale (Environment)

Atmosphere and humidity

- The atmospheric condition for this test is in air.
- HG-SCC is reduced in dry conditions, and 85% of humidity is required for this test.
- If the dew condensation water exists on the specimen, preferential corrosion occurs, during the test.



Ogawa. T, et al. Effect of Chemical Composition and Relative Humidity on the Humid Gas Stress Corrosion Cracking of Aluminum Alloys.
Pressure technology, High Pressure Institute of Japan (2018)

Rationale (Mechanics)

Test specimen

- Specimens for this test were cut from the wrought aluminum alloy products (plate, extruded and forged products).

  →It is difficult to cut the specimens from pressure vessels

- The face of specimen shall be processed to make the crack detectable and its length measurable.

Rationale (Mechanics)

Loads apply

- Both constant load condition and constant displacement condition is permitted in this test.
- A constant load condition is preferable to a constant displacement condition in this test.
- it seems that there is no difference in both condition when cracks do not propagate.

If the monitored load is less than 95% the test specimen should be rejected without waiting for the final qualification of materials. It has been confirmed that the crack length extension by HG-SCC exceed 0.16 mm

Rationale (Crirerion)

Acceptance Criterion

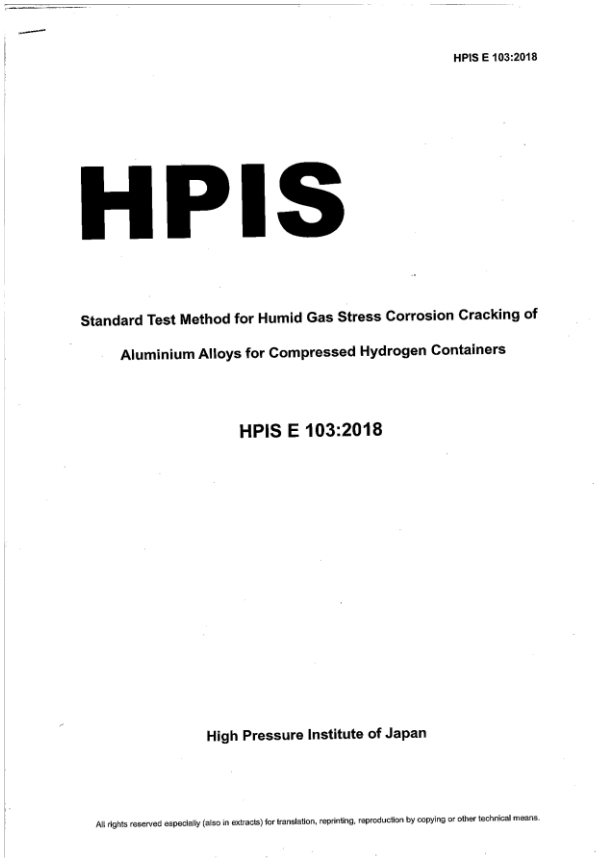
The crack extension of 0.16mm in 90days means that crack growth rate is less than $2X10^{-11}$m/s

➡ This value is lower than general SCC criteria of 10^{-10}m/s.

Appendix

This HG-SCC test method is based on HPIS E 103:2018.

(HPI : High Pressure Institute of Japan)



HPIS E 103:2018

HPIS

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

HPIS E 103:2018

High Pressure Institute of Japan

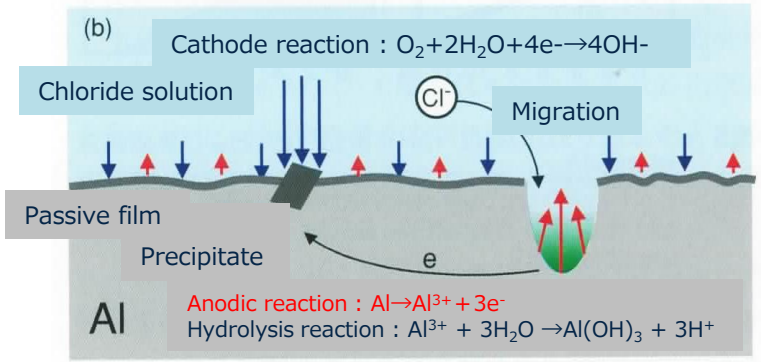
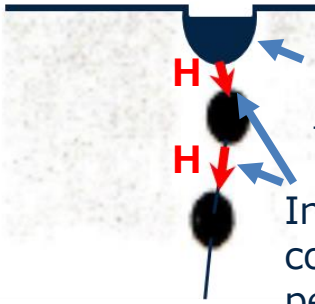
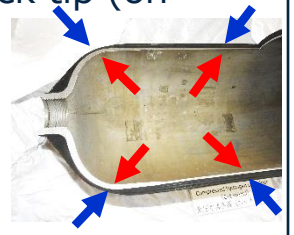
http://www.hpij.org/publication/?action=common_download_main&upload_id=1946

Thank you for your kind attention

Understanding on corrosion phenomena of aluminum alloys

■ 2 types of corrosion phenomena in aluminum alloys

SLC : Sustained Load Cracking
SCC : Stress Corrosion Cracking

Type	Anodic dissolution	SCC in humid gas environment
Principle	Electrochemical corrosion by salt water	SCC by the reaction of metallic Al and H_2O
		 Hydrogen atom generated by reaction of water on fresh metallic surface Intergranular cracking by continuous hydrogen atom penetration
Reaction	Anodic reaction : $Al\rightarrow Al^{3+}+3e^-$ Cathode reaction : $O_2+2H_2O+4e^-\rightarrow 4OH^-$	$2Al+3H_2O\rightarrow Al_2O_3+$**6H**
Characteristics	• Need oxygen and solution • Need Cl^- (break passive film) • Not occur in high pressure H_2 (no oxygen and no solution) ⇒ **Occur only outside of containers** 	• Occur under the presence of H_2O • Crack growth by accumulation of hydrogen atoms at the crack tip (on fresh metallic surface), not by dissolution of metal into ion ⇒ **Occur both outside and inside of containers** 
Evaluation	Current test method applied by each car OEM	※**HG-SCC test method (Improved SLC test) proposed by Japan for GTR13**

※ HG-SCC test method is different from H_2 compatibility test method in high pressure hydrogen gas environment.