• Rationale for humid gas stress corrosion cracking test method proposed for aluminum alloys

The compressed hydrogen storage and containment systems must be compatible with gaseous hydrogen over the entire applicable pressure and temperature ranges. Hydrogen embrittlement is a major problem for materials used in these systems. Aluminum alloys show good hydrogen embrittlement resistance and are possible materials for this system. However, some kinds of aluminum alloy show stress corrosion cracking (SCC) in humid gas conditions. The difference in mechanism of anodic dissolution type SCC and humid gas SCC(HG-SCC) is shown in Table 1.

Туре	Anodic dissolution	SCC in humid gas environment
	Electrochemical corrosion by salt water	SCC by the reaction of metallic AI and $\rm H_2O$
Princi ple	(b) Cathode reaction : $O_2+2H_2O+4e-\rightarrow 4OH$ - Chloride solution CP Migration Passive film Precipitate e Al Anodic reaction : $AI\rightarrow AI^{3+}+3e^{-}$ Hydrolysis reaction : $AI^{3+}+3H_2O\rightarrow AI(OH)_3 + 3H^{+}$	Hydrogen atom generated by reaction of water on fresh metallic surface Intergranular cracking by continuous hydrogen atom penetration
Reacti on	Anodic reaction : $AI \rightarrow AI^{3+} + 3e^{-}$ Cathode reaction : $O_2 + 2H_2O + 4e^{-} \rightarrow 4OH^{-}$	2AI+3H <sub>2</sub> O→Al <sub>2</sub> O <sub>3</sub> + <b>6H</b>
Chara cterist ics	<ul> <li>Need oxygen and solution</li> <li>Need Cl<sup>-</sup> (break passive film)</li> <li>Not occur in high pressure H<sub>2</sub> (no oxygen and no solution)</li> <li>⇒ Occur only outside of containers</li> </ul>	<ul> <li>Occur under the presence of H₂O</li> <li>Crack growth by accumulation of hydrogen atoms at the crack tip (on fresh metallic surface), not by dissolution of metal into ion</li> <li>⇒ Occur both outside and inside of containers</li> </ul>
Evalu ation	Current test method applied by each car OEM	*HG-SCC test method (Improved SLC test) proposed by Japan for GTR13

## Table 1 mechanism for SCC in humid gas environment

The outside of vessel is generally exposed to humid condition and impurity water is present in hydrogen gas. Therefore, this type of SCC occurs both outside and inside of containers under the presence of H<sub>2</sub>O. The crack growth test by constant load or constant displacement method is intended to demonstrate that the materials show adequate SCC resistance for anticipated service conditions. This kind of cracking was observed in scuba diving container. Seven accidents of the 6351 alloys scuba container seem to be caused by HG-SCC occurred in the USA, Australia, and New Zealand. Therefore, 6351 material have not used for this container and material changed to 6061 alloy. HG-SCC susceptibility depends on the chemical composition and the heat treatment condition. In the HG-SCC test specified in HPIS E103:2018 which is modified ISO 7866, 6351 alloy and 6082 alloy whose chemical composition is similar to 6351 were failed. On the other hand, 6061 alloy was passed in this test. [G. Itoh, A. Kurumada, S. Aoshima and T. Ogawa, Effect of alloying composition on humid-gas stress corrosion cracking behavior in Al-Mg-Si alloys, Proceedings of the 59<sup>th</sup> conference of metallurgists, COM2020, ISBM:978-1-926872-47-6] Materials with higher HG-SCC susceptibility will be selected by using this test. In the case of the expansion of available material in the future, the safety material will be identified by applying this test to the new alloy.

• Rationale for Section 1

This section defines the material for the testing.

*Materials definition* Materials for this test are aluminum alloys. In general, materials should be defined by a materials specification, which specifies compositional ranges and specifies minimum tensile properties yield strength (Sy), tensile strength (Su) and tensile elongation (El)). Allowable design stresses are often determined from the specified minimum strength properties of the material, while the elongation provides a qualitative assessment of damage tolerance. Verification that the material meets the materials definition can be based on the mill certification or based on testing by (or contracted for) the user. Verification tests are performed in laboratory air. For the purposes of this performance-based approach, the materials are assumed to sufficiently insensitive to materials variables, such as composition.

• Rationale for Section 2

This section defines the environmental conditions for the testing.

*Test temperature (section 2.1).* The environmental temperature range for the vehicle is generally considered to be 233 K to 358 K (-40°C to +85°C). While susceptibility for SCC at cold temperature is low, therefore, the test temperature for this test shall be at room temperature.

Atmosphere and humidity (section 2.2). SCC propagates by atomic hydrogen which generated by reaction of water and aluminum on fresh metallic surface as shown in Table 1. Therefore, the humidity shall be higher than 85% during the test period. SCC does not occur 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.

Test period (section 2.3). The test period is 90 days in accordance with B6.6 of ISO7886:2012.

Rationale for Section 3

*Test specimen (section 3.1, 3.2).* Specimens for this test were cut from the wrought aluminum alloy products (plate, extruded and forged products), It is recommended compact specimen (CS), or single edge bend (SE) specimen shall be used for this test. The geometry of the compact specimen and single edge bend specimen are shown in ISO7539-6:2011and ASTM E399-12.

Net width W, thickness B and half height H shall be measured within an accuracy of 0.1% of W along a line existing within 10% of W from crack plane.

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

*Fatigue pre-crack (section 3.3).* Fatigue pre-crack shall be introduced at room temperature in the atmospheric condition. Effective crack length *a* including the fatigue pre-crack shall fulfil the following equation for small scale yielding as specified in B.5 of ISO 7866:2012.

A, (W-a) 
$$\geq$$
 1270(K<sub>1APP</sub>/ $\sigma_{0.2}$ )<sup>2</sup>

Where

*a* : effective crack length (distance between fatigue pre-crack tip and load axis (mm))

W : specimen actual net width (mm)

 $K_{1APP}$  :stress intensity factor of a crack when a load was applied to the specimen (MPa $\sqrt{m}$ )

Loads apply and measurement (section 3.4, 3.5). 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, however, it seems that there is no difference in both condition when cracks do not propagate.

If the monitored load is less than 95% if applied load P, 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 when the threshold load decreases to less than 95% of applied load P.

Acceptance Criterion (section 3.9). The crack extension by HG-SCC is examined to exceed 0.16mm or not in 90 days test. This value means that crack growth rate is less than 2X10<sup>-11</sup>m/s and is lower than general SCC criteria of 10<sup>-10</sup>m/s.