HPIS

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

HPIS E 103:2024

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Foreword

This standard was authorized by the board of directors of High Pressure Institute of Japan through drafting by the HG-SCC Committee, consideration by the Pressure Equipment Codes and Standards Council and public comment.

This second edition cancels and replaces the first edition (**HPIS E 103**:2018).

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

Introduction

This standard is established at the request of automotive industry in order to evaluate the humid gas stress corrosion cracking (HG-SCC) susceptibility of aluminium alloys used in compressed hydrogen containers.

1 Scope

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6892-1, Metallic materials – Tensile testing – Part1: Method of test at room temperature

ISO 7539-6:2018, Corrosion of metals and alloys – Part 6: Preparation and use of precracked specimens for tests under constant load or constant displacement

ISO 7866:2012, Gas cylinders – Refillable seamless aluminium alloy gas cylinders – Design, construction and testing

ASTM E399-23, Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials

3 Terms and definitions

For the purpose of this document, the terms and definitions given in **ISO 7539-6**:2018 and the following apply.

HG-SCC (Humid Gas Stress Corrosion Cracking)

stress corrosion cracking in a humid gas environment

$\boldsymbol{K}_{\mathrm{I}}$

stress intensity factor of a crack subjected to opening mode displacements (mode I)

K_{IAPP}

stress intensity factor of a crack when a force was applied to the specimen at the beginning of the HG-SCC test

$\sigma_{0.2}$

average of the 0.2% proof stress of two specimens measured at room temperature in accordance with the procedures given in **ISO 6892-1**

small scale yielding condition

condition in which the plastic zone at the crack tip is so small in comparison to the dimensions of the specimen that the plastic zone does not significantly affect the stress distribution in the surrounding elastic zone, thus enabling the description of the field of stress at the crack tip in terms of stress intensity factor *K*

plane strain condition

condition in which the dimensions of the plastic zone at the crack tip are so small in comparison to the length of the crack and to the thickness of the specimen that plastic deformation along the thickness direction inside the plastic zone is restricted by elastic deformation in the surrounding elastic zone (due to difference in the Poisson's ratios), thus generating a tensile stress in the thickness direction and attaining a tri-axial tensile stress state

4 Principle

A fatigue pre-cracked specimen is loaded by a constant-force or constant-displacement method to a K_{IAPP} equal to a defined value. Then, the specimen is maintained in the loaded state at prescribed environment 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.

5 Specimens

5.1 Specimen geometries

The geometries of the HG-SCC specimen shall be as follows.

a) One of the specimen geometries, or a combination of them, shall be used for the tests.

  1) Compact specimen shown in **A.1**.

  2) Single-edge-notched bend specimen (SE specimen) shown in **B.1**.

  3) Double-cantilever-beam specimen (DCB specimen) shown in **Figure 4** of **ISO 7539-6**:2018.

  4) Modified wedge-opening-loaded specimen (modified WOL specimen) shown in **Figure 5** of **ISO 7539-6**:2018.

5) C-shaped specimen shown in **Figure 6** of **ISO 7539-6**:2018.

b) 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 the crack plane.

c) The faces of the specimen shall be processed so as to make the crack detectable and its length measurable.

5.2 Materials

Specimens are of wrought aluminium alloy products (plate, extruded and forged products).

5.3 Specimen orientation

The orientation of specimen sampling shall be as follows.

a) HG-SCC test specimens: Y-X orientation. Other orientations may be added when necessary.

b) Tensile test specimens: Y orientation.

NOTE The orientation of specimen is defined in **Figure 15** of **ISO 7539-6**:2018.

6 Fatigue pre-cracking

A fatigue pre-crack shall be introduced as follows (in accordance with **Clause 6** of **ISO 7539-6**:2018).

a) A fatigue pre-crack is introduced at room temperature in the atmosphere according to the following procedures.

1) Force control accuracy of the fatigue testing machine is within ± 2.5 % of the force applied.

2) The crack is measured on both surfaces of the specimen.

3) The crack is extended by 2.5 % of W or by 1.25 mm whichever is larger, on both surfaces.

4) The stress ratio R is from 0 to 0.1.

5) The crack may be started at K_I values higher than the expected K_{IAPP}; but, during the final 0.5 mm of crack extension, the fatigue pre-cracking shall be completed at a maximum stress intensity factor as low as possible (less than 60 % of K_{IAPP}).

6) To facilitate the introduction of a crack, the geometries of the notch tip are prescribed as shown in **Figure 16** of **ISO 7539-6**:2018.

7) At the initiation or during extension, the crack shall not deviate an angle exceeding 10° from the plane of the notch, and the difference between the crack lengths on both surfaces of the specimen shall not exceed 5 % of W.

NOTE See **FIG. 3** of **bibliography [2]** for the crack deviation angle from the plane of notch.

b) Effective crack length a including the fatigue pre-crack shall fulfill the following equation for small scale yielding as specified in **B.5** of **ISO 7866**:2012.

$$a,\ (W-a) \geq 1\,270\left(\frac{K_{IAPP}}{\sigma_{0.2}}\right)^2 \quad \text{...(1)}$$

where

a : effective crack length (mm)

W : specimen actual net width (mm)

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

$\sigma_{0.2}$: 0.2 % proof stress (MPa)

7 Test method

7.1 Loading

The loading test shall be conducted according to the following method.

a) A force is applied under constant-force or constant-displacement conditions.

b) Loading accuracy is attained as follows.

1) For constant-force condition, it is necessary to employ a testing machine capable of force accuracy control within ±1 % of the force applied, as defined in **7.6.3** of **ISO 7539-6**:2018.

2) For constant-displacement condition, the sensitivity of the displacement gauge shall be not less than 20 mV/mm so as to minimize the excess amplification of small signals. The linearity of the gauge is such that the deviations from the true displacements shall not exceed 3 μm (0.003 mm) for smaller displacements up to 0.5 mm and not exceed 1 % of recorded values for larger displacements. These conditions are in accordance with **7.5.3** of **ISO 7539-6**:2018.

c) The value of K_{IAPP} obtained by the following equation given in **B.6.2** of **ISO 7866**:2018 is loaded.

$$K_{IAPP} = 0.056\sigma_{0.2} \qquad \text{......(2)}$$

Additionally, the effective crack length a used in the calculations for the following **d)** or **e)** is the average value of the crack lengths measured on both surfaces of the specimen [see **a)7)** of **Clause 6**].

NOTE Because a fatigue pre-crack, in many cases, does not propagate symmetrically on the middle thickness plane of the specimen, a (the average value of the crack length measured on both surfaces of the specimen) is different from a_{pre} measured on the fracture surface (see **7.5**). Therefore, it is recommended to perform additional testings where K_{IAPP} is greater and/or smaller (e.g. 5 % for each) than that of equation (2) at the same time [see **c)** of **Clause 8**].

d) For constant-force method, the force (P) shall be defined as follows:

1) Compact specimen: use the equation shown in **A.2**.

2) SE specimen: use the equation shown in **B.2**.

3) DCB specimen: use the equation shown in **B.6.5** of **ISO 7866**:2012.

4) Modified WOL specimen: use the equation in **Figure 11** of **ISO 7539-6**:2018.

5) C-shaped specimen: use the equation in **Figure 14** of **ISO 7539-6**:2018.

e) For constant-displacement method, the force shall be applied as follows:

1) Compact specimen: use the equation shown in **A.3**.

2) SE specimen: use the equation shown in **B.3**.

3) DCB specimen: use the equation in **Figure 10** of **ISO 7539-6**:2018.

4) Modified WOL specimen: use the equation in **Figure 11** of **ISO 7539-6**:2018.

5) C-shaped specimen: use the equation shown in **B.6.4 b)** of **ISO 7866**:2012.

7.2 Test environment and period

The loaded specimens shall be tested under the following conditions.

a) Temperature: 25 °C ± 5 °C for the entire duration of the test.

b) Atmosphere and humidity: no generation of dew in air measuring 85 % or higher in relative humidity for the entire duration of the test.

c) Test period: 90 days (in accordance with **B.6.6** of **ISO 7866**:2012)

NOTE Typically, the specimens are set in the chamber and the sensors to measure temperature and relative humidity are located near the specimens to maintain the conditions **a)** and **b)**.

7.3 Measurement of force

7.3.1 For constant-displacement condition, the force shall be measured by the following method after the 90-day test period.

a) When the force is not monitored:

1) At the end of the test, the crack mouth opening displacement is measured before removal of the force.

2) The force is removed.

3) The force is reapplied until the crack mouth opening displacement attains the value in **1)** with a force measuring instrument.

b) When the force is monitored, the force at the end of the test is measured. It is also acceptable to calculate the force value from the values of elastic strain measured between the start and the end of the test.

7.3.2 If the force measured as **7.3.1** is less than 95 % of applied force *P*, the test specimen should be rejected without waiting for the final qualification of materials (see **Clause 8**).

NOTE It has been confirmed that the crack length extension by HG-SCC exceeds 0.16 mm [the threshold length defined in **a)** of **Clause 8**] when the measured force decreases to less than 95 % of applied force *P*.

7.4 Fatigue post-cracking and breaking

7.4.1 Fatigue post-cracking shall be introduced as follows.

a) For constant-force condition, a fatigue post-crack is introduced until the post-crack length is extended to 1 mm or more by fatigue loading with a force not exceeding 0.6 times the value obtained by **7.1 c)** and **7.1 d)**.

b) For constant-displacement condition, after the force measurement as **7.3.1** the loading is removed and a fatigue post-crack is introduced until the post-crack length is extended to 1 mm or more by fatigue loading with a force not exceeding 0.6 times the value obtained by **7.3.1**.

7.4.2 After the introduction of a fatigue post-crack, the specimen shall be broken open.

7.4.3 If it is possible to identify the HG-SCC fracture surface, the specimen may be broken by a method other than the introduction of a fatigue post-crack.

7.5 Measurement of crack length

After breaking of the specimen, the following aspects of crack length shall be measured using a scanning electron microscope (SEM) or other measuring instruments with an accuracy within ±0.01 mm.

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

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

The measurement shall be conducted from the direction perpendicular to the broken surface at the positions 25 %, 50 % and 75 % of the specimen thickness, and the average value of the measurements at these 3 points is selected as the effective crack length.

7.6 Validity of test

7.6.1 Fatigue pre-crack

Of the a_{pre} values for the positions 25 %, 50 % and 75 % of the specimen thickness, it shall be verified that the difference between the largest and smallest values does not exceed 5 % of net specimen width W.

7.6.2 Small scale yielding and plane strain conditions

It shall be verified that a, $(W - a)$ and B satisfy the following equation as specified in **B.6.7** of **ISO 7866**:2012.

$$a,\ (W-a),\ B\ \geq\ 1\,270\left(\frac{K_\mathrm{I}}{\sigma_{0.2}}\right)^2 \qquad \text{...(3)}$$

Where,

a, $(W - a)$ and K_I are as follows.

— For constant-force condition:

- In case of $(a_{scc} - a_{pre}) \leq 0.16$ (mm)

$$a = a_{scc}$$
$$(W-a) = (W - a_{scc})$$
$$K_\mathrm{I} = K_\mathrm{IAPP} \qquad \text{(provided that } a = a_{scc}\text{)}$$

- In case of $(a_{scc} - a_{pre}) > 0.16$ (mm)

$$a = a_{pre} + 0.16$$
$$(W-a) = \{W - (a_{pre} + 0.16)\}$$
$$K_\mathrm{I} = K_\mathrm{IAPP} \qquad \text{(provided that } a = a_{pre} + 0.16\text{)}$$

— For constant-displacement condition:

$$a = a_{\text{pre}}$$
$$(W - a) = (W - a_{\text{pre}})$$
$$K_{\text{I}} = K_{\text{IAPP}}$$

The units used in the above equations are same as the units used in Equation (1).

If test conditions do not satisfy these requirements, the test is invalid and shall not be used in the qualification of materials specified in **Clause 8**.

8 Qualification of materials

The applicability of materials shall be judged as follows.

a) The crack extension (a_{scc} - a_{pre}) by HG-SCC [see **7.5 a)** and **7.5 b)**] is examined to exceed 0.16 mm or not.

NOTE The threshold value of "0.16 mm" is specified in **B.7.3** of **ISO 7866**:2012.

b) The actual applied value of K_{IAPP}, which is defined as K_{IA}, is calculated by using a_{pre} and the force applied according to **7.1** for constant-force condition and **7.3** for constant-displacement condition.

c) The validity of materials is judged as shown in **Table 1**.

Table 1 Qualification of materials

Case	Crack extension	K_{IA} versus K_{IAPP}	Judgment [a]
I	$(a_{scc} - a_{pre}) \leq 0.16$ mm	$K_{IA} < K_{IAPP}$	invalid
II		$K_{IA} \geq K_{IAPP}$	pass
III	$(a_{scc} - a_{pre}) > 0.16$ mm	$K_{IA} \leq K_{IAPP}$	fail
IV		$K_{IA} > K_{IAPP}$	invalid

[a] Material shall be judged as follows.

pass : Materials that satisfy this requirement are judged to have applicable resistance to HG-SCC for compressed hydrogen containers as specified in **B.7.3** of **ISO 7866**:2012.

fail : Materials are judged to be failed for application for compressed hydrogen containers.

invalid : Materials cannot be judged in these conditions.
In case I, another testing is recommended in the condition K_{IA} equals to K_{IAPP} or is in some degree greater than K_{IAPP}.
In case IV, where K_{IA} is considerably greater than K_{IAPP}, another testing is recommended because materials may pass in the condition K_{IA} is a little greater than K_{IAPP}.

Annex A
(normative)
The compact specimen

A.1 Specimen geometries

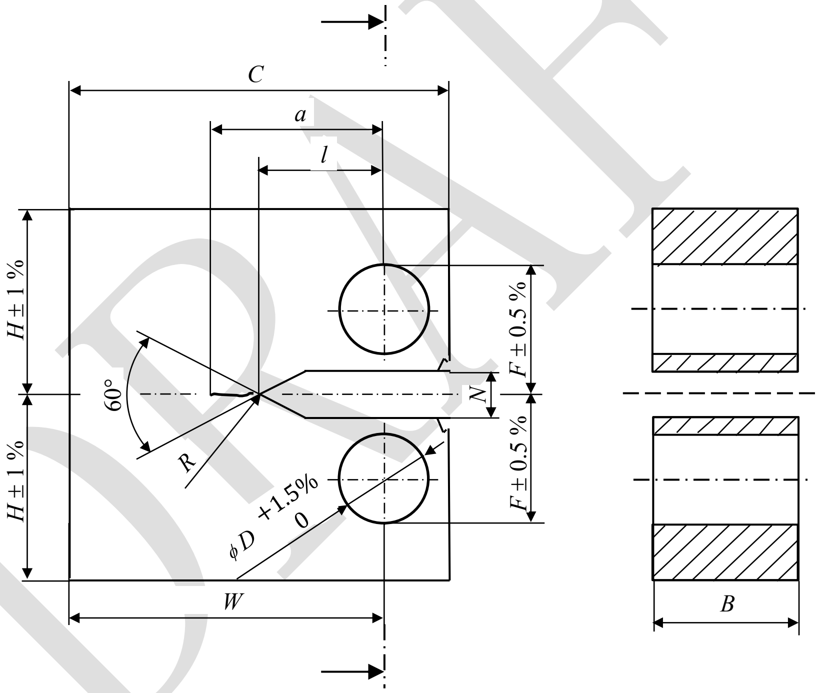
Geometry of compact specimen is given in **Figure A.1**, and a detail of knife edge is shown in **Figure A.2**.

NOTES

1 Compact specimens usually have a net width ranging from 25 mm to 50 mm.

2 **Figure A.1** is in accordance with **Figure 3** of **ISO 7539-6**:2018. However, the thickness B differs from that of **Figure 3** of **ISO 7539-6**:2018.

3 **Figure A.2** is in accordance with **Figure 8 a)** of **ISO 7539-6**:2018.



W : net width

C : total width = $1.25W$

B : thickness = $\frac{W}{20}$ to $\frac{W}{2}$

H : half-height = $0.6W$

D : hole diameter = $0.25W$

F : half-distance between outer edges = $1.6D$

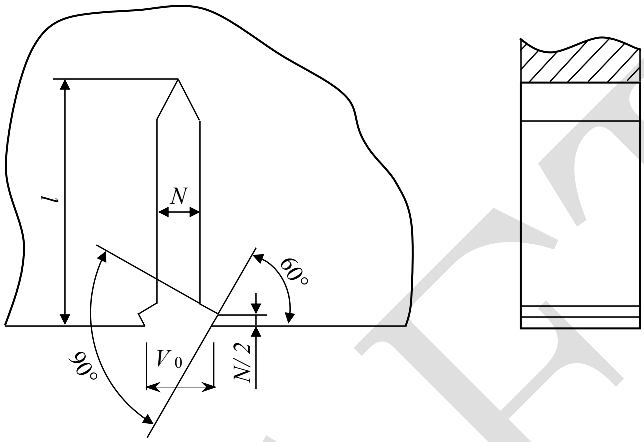
N : notch width = 0.065*W* maximum

R : notch radius = 0.1 mm maximum recommended

l : effective notch length = 0.25*W* to 0.40*W*

a : effective crack length = 0.45*W* to 0.55*W*

Figure A.1―Proportional dimensions for compact specimen



V_0 : knife edge spacing

Figure A.2―Knife edge for location of displacement gauges

A.2 Constant-force testing

For loading under constant-force conditions, force *P* shall be determined by the following equations specified in **Figure 13** of **ISO 7539-6**:2018 [see **7.1d)**].

$$P = \frac{31.62 K_{\mathrm{IAPP}} B \sqrt{W}}{Y} \quad \cdots\cdots \quad (\mathrm{A.1})$$

$$Y = \frac{(2+x)(0.886 + 4.64x - 13.32x^2 + 14.72x^3 - 5.6x^4)}{(1-x)^{3/2}}$$

$$x = \frac{a}{W}$$

Where

P : force (N)

K_{IAPP} : stress intensity factor of the tip of the crack (MPa√m)

B : thickness (mm)

W : net width (mm)

a : effective crack length (mm)

A.3 Constant-displacement testing

For loading under constant-displacement conditions, crack mouth opening displacement *V* shall be determined by the following equations given in **B.6.4 a)** of **ISO 7866**:2012 [see **7.1e)**].

$$V = \frac{K_{\mathrm{IAPP}}\sqrt{W}}{0.032Ef(x)} \quad \cdots\cdots \quad \text{(A. 2)}$$

$$f(x) = \frac{2.24(1.72 - 0.9x + x^2)(\sqrt{1-x})}{9.85 - 0.17x + 11x^2}$$

$$x = \frac{a}{W}$$

$$V = V_f - V_0$$

Where

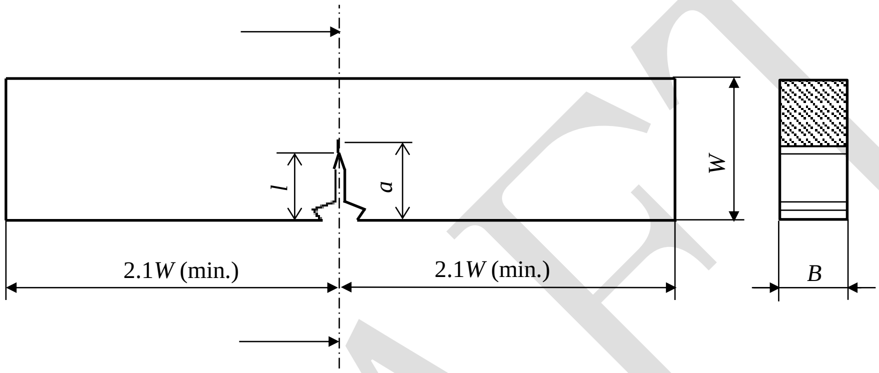
V	: crack mouth opening displacement (mm)
V_{f}	: knife edge spacing under loading (mm)
V_0	: knife edge spacing under no loading (mm)
K_{IAPP}	: stress intensity factor of the tip of the crack (MPa√m)
W	: net width (mm)
a	: effective crack length (mm)
E	: longitudinal elastic modulus (MPa)

Annex B
(normative)
The single-edge-notched bend specimen (SE specimen)

B.1 Specimen geometries

Geometry of SE specimen is given in **Figure B.1**.

NOTE **Figure B.1** is in accordance with **FIG.A.3.1** of **ASTM E399-23**. However, the thickness *B* differs from that of **FIG.A.3.1** of **ASTM E399-23**.



W : net width

B : thickness $= \frac{W}{20}$ to $\frac{W}{2}$

l : effective notch length = $0.25W$ to $0.40W$

a : effective crack length = $0.45W$ to $0.55W$

The faces of the specimen shall be processed as shown in **5.1c)**.
The geometry of the knife edge shall be in accordance with **Figure A.2**.
Notch radius should be 0.1 mm maximum.
Other conditions shall be in accordance with **NOTES** in **FIG. A3.1** of **ASTM E399-23**.

Figure B.1－**Proportional dimensions for SE specimen**

B.2 Constant-force testing

For loading under constant-force conditions, force *P* shall be determined by the following equations specified in **A3.5.3** of **ASTM E399-23** [see **7.1d)**].

A loading fixture of SE specimen is given in **A2.1** of **ASTM E399-23**.

$$P = 31.62 K_{\mathrm{IAPP}} \frac{BW^{3/2}}{Sf(x)} \quad \cdots\cdots \quad (\mathrm{B.1})$$

$$f(x) = 3\sqrt{x}\frac{1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)}{2(1+2x)(1-x)^{3/2}}$$

$$x = \frac{a}{W}$$

Where

- P : force (N)
- K_{IAPP} : stress intensity factor of the tip of the crack (MPa$\sqrt{\mathrm{m}}$)
- B : thickness (mm)
- W : net width (mm)
- a : effective crack length (mm)
- S : span of roller pins defined in **A2.1** of **ASTM E399-23** (mm)

B.3 Constant-displacement testing

For loading under constant-displacement conditions, crack mouth opening displacement V shall be determined by the following equations which are combined the equation (B.1) and the equation (A3.3) specified in **ASTM E399-23** [see **7.1e)**].

A loading fixture of SE specimen is given in **A2.1** of **ASTM E399-23**.

$$V = 31.62K_{\mathrm{IAPP}}\frac{\sqrt{W}}{Ef(x)}q(x) \quad \cdots\cdots\cdots\cdots \quad (\mathrm{B.2})$$

$$f(x) = 3\sqrt{x}\frac{1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)}{2(1+2x)(1-x)^{3/2}}$$

$$q(x) = 6x[0.76 - 2.28x + 3.87x^2 - 2.04x^3 + \frac{0.66}{(1-x)^2}]$$

$$x = \frac{a}{W}$$

$$V = V_{\mathrm{f}} - V_0$$

Where

- V : crack mouth opening displacement (mm)
- V_{f} : knife edge spacing under loading (mm)
- V_0 : knife edge spacing under no loading (mm)
- K_{IAPP} : stress intensity factor of the tip of the crack (MPa$\sqrt{\mathrm{m}}$)
- W : net width (mm)
- a : effective crack length (mm)
- E : longitudinal elastic modulus (MPa)

Bibliography

[1] **ISO 7539-1**:2012, Corrosion of metals and alloys – Part 1: General guidance on testing procedures

[2] **ASTM E647-13a**, Standard Test Method for Measurement of Fatigue Crack Growth Rates

[3] **ASTM E1681-03**, Standard Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials

[4] **ASTM E1820-13**, Standard Test Method for Measurement of Fracture Toughness

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

This explanation relates to the matters specified and/or described in **HPIS E 103** and the relevant matters, but does not constitute part of this standard.

1 Purpose of establishment

ISO 7866:2012 has been established as a standard for compressed hydrogen cylinders using aluminum alloys (see **Clause 2** of **HPIS E 103**), and its **Annex B** specifies "Test method to determine the sustained-load cracking resistance of aluminium alloy gas cylinders". It is known that depending on the type of alloy or temper of aluminum alloys, stress corrosion cracking (SCC) occurs when exposed to a moist environment under loaded force. Since **Annex B** does not specify environmental conditions, it is considered inappropriate to judge the suitability for cylinders based solely on the standard.

Therefore, the High Pressure Institute of Japan established **HPIS E 103**:2018 (first edition), which specifies test methods and judgment conditions in a humid environment, based on **ISO 7866**. This standard is intended to be applied not only to containers, but also to the evaluation of materials before they are processed into containers.

2 Contents of the amendment

HPIS E 103:2024 (second edition) has the following amendments.

a) In “**2 Normative references**”, the latest editions of **ISO 7539-6** and **ASTM E399** are cited. It has been confirmed that the provisions cited by **HPIS E 103** from these standards have not changed from the old editions.

b) The provision of **a)7)** of **Clause 6** is revised from recommendation (should) to requirement (shall), because **Clause 6** as a whole is a requirement.

c) In **b)** of **Clause 6**, *a* (effective crack length) was defined as “distance between fatigue pre-crack tip and load axis in mm” in the first edition, which was intended for compact type specimens. Since this standard applies to specimens of various type [see **a)** of **5.1**], this definition statement has been deleted.

d) Regarding "**—** For constant force condition" in "**7.6.2** Small scale yielding and plane strain conditions", the first edition stated that the test was invalid if the small scale yielding and plane strain conditions were no longer met at the end of the test. In this second edition, the provisions are divided into (1) “In case of $(a_{\mathrm{scc}} - a_{\mathrm{pre}}) \leq 0.16$ (mm)” and (2) “In case of $(a_{\mathrm{scc}} - a_{\mathrm{pre}}) > 0.16$ (mm)” (0.16 mm is the value at which HG-SCC extension is judged to pass). This is because the test is judged to be valid if the small scale yielding and plane strain conditions are met at the length of HG-SCC extension in (1) and 0.16 mm of HG-SCC extension in (2).

e) In accordance with **ISO** and **ASTM** standards, a distinction has been made between force and load throughout this standard. Also, the related expressions are revised.

List of Members of the HG-SCC Committee

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