Hydrogen Compatibility of Materials

1. Materials definition

1.1. The material under consideration shall be defined by a materials specification – the specification can be a nationally-recognized standard or a company-defined specification. The materials specification shall include requirements for the following:

1.1.1. allowable compositional ranges;

1.1.2. specified minimum tensile yield strength, Sy;

1.1.3. specified minimum tensile strength, Su;

1.1.4. specified minimum tensile elongation, El.

1.2. The material should be tested in the final product form whenever possible, or with a nominally equivalent microstructure.

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. The measured tensile strength is denoted S* (average value from at least two tests at room temperature in air or from the mill certification) and is used to define the maximum stress for fatigue testing.

1.4. Welds and metallurgically-bonded materials

1.4.1. When materials are welded (or metallurgically-bonded) and the joint is exposed to hydrogen gas, weld specimens shall be tested in conjunction with the base materials. However, the specified minimum tensile properties of the weld specimens may be different than the base materials.

1.4.2. Welds and metallurgically-bonded materials shall be defined by a welding procedure specification (WPS) that defines the joining procedure as well as the composition and specified minimum tensile requirements (Sy, Su and El) of the joined structure (e.g., weld metal).

1.4.3. Test specimens should be extracted from the joined structure whenever possible. Representative joints can be prepared, if test specimens cannot be extracted from the joined structure.

1.4.4. Weld test specimens shall be measured in gaseous hydrogen and shall satisfy the requirements of the WPS as well as the fatigue life requirements in section 3.2.

JAMA/JARI)Welds are not studied in Japan. The use of welding structures has not been permitted for the high pressure containers in Japanese regulations. However in the GTR, we expect a detailed discussion. (e.g, detailed definition of specimen, evaluation methods for heat affected zone (HAZ), any inspections). CS reply: definition of welds becomes very complicated. I propose either keeping vague or disallow welds. Alternative/additional proposal ideas and language are welcome.

2. Environmental test conditions

2.1. Gas purity: the purity of the gaseous hydrogen from the testing chamber (referred to as the sampled gas) shall be verified to be satisfy the requirements from Table 2.1. The hydrogen source gas shall meet the requirements of applicable fueling standards or the values in Table 2.1.

2.1.1. If three consecutive tests of the sampled gas meet the oxygen and water vapor requirements in Table 2.1, the gas may be sampled periodically at an interval not exceeding 6 months. If the sampled gas does not meet the requirements, the test system is modified, the purging procedures are changed, or the gas sampling interval exceeds six months, three consecutive gas samples shall be evaluated to demonstrate that the test system and procedures meet the requirements of Table 2.1.

JAMA/JARI)We propose not to specify the requirements for sampled gas, because it is difficult to measure the purity after every testing. CS reply: the composition of the test gas is very important. It is not difficult to measure. Existing standards require testing, so I believe that we should keep. We can add language that similar to CSA CHMC1 that requires only periodic testing.

Table 2.1. Gaseous hydrogen purity requirements in parts per million by volume (except where noted). (We need Unit.)*CS reply: untis are above = v/v ppm*

Species	Source gas requirements	Sampled gas requirements
H_2	99.999% min	–
O_2	≤ 1	< 2
H_2O	≤ 3.5	< 10
$CO + CO_2$	≤ 2	–

2.2. Pressure

2.2.1. *The* testing in gaseous hydrogen shall be performed at a minimum hydrogen pressure as follows: ~~1.25xNWP *the following*~~

2.2.2. For slow strain rate tensile (SSRT) test, the pressure shall be 1.5xNWP.

2.2.3. For fatigue life test, the pressure shall be 1.25xNWP.

JAMA/JARI)We propose to specify the maximum pressure of 1.5xNWP for SSRT test as the performance requirement based on the hydrogen station failure . CS reply: 1.5xNWP seems inconsistent with the other requirements, such as the full-scale tank testing. While I would propose keeping 1.25xNWP, 1.5xNWP is unlikely to change the results of the testing.

2.3. Temperature

2.3.1. The specimen temperature for ~~tensile testing~~ slow strain rate tensile (SSRT) test in hydrogen shall be as indicated below:

JAMA/JARI)We propose the correct test name instead of "tensile testing". The original expression may lead to a misunderstanding and a confusion. For example, reader may misunderstand that tensile testing can be used instead of SSRT testing. CS reply: agreed.

2.3.1.1. For austenitic stainless steels, the temperature shall be 228 ±5 K;

2.3.1.2. For aluminum alloys, the temperature shall be 293 ±5 K;

2.3.1.3. For all other materials, testing shall be conducted at the temperature where the material shows a minimum of tensile ductility in gaseous hydrogen within the range of 228 to 363 K.

2.3.2. The specimen temperature for fatigue testing in hydrogen shall be the same as for ~~tensile testing~~ slow strain rate tensile (SSRT) test, except:

2.3.2.1. For austenitic stainless steels, the temperature shall be 293 ±5 K

CS reply: the available data suggest that the limiting fatigue behavior of austenitic stainless steels occurs at room temperature (see Iijima etal ASME PVP2018-84267)

3. Testing requirements

3.1. Slow strain rate ~~tension~~ tensile (SSRT) test

3.1.1. Smooth tensile test specimens shall be used in accordance with internationally-recognized standards. A minimum of three specimens shall be tested in the environmental conditions described in section 2 to demonstate that the material strength properties at the specified temperature in H_2 (cf. section 2.3) shall be greater than or equal to the specified material criteria and the tensile elongation shall exceed 12%.

JAMA/JARI)We propose to add the above sentence to clarify the meaning of the SSRT test. CS reply: we should motify the words since the requirement for elongation is different than the specified material criteria.

3.1.2. Displacement during the test shall be measured on the specimen over a conventional gauge length (3-5 times the diameter of the specimen). Normally, this is an extensometer attached directly to the specimen, but other equivalent methods are acceptable. The measured strain rate (between the yield force and the maximum force) shall be $\leq 5 \times 10^{-5}$ s^{-1}

3.1.3. Requirements (for each test)

3.1.3.1. The measured yield strength shall be greater than or equal to the specified minimum tensile yield strength (Sy) from the materials specification.

3.1.3.2. The measured tensile strength shall be greater than or equal to the specified minimum tensile strength (Su) from the materials specification.

3.1.3.3. The strain hardening capacity as measured by SSRT, defined as the ratio of measured tensile strength to the measured yield strength for a given test, shall be greater than 1.07.

3.1.3.4. The measured elongation~~(GL:25 mm)~~ shall be greater than 12% (for gauge length ≥ 12 mm).

3.2. Fatigue life test

3.2.1. Two general types of fatigue life test specimens are allowed.

(a) Smooth specimens in accordance with internationally-recognized standards.

(b) Notched tensile specimens with an elastic stress concentration factor (Kt) of greater than or equal to 3.

A minimum of three specimens shall be tested in the environmental conditions described in section 2 to demonstrate sufficient durability of the materials. The objective of the fatigue life test using the notched specimens is to show that the fatigue life in the presence of a stress concentration at the specified temperature and H_2 pressure exceeds 9 times the maximum pressure cycle life for a stress of 1/3 x S* (i.e., 9 x 11,000 cycles, or greater than 10^5 cycles). The objective of the fatigue life test using smooth specimens is to evaluate that the fatigue limit at the specified temperature in H_2 ~~(cf. section 2.3)~~ shall be greater than or equal to the fatigue limit at room temperature in air.

JAMA/JARI)We propose to add the above sentence to clarify the meaning of the fatigue life test.

3.2.2. Force-controlled fatigue life tests shall be performed with a constant load cycle in accordance with internationally-recognized standards. The stress at maximum load during fatigue cycling shall be greater than or equal to 1/3 of S* (the average tensile strength measured at room temperature in air). The stress for both smooth and notched specimens is defined as the load divided by the net-section stress (i.e., minimum initial cross sectional area of the specimen). Either smooth or notched specimens shall be tested with the load ratio (R) as indicated:

(a) smooth specimens with R = -1, or

(b) notched specimens with R = 0.1,

where $R = S_{min}/S_{max}$ (S_{min} is the minimum net-section stress and S_{max} is the maximum net-section stress).

3.2.3. The frequency shall be 1 Hz or lower.

3.2.4. Requirements (for each test)

3.2.4.1. Either (a) or (b) shall be satisfied.

(a) For smooth-specimen fatigue testing, the number of applied cycles (N) shall be greater than $2x10^5$ cycles for each tested specimen.

(b) For notched-specimen fatigue testing, the number of applied cycles (N) shall be greater than 10^5 cycles for each tested specimen.

CS comments: I would prefer for both test methods to use 100,000 cycles as the requirement. The 200,000 cycles was a compromise for the JAMA/JARI perspective to invoke the fatigue limit. But in both cases the requirement greatly exceeds the pressure cycle life of 5,500 to 11,000 cycles. Increasing the notched requirement to 200,000 cycles makes it unnecessarily conservative.

Also if necessary, we can leave the choice of the two options to the contracting party.

4. Summary of tests and requirements

Table 4. Summary of tests and requirements for hydrogen compatibility of materials.

	SSRT	Fatigue
Test conditions	• H2 pressure = ~~1.25~~1.5 NWP • Temperature (see 2.3) • Displacement rate ≤ $5x10^{-5}$ s^{-1}	• H2 pressure = 1.25 NWP • Temperature (see 2.3) • Net section stress ≥ 1/3 S* • Frequency = 1 Hz
Number of tests	3	3
Requirements for each test	(1) Yield strength ≥ Sy (2) Tensile strength ≥ Su (3) Tensile/yield > 1.07 (4) Elongation > 12%	(1) either (a) or (b) (a) smooth: N > $2x10^5$ (b) notched: N > 10^5