Development of Standards for Evaluating Materials for Service in High-Pressure Gaseous Hydrogen

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Objectives and Outline of Presentation

- Provide brief *background* and summarize existing *guidance* relevant to materials selection for hydrogen service
- Identify *existing standards* for qualifying materials and designs for hydrogen service and their *limitations*
- Describe *general test standard* for qualification of materials for hydrogen service (CSA CHMC1)
Several physical processes affect observations of hydrogen-assisted fracture

1) **Hydrogen-surface interactions**: molecular adsorption and dissociation producing atomic hydrogen chemisorbed on the metal surface

2) **Bulk metal-hydrogen interactions**: dissolution of atomic hydrogen into the bulk and segregation to defects in the metal (i.e., transport and trapping)

3) **Hydrogen-assisted cracking**: interaction of hydrogen with defects changes local properties of the metal leading to embrittlement and possibly failure

Science-based understanding of embrittlement essential for ensuring safety and reliability of hydrogen technology
Definitions

Hydrogen Compatibility: *materials evaluation*
(commonly described as **Materials Compatibility**)
*•* Standardized materials testing to determine materials properties for design

Hydrogen Suitability: *component evaluation*
*•* Generally used in the context of a component level test with gaseous hydrogen
*•* Can also be design qualification using hydrogen compatibility data
Standard practice for testing and materials selection

- **Guidance on testing in high-pressure gaseous hydrogen**
  - CSA Group: **CHMC1-2012**
  - ASTM International: G142 (and G129)

- **Guidance on materials selection for hydrogen service**
  - American Society of Mechanical Engineers (ASME)
    - B31.12 Hydrogen Piping and Pipelines
    - Hydrogen Standardization Interim Report for Tanks, Piping and Pipelines (STP/PT-003)
  - European Industrial Gases Association (EIGA)
    - IGC Doc 100/03/E Hydrogen Cylinders and Transport Vessels
    - IGC Doc 121/04/E Hydrogen Transportation Pipelines
  - NASA/AIAA (American Institute of Aeronautics and Astronautics)
  - Sandia National Laboratories (compilation of data measured in hydrogen)
    - SAND2012-7321 Technical Reference for Hydrogen Compatibility of Materials
Standards that include materials qualification in high-pressure gaseous hydrogen

- **ISO 11114-4** (International Organization for Standardization)
  - Three options for evaluating *compatibility in gaseous hydrogen*
  - Pass-fail criteria
  - Specific to high-strength steels for pressure vessels

- **ASME KD-10** (American Society of Mechanical Engineers)
  - Design method using *fracture and fatigue properties measured in gaseous hydrogen*
  - Specific to low-strength steels for vessels steels with high pressure
  - Also adopted for piping and pipelines in ASME B31.12

- **SAE J2579** (Society of Automotive Engineers)
  - Several options for materials selection in appendices
  - One option includes materials qualification testing: *fatigue properties measured in gaseous hydrogen*
  - Specific to automotive fuel systems
Critical assessment shows need for further development of testing protocols

Sustained load cracking, measured according to guidance from ASME Article KD-10 (open symbols); ASTM E1681

Elastic-plastic fracture, measured using ASTM E1820 (closed symbols)

Sustained load procedures for determining fracture resistance in gaseous hydrogen appear to be non-conservative for low-strength steels

Open symbols = crack arrest threshold
Closed symbols = crack initiation threshold
Ref. SAND2010-4633 (also Nibur et al. Metall Mater Trans 44A p.248)
Efficient methods for measuring fatigue crack growth in gaseous hydrogen are necessary.

- Duration of 10 Hz test: 40 hrs
- Duration of equivalent test at 0.1 Hz test: estimated at 4000 hrs!! (or 5-6 months)
General standards for qualifying materials for hydrogen service

- **CSA CHMC1 revision** (CSA Group)
  - Methodology using *fatigue properties measured in gaseous hydrogen*
  - *Not specific* to application or component
  - Design approach is not specified (provides flexibility)
  - One testing option provides hydrogen safety factor
    - Multiplicative factor incorporated in design safety factors
  - Other testing options require properties measured in hydrogen be used in design
  - Rules for qualification of materials specifications
    - Requires comprehensive definition of material
    - Bounds qualification activity
Test method for evaluating material compatibility in compressed hydrogen applications: Phase 1 - metals

First edition – **published**: definition of procedures for mechanical property evaluation in gaseous hydrogen

Revised document – **draft**: methods for materials qualification

- **Screening tests** to determine compatibility without special design requirements for hydrogen service
  - Acceptable for aluminum alloys and austenitic stainless steels
- **Safety Factor Multiplier Method**
  - Fatigue testing determine additional safety factor for hydrogen for wide range of cycle life
- **Design qualification method**
  - Allows other documented fatigue design methods (eg ASME BPVC) with appropriate testing in gaseous hydrogen
Metal or alloy of interest

YES

γ-SS steel or Al alloy

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RNTS ≥ 0.90 or RRA ≥ 0.90

NO

RNTS ≥ 0.50

YES

Conduct additional testing to determine design constraints for hydrogen service

Material is suitable for hydrogen service without design modification

Material is NOT suitable for hydrogen service

RNTS = Notch Tensile Strength Ratio
RRA = Reduction of Area Ratio (smooth tensile)
CSA CHMC1 Level 2: Safety Factor Multiplier Method

Notch Tensile Fatigue Tests

- Measure Wohler curves and determine stress amplitude (S) for number of cycles to failure (N) of $10^3$, $10^4$ and $10^5$ in hydrogen and reference environments
  - $\text{SF}_3 = \frac{S3_R}{S3_H}$
  - $\text{SF}_4 = \frac{S4_R}{S4_H}$
  - $\text{SF}_5 = \frac{S5_R}{S5_H}$
  - $\text{SF}_0 = \frac{\text{NTS}_R}{\text{NTS}_H}$
- Hydrogen safety factor: $\text{SF}_H = \max(\text{SF}_0, \text{SF}_3, \text{SF}_4, \text{SF}_5)$

Safety factor for design $\Rightarrow \text{SF}_{\text{design}} = \text{SF}_{\text{component}} \times \text{SF}_H$
Schematic representation of Safety Factor Multiplier Method

\[ SF_0 = \frac{NTS_R}{NTS_H} \]
\[ SF_3 = \frac{S_R}{S_H} \]
\[ SF_4 = \frac{S_R}{S_H} \]
\[ SF_5 = \frac{S_R}{S_H} \]

In this example: \( SF_H = SF_0 > SF_3 > SF_4 > SF_5 \)
Summary of CSA CHMC1

- True material qualification test
  - Not specific to component or application
  - Specific to environment and material form
- Three routes to qualify materials for hydrogen service
  - Screening method
  - Safety Factor Multiplier Method (stress-based fatigue method)
  - Other fatigue design methods allowed with appropriate data
- Qualification of materials from different sources requires a materials specification that defines the material
  - Compositional ranges
  - Mechanical properties, minimum and maximum values
  - Product form, processing route, etc
- Qualification of the materials specification requires testing of materials from 3 sources (or heats)
  - Additional testing is required when the materials specification changes
Summary of standards for qualifying materials for hydrogen service

• Several standards exist for hydrogen pressure vessels
  • ISO 11114-4 and ASME BPVC VIII.3 KD-10
    • Limited scope
    • Opportunity to improve test methods; existing methods may not result in conservative design values

• Standard for fuel systems on vehicles: SAE J2579
  • Limited scope; does not define material
  • Provides framework for materials testing and metrics for evaluating testing results

• General standard for qualifying materials for hydrogen service: CSA CHMC1
  • General rules for qualifying materials
  • Specific requirements: safety factor multiplier method
  • Includes qualification of material specifications