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

Chris San Marchi

Sandia National Laboratories, Livermore CA

and

B.P. Somerday, SNL/CA

K.A. Nibur, Hy-Performance Materials Testing, LLC



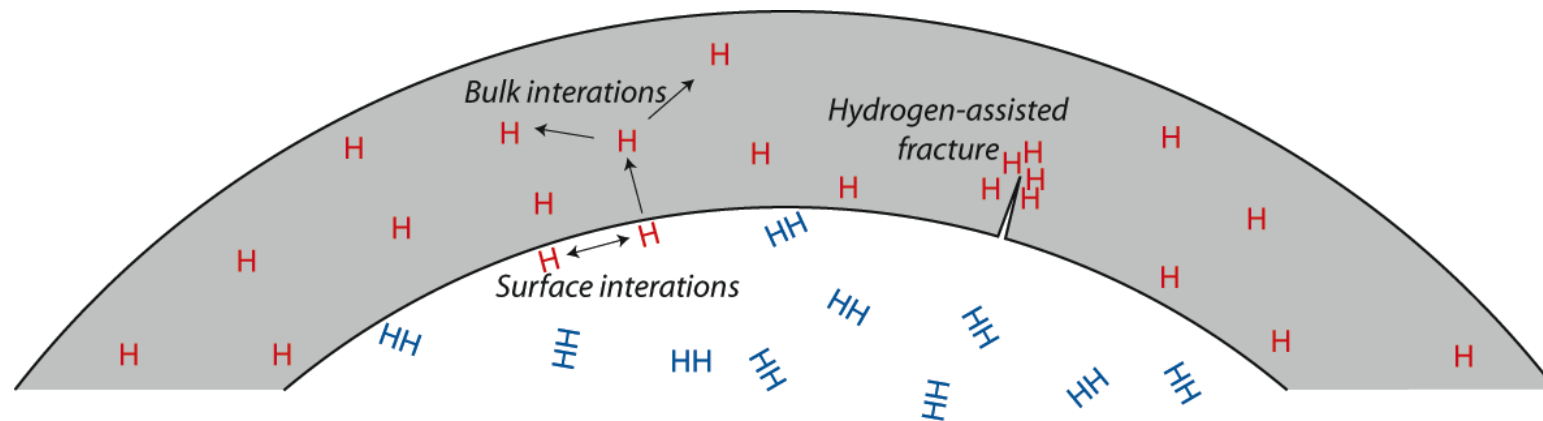
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Objectives and Outline of Presentation

- 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

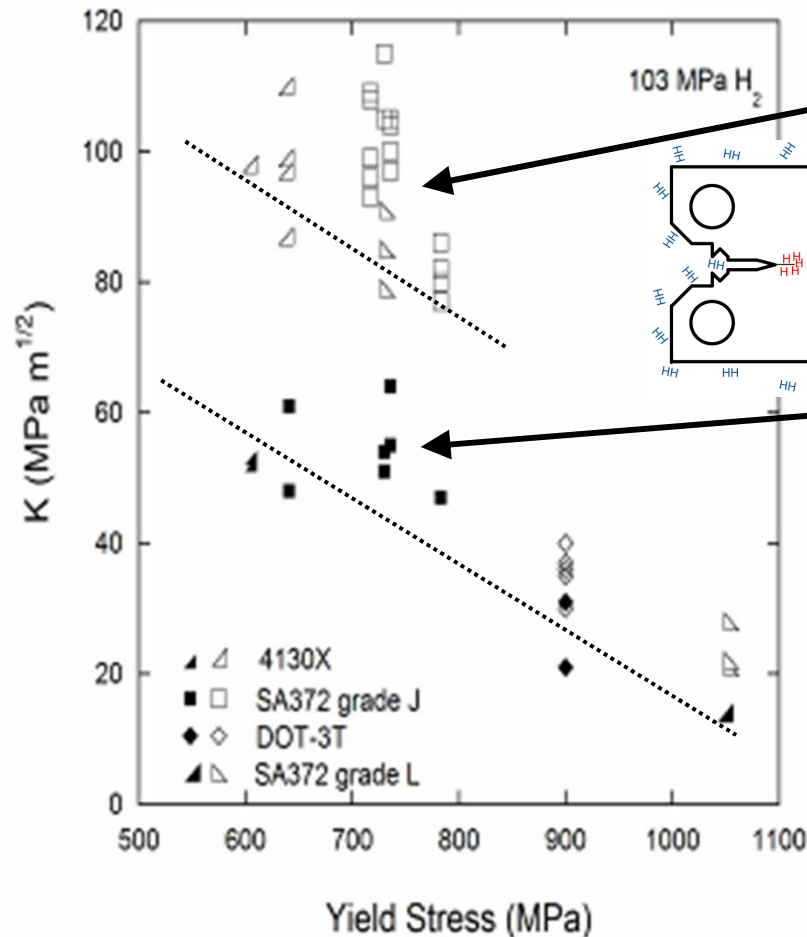
Standard practice for testing and materials selection

- ***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)
 - AIAA G-095 Guide to Safety of Hydrogen and Hydrogen Systems
 - 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)

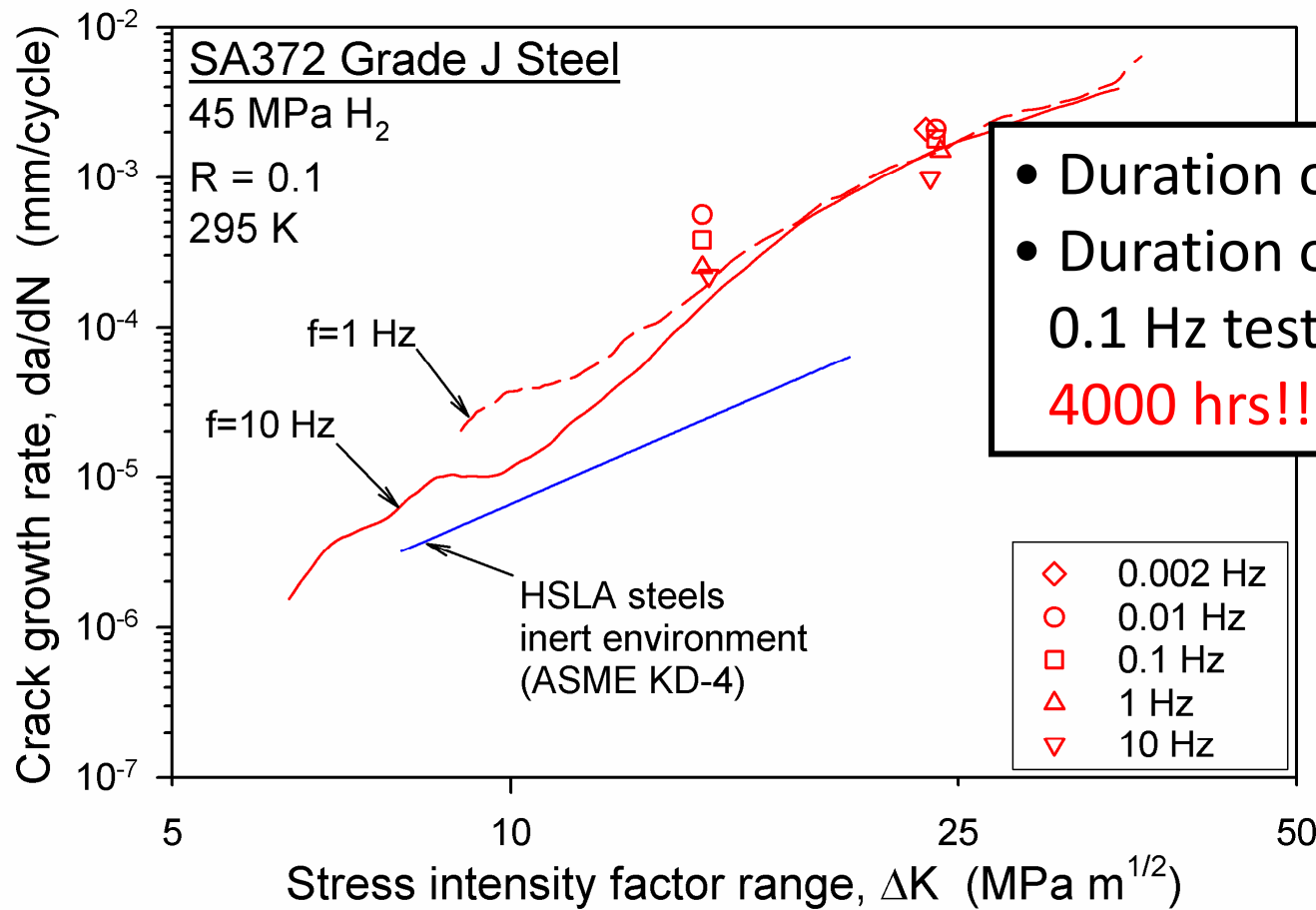
Sustained load procedures for determining fracture resistance in gaseous hydrogen appear to be non-conservative for low-strength steels

Open symbols = crack arrest threshold

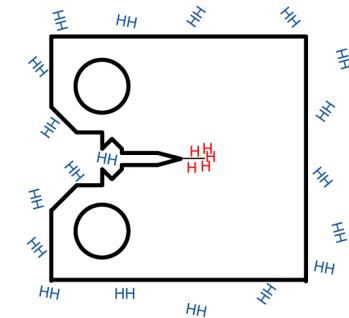
Closed symbols = crack initiation threshold

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

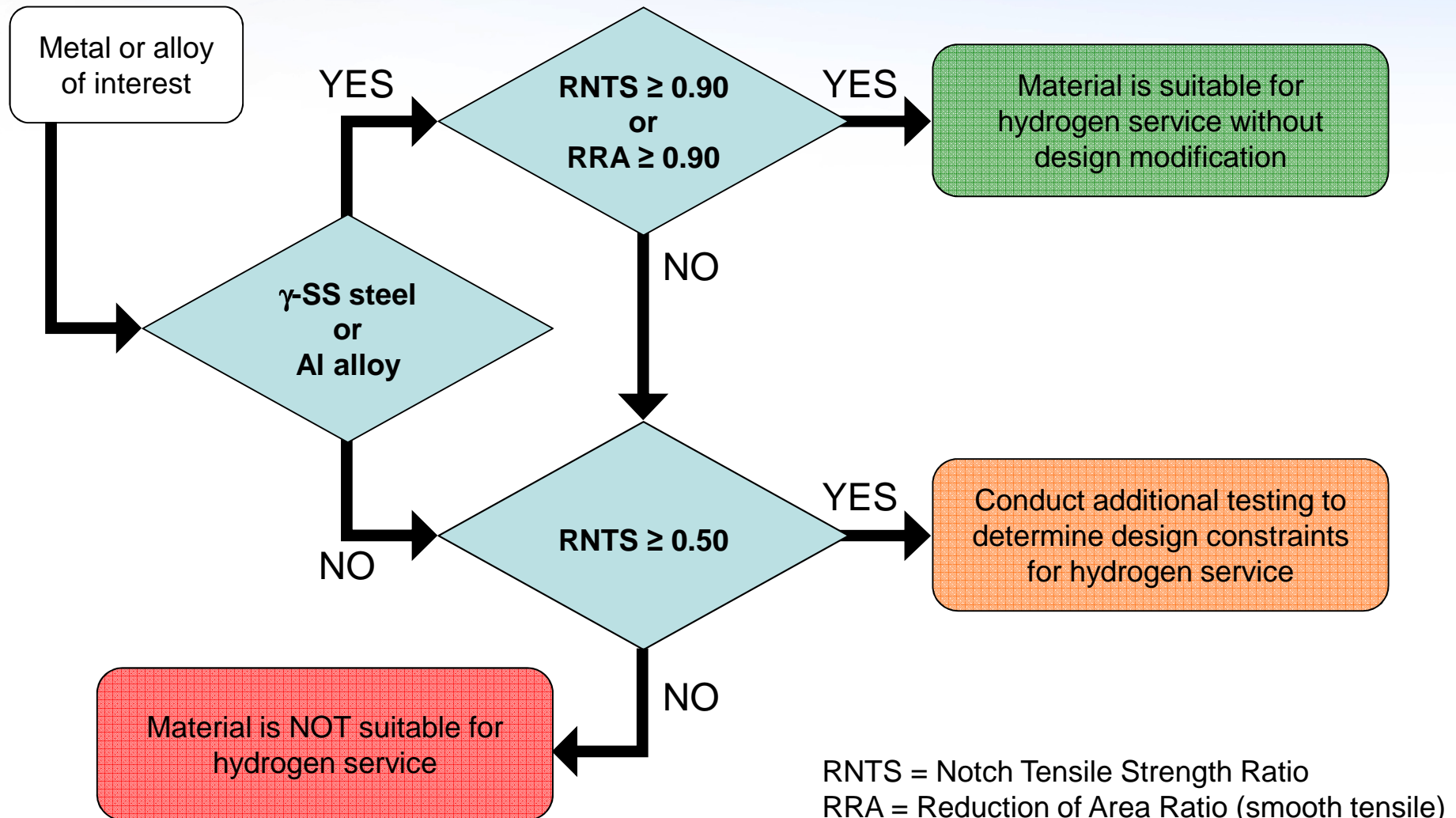
Test method for evaluating material compatibility in compressed hydrogen applications: Phase 1 - metals

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

CSA CHMC1: Logic Diagram



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 10³, 10⁴ and 10⁵ in hydrogen and reference environments

- $SF_3 = S3_R / S3_H$
- $SF_4 = S4_R / S4_H$
- $SF_5 = S5_R / S5_H$
- $SF_0 = NTS_R / NTS_H$

S3 = stress amplitude for failure at N = 10³

S4 = stress amplitude for failure at N = 10⁴

S5 = stress amplitude for failure at N = 10⁵

R = reference environment

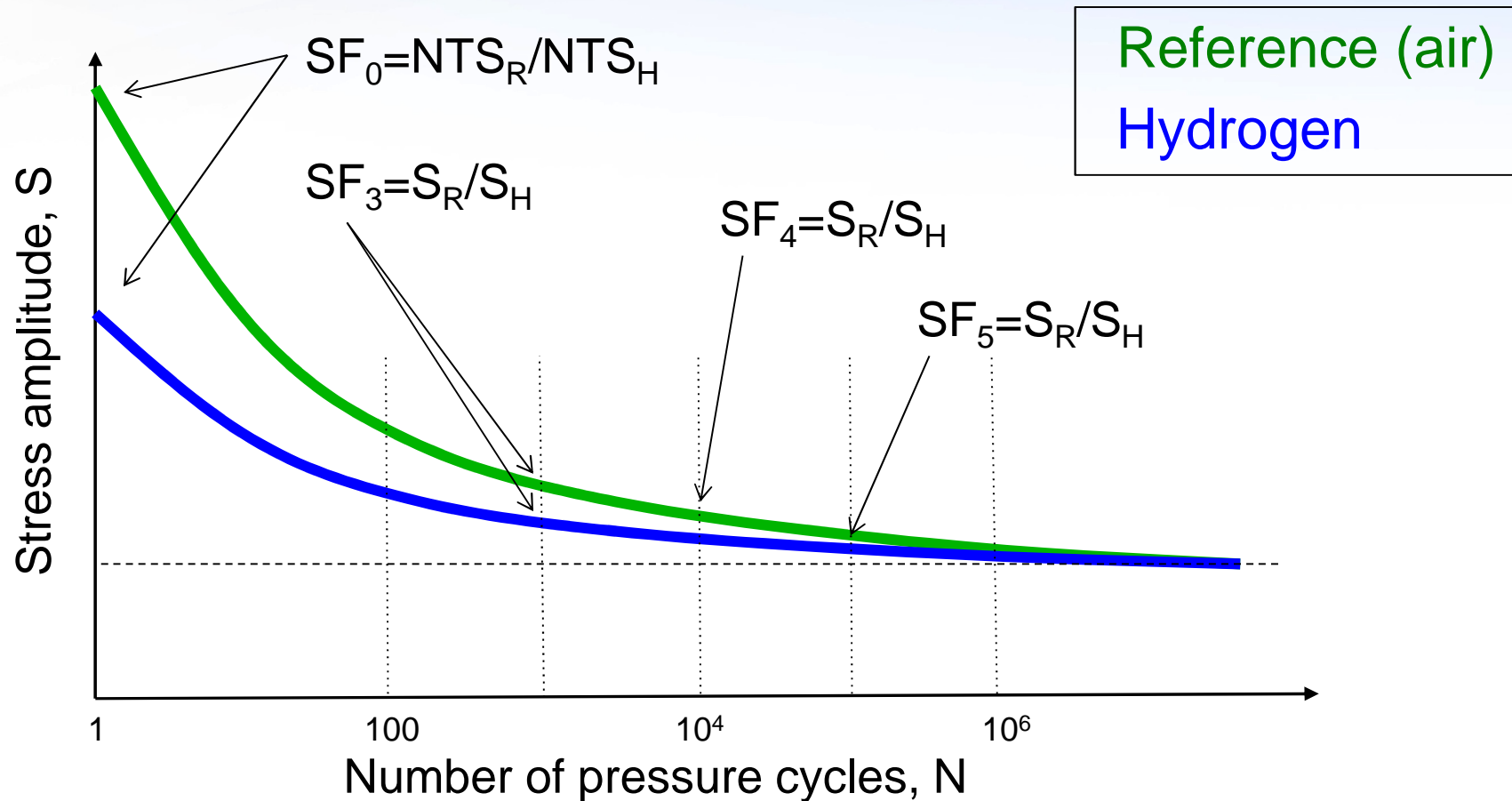
H = hydrogen environment

- Hydrogen safety factor: $SF_H = \max(SF_0, SF_3, SF_4, SF_5)$

Safety factor for design →

$$SF_{\text{design}} = SF_{\text{component}} \times SF_H$$

Schematic representation of Safety Factor Multiplier Method



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