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

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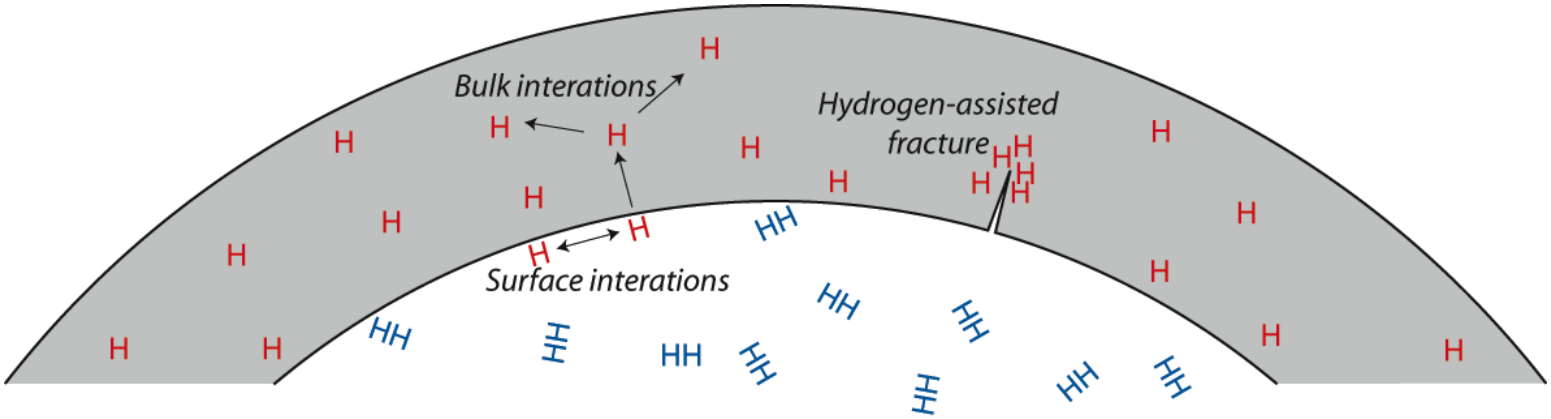
Objectives and Outline of Presentation

- 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)
    - 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

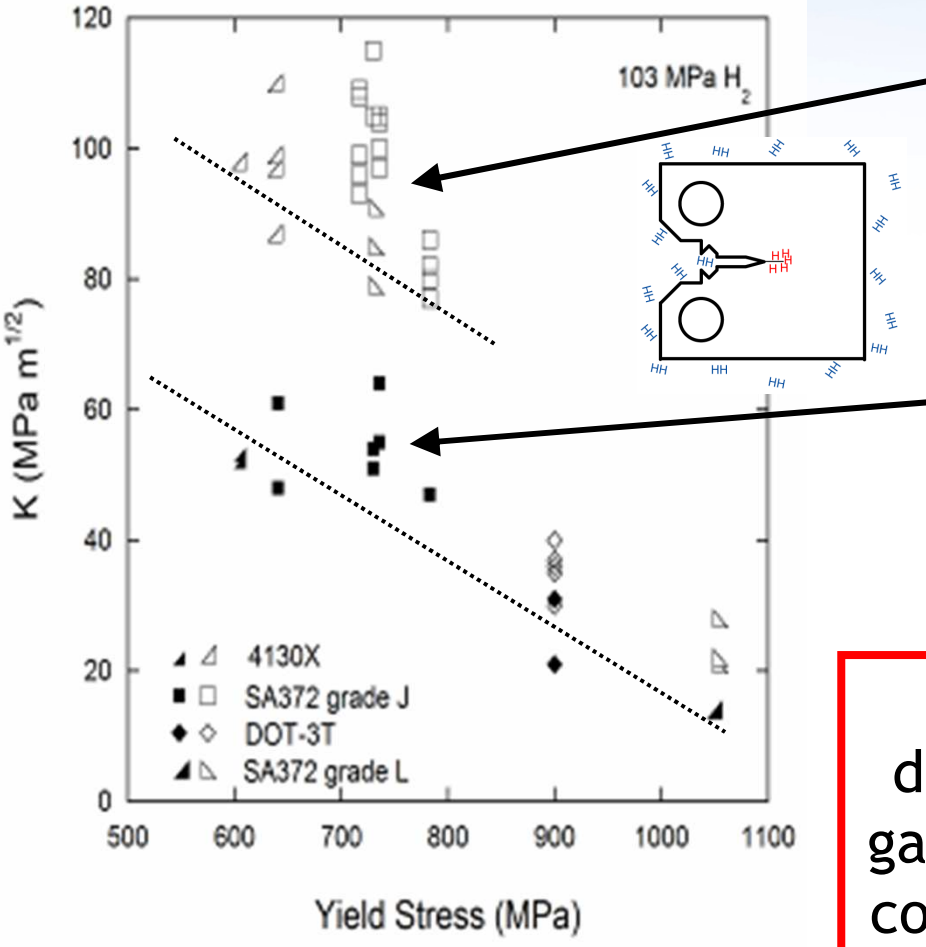
Standards that include materials qualification in high-pressure gaseous hydrogen

- ***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)

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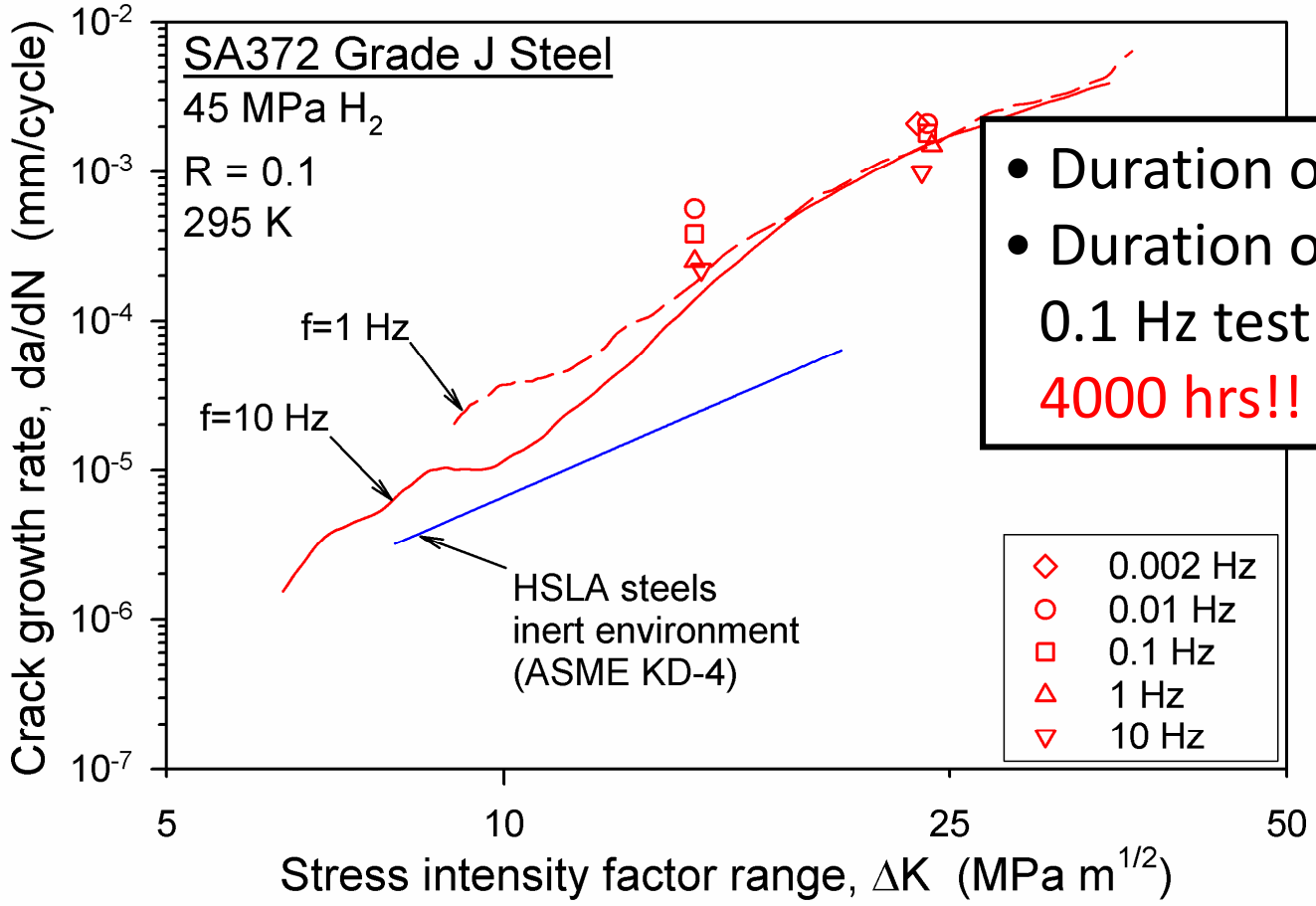
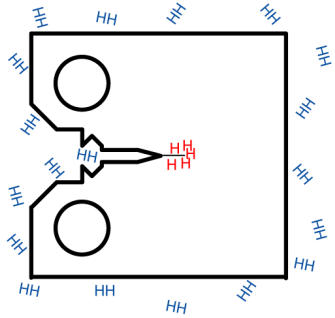
Efficient methods for measuring fatigue crack growth in gaseous hydrogen are necessary



- 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

CSA CHMC1

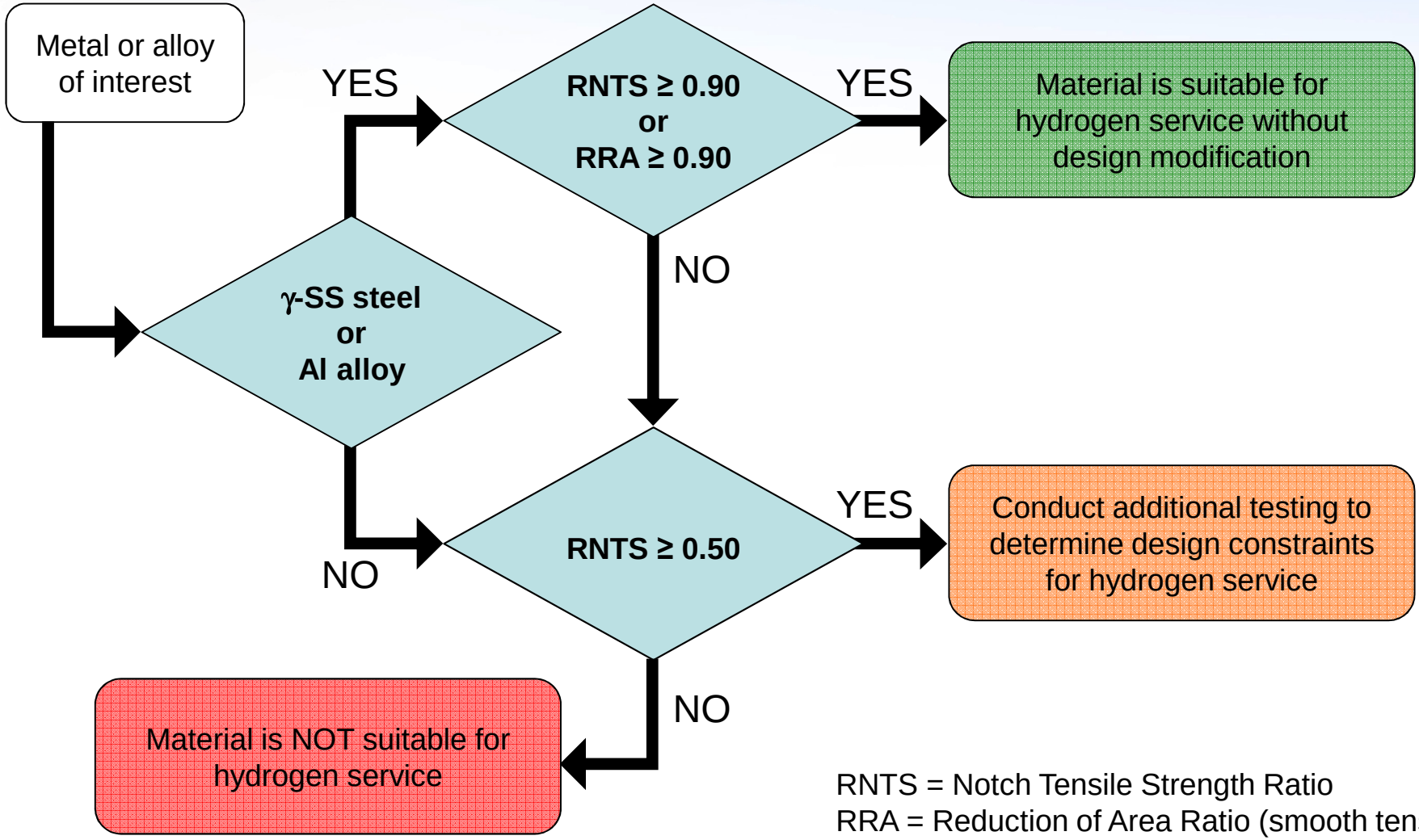
Test method for evaluating material compatibility in compressed hydrogen applications: Phase 1 - metals

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



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

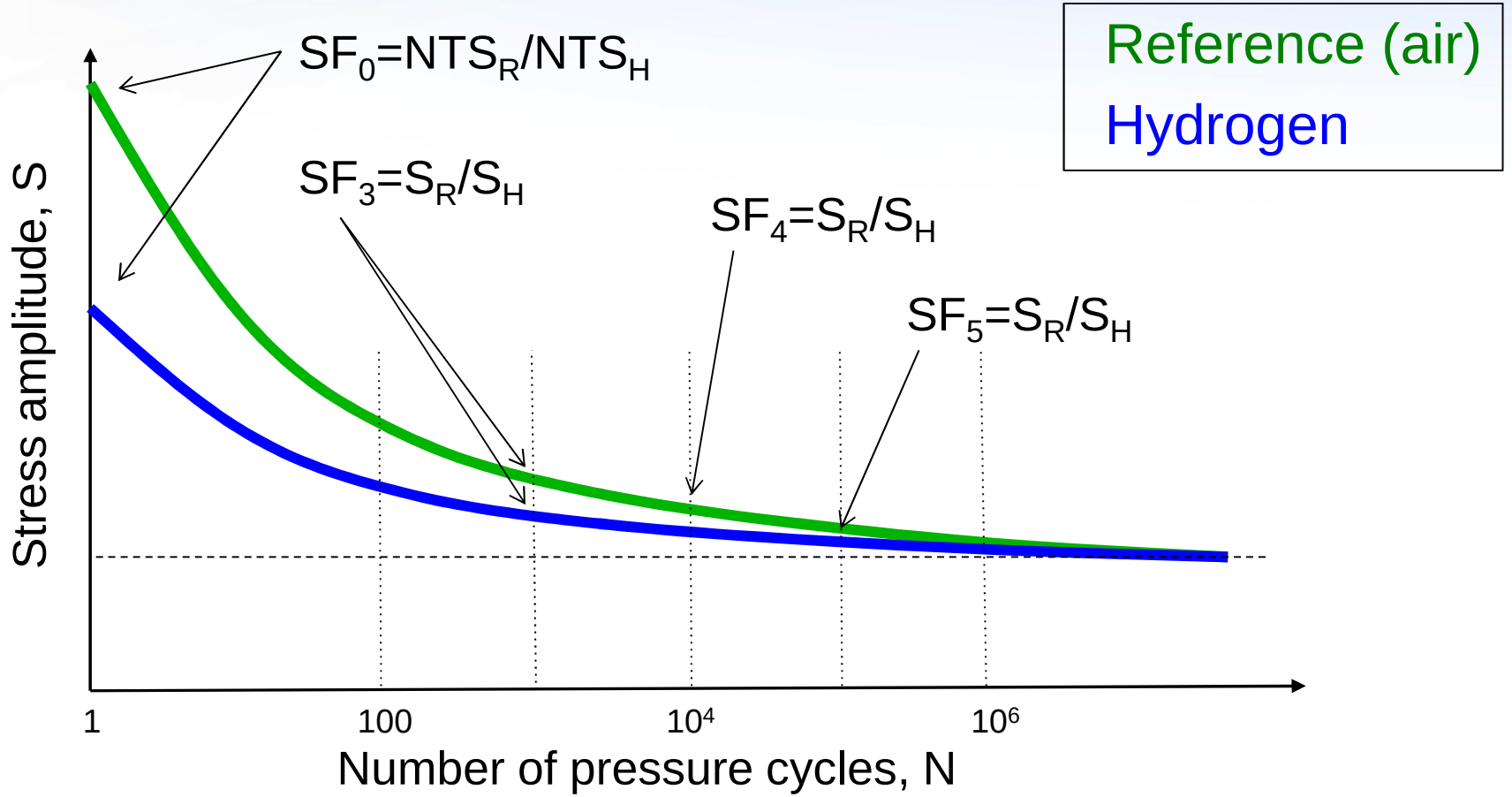
S3 = stress amplitude for failure at N = 10^3
S4 = stress amplitude for failure at N = 10^4
S5 = stress amplitude for failure at N = 10^5

$_R$ = reference environment
$_H$ = hydrogen environment

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

Safety factor for design ➔ $SF_{design} = SF_{component} \times SF_H$

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