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Measuring fracture properties in gaseous hydrogen

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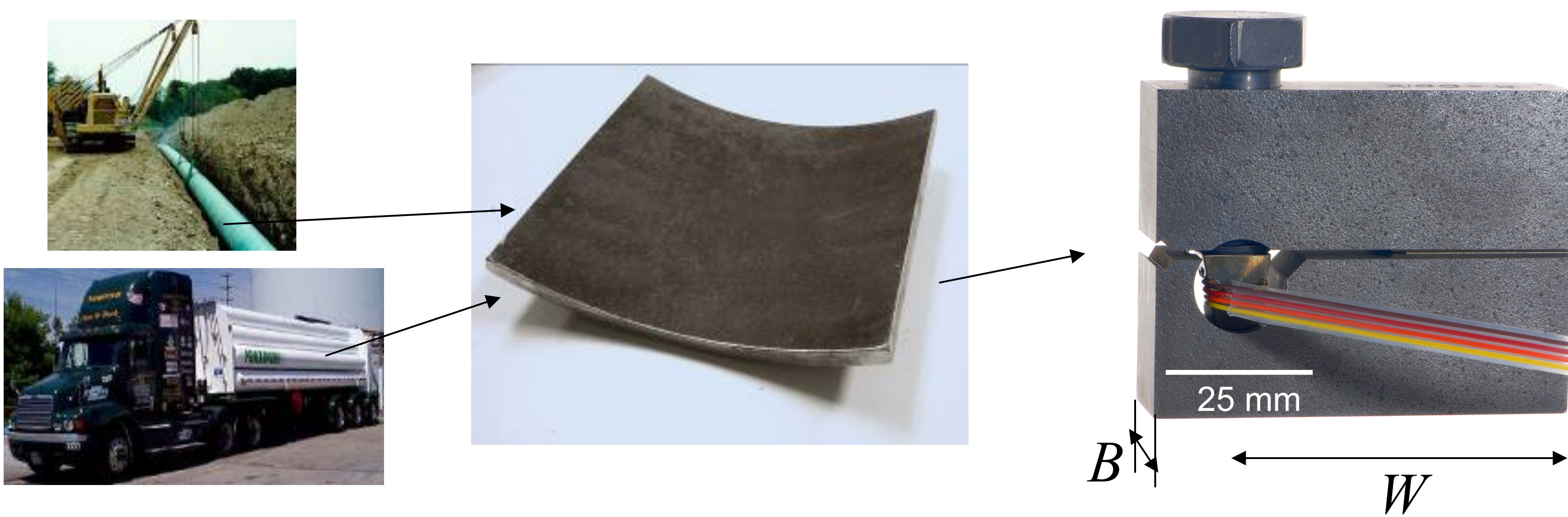
Motivation and background of fracture resistance testing

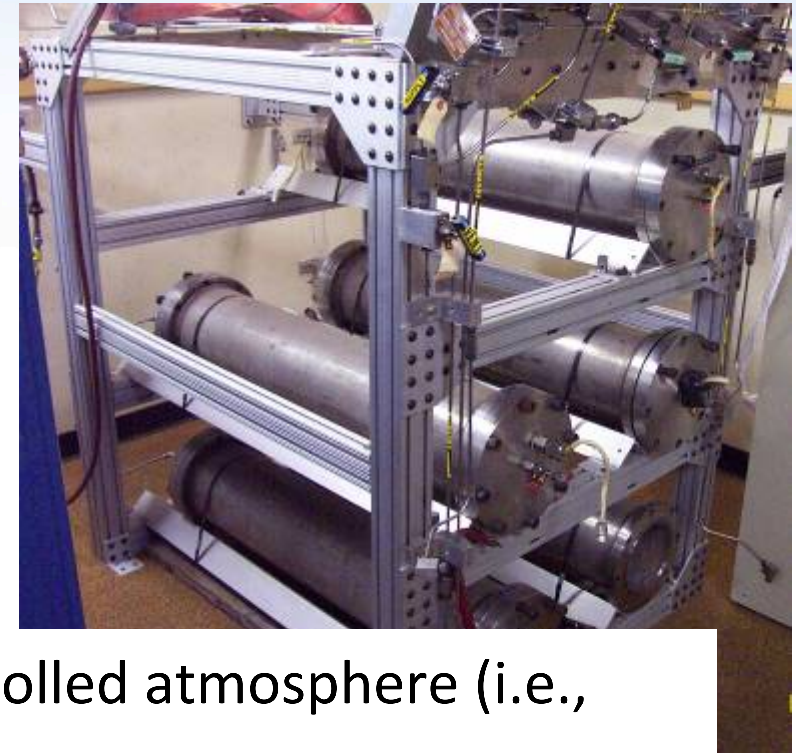
- ASME recently published article KD-10 in Section VII Division 3 of the Boiler and Pressure Vessel Code (BPVC)
  - Applies to high-pressure hydrogen storage vessels
  - Also considered in ASME piping code for hydrogen: B31.12
  - Includes fracture and fatigue testing in gaseous hydrogen
- Sandia test program developed to exercise and evaluate test methods for hydrogen compatibility testing
  - Primary interest is low-strength, low-alloy steels for pressure vessels as well as carbon steels pipeline steels
  - Assessment of methods for evaluating hydrogen-assisted fracture illuminates important differences between constant-displacement and rising-displacement testing methodologies

ASME low-alloy pressure vessel steels: 11 heats tested

- Commercially produced Cr-Mo and Ni-Cr-Mo steel
  - 641-1050 MPa yield strength
- Lower strength C-Mn linepipe steels also tested (X70 and X80)
- Thickness: $B \leq 22$ mm (7/8 inch)
- Width: $W = 57$ mm (2.24 inch)



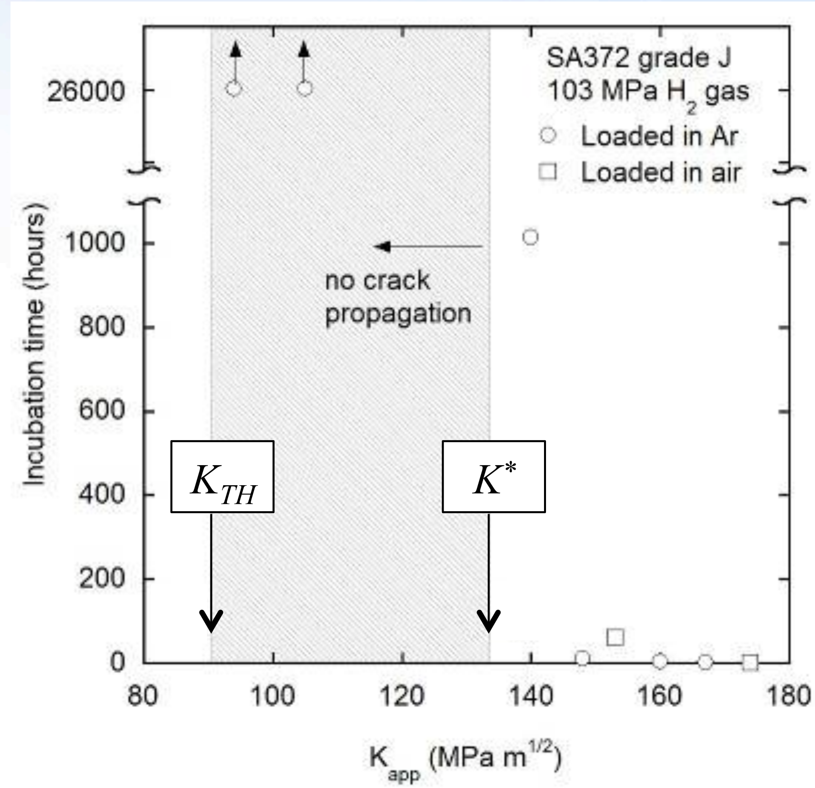
Procedures designed to minimize testing unknowns

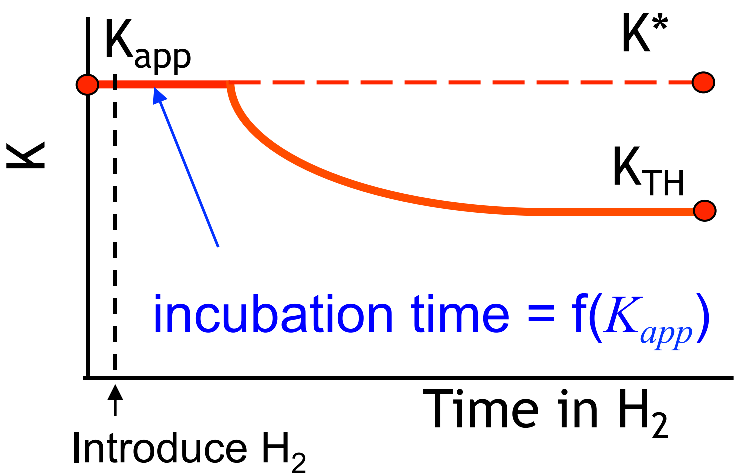


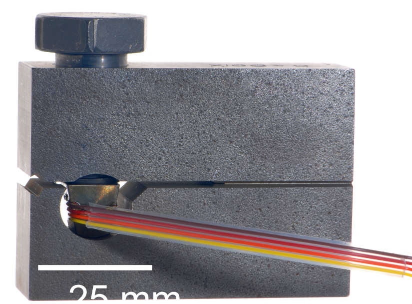
- Load applied to specimen in controlled atmosphere (i.e., glovebox)
  - ~1ppm O_2, ~5 ppm H_2O
- Transferred to pressure vessel in glovebox
- Testing in 99.9999% hydrogen gas at pressure of 103 MPa

Two thresholds identified from constant displacement tests

- Two thresholds identified:
  - Crack initiation: K^*
  - Crack arrest: K_{TH}
  - Both are allowed by ASME KD-10
- K^* always greater than K_{TH}
- Long final crack lengths observed
- K_{TH} values are quantitative (i.e., all initiated cracks arrest at K_{TH})







Difference between K_{TH} and K^* is not related to testing anomalies

- Metallographic cross sections reveal no crack extension for $K_{app} < K^*$ (and when $K_{app} > K_{TH}$)
- FEM demonstrates K-dominance at crack arrest for all σ_{YS} and all crack arrest positions (a_f)
- Elastic-plastic analysis suggest K_{app} is representative of initial crack driving force (even if K-dominance is not maintained at K_{app})
- Varying specimen geometry (to alter crack arrest position) indicates no correlation between K_{TH} remaining ligament length (b_f)
- These observations suggest that there is an intrinsic source for the difference between K_{TH} and K^*

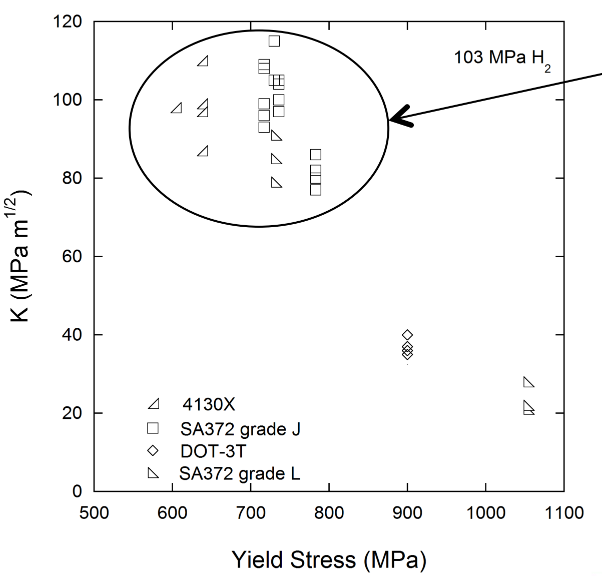
Why is $K^* > K_{TH}$?

- Important to recognize:

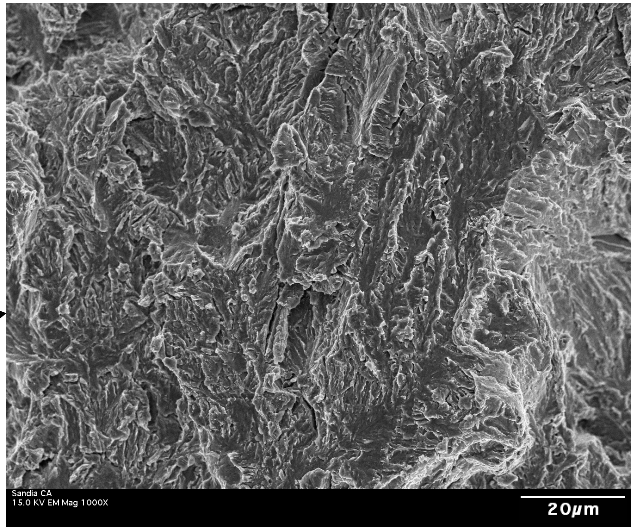
  Fracture in low-strength steels tends to be strain-controlled even for gaseous hydrogen-assisted fracture

  – K^* affected by sequence of H_2 exposure and accumulation of crack tip strain

  – K_{TH} on the order of 80-100 MPa $m^{1/2}$



- For low-strength steels fracture resistance in H_2 remains relatively large
- Fracture process involves plasticity (i.e., strain can be important)



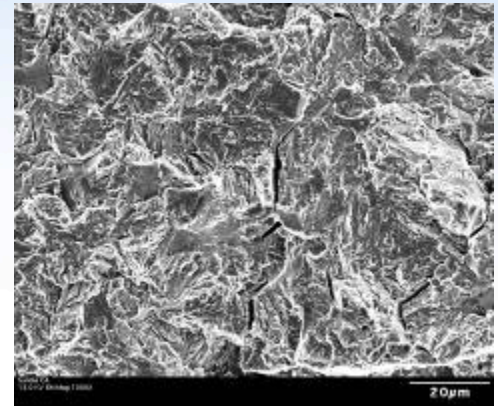
Hydrogen reduces critical continuum strain for failure

Test in air
Strain incompatibility at inclusions initiates fracture

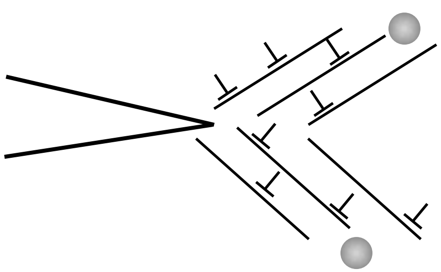
$$K_{air} \propto 6\sqrt{E\sigma_0 l^* \varepsilon^*}$$

Test in H_2
No inclusions observed on fracture surface

$$K_H \propto 6\sqrt{E\sigma_0 l^* \varepsilon_H^*}$$



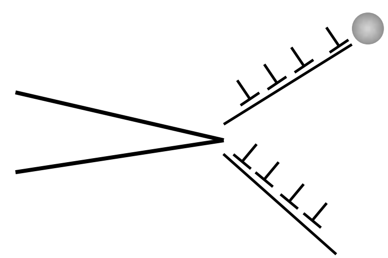
- Hydrogen alters deformation at crack tip (localized deformation)
- Microcrack formation results from strain incompatibilities associated with localized deformation
- Crack extension preempts accumulation of strain to ε^*
- $\varepsilon^*_H < \varepsilon^*$, where ε^*_H is the critical continuum strain for hydrogen assisted cracking



Without strain localization

$$K_{air} > K_H$$

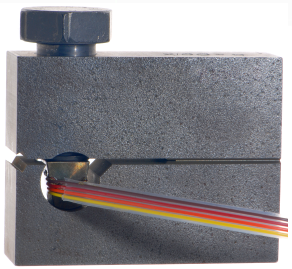
$$\therefore \quad \varepsilon^* > \varepsilon_H^*$$

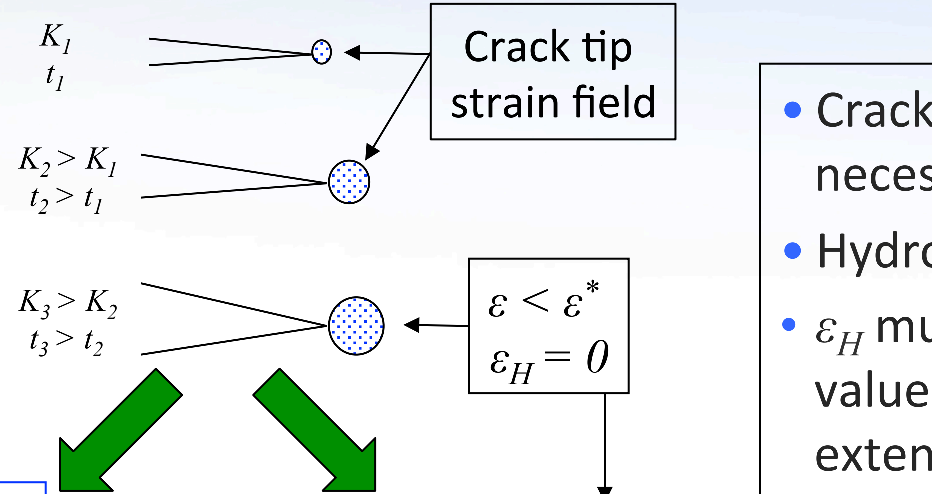


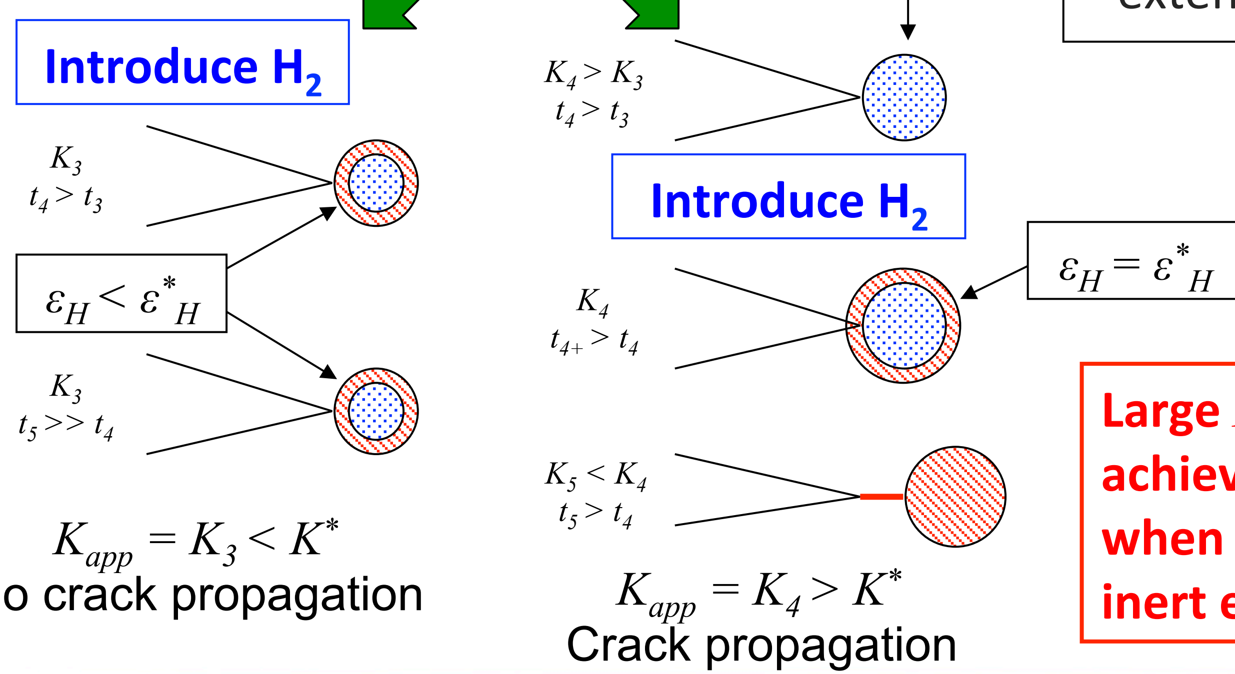
Influence of strain localization

Hydrogen activated strain (ε_H) develops when exposed to hydrogen under load

Constant displacement applied in Ar followed by H_2 exposure



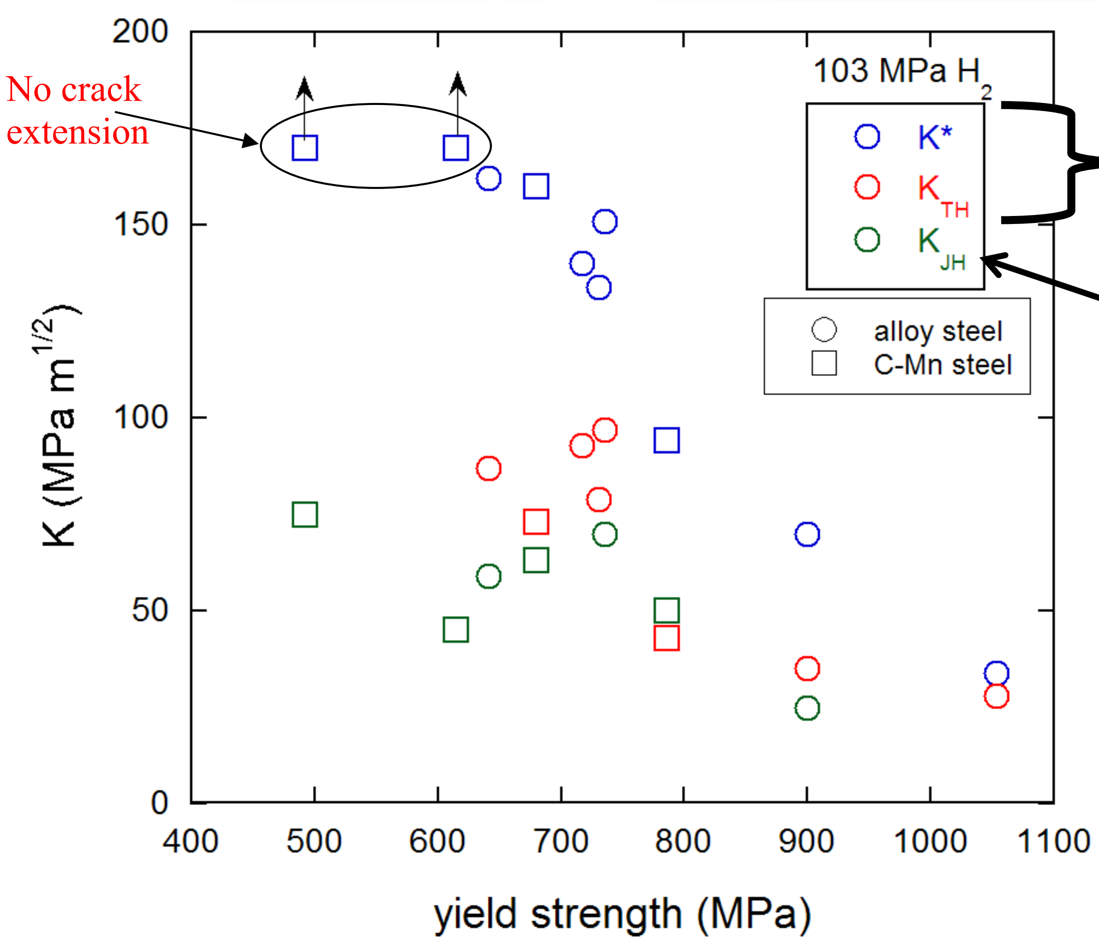




- Crack tip strain is necessary during SCC
- Hydrogen induces strain
- ε_H must exceed a critical value (ε^*_H) for crack extension to occur

Large K_{app} is necessary to achieve crack initiation when load is applied in an inert environment

Considering strain-controlled fracture: does K_{TH} represent the limiting fracture resistance?



Constant displacement tests (bolt-loaded WOL)

Rising-displacement fracture resistance measurements

Rising-displacement tests result in lower bound fracture resistance

- K_{TH} is non-conservative

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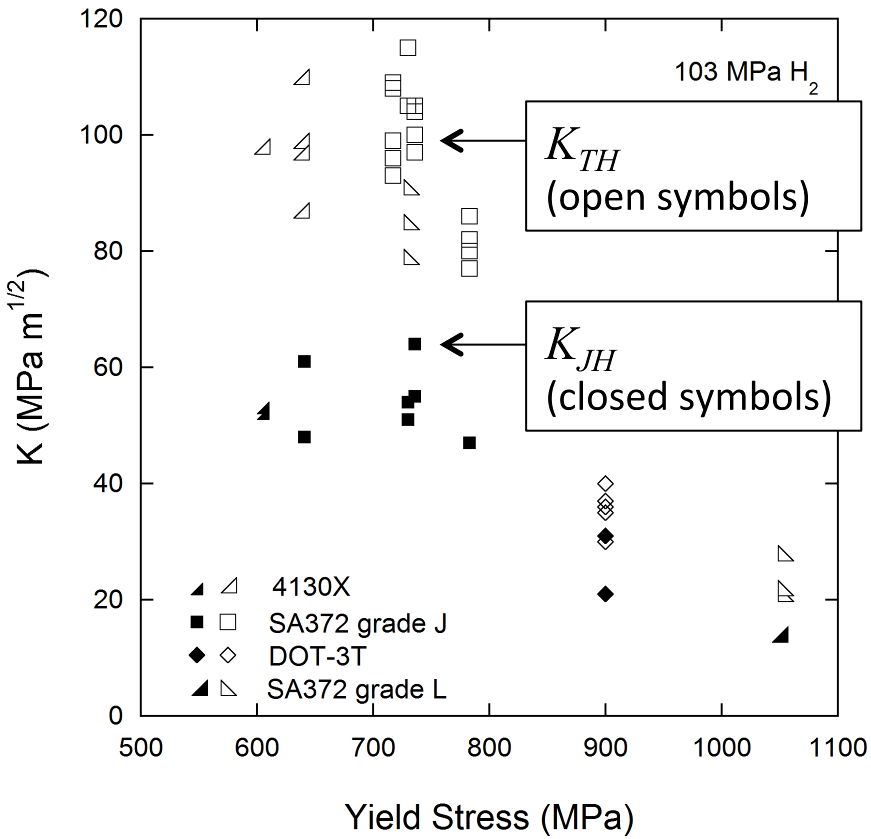
What are the differences/similarities between K_{TH} and K_{JH} ?

Differences:

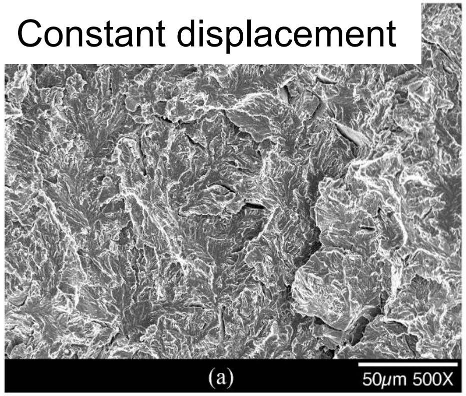
- $K_{TH} > K_{JH}$



Similarities:

- Both thresholds increase with decreasing strength
- Consistency of fracture surface appearance suggests fracture mechanism is the same

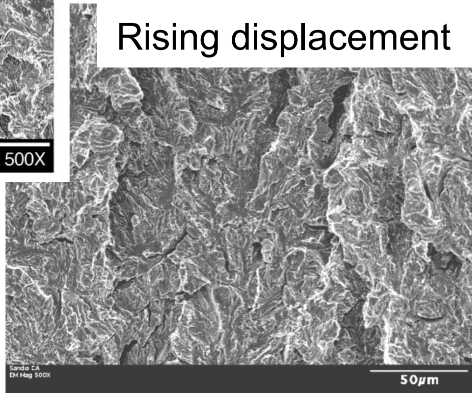
Constant displacement



4130X

103 MPa H_2

Rising displacement

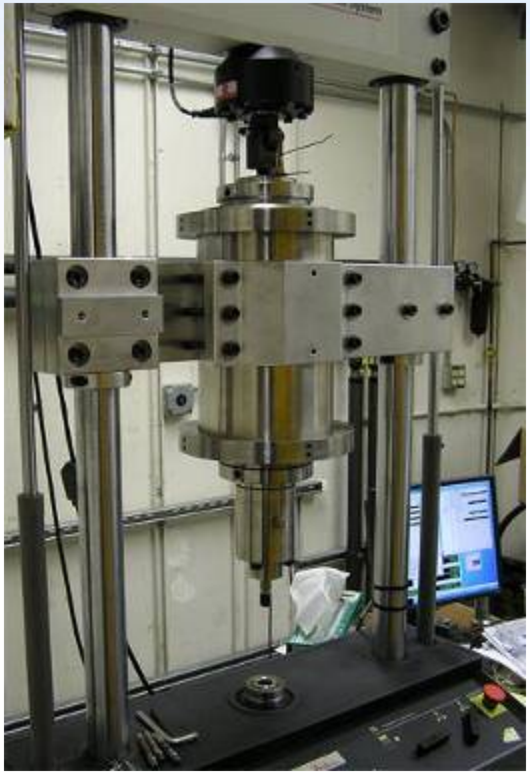


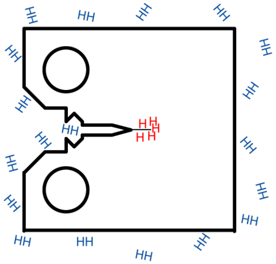
Rising-displacement fracture resistance measurements

- Measure J_{IC} following ASTM E1820 and E1737
  - Elastic-plastic fracture mechanics
- Tests were conducted in custom chamber at 103 MPa H_2 gas pressure
- Testing rates 0.3 to 3 MPa $m^{1/2}$/minute
- Accurate measurement of J and crack-length
  - Load and displacement sensors internal to pressure vessel
  - Crack-length monitored with direct current potential drop (DCPD)
  - Crack-growth resistance (J-R) curves can be generated

$$K_{JH} = \sqrt{J_{IC} E'}$$

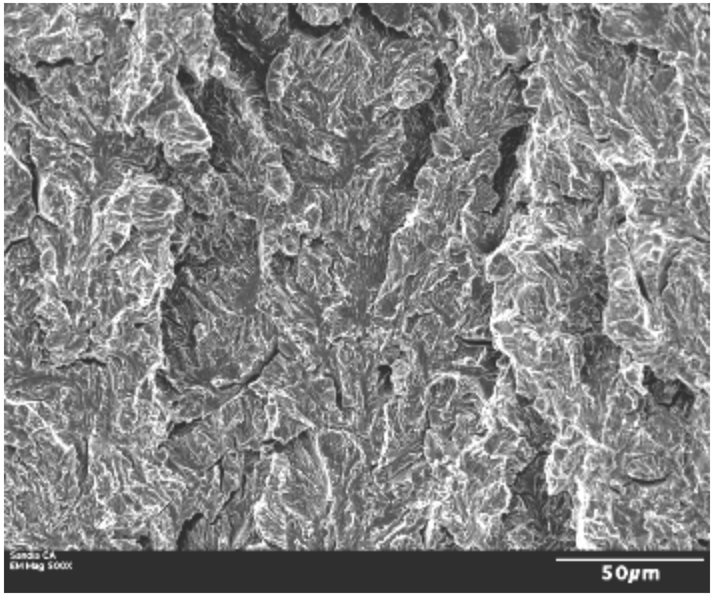
K_{JH} is a threshold measurement from a rising displacement test

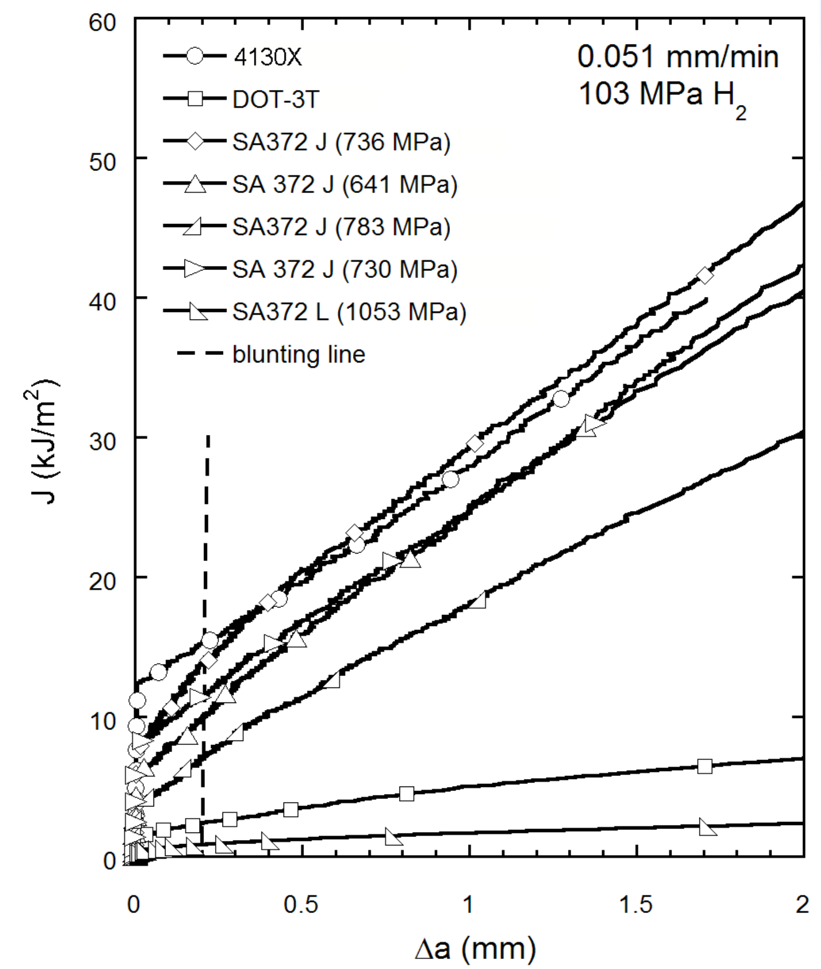




Fracture mechanism is strain controlled

- R-curve behavior in gaseous hydrogen
- Evidence of plasticity on fracture surface
- Consensus in the literature
  - e.g., Takeda and McMahon, *Met Trans A* 1981





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Critical strain criteria for K_{TH} and K_{JH}

- When fracture involves plasticity (e.g. $\varepsilon^*_H > \varepsilon_{yield}$) strain-controlled fracture criterion may be invoked
  - Ritchie and Thompson* described critical strain criterion for extension of a stationary crack based on the HRR fields

$$\varepsilon \propto \frac{1}{r}$$

  - resulting criterion for K_{JH}, K_{IC}, K^*, etc

$$K_{JH} \approx \sigma_0 \sqrt{l^*} \sqrt{\frac{\varepsilon^*_H}{\varepsilon_0}}$$

- K_{TH} occurs when a propagating crack arrests
  - Critical strain criterion must consider the strain field of a propagation crack
  - Rice *et al*** showed the strain ahead of a propagating crack decays as:

$$\varepsilon \propto \ln\left(\frac{1}{r}\right)$$

* Ritchie Thompson *Met Trans* 1985
** Rice Drugan Sham ASTM STP700, 1980

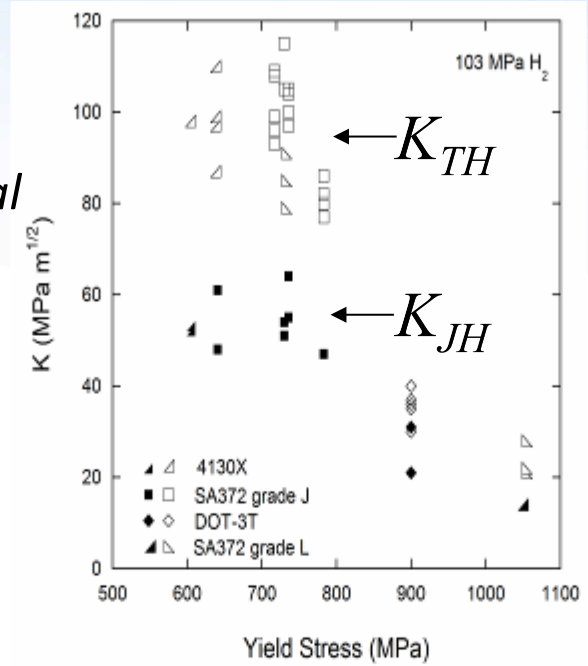
Crack tip mechanics-based model supports $K_{TH} > K_{JH}$ for strain-controlled fracture

$$K_{TH} \approx \sigma_0 \sqrt{l^*} \exp\left(\frac{\varepsilon_H^*}{\varepsilon_0}\right)$$

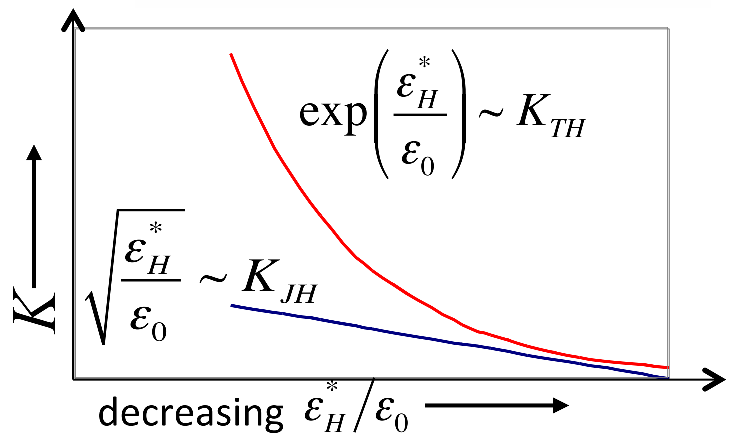
Derived from Rice *et al* strain field for propagating crack

$$K_{JH} \approx \sigma_0 \sqrt{l^*} \sqrt{\frac{\varepsilon_H^*}{\varepsilon_0}}$$

Derived from HRR strain field for stationary crack



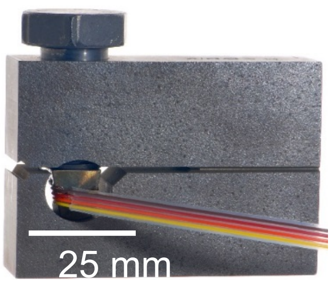
$K_{TH} \approx K_{JH}$ only when strains associated with fracture are small

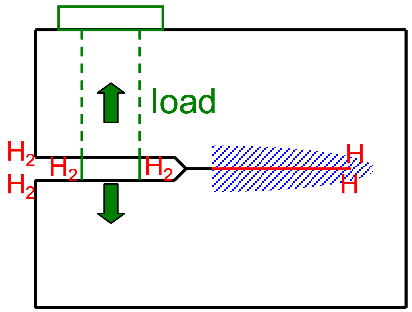


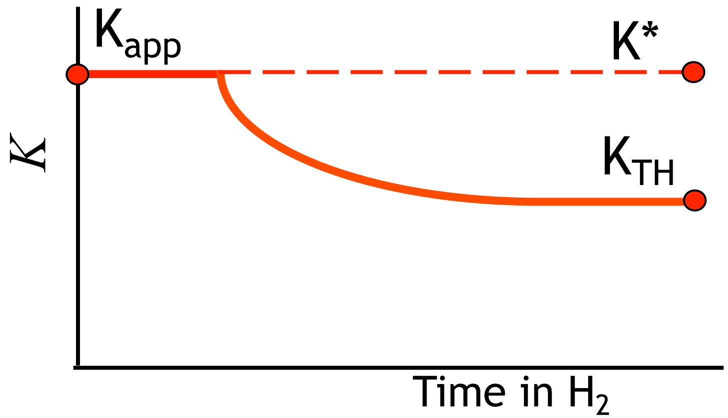
Three methods to measure fracture resistance in gaseous hydrogen

Constant Displacement (E1681)

(1) K^* - measured at crack initiation
(2) K_{TH} - measured at crack arrest

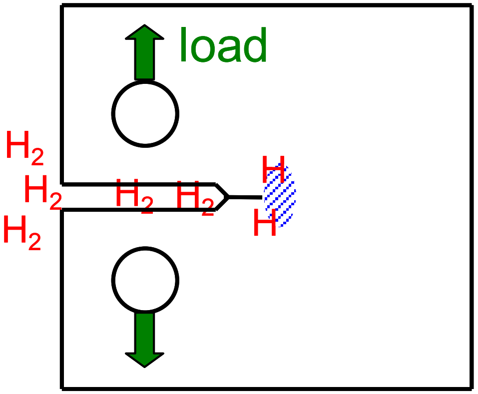






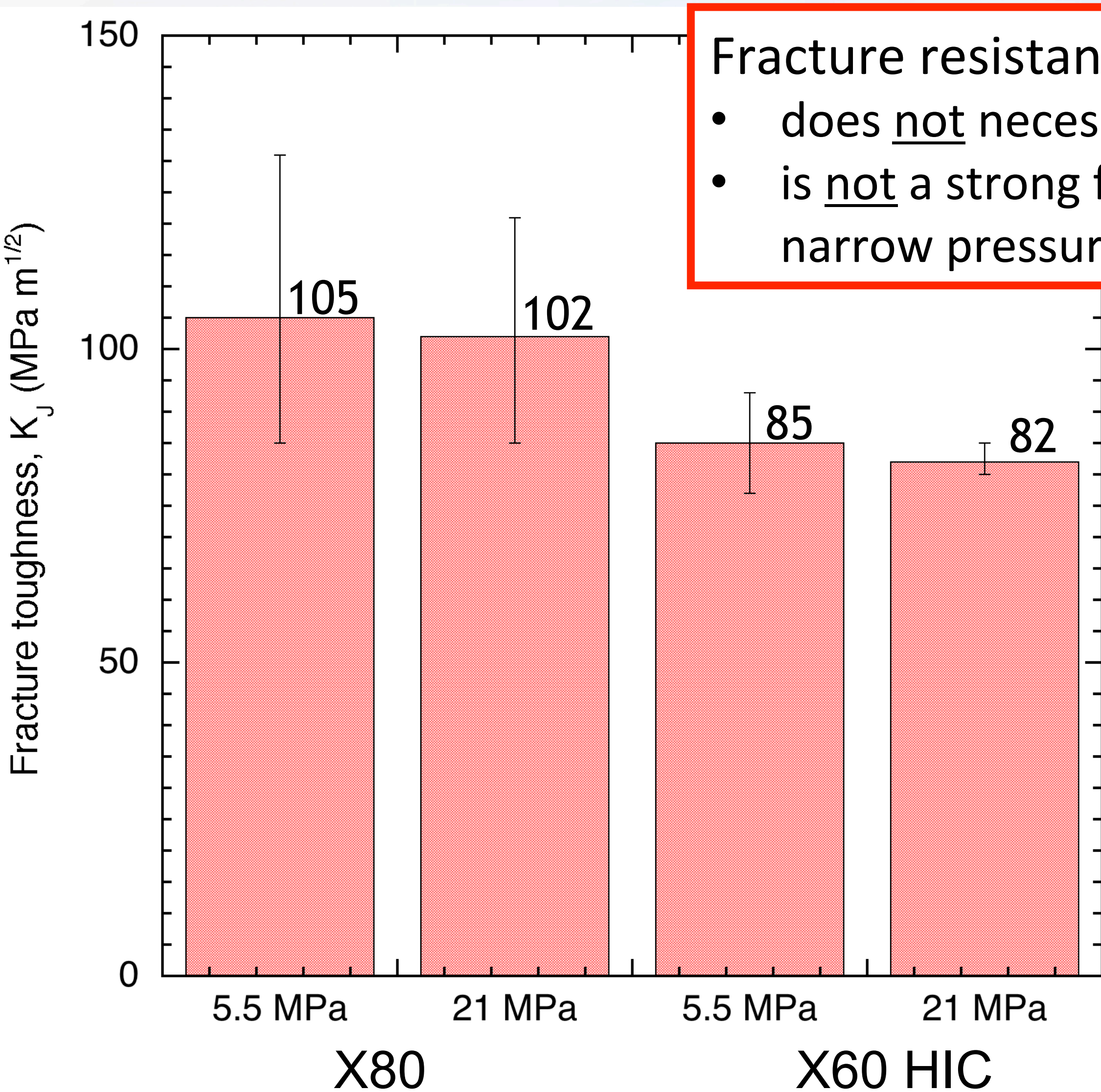
Rising Displacement (E1820)

(3) K_{JH} - measured at crack initiation; using elastic-plastic J-Integral



Rising-displacement fracture resistance is most conservative due to limited strain history

Fracture resistance (K_{JH}) of pipeline steel is typically >75 MPa $m^{1/2}$ for $P_{H2} \leq 20$ MPa

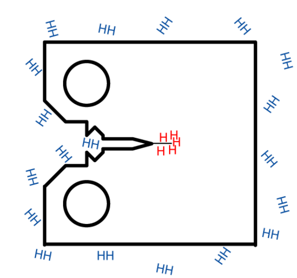


Fracture resistance:

- does not necessarily follow strength trend
- is not a strong function of pressure in narrow pressure range

Average of at least four measurements

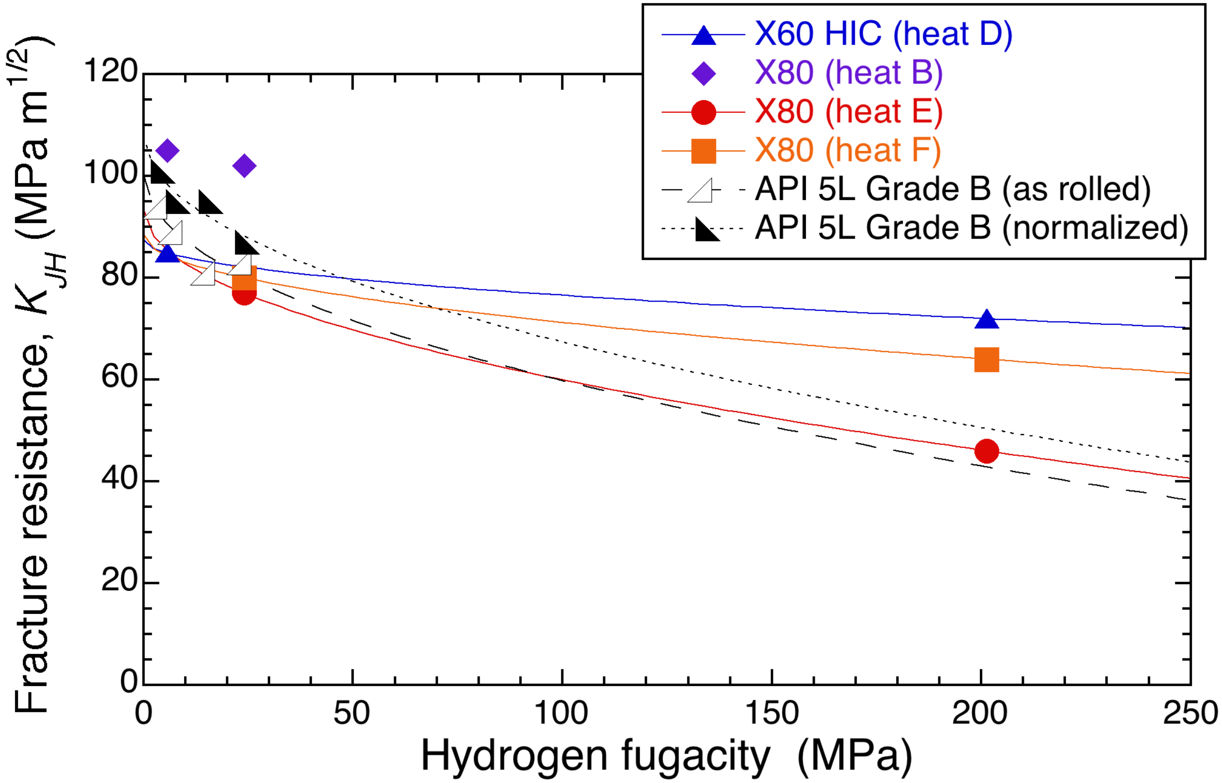
Error bars represent minimum and maximum measured values

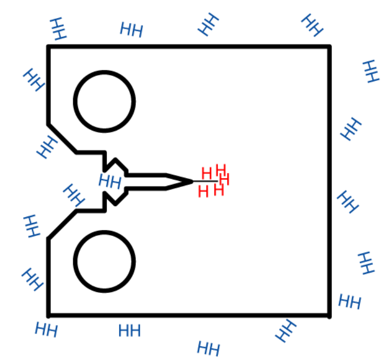


from: ASME PVP2010-25825

Fracture resistance (K_{JH}) in gaseous hydrogen depends on hydrogen fugacity

- Curves represent empirical fit assuming square root dependence on fugacity ($K \propto f^{1/2}$)
- API 5L Grade B: data from literature

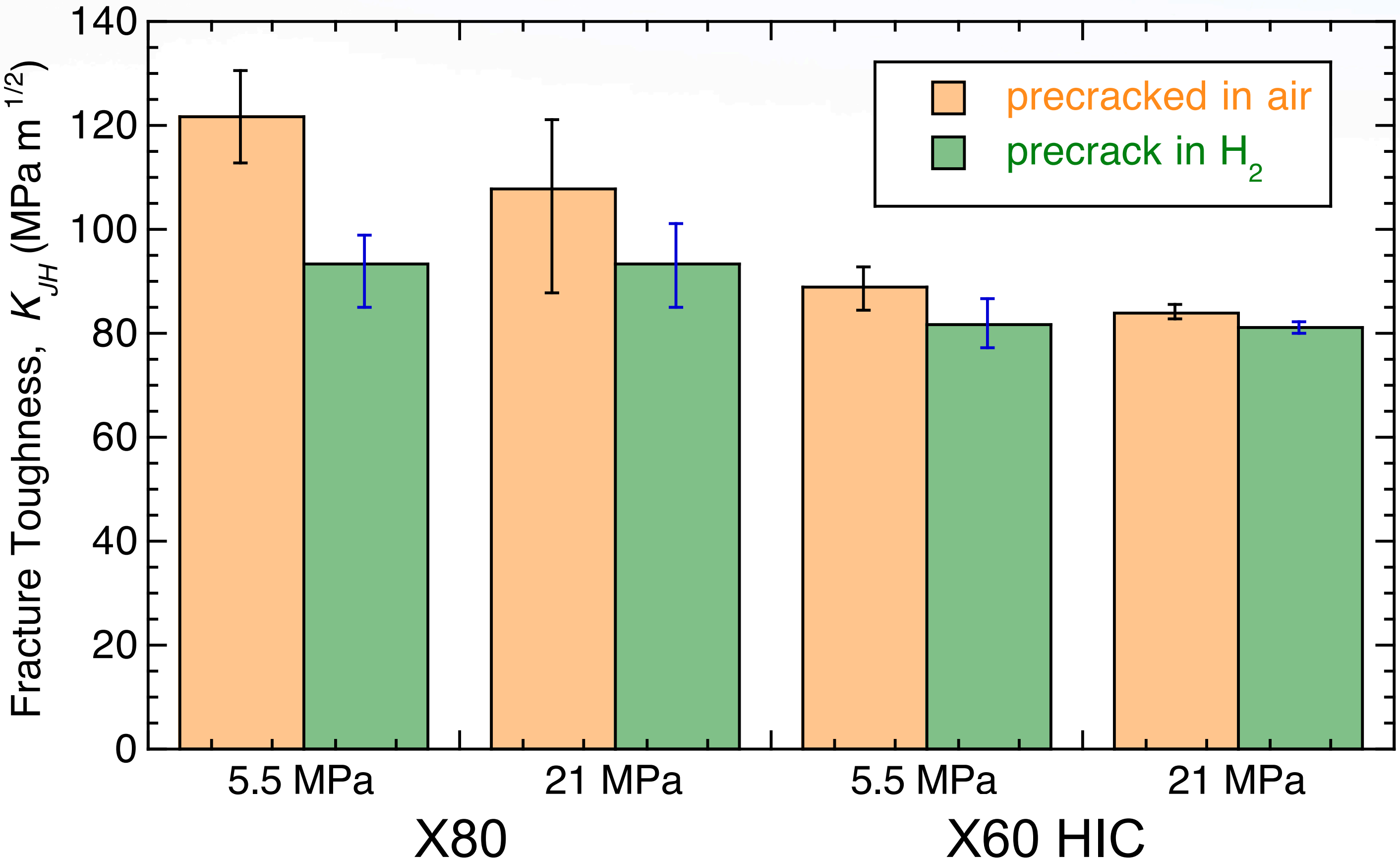


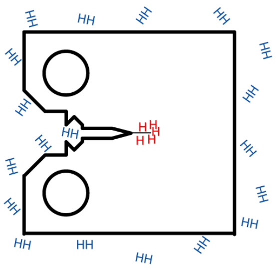


$$f = P \exp\left(\frac{Pb}{RT}\right)$$

from: ASME PVP2011-57684

Fracture resistance (K_{JH}) can be measured after fatigue crack growth testing





from: ASME PVP2010-25825

Summary

- Two fracture thresholds can be identified from constant-displacement fracture tests
  - K^* : stress intensity factor necessary to initiate fracture
  - K_{TH} : stress intensity factor at which a propagating crack arrests
  - $K_{TH} < K^*$
  - Both K_{TH} and K^* are non-conservative with respect to a stationary crack subjected to a dynamic load
- Standard elastic-plastic fracture measurements in gaseous hydrogen (K_{JH}) provide a conservative measure of fracture resistance
  - Differences in fracture measures can be related to the mechanics at the crack tip of stationary and propagating cracks respectively
- Fracture resistance of steels is greatly reduced by *in situ* exposure to gaseous hydrogen
  - Effects are significant, even for low-pressure exposure
  - However, pipeline steels commonly remain ductile: K_{JH} >75 MPa $m^{1/2}$