SAND2015-8337C

# Hydrogen-Accelerated Fatigue Crack Growth in Pipeline Steels and Their Welds

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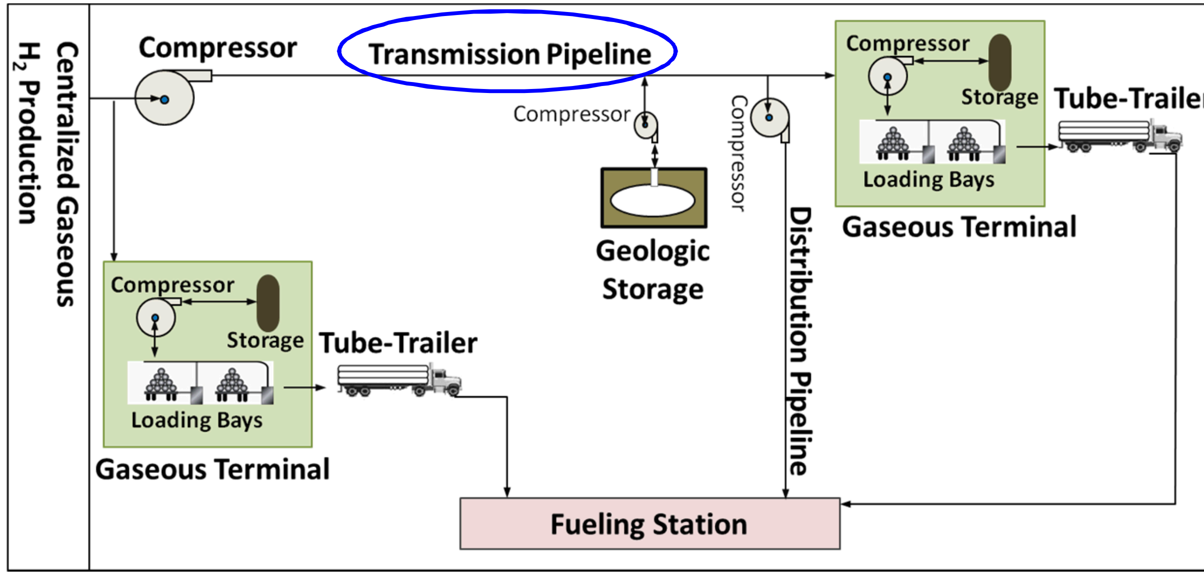
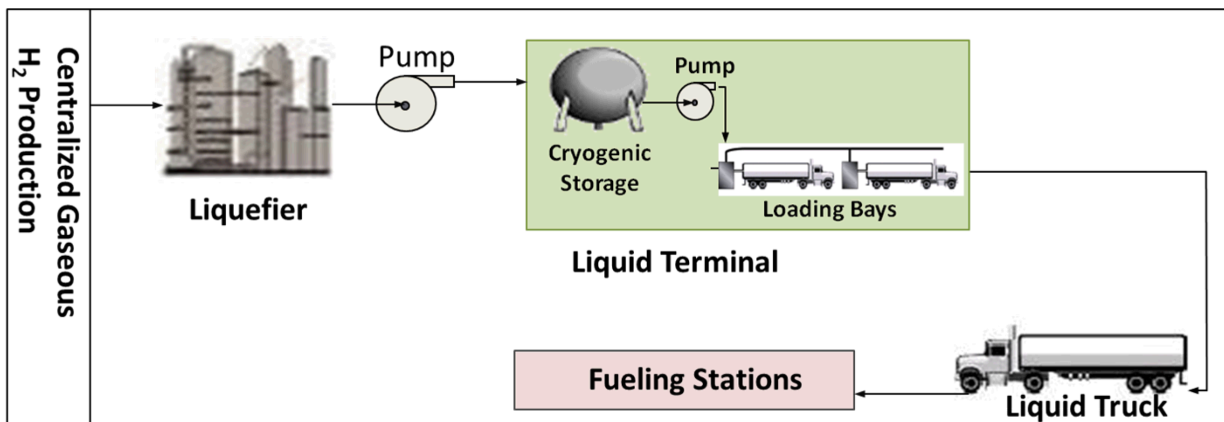
# Structural materials are central focus for cost reduction and reliability of $H_2$ fuel infrastructure

Gaseous Delivery Pathways




Liquid Delivery Pathway




A. Elgowainy, ANL

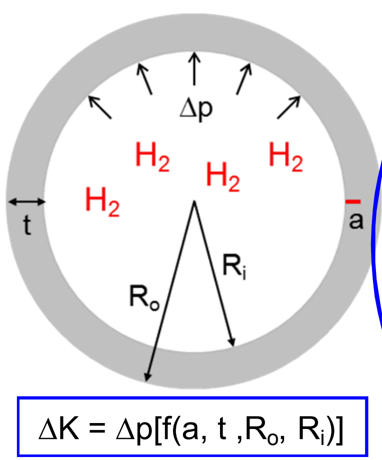
***Hydrogen embrittlement recognized as potential reliability issue for steel $H_2$ pipelines***

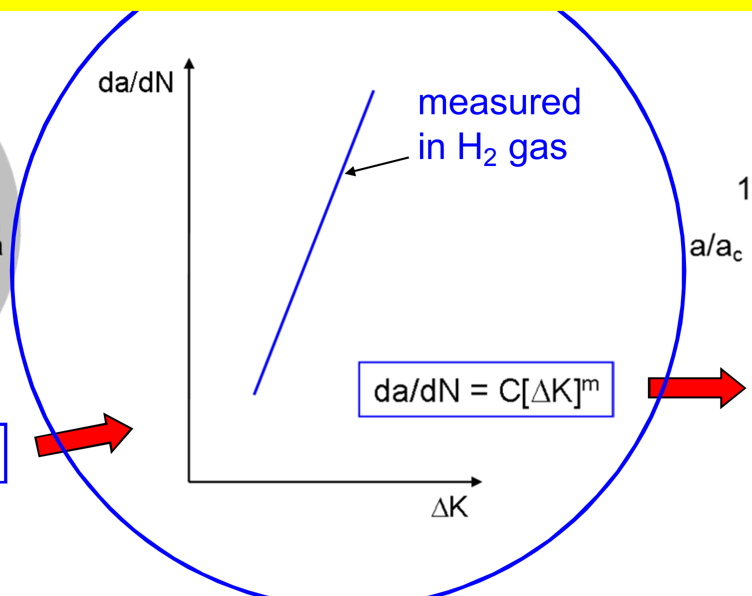
# *Motivation*: Address hydrogen embrittlement in reliability assessment of steel $H_2$ pipelines

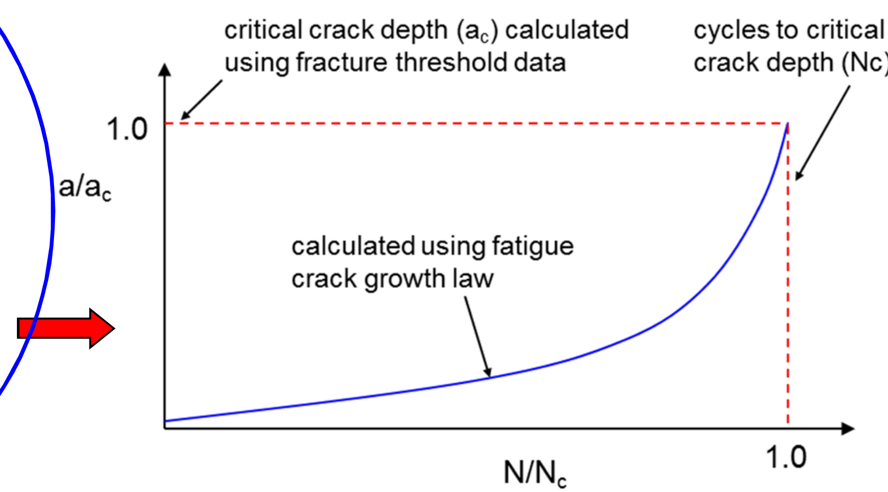
- *Objectives*: Measure fatigue crack growth relationships for relevant pipeline steels in service environment, i.e., $H_2$ gas
  - ***What is effect of microstructure anisotropy on $H_2$-accelerated crack growth in base metal?***
  - ***Are girth welds more susceptible to $H_2$-accelerated crack growth compared to base metal?***
  - ***What is effect of $O_2$ impurities on $H_2$-accelerated crack growth?***

ipelines for

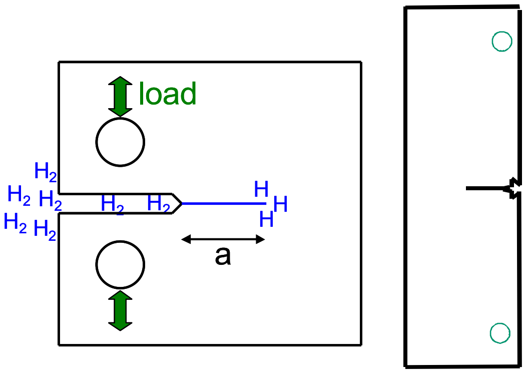
ploy damage-
in $H_2$ gas

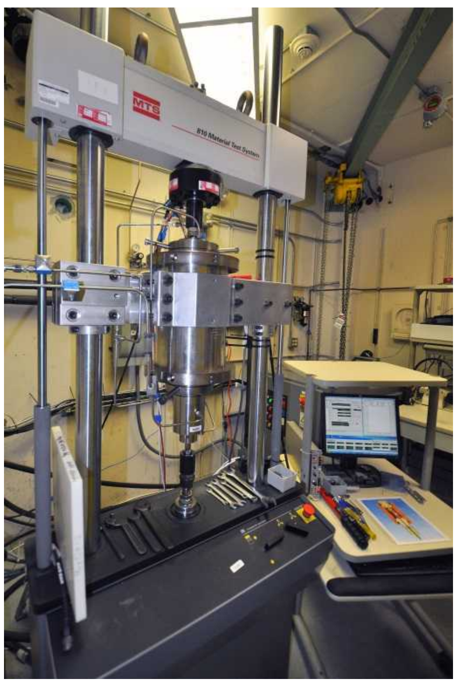


# Fatigue crack growth relationships measured in high-pressure $H_2$ gas



- Materials
  - X65 base metal and gas metal arc weld
  - X52 base metal
- Instrumentation
  - Internal load cell in feedback loop
  - Crack-opening displacement measured internally using LVDT
  - Crack length calculated from compliance
- Mechanical loading
  - Triangular load-cycle waveform
  - Constant load amplitude or constant $\Delta K$
- Environment
  - Supply gases: 99.9999% $H_2$
    $H_2$ with 10-1000 vppm $O_2$
  - Pressure = 21 MPa
  - Room temperature

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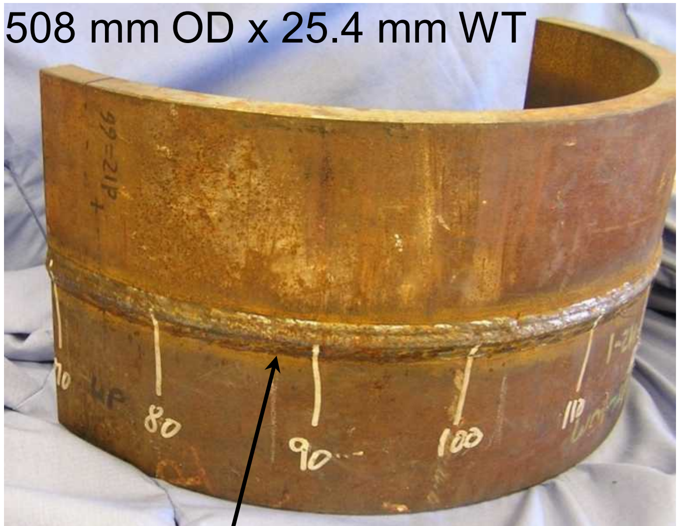
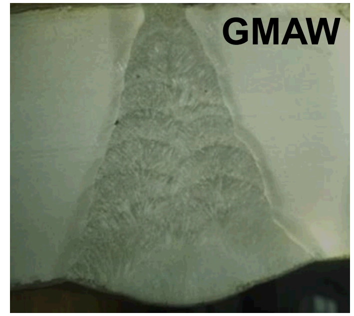
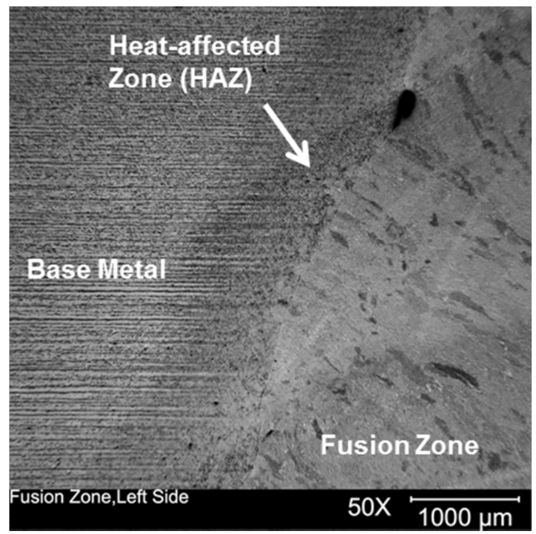
# Measurements performed on technologically relevant pipe: API 5L X65 steel with GMAW

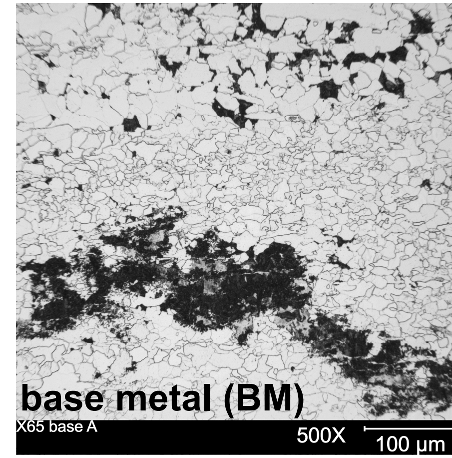


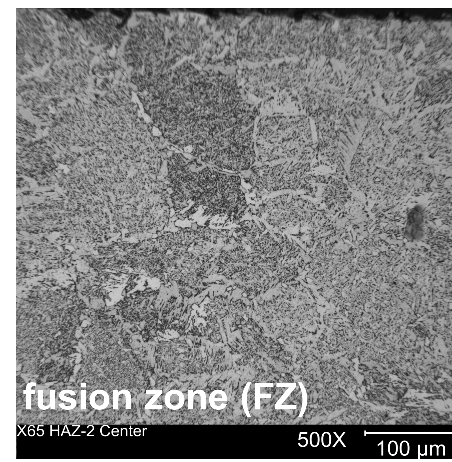

gas metal arc weld (GMAW)

| Material | YS (MPa) | UTS (MPa) |
|---|---|---|
| Base Material | 478 | 564 |
| GMAW | 591 | 662 |




Base Metal Chemical Composition (wt%)

| C | Mn | P | S | B | Si | Cu | Ni | Nb | Ti |
|---|---|---|---|---|---|---|---|---|---|
| 0.08 | 1.53 | 0.01 | 0.001 | 0.002 | 0.32 | 0.024 | 0.038 | 0.039 | 0.002 |

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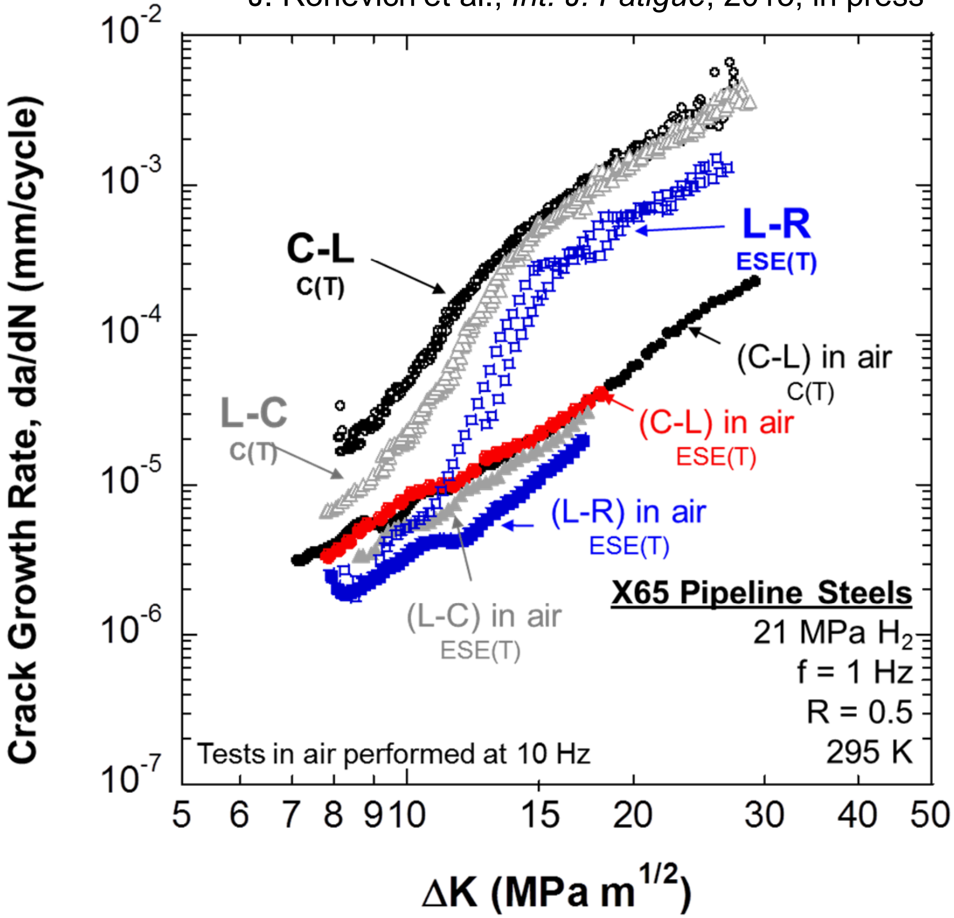
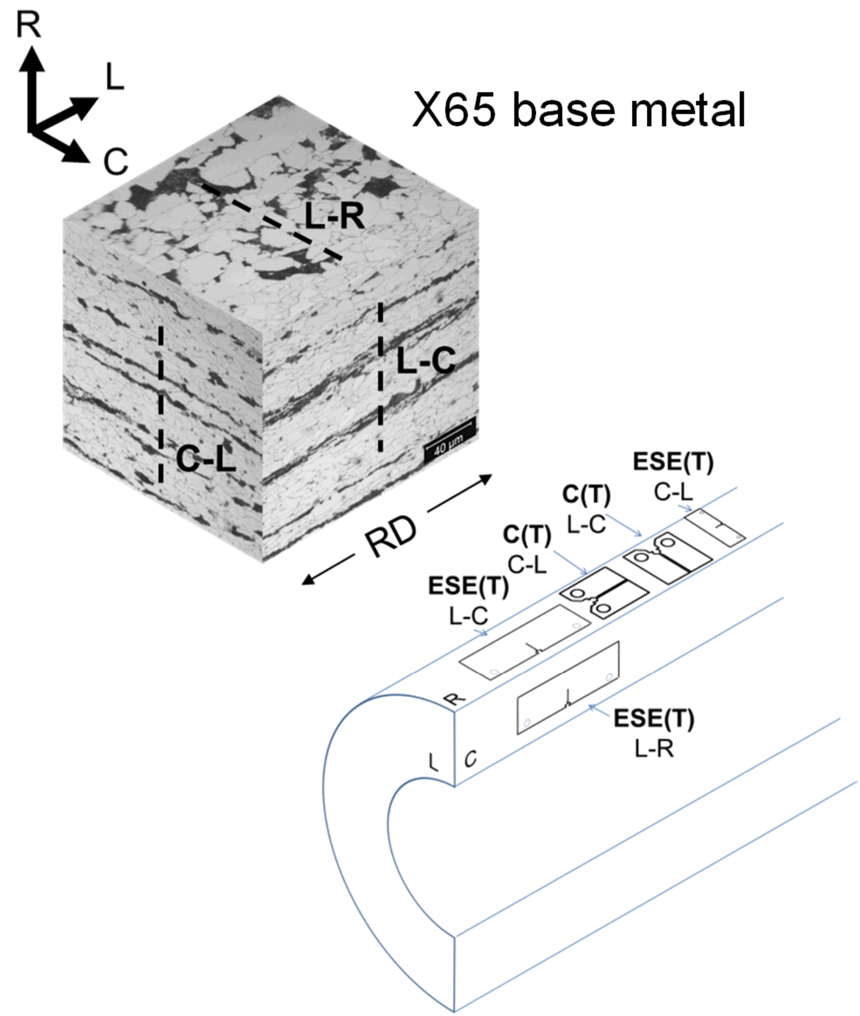
# Fatigue crack growth rates in X65 base metal depend on specimen orientation

J. Ronevich et al., *Int. J. Fatigue*, 2015, in press

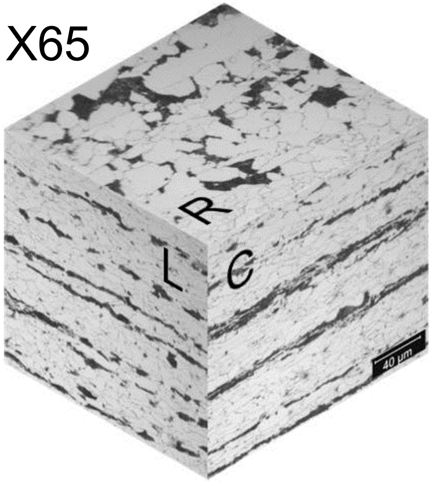


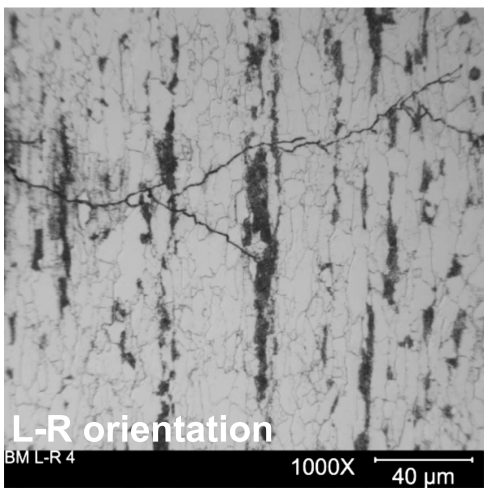




***Does banded ferrite-pearlite microstructure reduce crack growth rates in L-R orientation?***

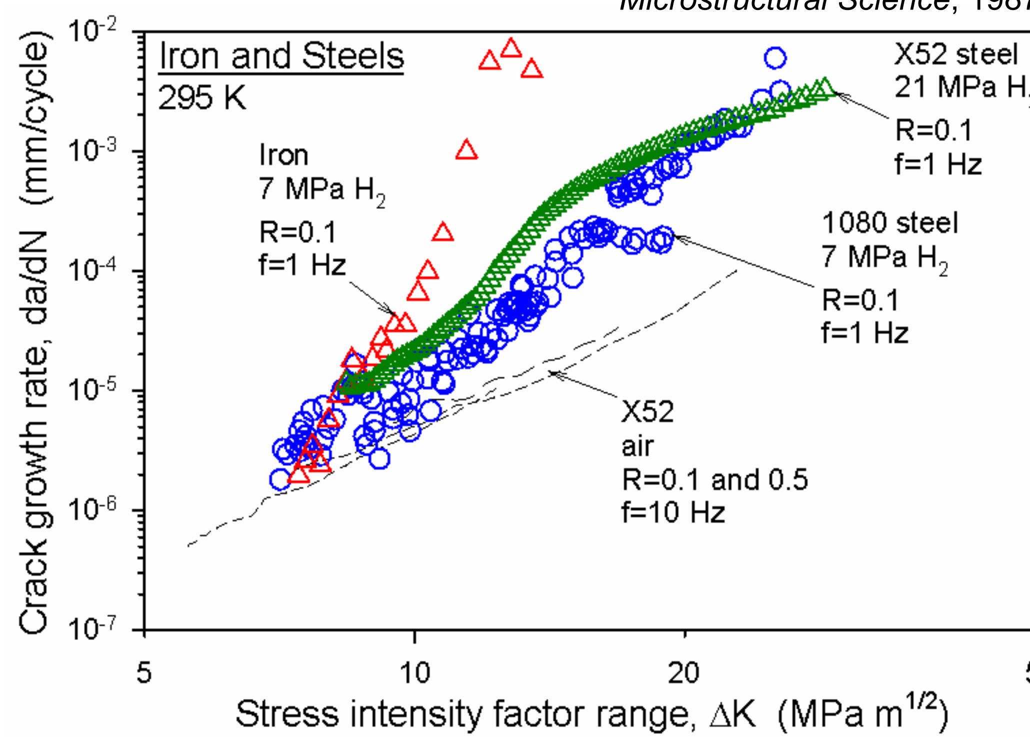
# Reduced crack growth rates in L-R orientation attributed to banded ferrite-pearlite

X65




H. Cialone and J. Holbrook, *Microstructural Science*, 1987




- ***Pearlite bands induce crack branching***
- ***$H_2$-assisted crack growth rates lower through pearlite bands***

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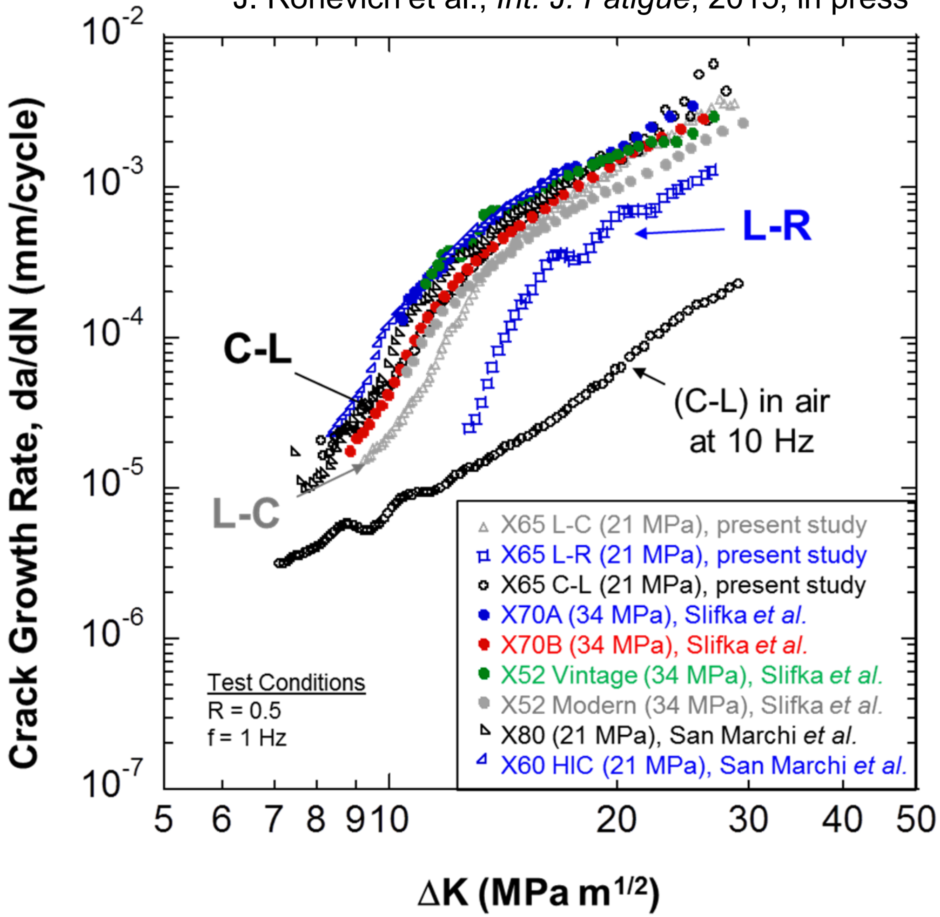
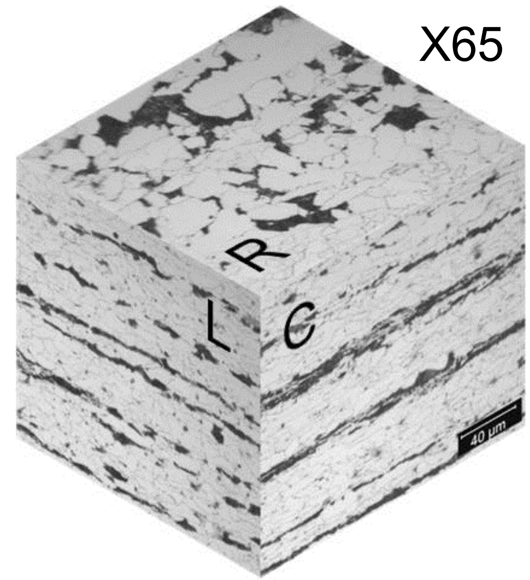
# $H_2$-assisted fatigue crack growth generally not sensitive to microstructure in pipeline steel BM

J. Ronevich et al., *Int. J. Fatigue*, 2015, in press




X60 and X80 data:
San Marchi et al., ASME PVP, 2010

X52 and X70 data:
Slifka et al., ASME PVP, 2014
Drexler et al., Proceedings of SteelyHydrogen, 2014

***Most pronounced microstructure effect is banded ferrite-pearlite in L-R orientation***

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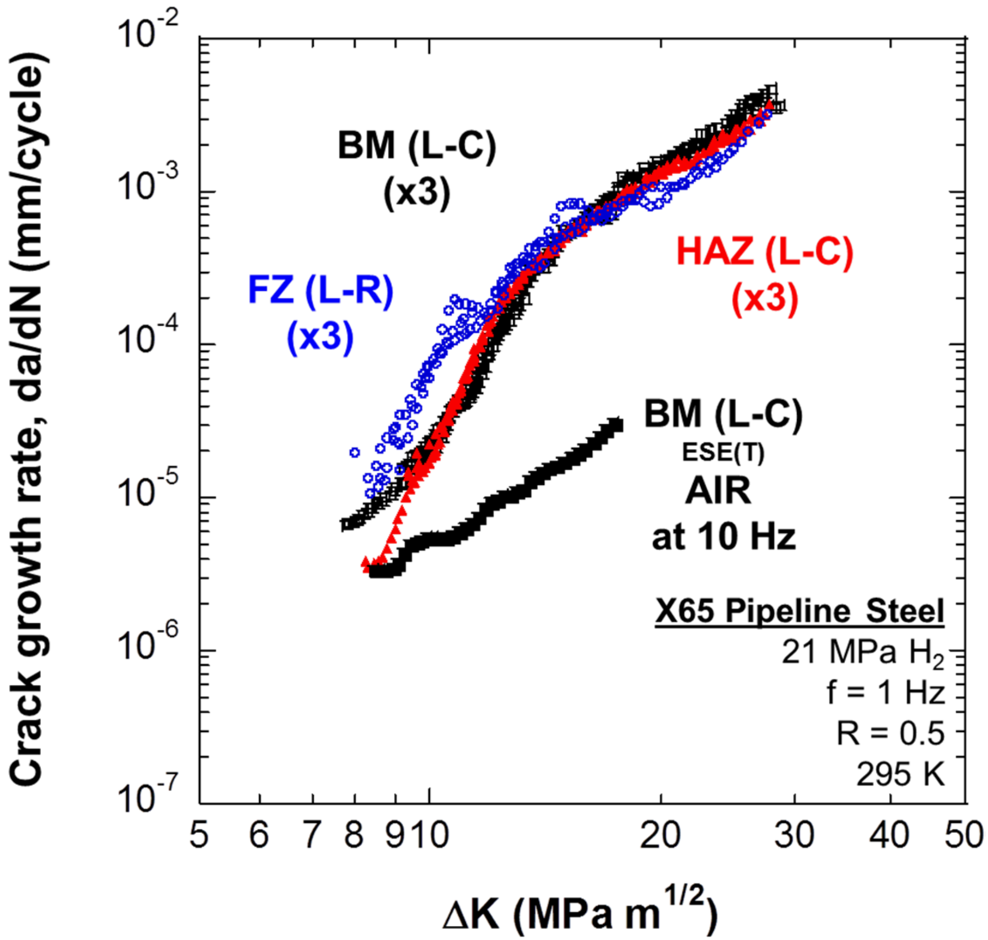
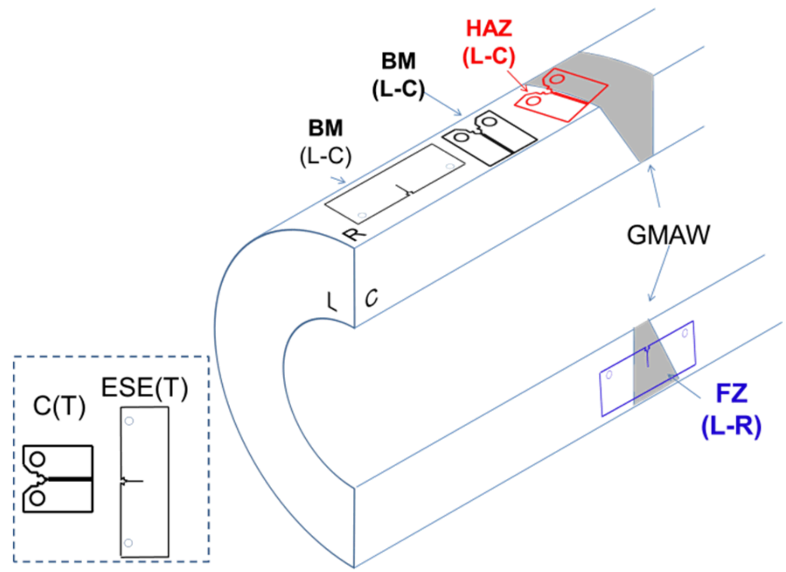
# Measurements of $H_2$-assisted fatigue crack growth repeatable for X65 GMAW

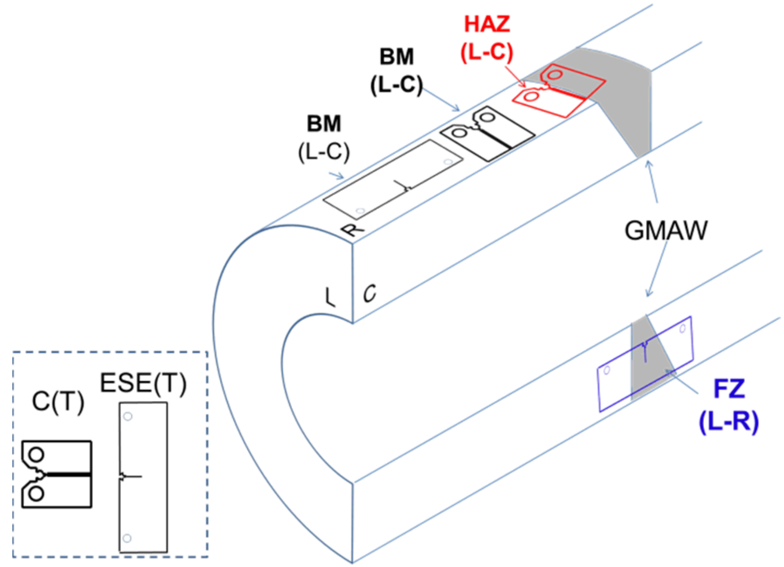


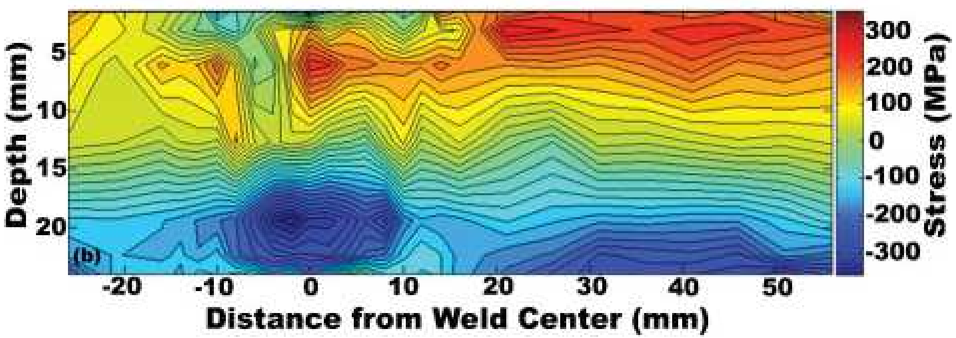

***Measurements for weld may not reflect intrinsic material behavior because of residual stresses***

# Residual stresses quantified in X65 GMAW through neutron scattering measurements

pipe longitudinal direction



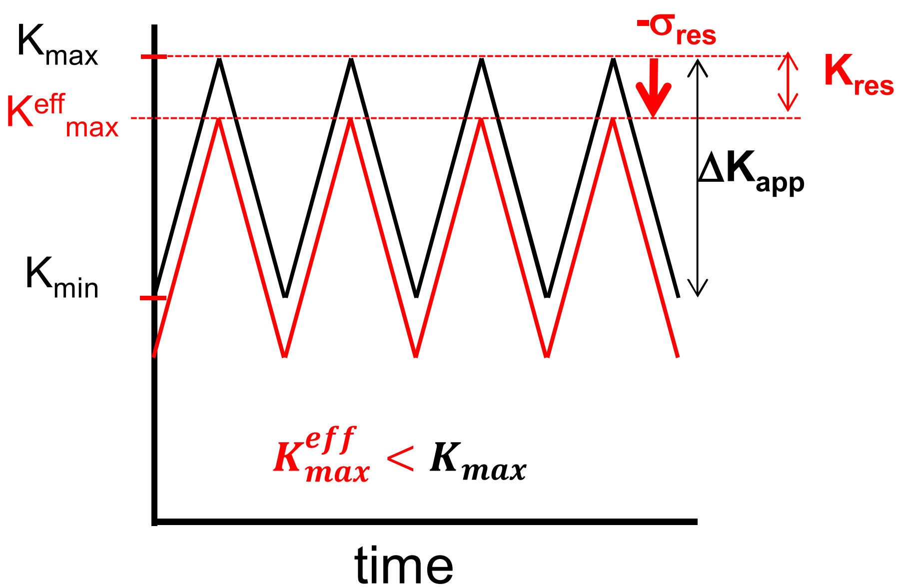

longitudinal stress



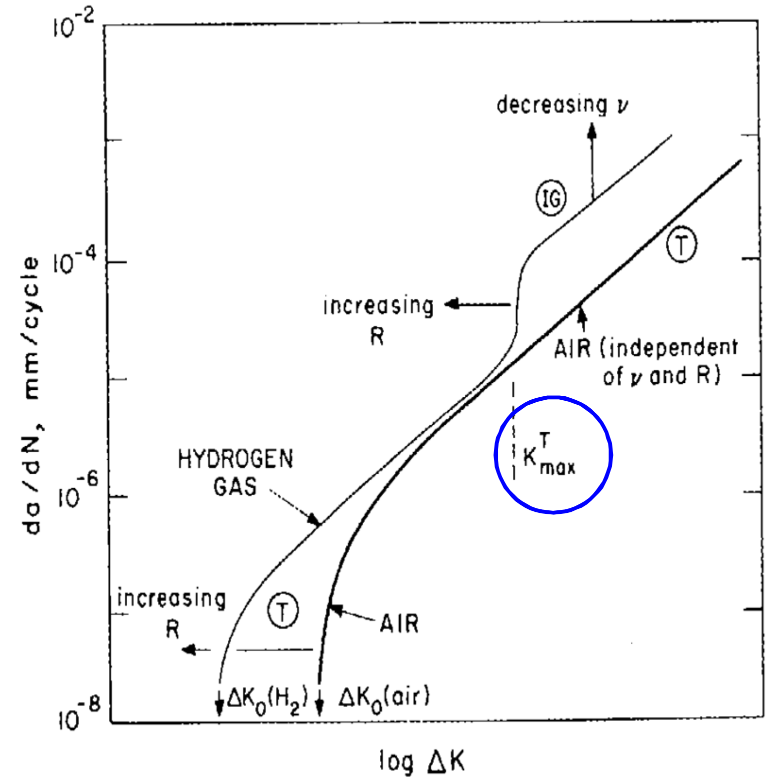

T. Neeraj, *Science and Technology of Welding and Joining*, 2011

***Significant residual stress gradient through X65 pipe wall at weld***

# Residual stresses can modify applied $K_{max}$




S. Suresh and R.O. Ritchie, *Metal Science*, 1982

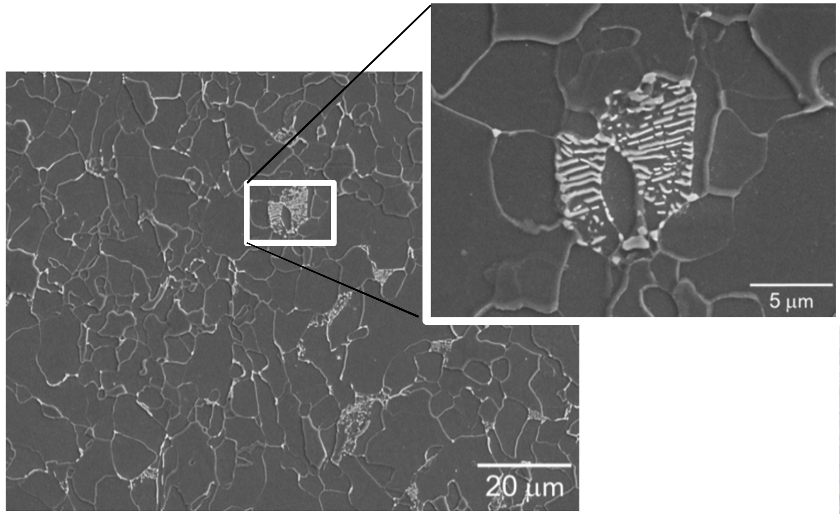



***Residual stresses can alter onset of $H_2$-accelerated crack growth by modifying $K_{max}$***

I²CNER

# Measurements performed on technologically relevant steel: API 5L X52 (PSL 2)

ferrite + 8 vol% pearlite





324 mm OD x 12.7 mm WT

- Tensile properties
  - Yield strength: 428 MPa
  - Ultimate tensile strength: 483 MPa

- Alloy composition

| C | Mn | P | S | Si | Cu | Ni | Cr | V | Nb | Al | CE |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.06 | 0.87 | 0.011 | 0.006 | 0.12 | 0.03 | 0.02 | 0.03 | 0.002 | 0.03 | 0.034 | 0.11 |

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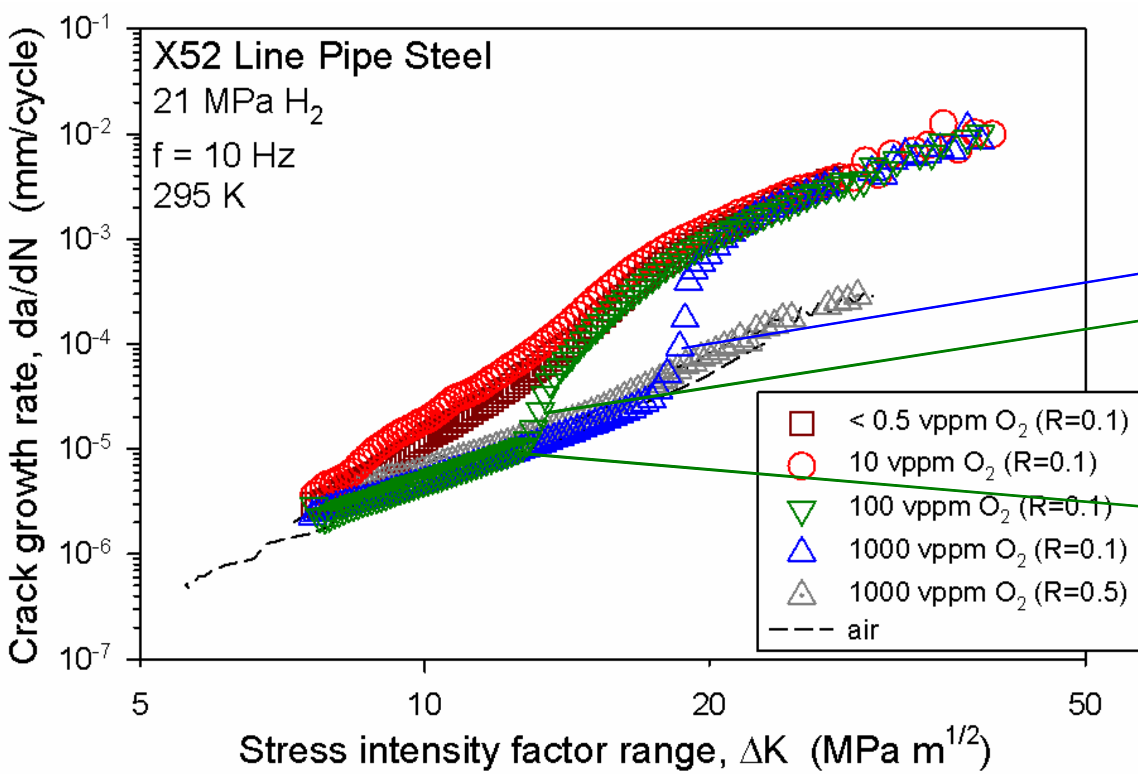
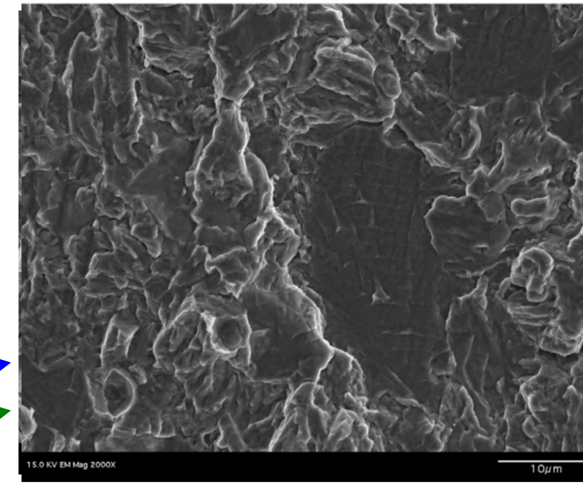
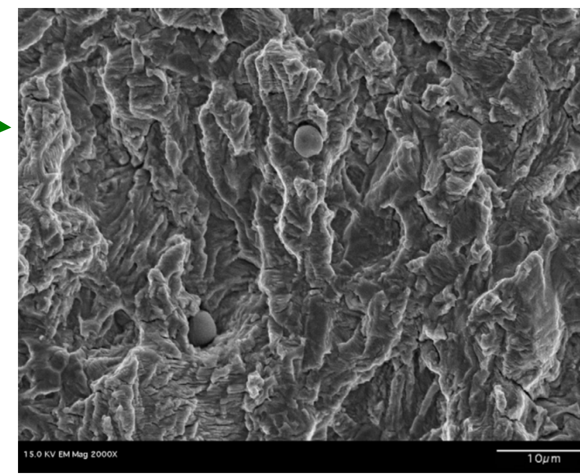
B. Somerday et al., *Acta Materialia*, 2013

# *da*/*dN* vs. Δ*K* measured in $H_2$ gas as function of $O_2$ content at fixed frequency and *R* ratio

B. Somerday et al., *Acta Mater*, 2013



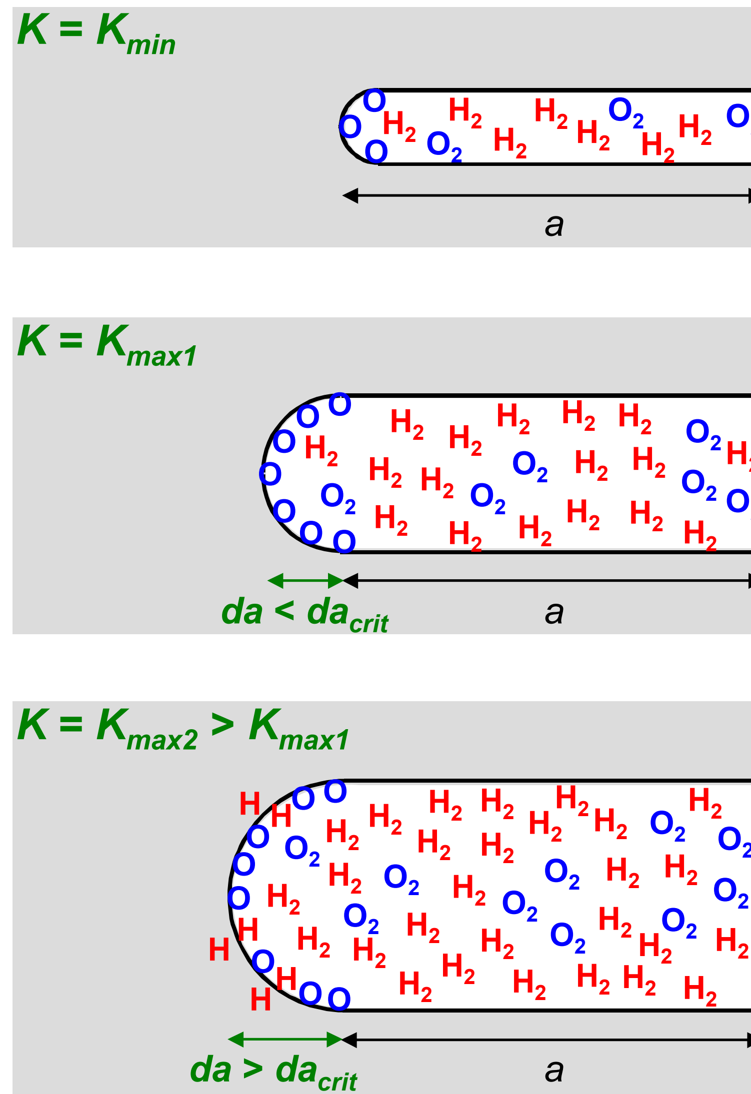


***Onset of $H_2$-accelerated crack growth delayed to higher ΔK level as $O_2$ concentration increases***

# Assumption: measured fatigue crack growth trends governed by $O_2$ inhibition of H uptake

- Initial inert-environment crack growth modeled by blunting-resharpening
- Oxygen out-competes hydrogen for adsorption sites on freshly exposed crack-tip surface
- Extent of oxygen adsorption depends on crack-tip area, proportional to crack-growth increment (*da*)
  - when $da < da_{crit}$, crack tip *fully passivated* by oxygen
  - when $da > da_{crit}$, crack tip *not fully passivated* → H uptake
- Develop model that quantitatively relates adsorbed oxygen (H uptake) to mechanical and environmental variables

B. Somerday et al., *Acta Mater*, 2013

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# Summary

- Fatigue crack growth relationships measured for pipeline steel base metal and girth weld in $H_2$ gas
  - Orientation-dependent fatigue crack growth rates in base metal attributed to pearlite banding
  - Weld microstructures not inherently more susceptible to $H_2$-accelerated crack growth compared to base metal
- Effect of trace $O_2$ on $H_2$-accelerated fatigue crack growth quantified for pipeline steel
  - Measurements reveal notable effects of $O_2$ concentration, load-cycle frequency, and *R* ratio
  - Analytical model accurately captures interplay between $O_2$ concentration, $da/dN$, frequency, and *R* ratio

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# Acknowledgments

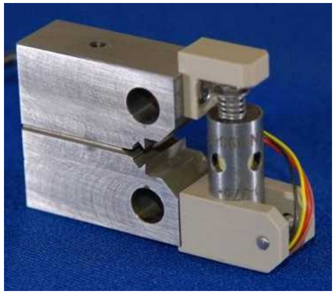
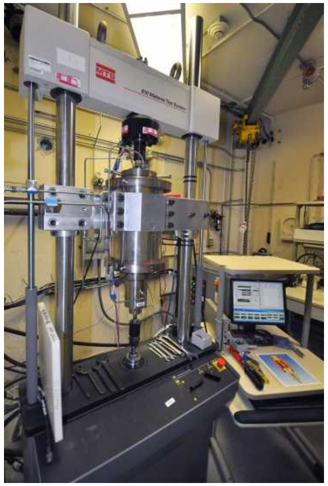

- Sandia National Laboratories, Hydrogen Effects on Materials Laboratory team
  - Chris San Marchi
  - Ken Lee
  - Jeff Campbell
  - Mark Zimmerman

- International Institute for Carbon Neutral Energy Research
  - Prof. Alex Staykov (Kyushu University)
  - Prof. Petros Sofronis (University of Illinois)
  - Prof. Reiner Kirchheim (University of Göttingen)

# SNL core capability in hydrogen embrittlement features Hydrogen Effects on Materials Lab

- Static-loading crack-growth system
  - Wedge opening load (WOL) and double cantilever beam (DCB) specimens
  - $H_2$ pressure up to 200 MPa
  - Temperature -70 to 170 $^oC$
- Dynamic-loading crack-growth system
  - Compact tension (CT) and single edge notch (SEN) specimens
  - $H_2$ pressure up to 138 MPa
  - New pressure vessel design with target temperatures -100 to 200 $^oC$




***Materials testing in $H_2$ supports technology development in several mission areas***

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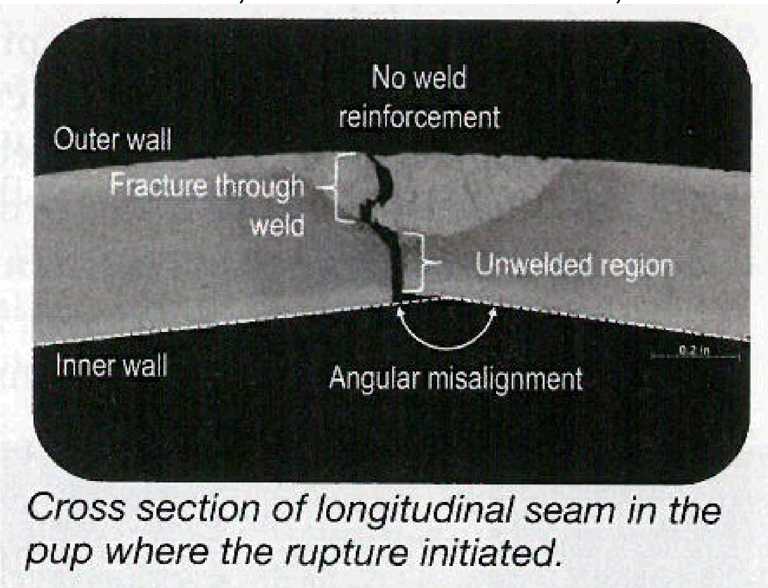
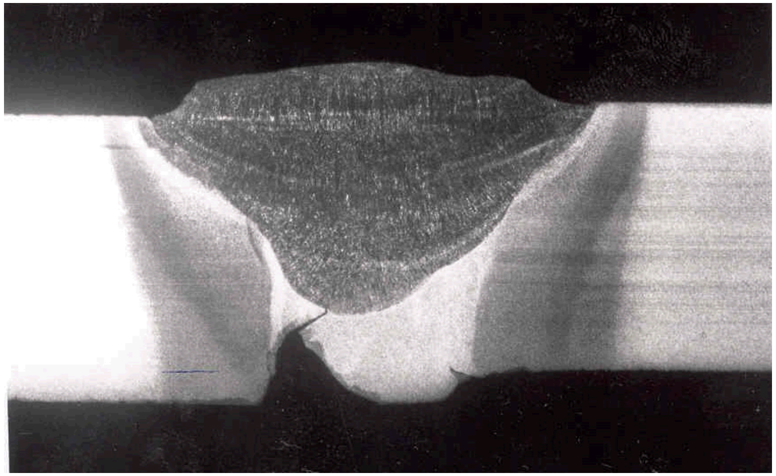
# Structural integrity management for hydrogen pipelines must focus on welds

F. Richards, *Adv Mat & Processes*, 2013



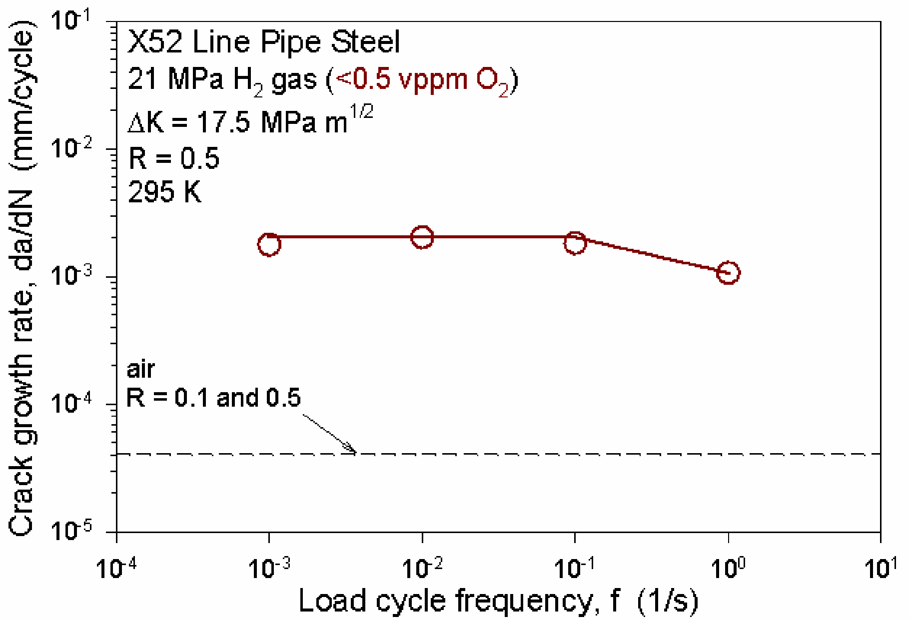

I. Alliat, NATURALHY EC project, 2007

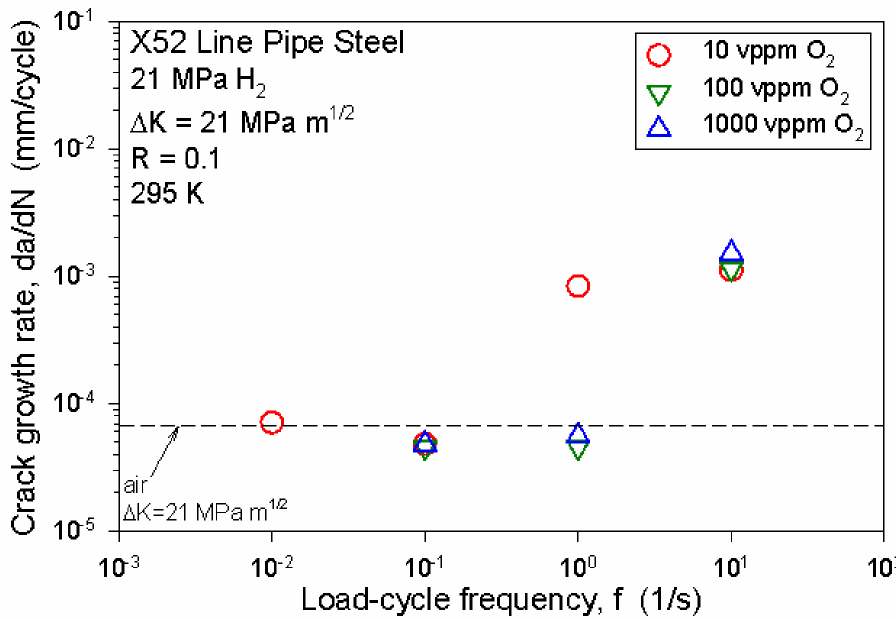


- Welding can create defects, increasing probability of crack growth in these regions
- ***Are weld microstructures (fusion zone, heat-affected zone) more susceptible to $H_2$-assisted fatigue crack growth?***

# *da/dN* measured as function of frequency in $H_2$ gas over range of $O_2$ concentrations

B. Somerday et al., *Acta Mater*, 2013




- ***In $H_2$ gas with $O_2$ impurities, da/dN decreases to rates in air as frequency decreases***
- ***Frequency at transition from accelerated da/dN to rates in air depends on $O_2$ concentration***

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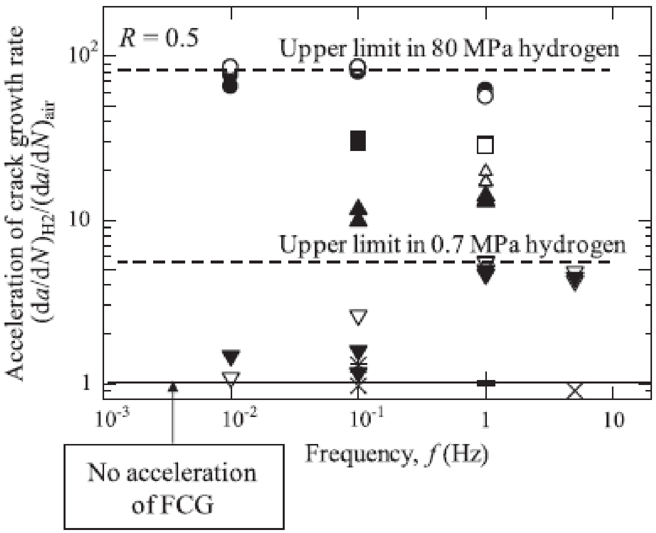
# Model can guide interpretation of $H_2$-assisted fatigue crack growth data

Macadre et al., *Engineering Fracture Mechanics*, 2011




Hydrogen atmosphere

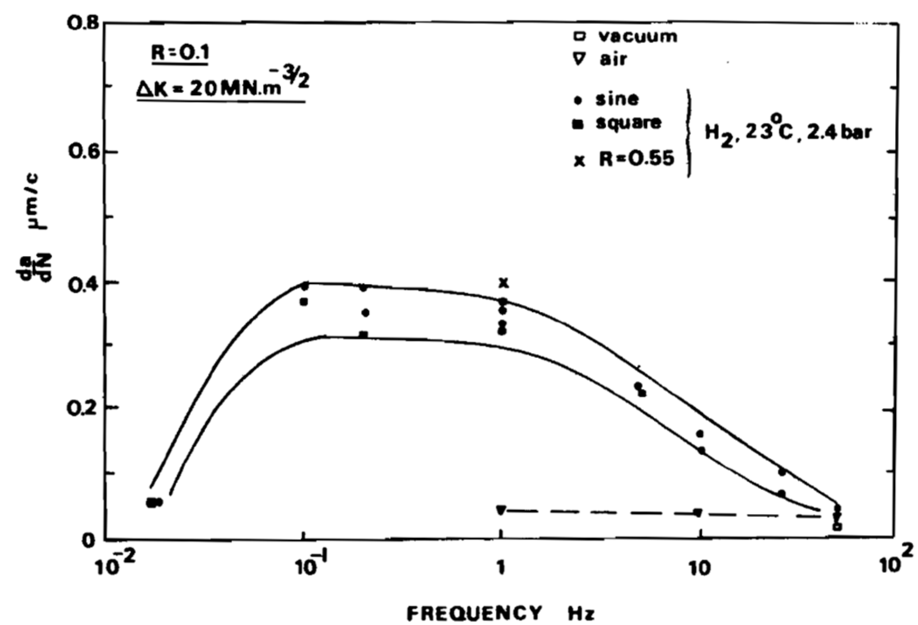
| Specimens | 0.7 MPa | 10 MPa | 40~45 MPa | 90 MPa |
|---|---|---|---|---|
| Circumferential (SCI, SCO) | ▼ | ▲ | ■ | ● |
| Longitudinal (SLI, SLO) | ▽ | △ | □ | ○ |

Nitrogen atmosphere

| Specimens | 0.7 MPa $N_2$ | 90 MPa $N_2$ |
|---|---|---|
| Longitudinal (SLI, SLO) | × | + |

$$f\big|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{(da/dN)\pi S\theta_{crit} R_g T E\sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$

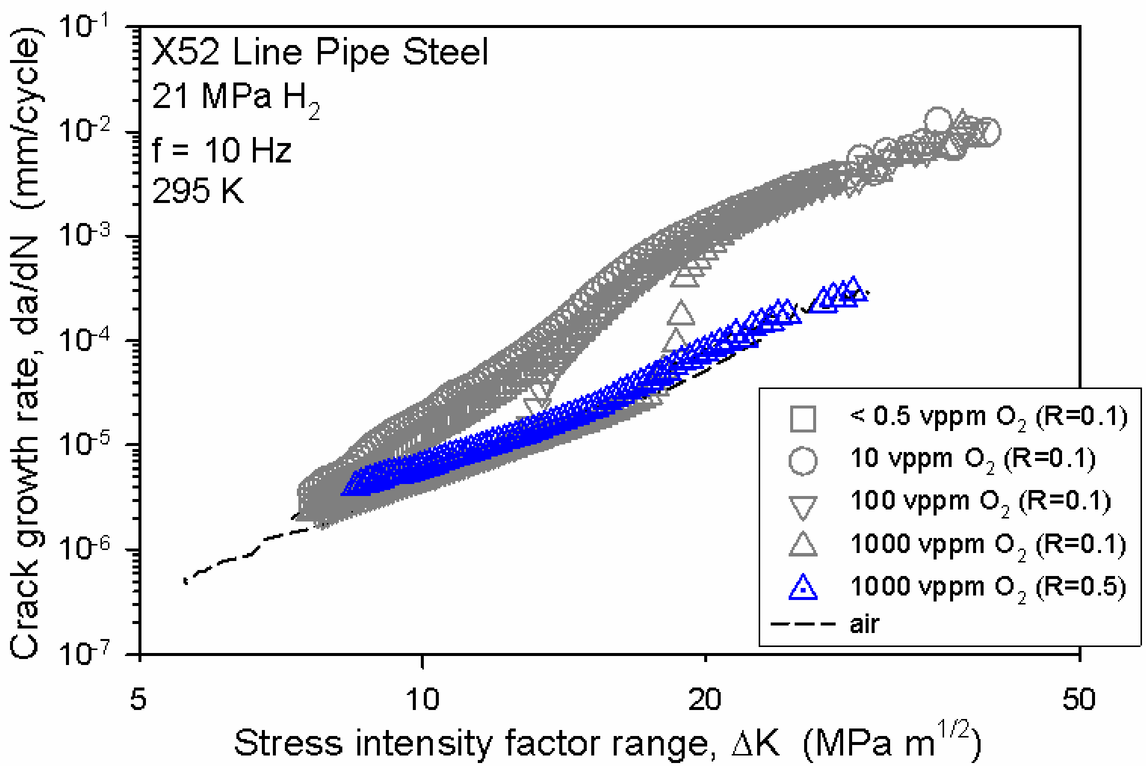
Stewart, *Mechanisms of Environment Sensitive Cracking of Materials*, 1977




***Model indicates that decreasing da/dN at lower frequency could be attributed to $O_2$ impurities***

# $da/dN$ vs. $\Delta K$ measured at higher $R$ ratio in $H_2$+1000 vppm $O_2$

Somerday et al., *Acta Mater*, 2013

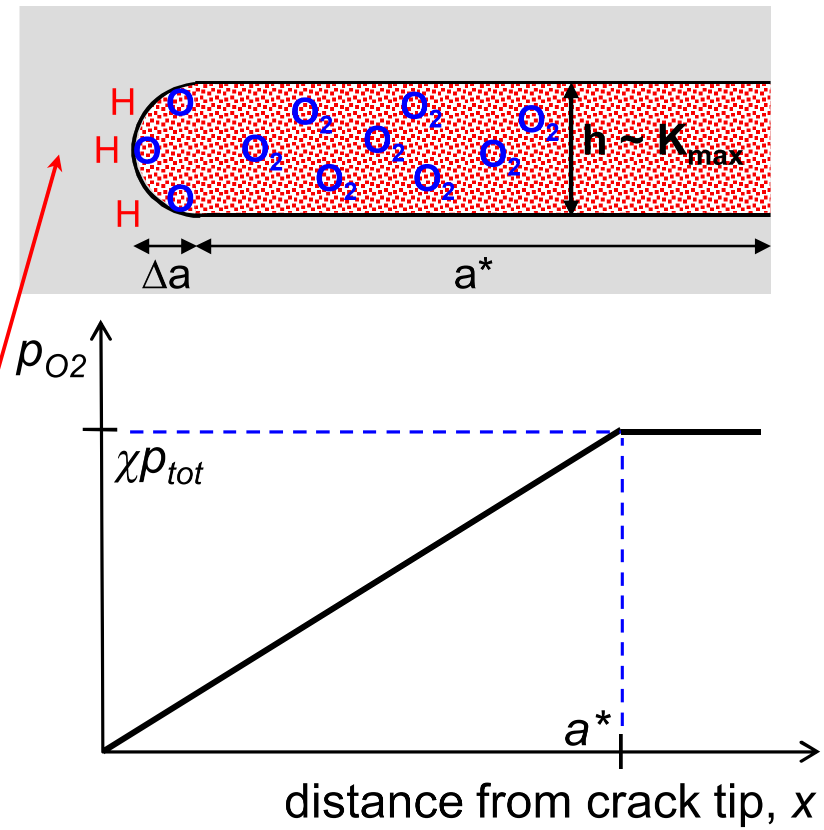



**In $H_2$+1000 vppm $O_2$, accelerated crack growth not observed at higher R ratio**

# Model developed based on idealized crack geometry and diffusion-limited oxygen adsorption

Somerday et al., *Acta Mater*, 2013

- *Goal:* quantify amount of adsorbed oxygen ($n$) during load-cycle time ($\Delta t$)
- *Key assumption: adsorption rate-limited by $O_2$ diffusion in crack channel*
  - constant crack-channel height ($h$) during diffusion
  - steady state $p_{O2}$ profile
- Model foundation: oxygen delivered to crack tip ($Jh\Delta t$) = oxygen adsorbed on crack tip ($S\theta\pi\Delta a$)




$$J = \text{flux} = D\frac{\chi p_{tot}}{R_g T a^*}$$

$$h = \text{channel height} = 0.6(1-\nu^2)\frac{\sigma_0}{E}\left(\frac{\Delta K}{\sigma_0(1-R)}\right)^2$$

$$\Delta t = 1/f$$

$\theta$ = oxygen coverage  $S$ = surface site density

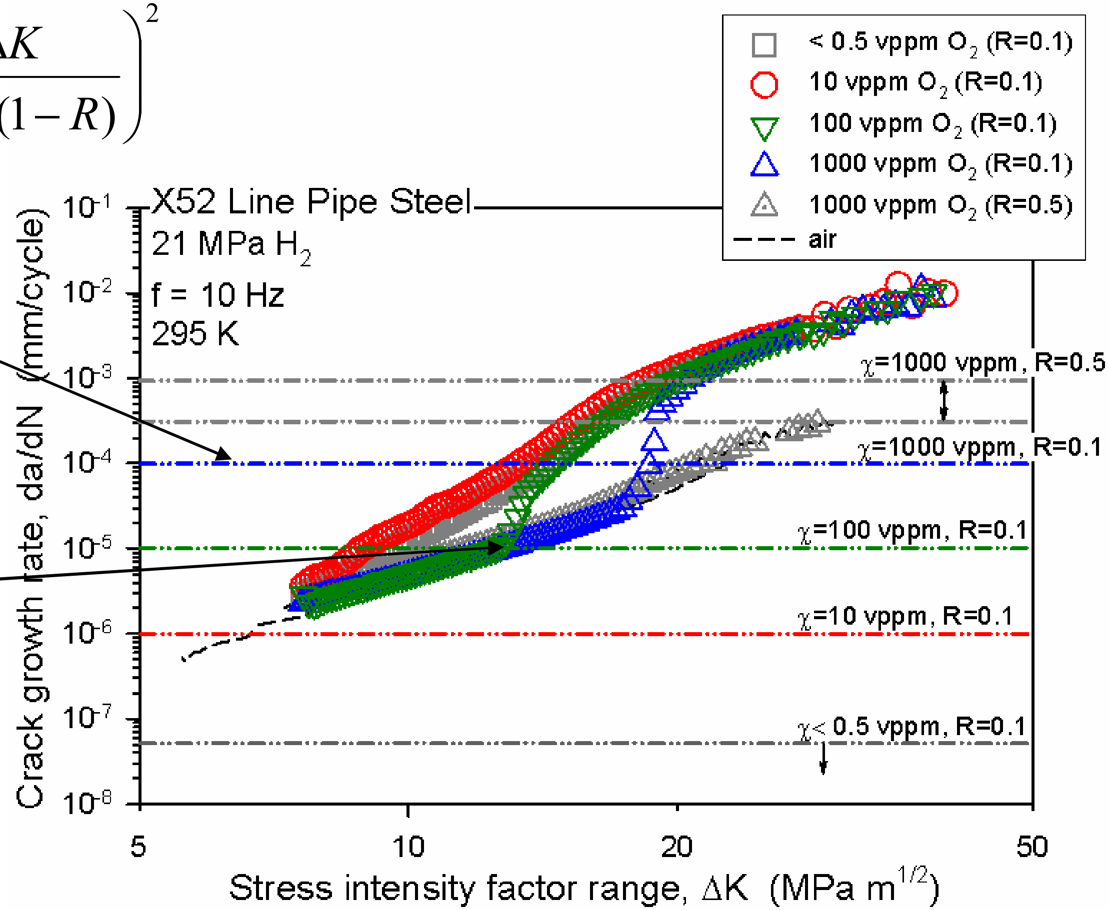
**H uptake and accelerated crack growth when $\theta = \theta_{crit}$**

$$(\Delta a)f\Big|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{\pi S\theta_{crit} R_g T E\sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$

# Model predictions consistent with *da/dN* vs. $\Delta K$ data measured in $H_2$+$O_2$ gas at $R$ = 0.1

$$\left.\frac{da}{dN}\right|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{f\pi S\theta_{crit} R_g T E \sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$

$S\theta_{crit}$ calculated from measured *da/dN* at onset of accelerated crack growth for $H_2$+100 vppm $O_2$



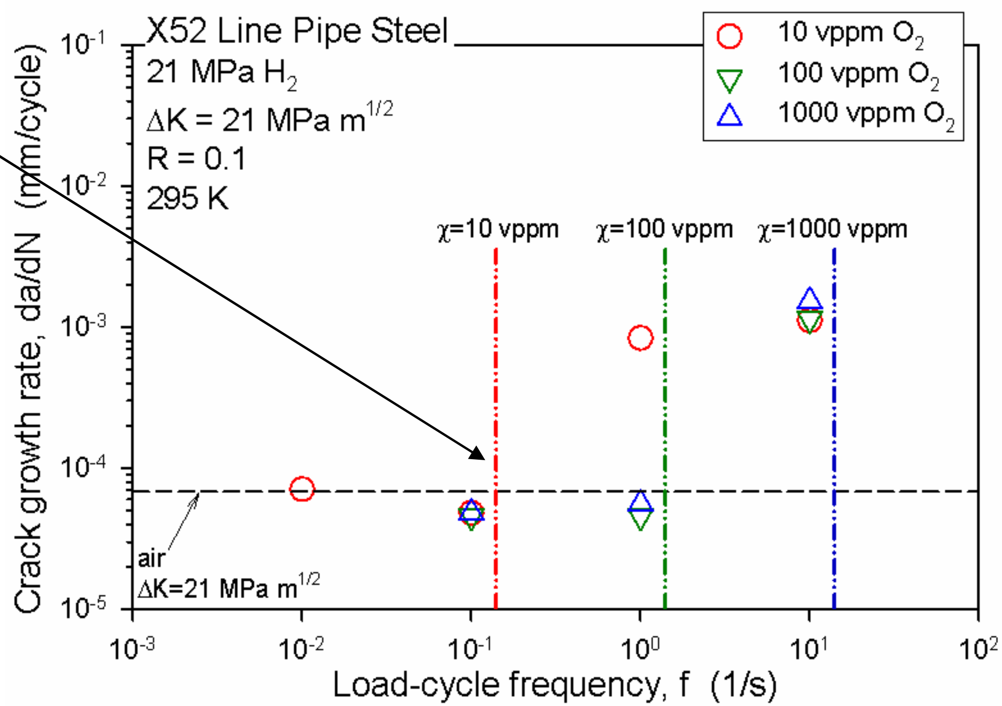

***Model reasonably predicts measured da/dN at onset of accelerated cracking for 10 and 1000 vppm $O_2$***

Somerday et al., *Acta Mater*, 2013

# Model predictions consistent with *da/dN* vs. frequency data measured in $H_2$+$O_2$ gas

$$f\big|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{(da/dN)\pi S\theta_{crit} R_g T E \sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$

$S\theta_{crit}$ from measured *da/dN* vs. $\Delta K$ data in $H_2$+100 vppm $O_2$



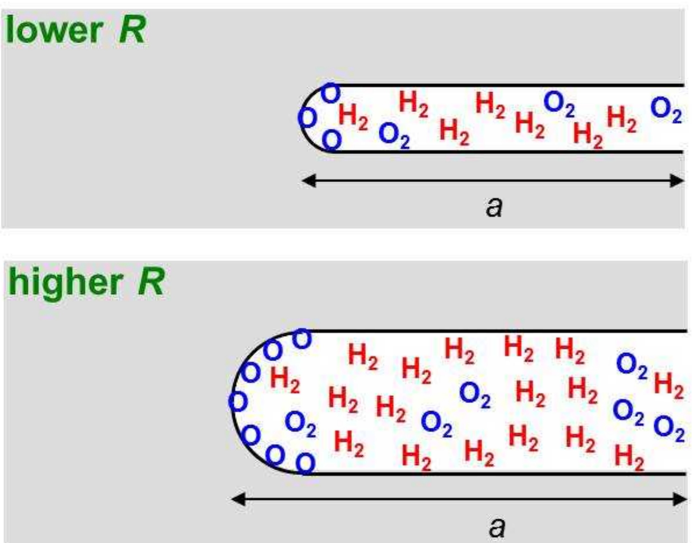

***Predicted frequency for transition to accelerated da/dN consistent with measurements at 10 and 100 vppm $O_2$***

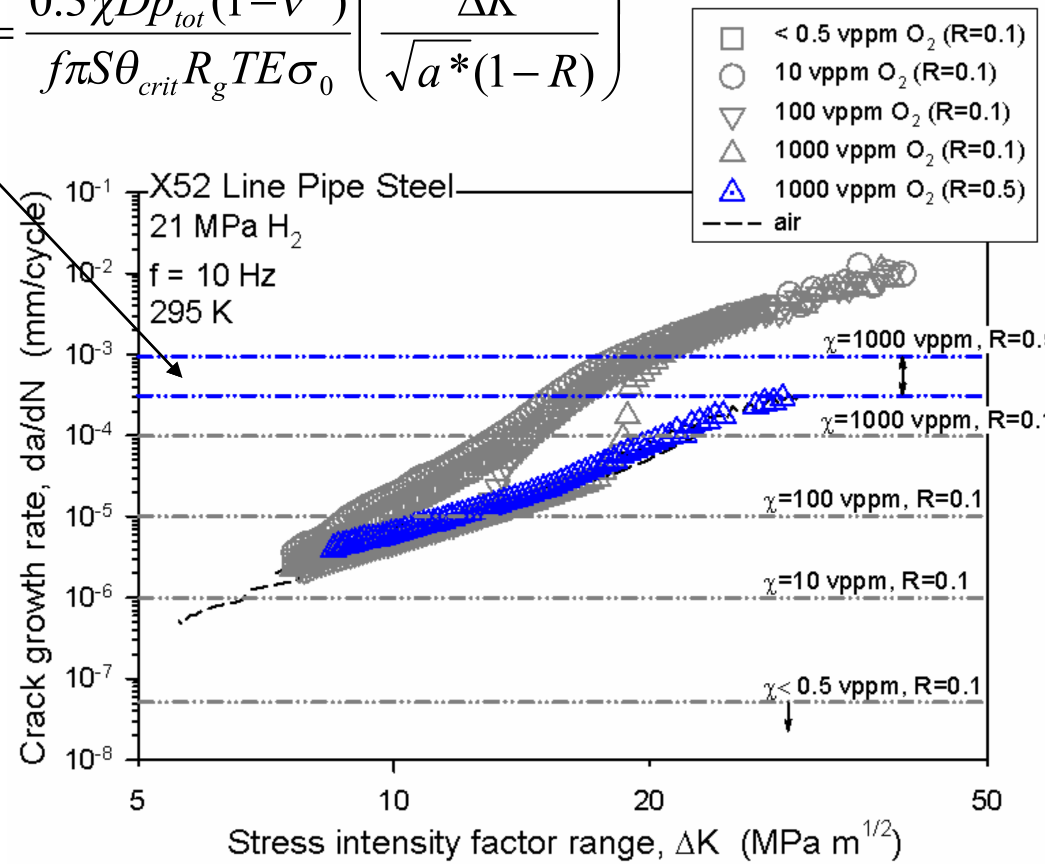
Somerday et al., *Acta Mater*, 2013

# Model predicts inhibition of accelerated crack growth in $H_2$+$O_2$ gas at higher *R* ratio

$S\theta_{crit}$ from measured *da/dN* vs. $\Delta K$ data in $H_2$+100 vppm $O_2$

$$\left.\frac{da}{dN}\right|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{f\pi S\theta_{crit} R_g T E \sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$



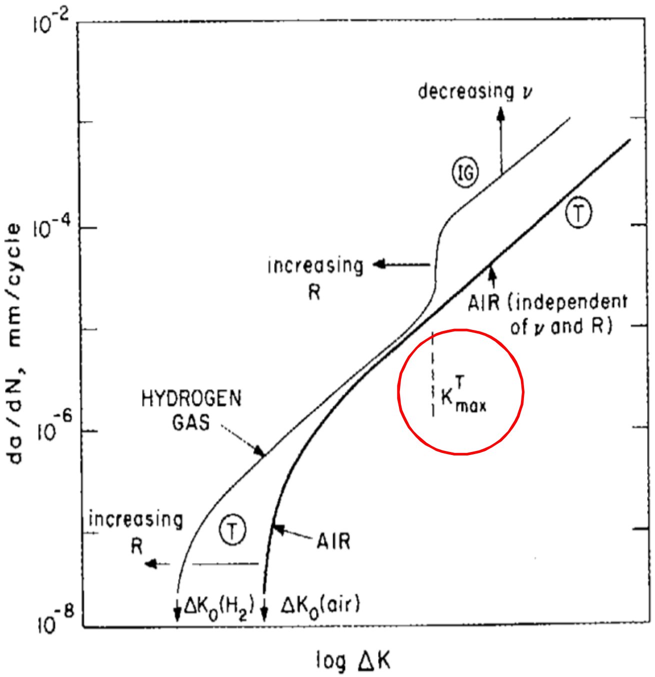

***Enhanced inhibition at higher R ratio associated with effect of crack channel height on $O_2$ transport***

Somerday et al., *Acta Mater*, 2013

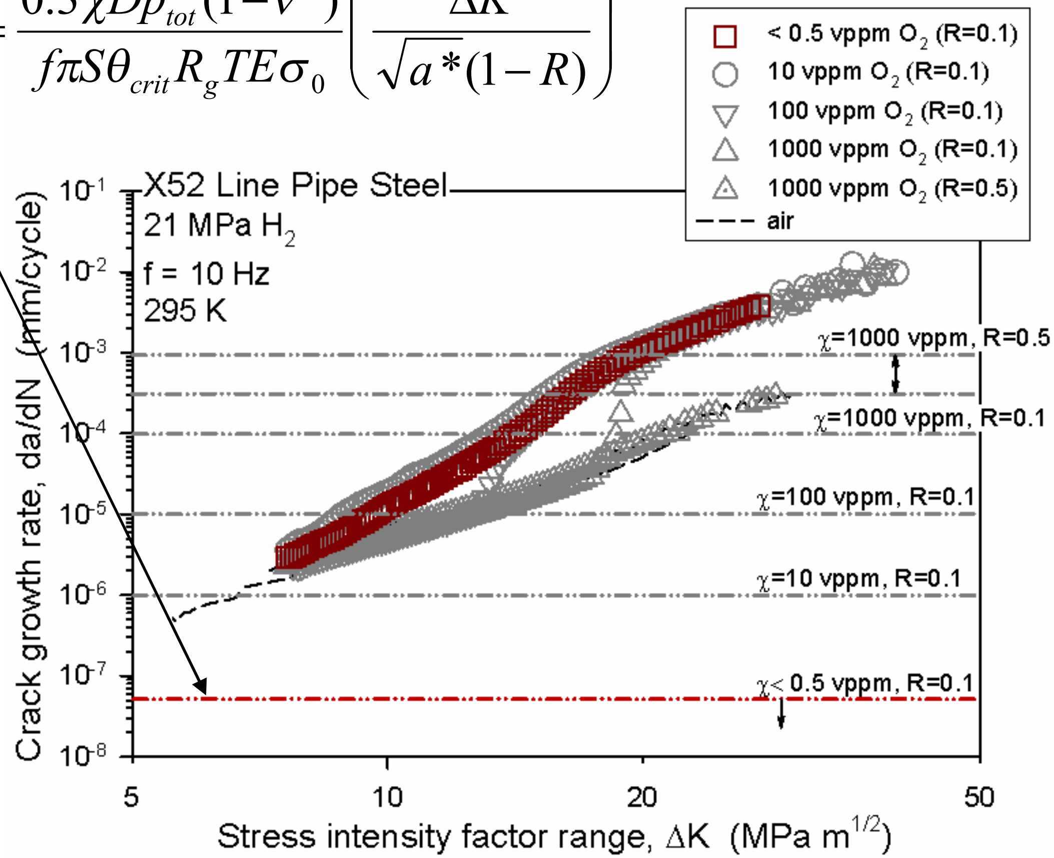
# Model does not predict onset of accelerated crack growth for high-purity $H_2$ case

$S\theta_{crit}$ from measured $da/dN$ vs. $\Delta K$ data in $H_2$+100 vppm $O_2$

$$\left.\frac{da}{dN}\right|_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{f\pi S\theta_{crit} R_g T E \sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$




Suresh and Ritchie, *Metal Science*, 1982




***Rationale: onset of accelerated crack growth dictated by threshold values of both da/dN and $K_{max}$***

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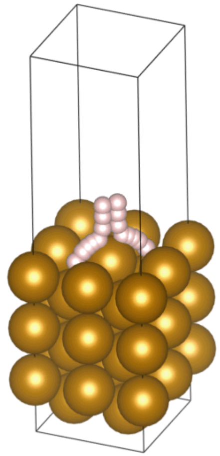
Somerday et al., *Acta Mater*, 2013

# Density functional theory (DFT) simulations reveal effect of $O_2$ on $H_2$ dissociation

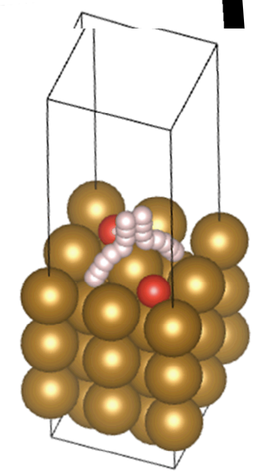
Potential energy surface scan for $H_2$ approaching Fe(100) surface

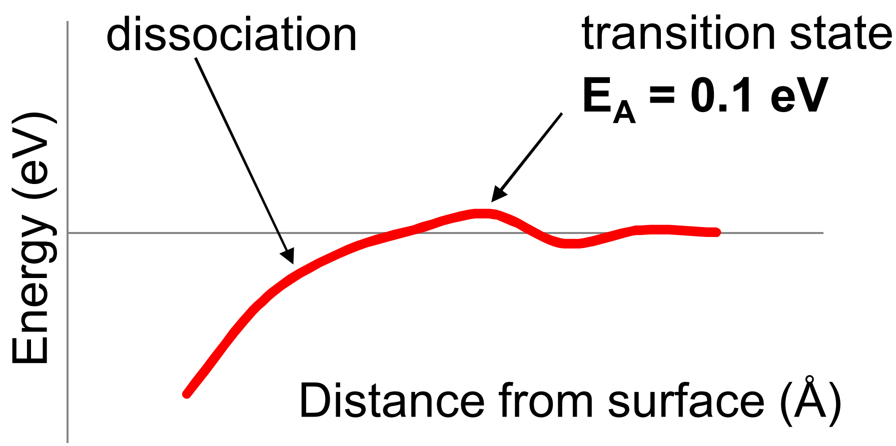
$H_2$ molecule approaches directly on top Fe atom

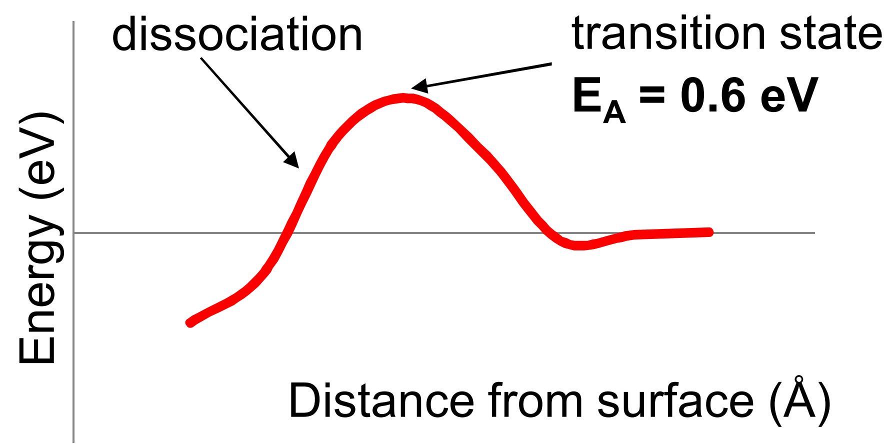


Potential energy surface scan for $H_2$ approaching Fe(100) surface with preadsorbed O atoms

$H_2$ molecule approaches directly on top Fe atom






Staykov et al., *Int J Quantum Chemistry*, 2014

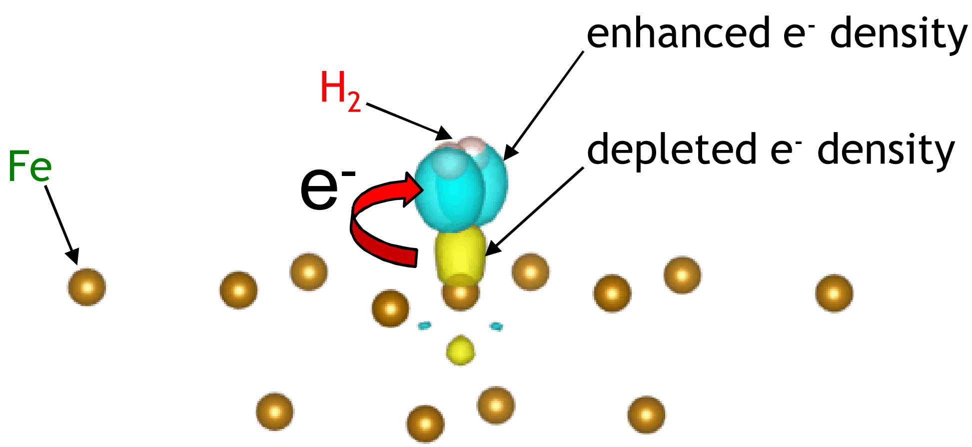
***Preadsorbed oxygen on iron surface raises activation energy for $H_2$ dissociation***

Sandia National Laboratories

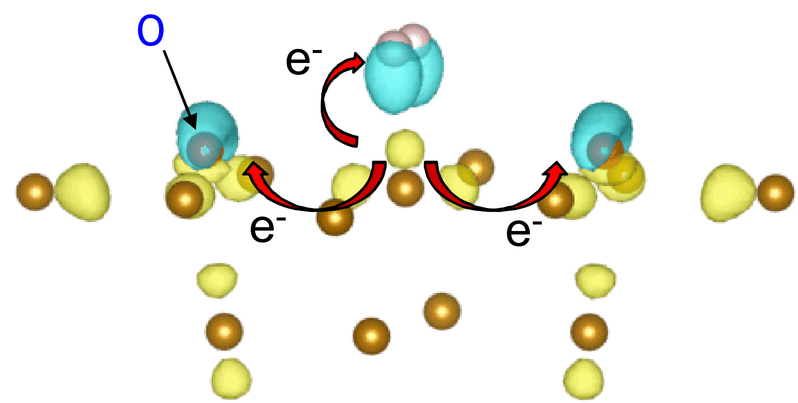
# Electron density difference method provides insight into dissociation inhibition mechanism

Staykov et al., *Int J Quantum Chemistry*, 2014

$H_2$ approaching Fe(100) surface




$H_2$ approaching Fe(100) surface with preadsorbed O atoms

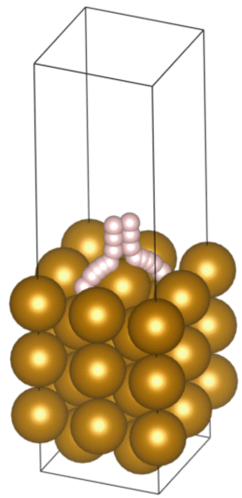



- *Oxygen atoms on surface localize $e^-$ density, reducing ability of neighboring Fe atoms to transfer $e^-$ to $H_2$*
- ***Less $e^-$ density available for $H_2$ activation: dissociation hindered***

# DFT simulations provide rationale for preferential adsorption of $O_2$ in mixed $H_2$+$O_2$ gas

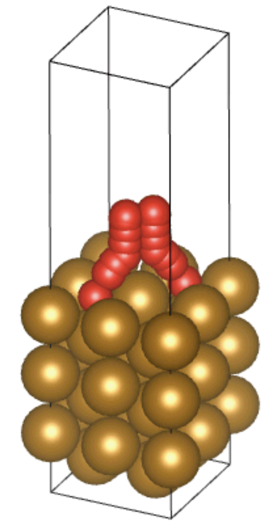
Potential energy surface scan for $H_2$ approaching Fe(100) surface

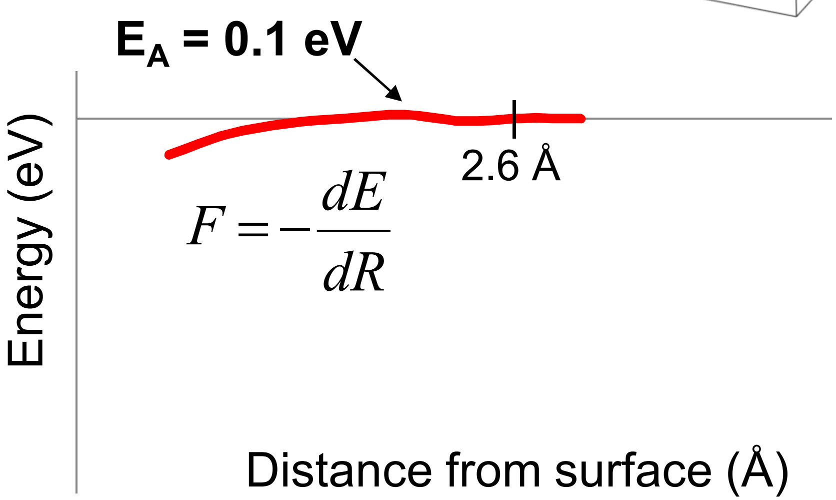
- $H_2$ is detected at 2.6 Å
- Weak attractive force
- Activation barrier: not all $H_2$ molecules dissociate

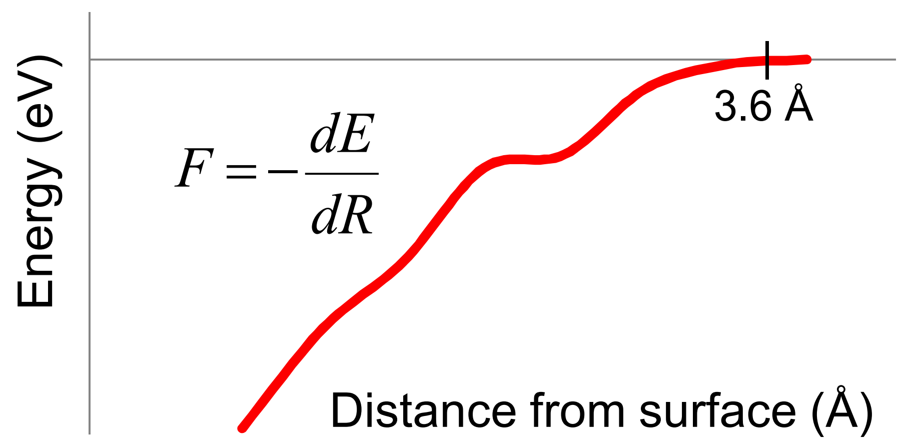


Potential energy surface scan for $O_2$ approaching Fe(100) surface

- $O_2$ is detected at 3.6 Å
- Strong attractive force
- No activation barrier: all $O_2$ molecules dissociate






Staykov et al., *Int J Quantum Chemistry*, 2014

***Strong attractive force and absence of activation barrier allow $O_2$ to out-compete $H_2$***

# DFT simulations define potential scenario for $O_2$ inhibition of H uptake

Staykov et al., *Int J Quantum Chemistry*, 2014

**Key elements in $O_2$ inhibition scenario:**

- *$O_2$ detected deeper in gas volume compared to $H_2$*
- *Force on $O_2$ >> force on $H_2$*
  - *$O_2$ can out-compete $H_2$ for adsorption sites*
- *Adsorbed O leads to repulsive force on $H_2$*