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Understanding Hydrogen Enhanced DEcohesion through micromechanical fracture tests

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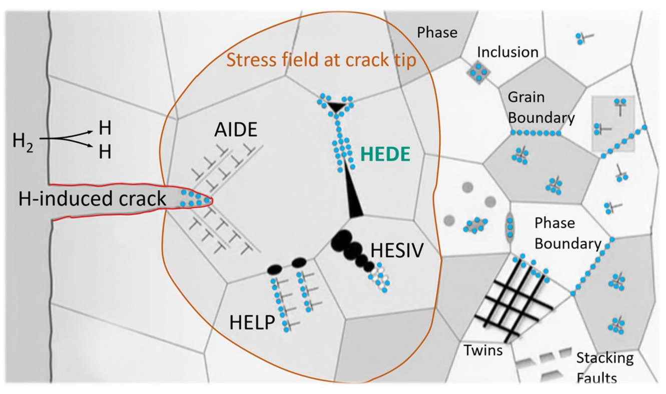
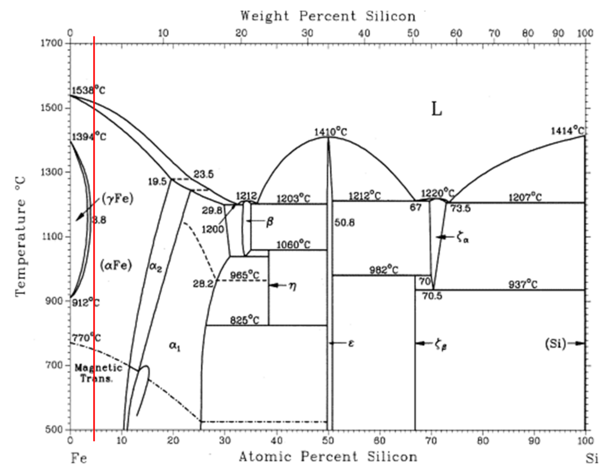
Motivation

- **H**ydrogen **E**nhanced **DE**cohesion (HEDE) suspected to be one of the fundamental mechanisms in hydrogen embrittlement
- Quantifying fracture toughness with embrittlement mechanisms requires in-situ micromechanical testing and local resolution of hydrogen
- Why use Fe3wt%Si?
  - Ferritic steels common candidates for hydrogen storage and transportation
  - No phase transformations in wide temperature-range → single and bi-crystals can be grown
  - TRITIME-Approach applicable



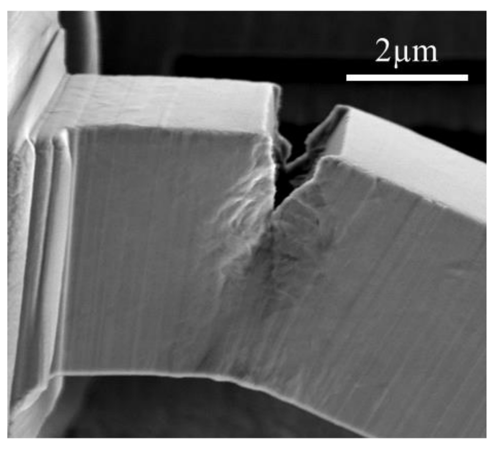
Modified from: https://www.mpie.de/4604692/hydrogen_embrittlement



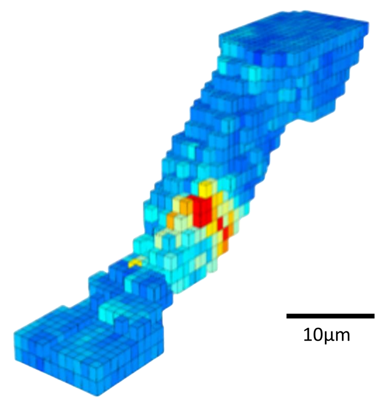
Yuan et al. Materials Characterization, 58 (4), 2007, 376-379

TRITIME**- Approach **(TRITIum based microMEchanics)

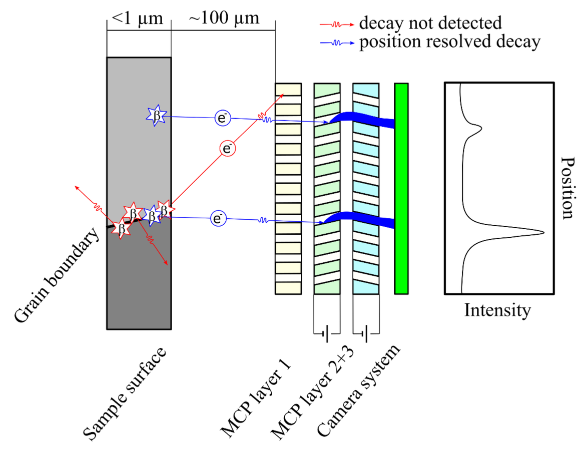
- In-situ micromechanics
- Focused Ion Beam (FIB)
- Fs-laser
- SEM
- µLaue
- 3D-DAXM (differential-aperture X-ray microscopy)
- Micro channel plate (MCP)
- tof-SIMS (time-of-flight secondary ion mass spectroscopy)
- APT (atom probe tomography)



ISOLATE



OBSERVE



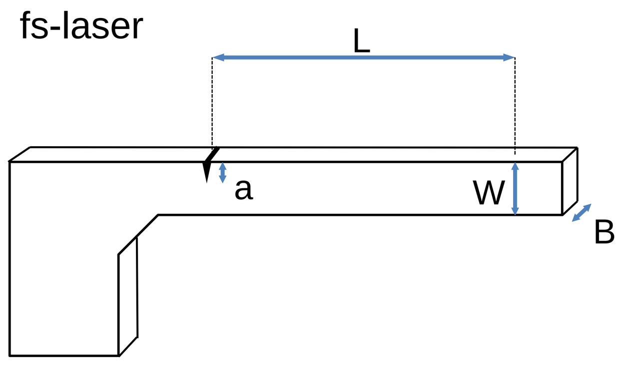
QUANTIFY

C. Kirchlechner, ERG CoG TRITIME

Can we comprehend the HEDE mechanism by evaluating the fracture-mechanical behavior?

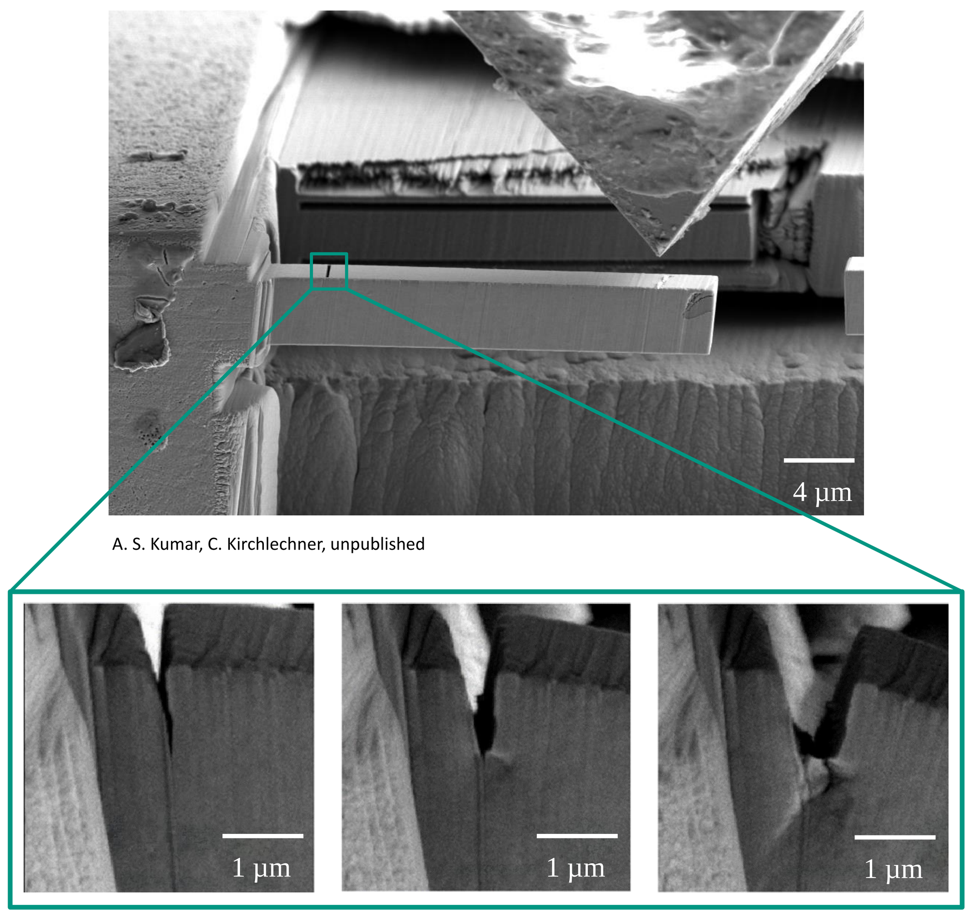
Procedure

1. **Estimation** of required sample size for reliable fracture mechanical results
2. **Fabrication** of notched micro cantilevers in single crystals:
   - FIB
   - fs-laser



3. **Charging** samples with tritium (^3H) and probing content – Why tritium?
   → See Poster „Probing hydrogen with high spatial resolution" by Dr. M. Vrellou and "Measurement of tritium content with a novel detector system" by J. Müller

4. **Fracture-mechanical analysis** of uncharged/charged samples in SEM via cantilever bending (same indenter system in/outside controlled area)



A. S. Kumar, C. Kirchlechner, unpublished

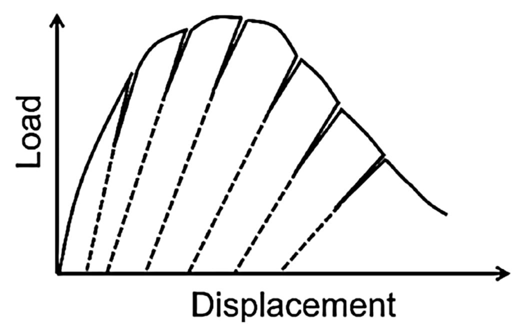
Modified from: „Micro fracture investigations of white etching layers", Saxena et al. Materials & Design, 180, 2019

5. **Calculating** the fracture toughness of uncharged and charged samples:
   - Fe3wt%Si too ductile for linear elastic approach
   - → Estimation of fracture toughness via J-Integral:

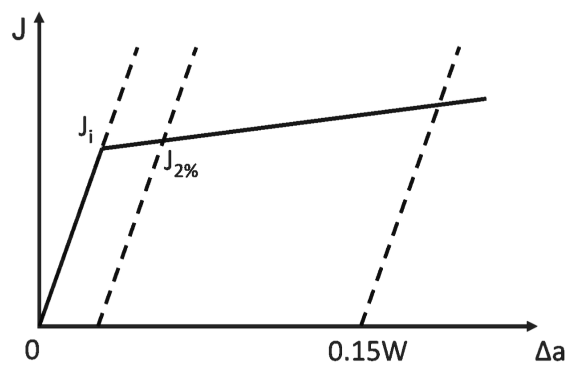
$$J^{(i)} = J^{(i)}_{elastic} + J^{(i)}_{plastic}$$

$$J^{(i)}_{elastic} = \left(K^{(i)}_{IQ}\right)^2 \frac{(1-\nu^2)}{E} \qquad J^{(i)}_{plastic} = \int \frac{\eta A_{Work}}{B(W-a_i)}$$

$$K^{(i)}_{IQ} = \frac{F^{(i)}_Q L}{B W^{\frac{3}{2}}} f\left(\frac{a^{(i)}}{W}\right) \text{(Matoy)} \qquad W - a_i = \sqrt[3]{\frac{4kL^3}{BE}}$$



Modified from: „Experimental conditions affecting the measured fracture toughness at the microscale: Notch geometry and crack extension measurement", Saxena et al. Materials & Design, 191, 2020



Modified from: „Fracture mechanics of micro samples: Fundamental considerations", Pippan et al. Materials & Design, 159, 2018

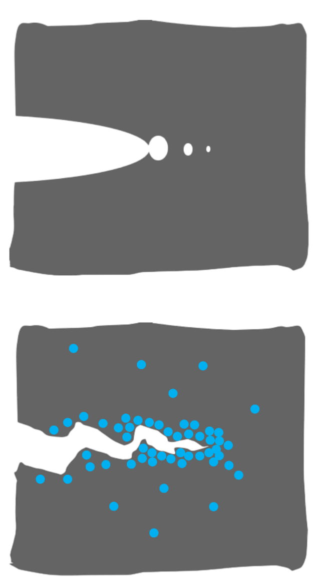
- For macroscopic samples: $J_{0,2}$
- For microscopic samples:
  - → shift blunting line to Δa = 0,02 · W
  - → intersection with tearing regime: $J_{2\%}$

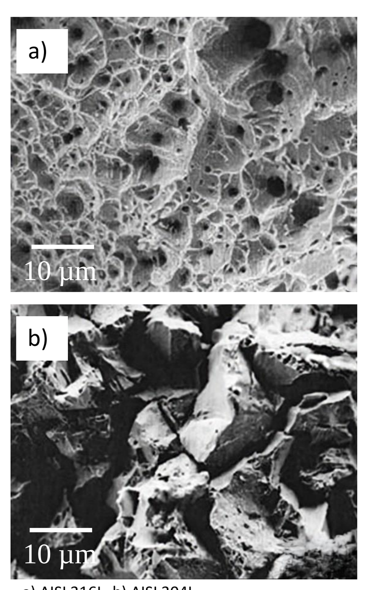
$$\rightarrow K^{(i)}_{IQ} = \sqrt{J_{IQ} \frac{E}{(1-\nu^2)}}$$

Challenges

- **Reliable** fracture mechanical results with "ductile steel"
- ^1H / ^3H concentration **sufficient enough** for embrittlement?
- **Correlation** of hydrogen/tritium content and **actual** embrittlement effects
- **Separation** of **HEDE** and **HELP** in elastoplastic fracture mechanics (EPFM)

Outlook

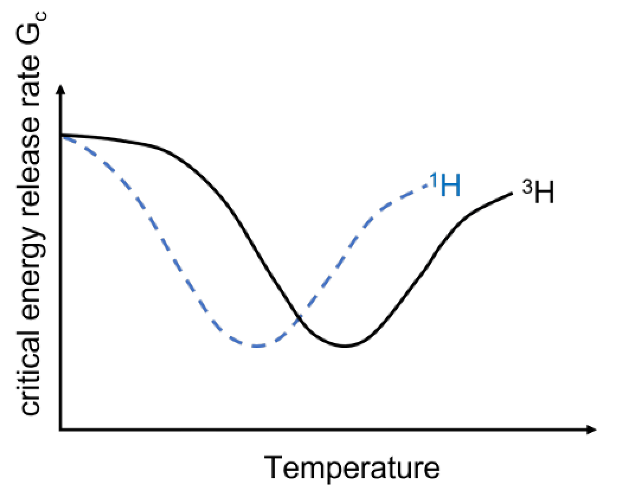




a) AISI 316L, b) AISI 304L

Modified from: Lecture „Hydrogen in Materials", A. Pundt

- Micro-fracture at grain boundaries with ^1H / ^3H in the vicinity
- Influence of temperature on (grain boundary) toughness
- Influence of different ^1H / ^3H concentrations



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