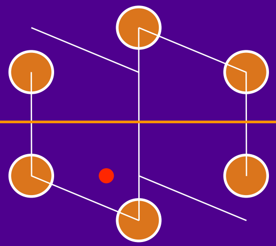
# Towards Safe Hydrogen Transport

*A Multiscale Assessment of Hydrogen Embrittlement in Pipeline Steels*



**Mahdieh Safyari** | Assistant Professor / Academy Research Fellow

Metals Technology Group
Tampere University

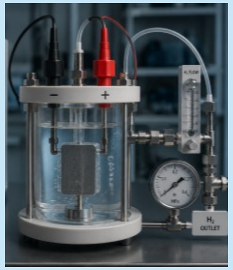
# When does hydrogen embrittlement happen in pipelines?

**HYDROGEN**

Gaseous

Pressure
Temperature
Composition

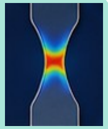
Electrochemical charging



**LOADING**

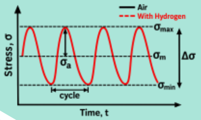
Tensile loading

- Stress level
- Starin rate
- Internal/external $H_2$



Cyclic loading (Fatigue)

- Strain range
- Stress ratio (R)
- Frequency
- Internal/External $H_2$
- Mean stress

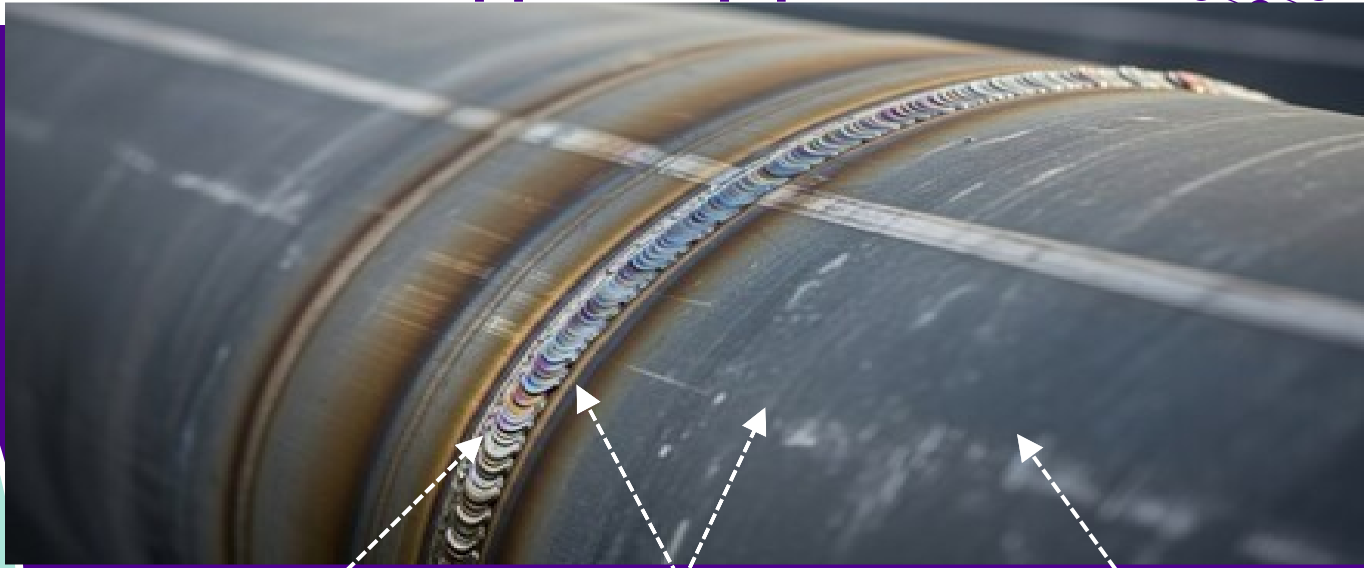


Env.
HE
Load
Material

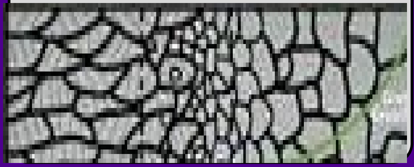
**MATERIAL**

Base Metal
Hydrogen trapping
Hydrogen diffusion
Residual Stress

Weld Metal
Hydrogen trapping
Hydrogen diffusion
Microstructural gradient
Weld/CG-HAZ/ FG-HAZ
Residual Stress
Welding type



**-Welding type**
**-Welding parameters**
**-Defects**
**-microstructural gradient**
**-FCGR**

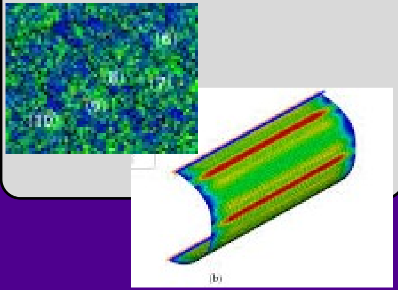


**-Strain localization**

-Thermomechanical processing

-Welding

-H induced stress localization

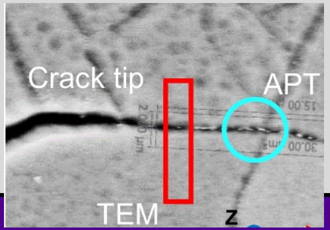


**-Microstructural Trapping**

Mitigate or Exacerbate?

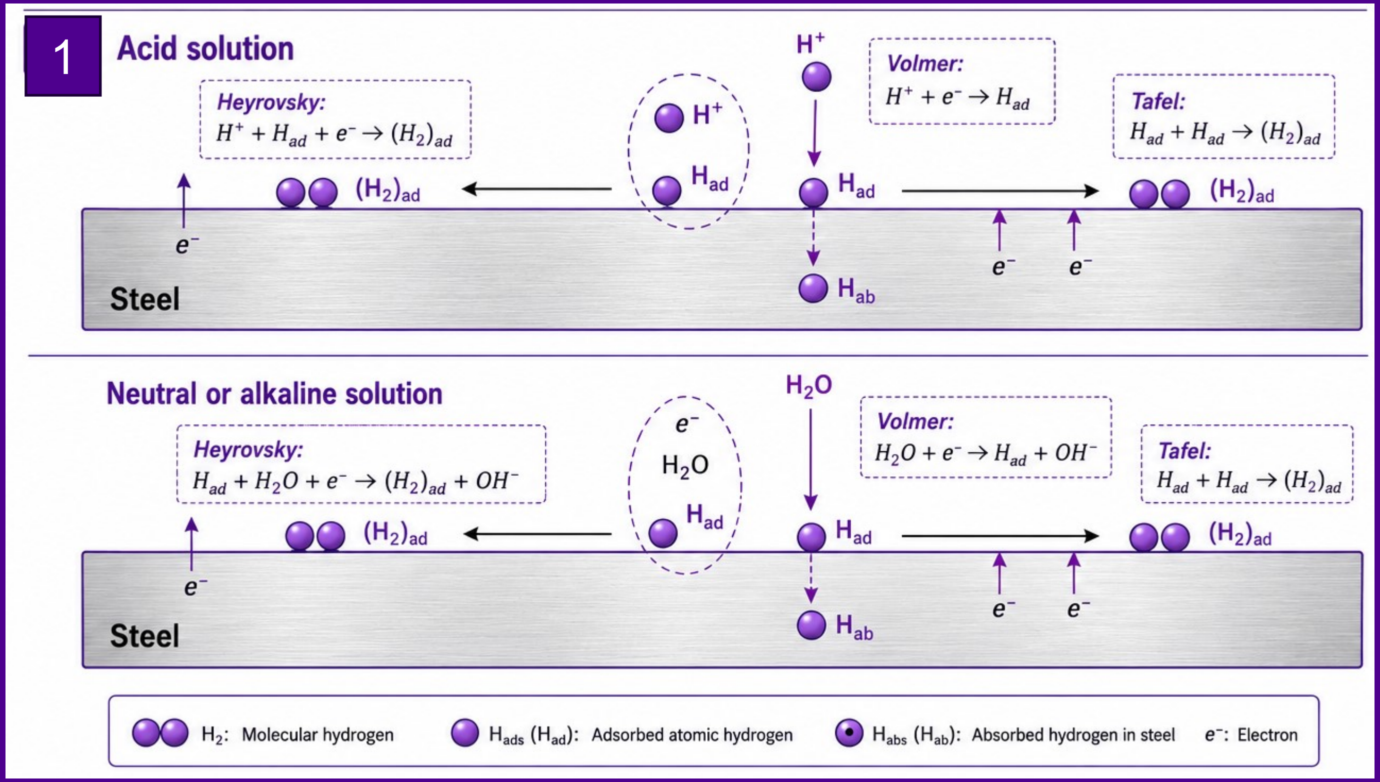
**-HE Mechanism**

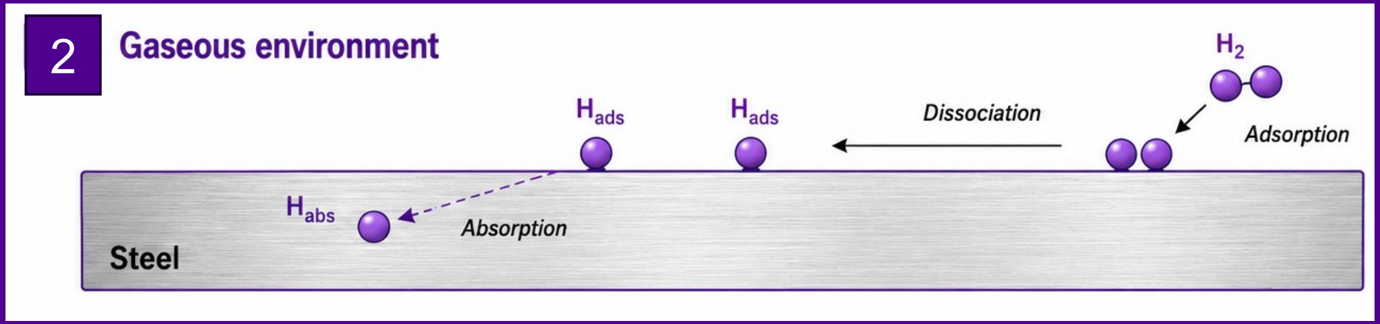
Different metal structures react uniquely to H.



# Environment: Comparing laboratory charging methods for pipeline steel research

## Two Approaches to Hydrogen Charging





Unrealistic Concentration

**ELECTROCHEMICAL CHARGING**

- Atomic H generated at surface:
  $2H^+ + 2e^- \rightarrow 2H$
- Higher surface H concentration
- May cause corrosion, pitting &surface damage

VS

Realistic Environment

**GASEOUS CHARGING**

- Pressurized $H_2$ gas environment
- $H_2 \rightarrow 2H$ surface dissociation before diffusion into steel
- Follows Sieverts' law: $C_H \propto \sqrt{PH_2}$
- Reproduces real pipeline conditions
  Diffusion, trapping
- hydrogen-assisted cracking

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# Materials Contribution to Hydrogen Embrittlement of Pipeline Steels

**Among all factors, materials can be engineered & optimized → Most powerful lever against HE**

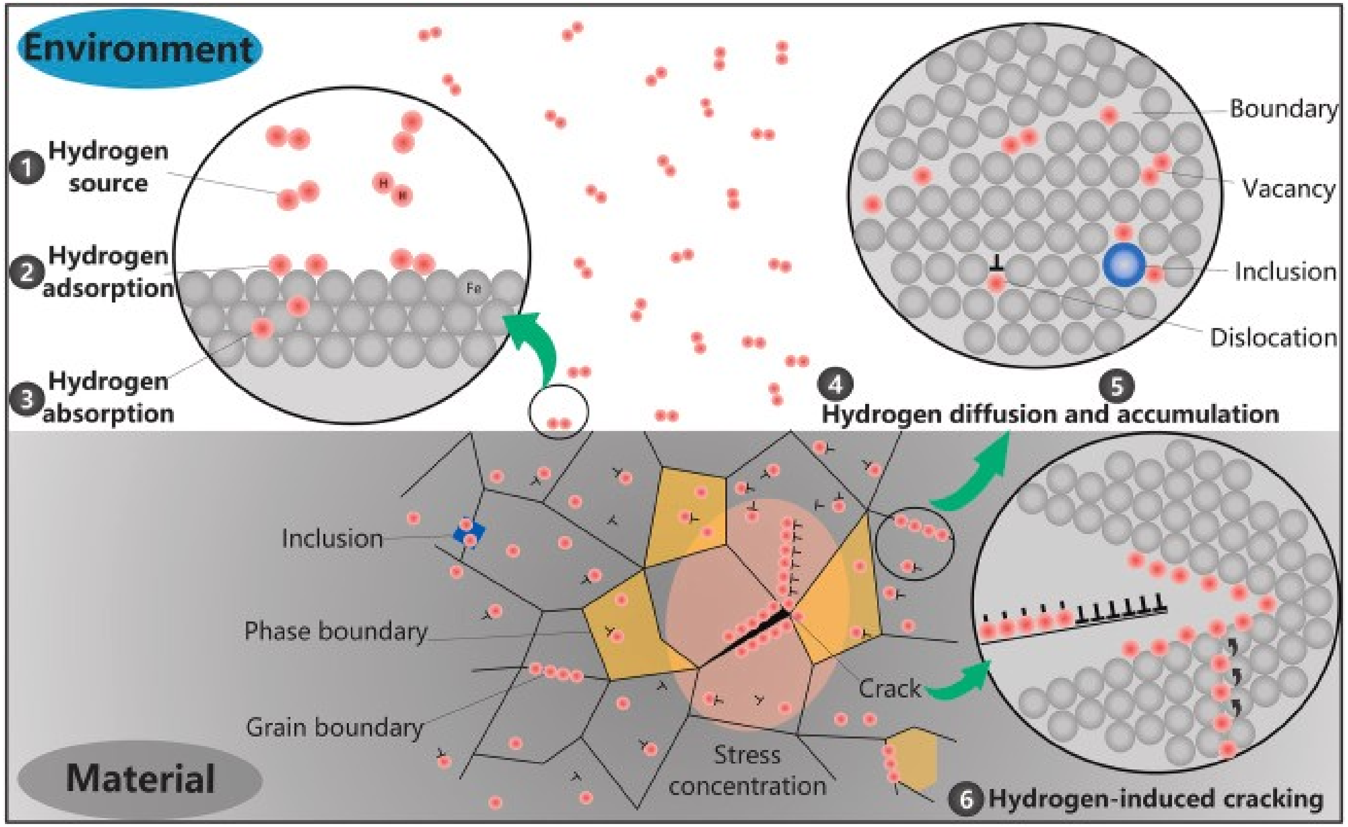
## Role of materials in HE:

1 **Absorption & Diffusion**
Controls H uptake rate and transport through the microstructure

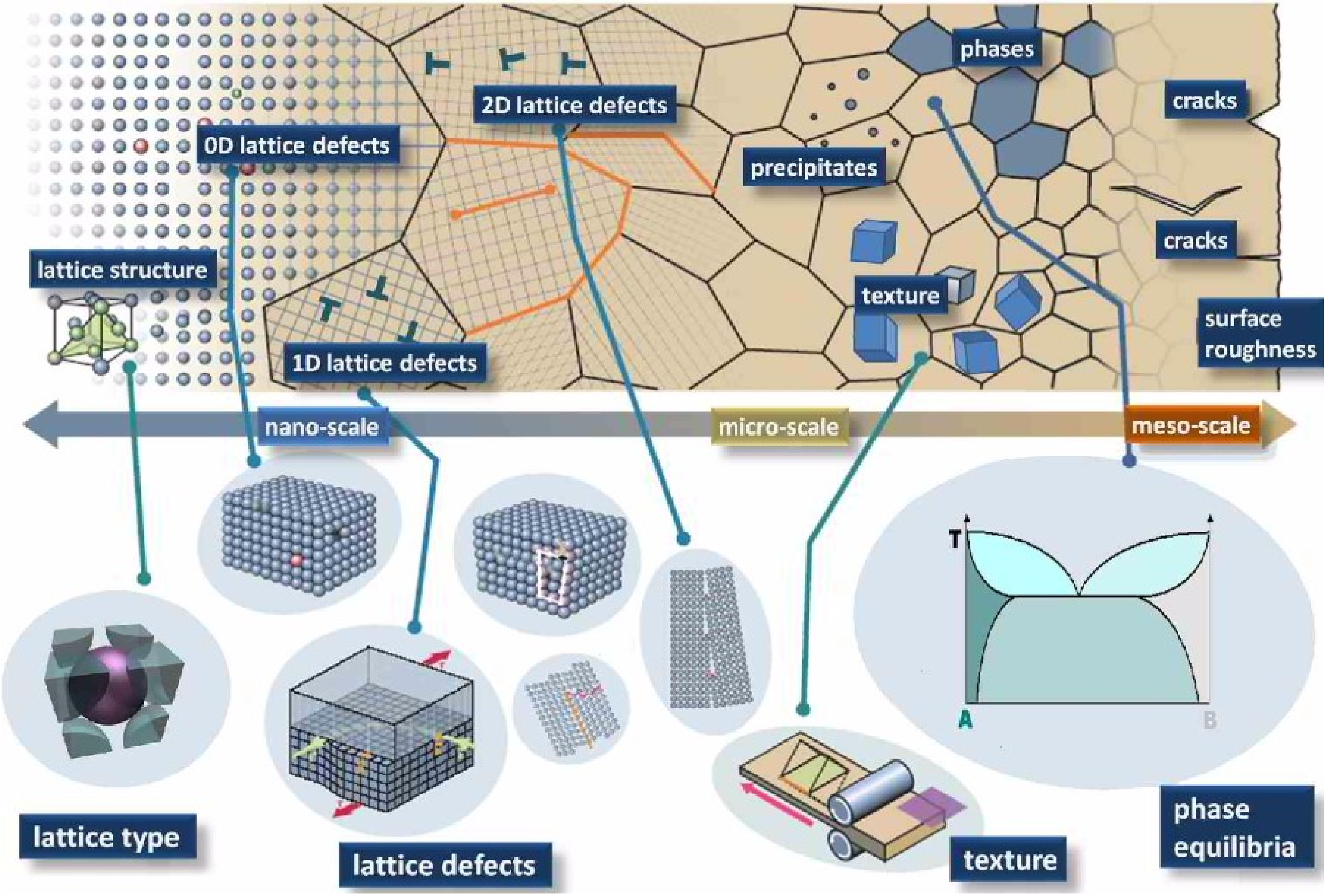
2 **Trapping Behavior**
Reversible & irreversible trap sites govern local H concentration

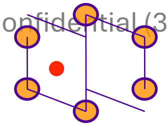
3 **Crack Initiation & Growth**
Crack nucleation sites and crack-growth resistance

4 **HE-Toughness Balance**
Optimal alloy design balances HE and fracture toughness



# Microstructure /Hydrogen/ Performance Relationship





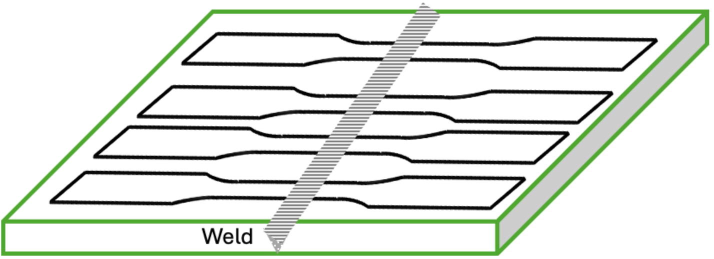
# Mechanical Tests for investigation of HE Susceptibility

- Slow Strain Rate Testing (SSRT)
- Fatigue Fracture Toughness Testing
- Step-Loading or Constant Load Tests
- Fatigue Crack Growth Rate Testing

(3Y)

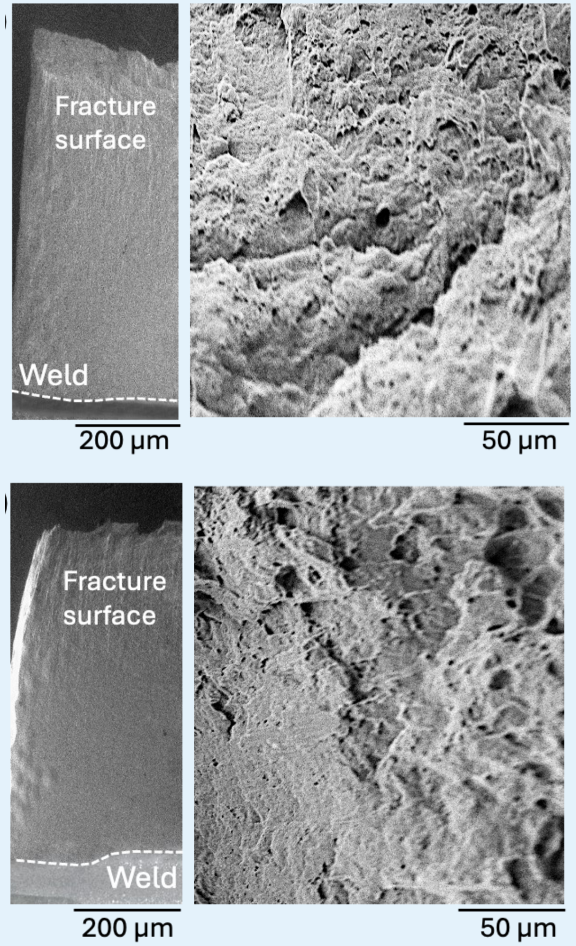
# Is SSRT a suitable method for evaluating the hydrogen behavior of welded pipelines?

Cut layout of welded specimen for tensile test

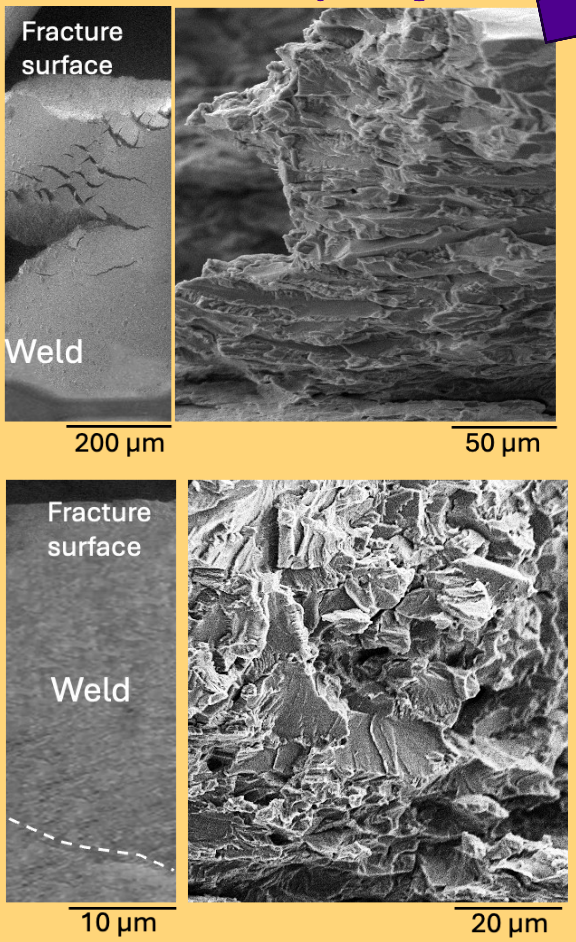


Both repeated tests, fracture occurs in the base metal.

Tested in Air



Tested in Hydrogen



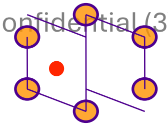
In one specimen, the fracture occurs in the base metal, while in the other, it occurs in the weld

Comparison of hydrogen embrittlement in weld and base

- No **pass/fail criteria**, only comparative assessment
- Highly dependent on **strain rate and hydrogen charging conditions**.

SSRT is not standard & applicable technique for Industrial applications

Focusing on other methods in TAU



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- Slow Strain Rate Testing (SSRT)
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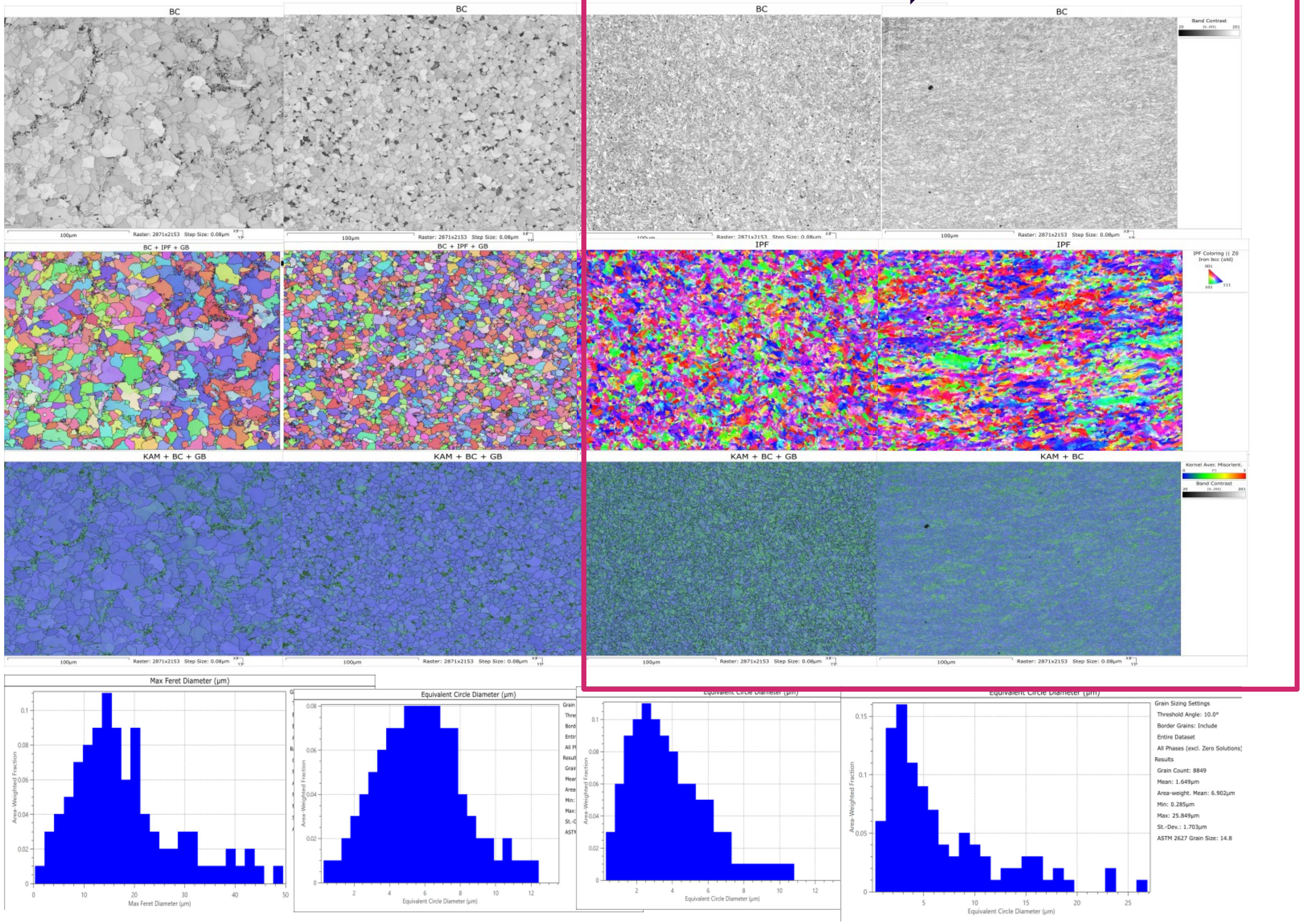
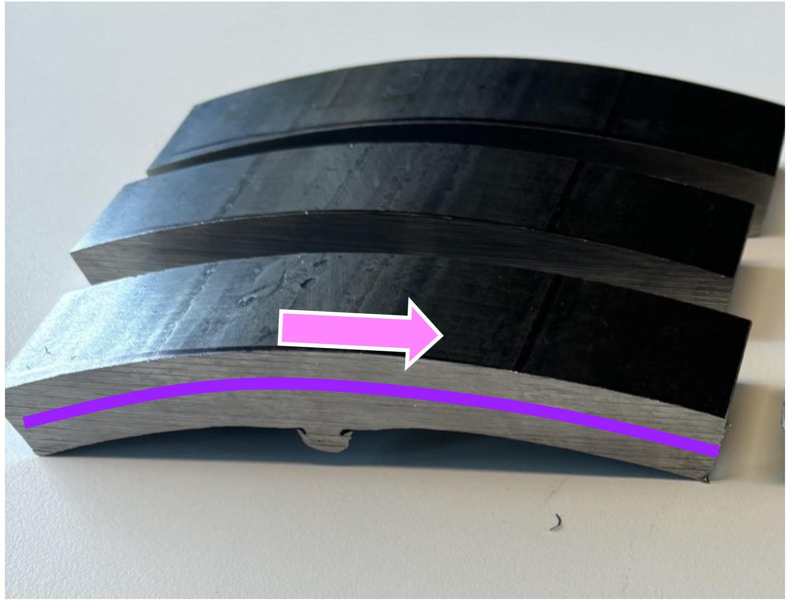
# Gaseous hydrogen embrittlement in welded X70 pipeline steels

## Micro-scale investigation:

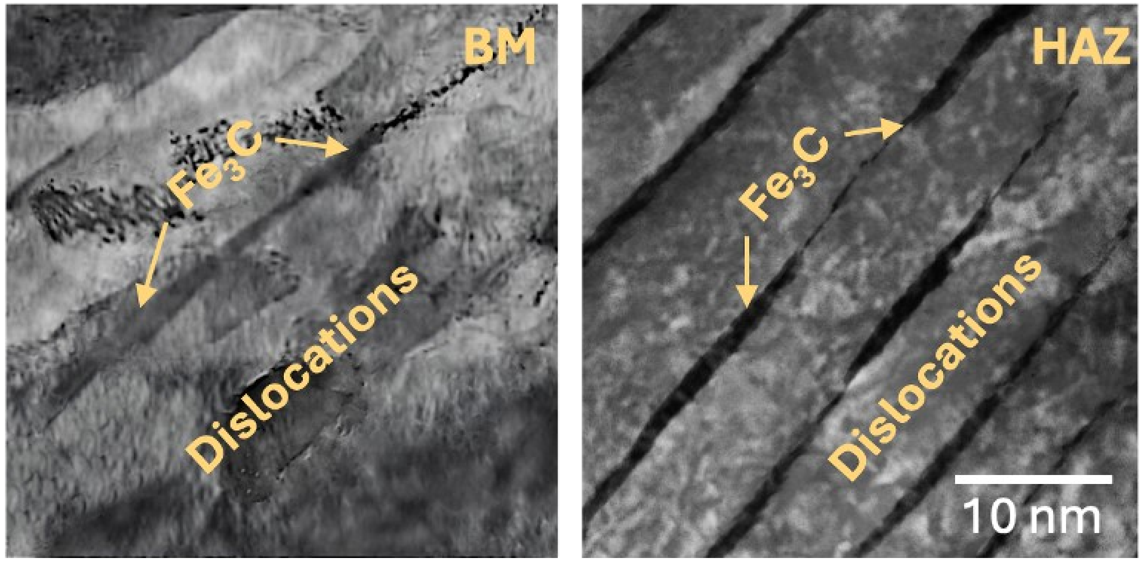
Weld → Base

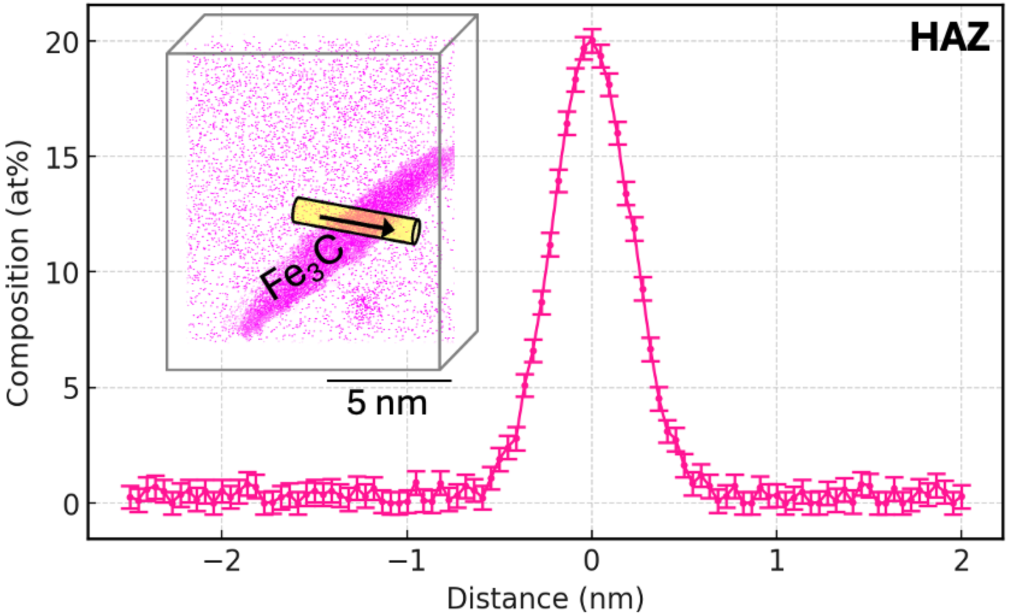


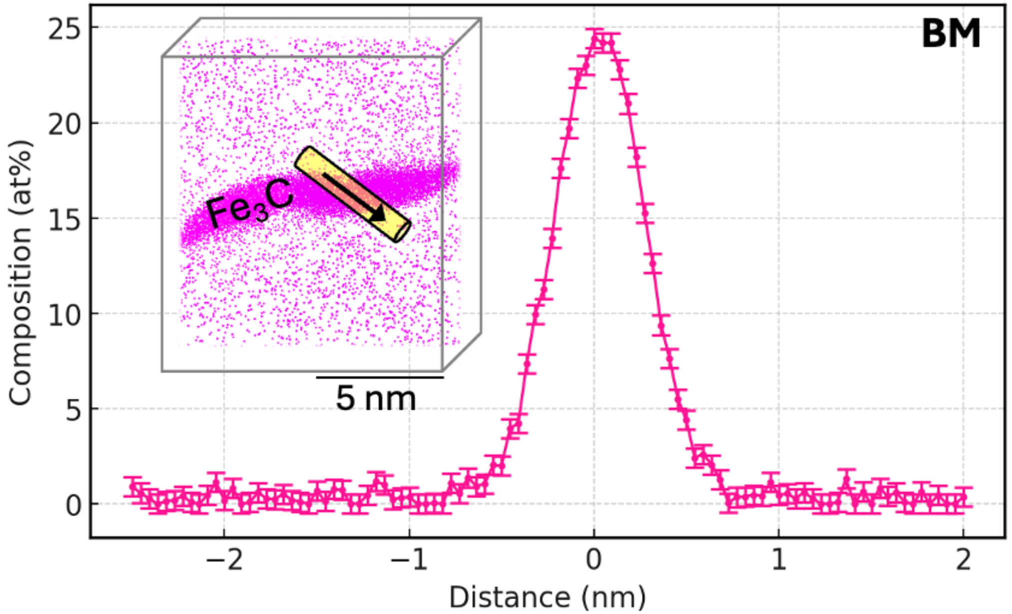
Grain Sizing Settings
Threshold Angle: 10.0°
Border Grains: Include
Entire Dataset
All Phases (excl. Zero Solutions)
Results
Grain Count: 8849
Mean: 1.649µm
Area-weight. Mean: 6.902µm
Min: 0.285µm
Max: 25.849µm
St.-Dev.: 1.703µm
ASTM 2627 Grain Size: 14.8



# Atomic-scale investigation:



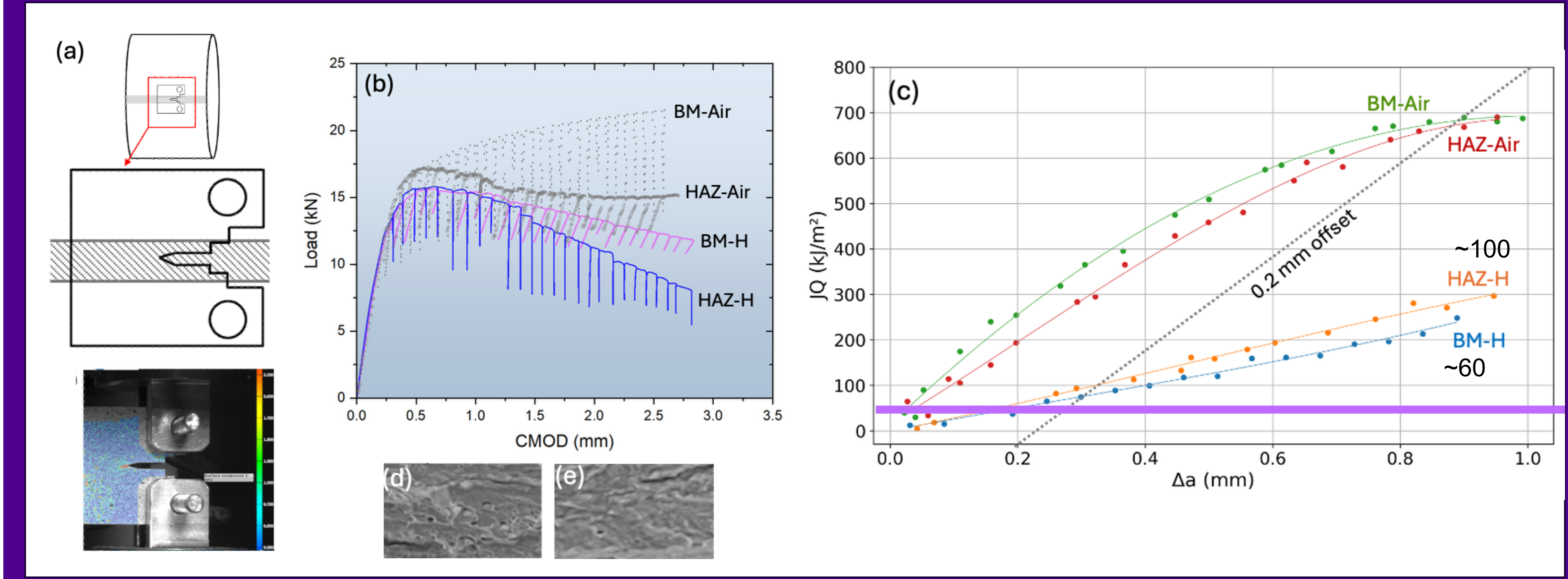




**Faster cooling in HAZ refines ferrite–bainite laths and generates off-stoichiometric cementite (carbon-deficient).**

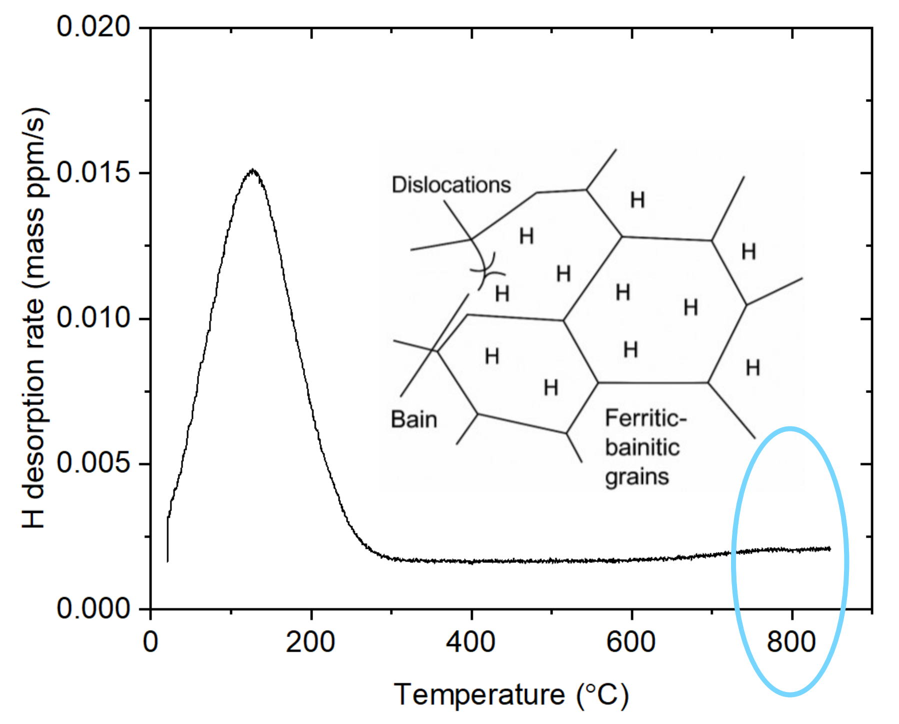
# Fracture properties of API X70 pipeline steel under the effect of an environment containing hydrogen: FGHAZ and Base metal

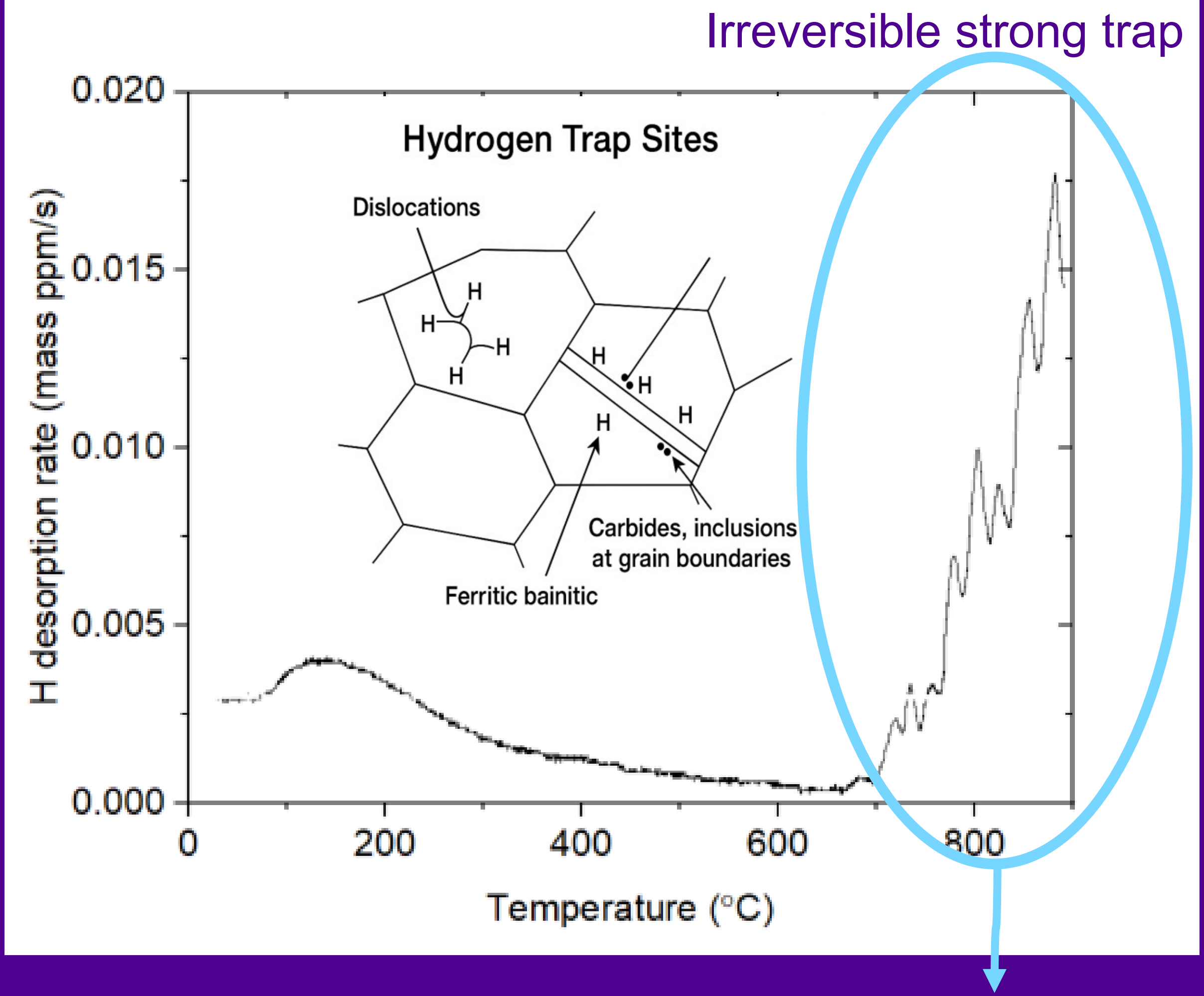
Force vs CMOD curves of base metal in air and in hydrogen and of HAZ in 100 bar hydrogen



ASME Criteria

# Investigation of Hydrogen Distribution in Materials Using Thermal Desorption Spectroscopy (TDS)





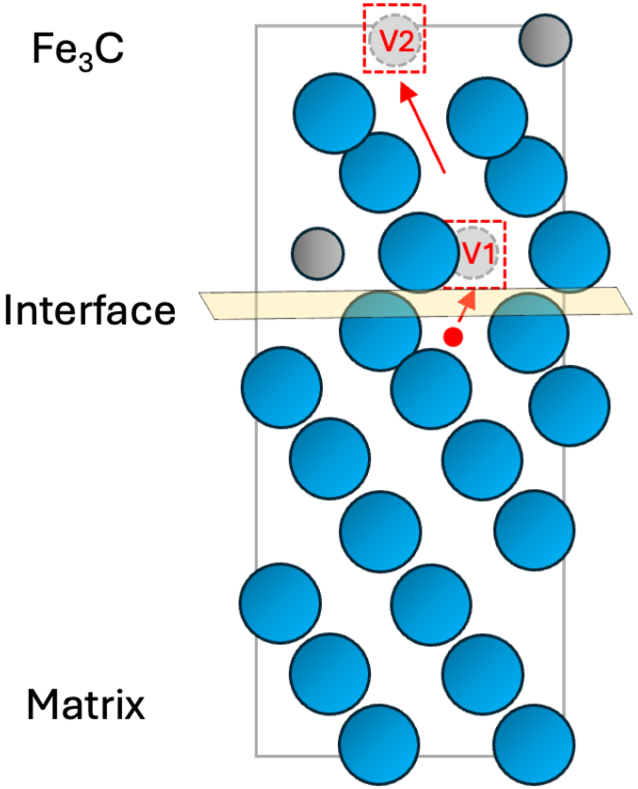
Irreversible strong trap

**lath-like ferritic bainitic packets.**

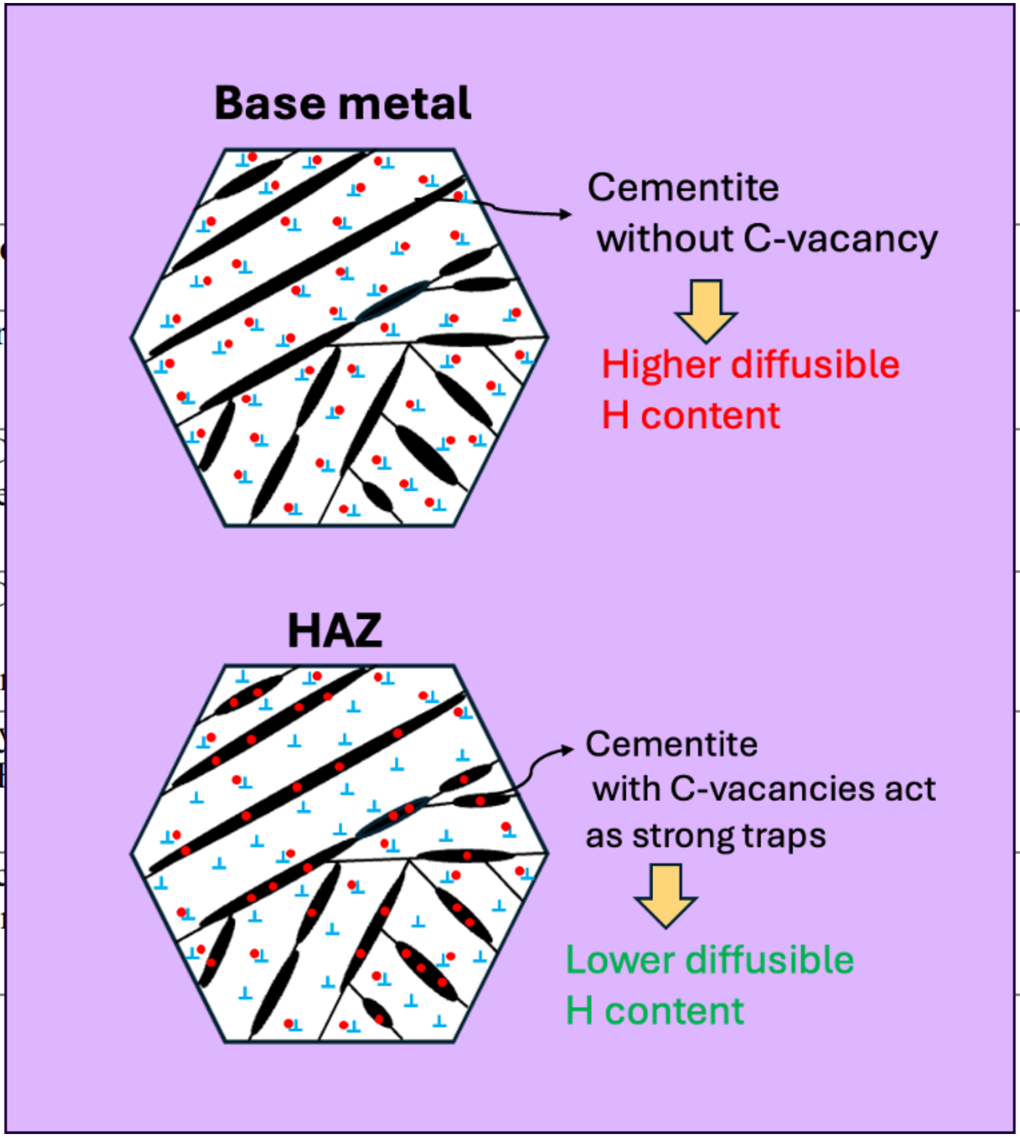
**Higher amount of diffusible H**

Excellent Hydrogen Resistance

# DFT provides atomic-scale insight into H-induced damage mechanisms



| Vacancy (Fe$_3$C) | $E_{sol}(4H)$ | $E_{sol}(5H)$ |
|---|---|---|
| Bulk inter vacancy) | - | - |
| Isolated C planes (Fe | 0.00 eV | +0.10 eV |
| Isolated C 4c planes surroundi | +0.05 eV | +0.10 eV |
| C-vacancy an $\alpha$-Fe/I interface | -0.05 eV | 0.00 eV |
| Two adjac (cluster) i | -0.10 eV | -0.05 eV |



**Base metal**

Cementite without C-vacancy

Higher diffusible H content

**HAZ**

Cementite with C-vacancies act as strong traps

Lower diffusible H content

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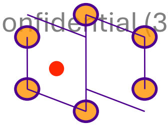

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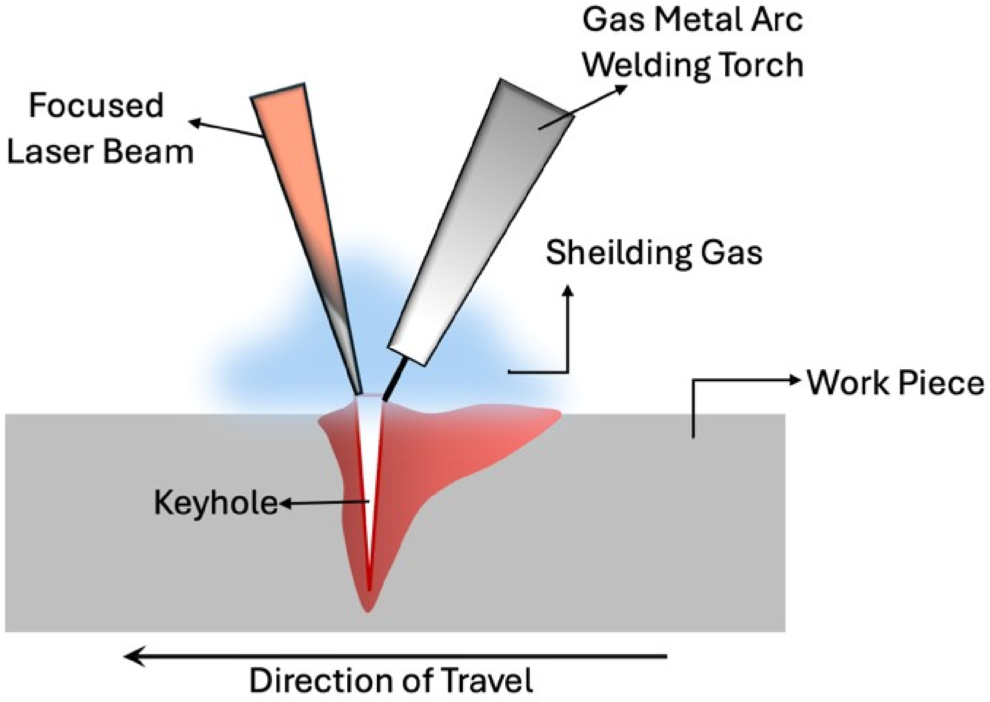
# Fracture Toughness of SENT Specimens in Air and Hydrogen:
# Welding Design & HE Assessment

## Materials used in this study

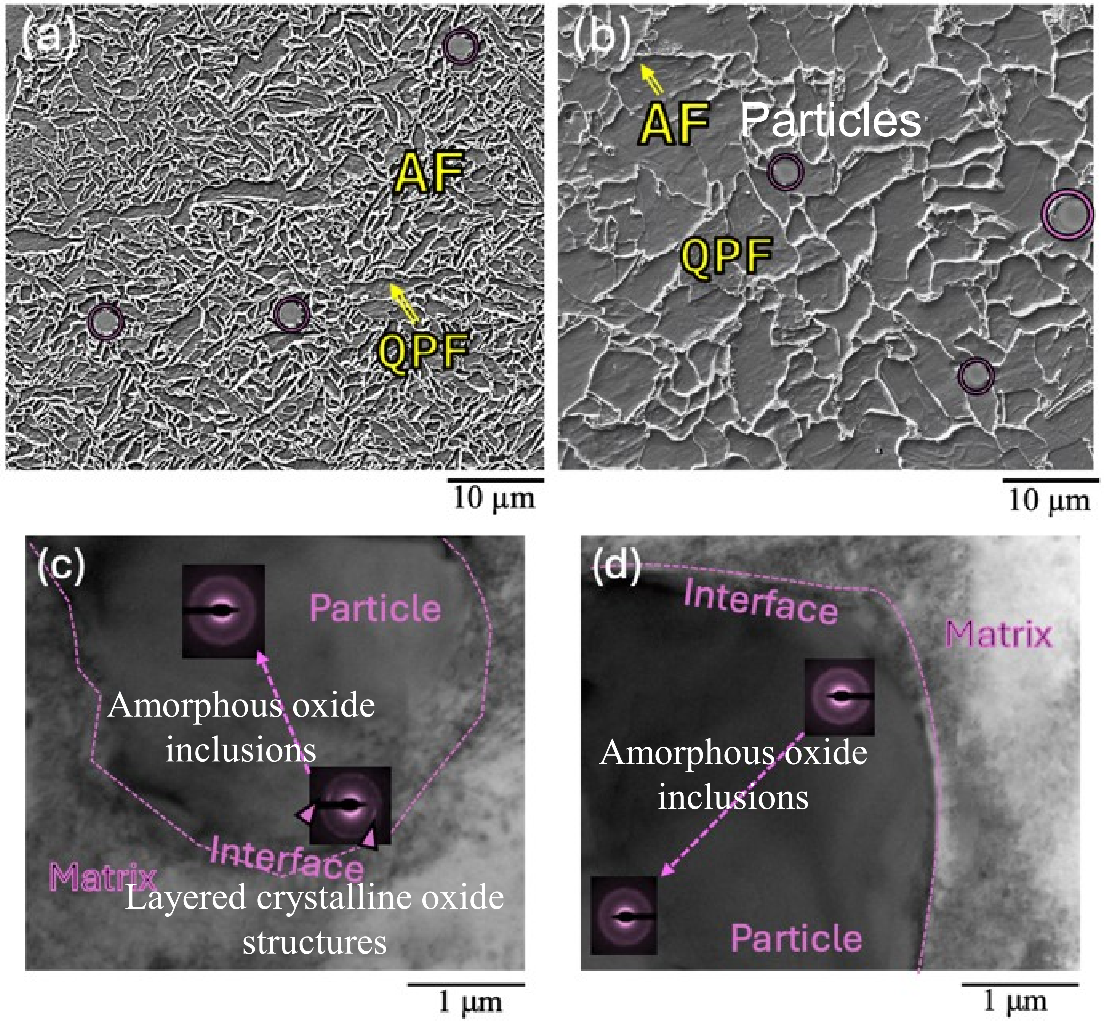
| | C | Mn | Si | P | S | Al | Nb | Cu | Cr | Ni | Ti |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **X65** | 0.08 | 1.38 | 0.24 | 0.01 | 0.002 | 0.03 | 0.03 | 0.09 | 0.02 | 0.47 | 0.001 |
| **Filler metal** | 0.07 | 1.40 | 0.51 | 0.01 | 0.003 | - | - | - | - | - | 0.2 |

## Methods used in this study

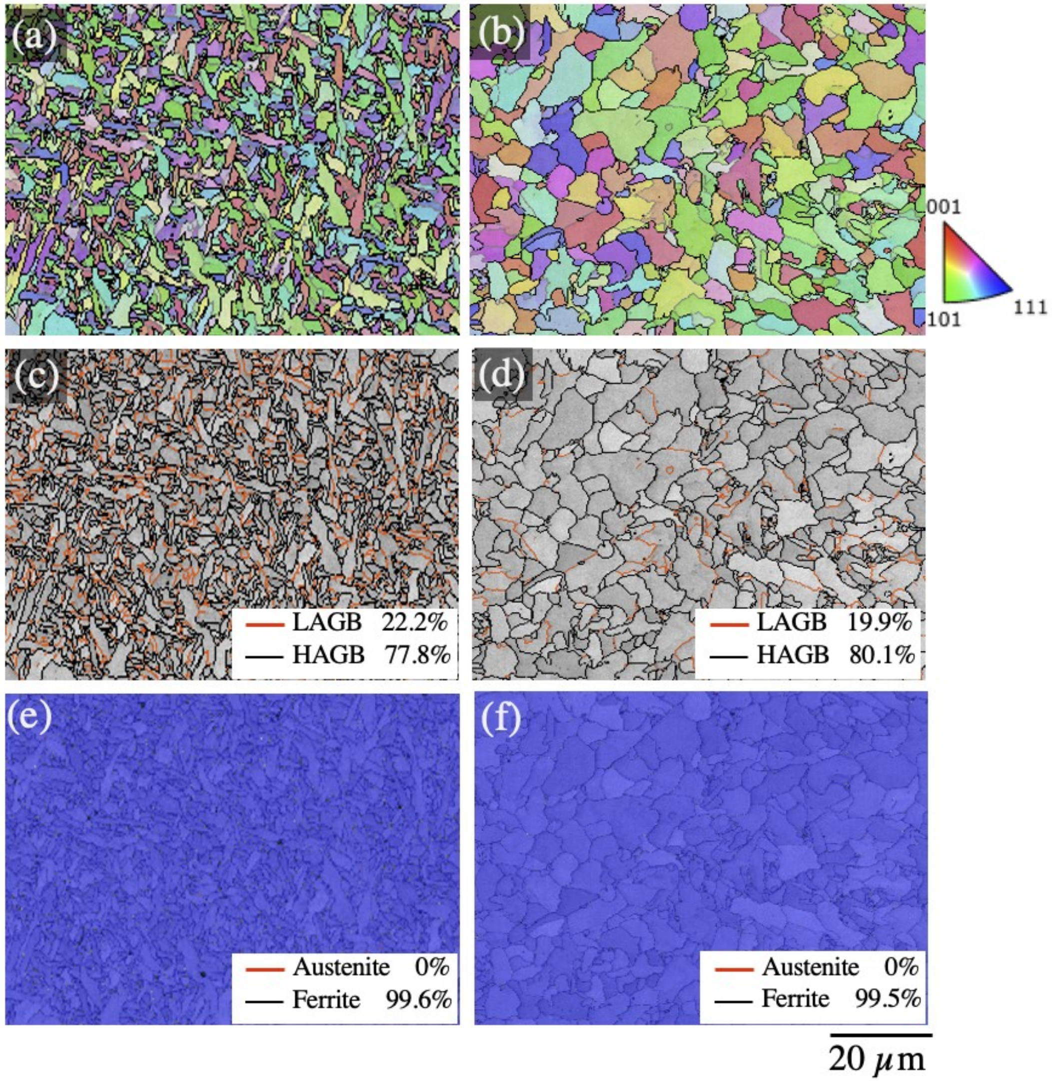


# Microstructural Evolution in Conventional and Hybrid Welding Processes:

**HLAW** **MAG**

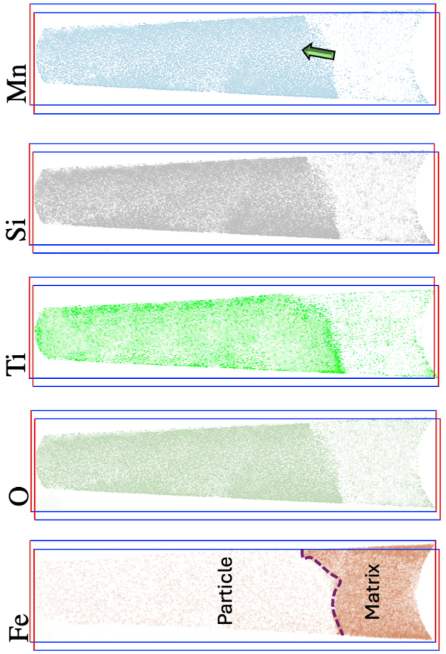


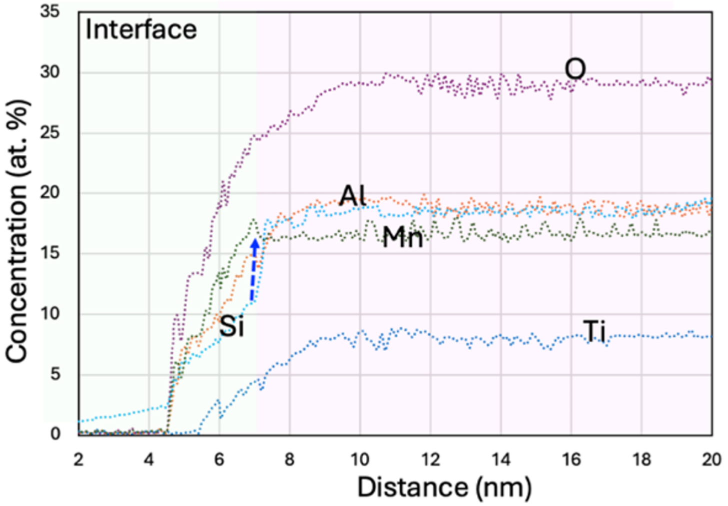
**HLAW** **MAG**



# Nano-structural Evolution in Conventional and Hybrid Welding Processes:

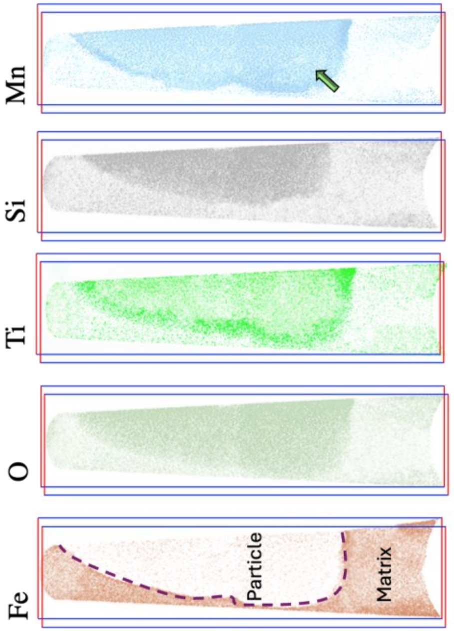
## MAG

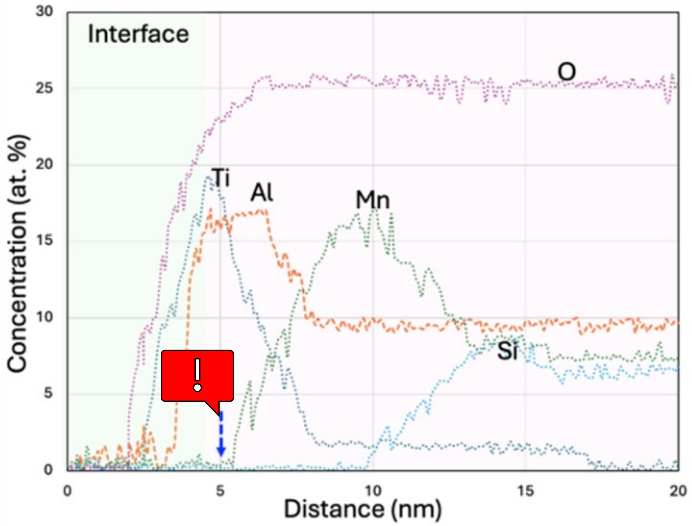




Uniform elemental distribution at the particle/Matrix interfaces

## HLAW





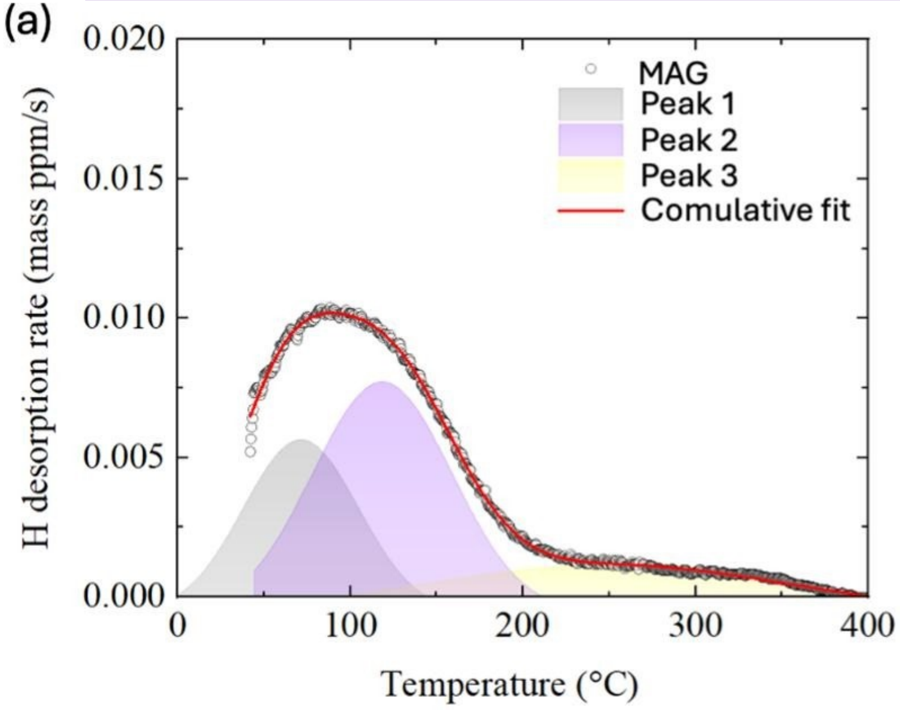
Non-uniform distribution of elements at particle interfaces

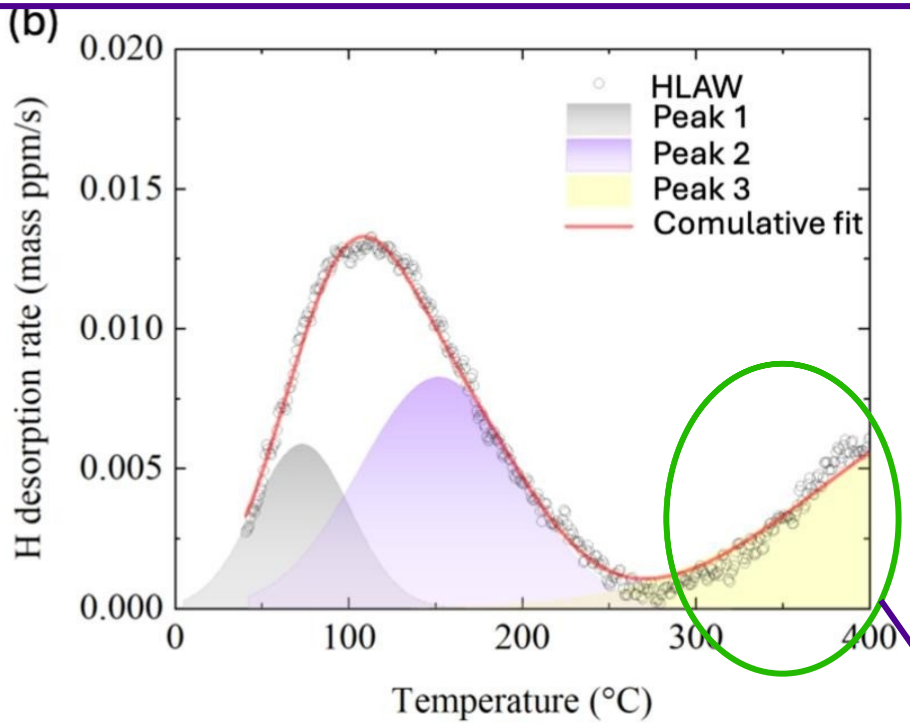
!

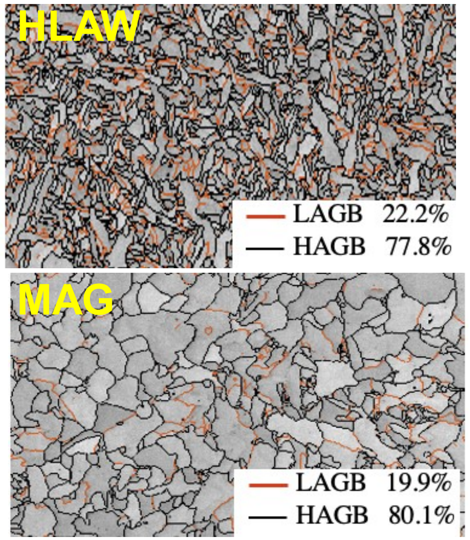
Mn → austenite stabilizer

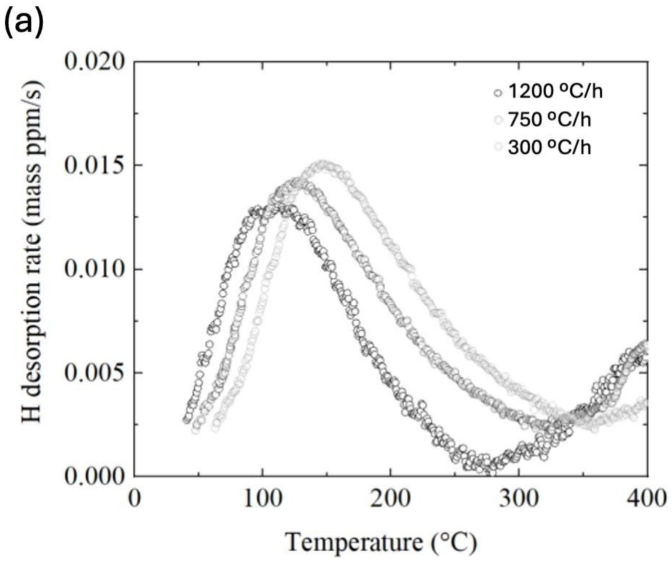
Local Mn depletion near the interfaces → reduced austenite stability → promoted ferrite formation → finer microstructure → HAGBs

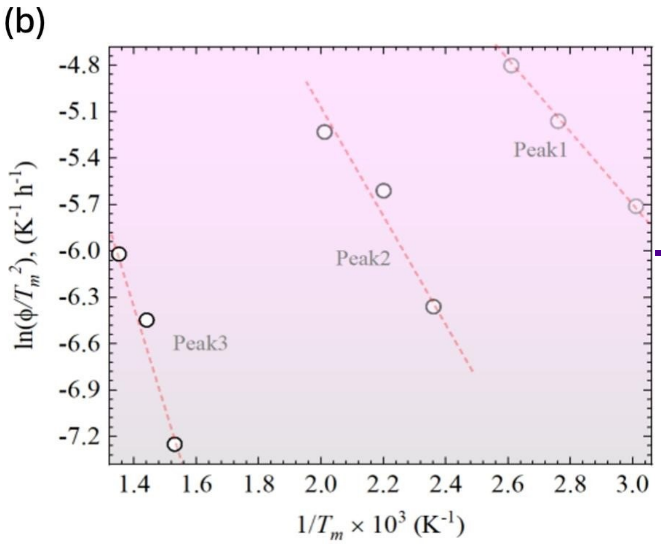
# Thermal Desorption Spectroscopy (TDS)











50 kJ/mol → Strong Trap

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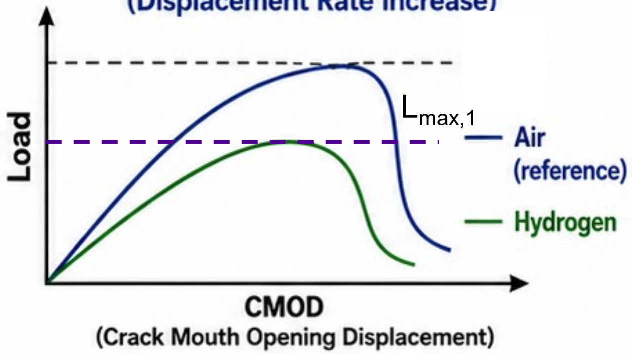
# Fracture Toughness of SENT Specimens in Air and Hydrogen

## STEP 1
## MAXIMUM LOAD MEASUREMENT ($L_{max,1}$)

- SENT specimens were tensile tested in both air and hydrogen environments under a constant displacement rate increase to determine baseline fracture performance.
- The air test provided reference data, while the hydrogen test determined the $L_{max,1}$.

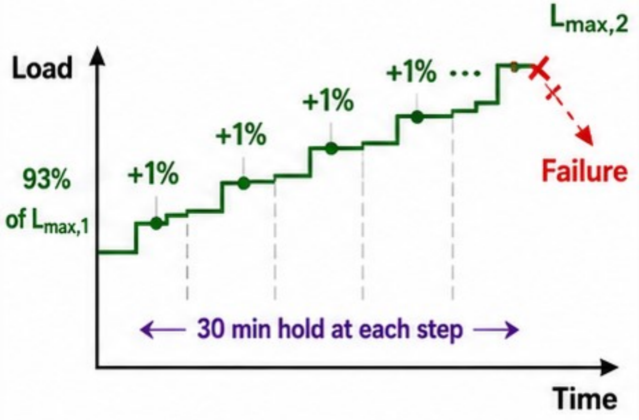
**Load–CMOD Curve (Displacement Rate Increase)**



**OUTPUT: $L_{max,1}$**
**(Maximum load in hydrogen)**

## STEP 2
## STEPWISE LOAD INCREASE

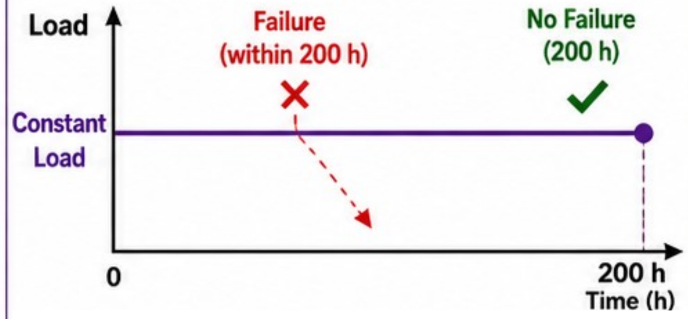
- Starting at 93% of $L_{max,1}$, specimens were subjected to incremental load increases of 1%.
- 30-minute hold at each step until failure occurred.
- The load, CMOD, maximum load in this stage ($L_{max,2}$), and total elongation at failure were used for the next stage.



**OUTPUT: $L_{max,2}$**
**(Maximum load in this stage) and total elongation at failure**

## STEP 3
## CONSTANT LOAD TEST

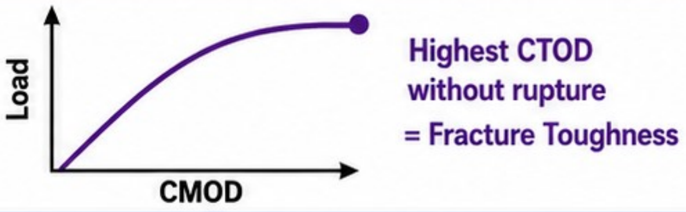
- The obtained $L_{max,2}$ served as the basis for longer-term constant load tests.
- If the specimen failed within the set time limit (200 hours), the load was reduced for the next test.
- If the specimen withstood the load, the test was aborted, and a higher load was applied.
- The fracture toughness was defined as the highest CTOD observed without rupture after the target hold time.



## FINAL RESULT

### FRACTURE TOUGHNESS IN HYDROGEN ENVIRONMENT

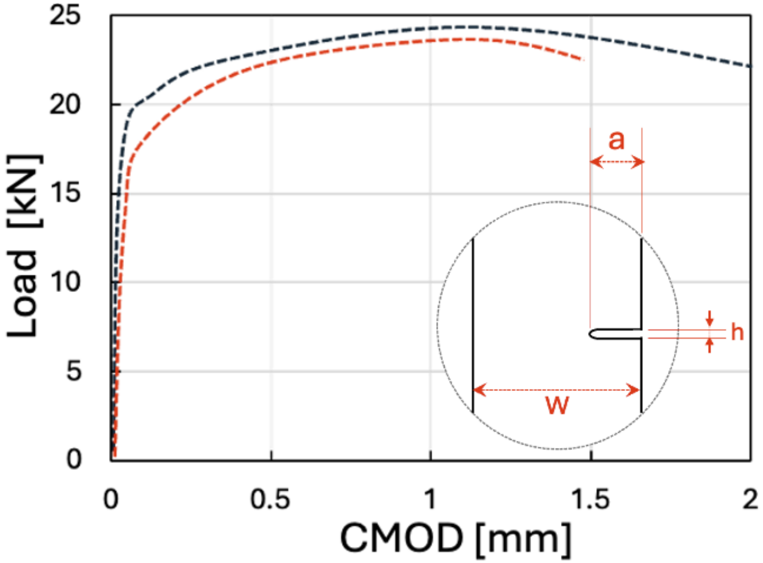
Defined as the highest CTOD observed without rupture after the target hold time (200 hours).



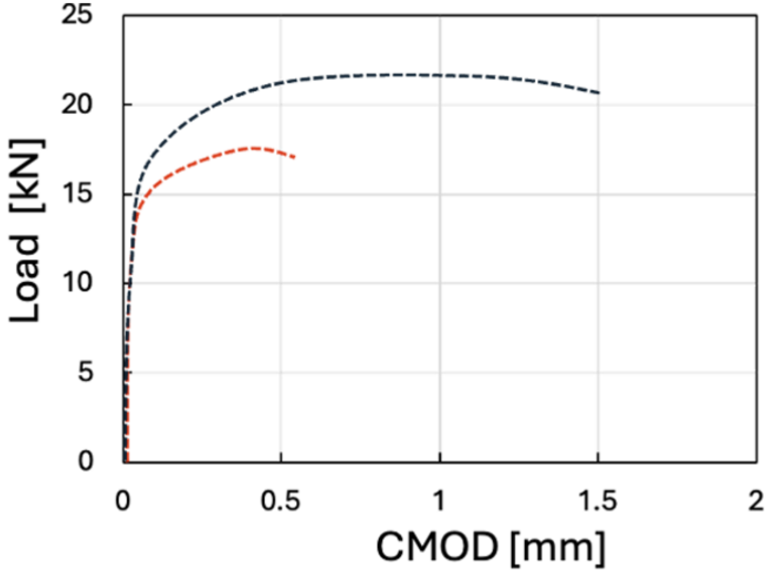
# Fracture Toughness of SENT Specimens in Air and Hydrogen

Step (i)

**HLAW**



**MAG**

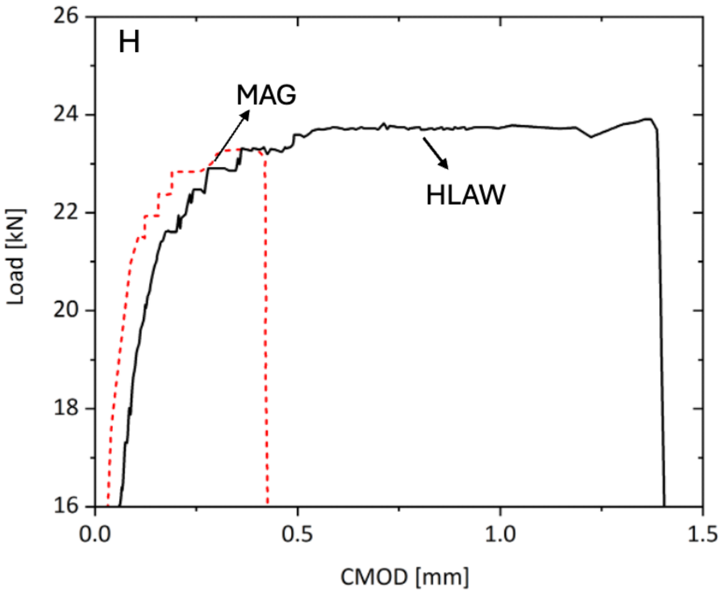


| Step (i) | Maximum Load (kN) | | Maximum CTOD (mm) | |
|---|---|---|---|---|
| | HLAW specimen | MAG specimen | HLAW specimen | MAG specimen |
| **Air** | 24.5 | 22.2 | 0.23 | 0.17 |
| **Hydrogen** | 23.2 | 17.8 | 0.17 | 0.06 |

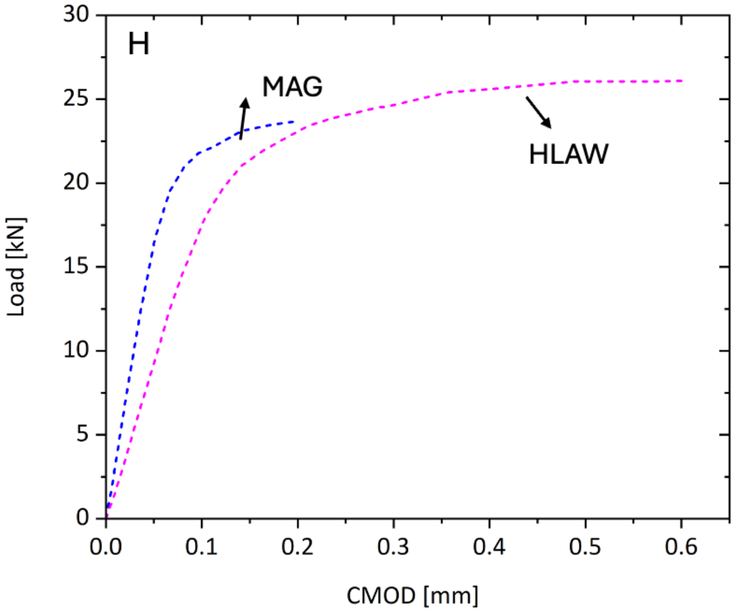
# Fracture Toughness of SENT Specimens in Air and Hydrogen

## Step (ii) and (iii)

### Step (ii)



### Step (iii)



| | Maximum Load (kN) | | Maximum CTOD (mm) | |
|---|---|---|---|---|
| | HLAW specimen | MAG specimen | HLAW specimen | MAG specimen |
| **Step (ii)** | 24.1 | 23.1 | 0.65 | 0.18 |
| **Step (iii)** | 25.3 | 24.2 | 0.28 | 0.09 |

Enhanced fracture toughness of HLAW in both hydrogen and air environments

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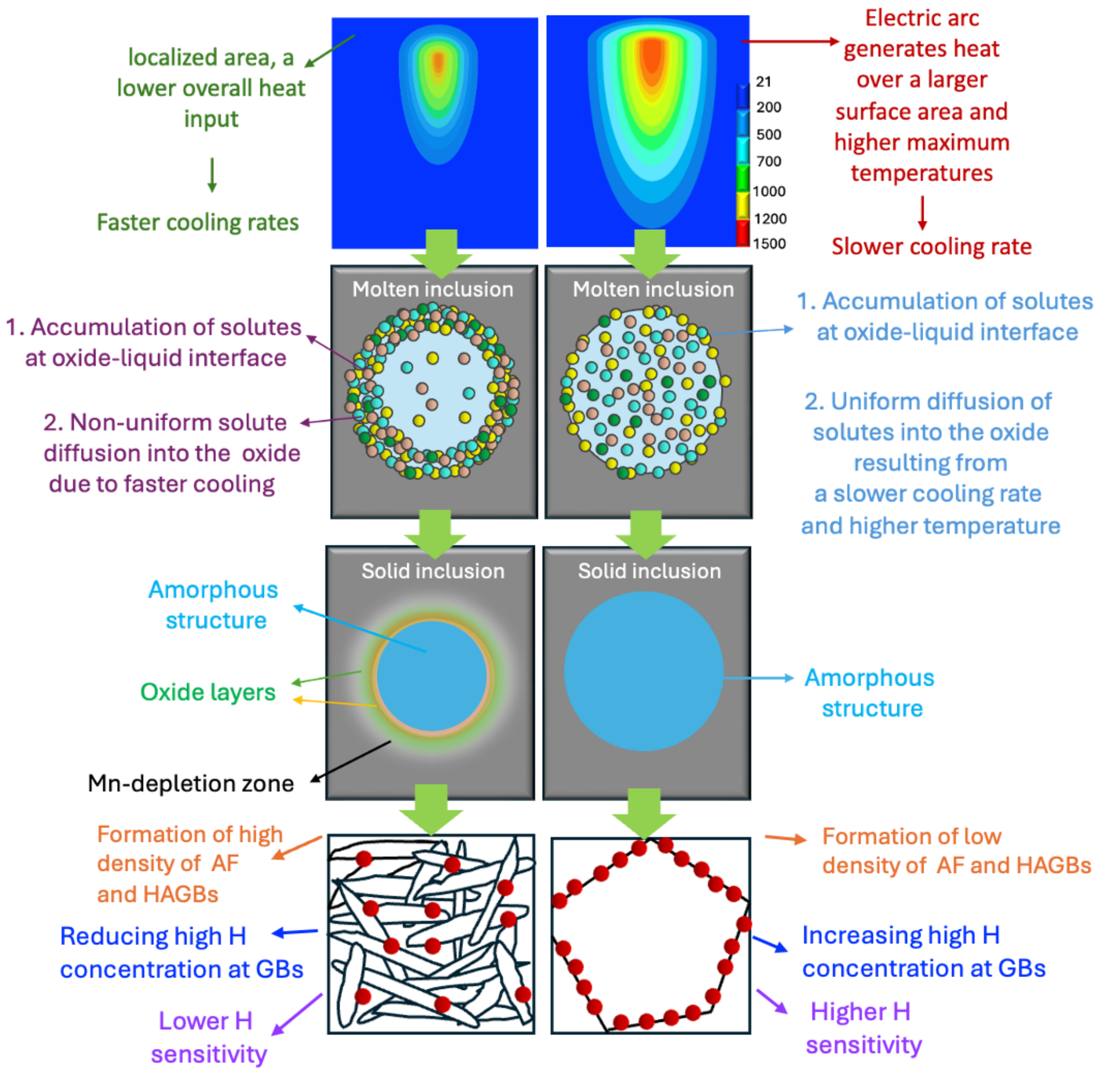
# An overview of the influence of different welding methods on microstructure and fracture toughness in hydrogen environment



localized area, a lower overall heat input

↓

Faster cooling rates

Electric arc generates heat over a larger surface area and higher maximum temperatures

↓

Slower cooling rate

1. Accumulation of solutes at oxide-liquid interface

2. Non-uniform solute diffusion into the oxide due to faster cooling

1. Accumulation of solutes at oxide-liquid interface

2. Uniform diffusion of solutes into the oxide resulting from a slower cooling rate and higher temperature

Amorphous structure

Oxide layers

Mn-depletion zone

Amorphous structure

Formation of high density of AF and HAGBs

Reducing high H concentration at GBs

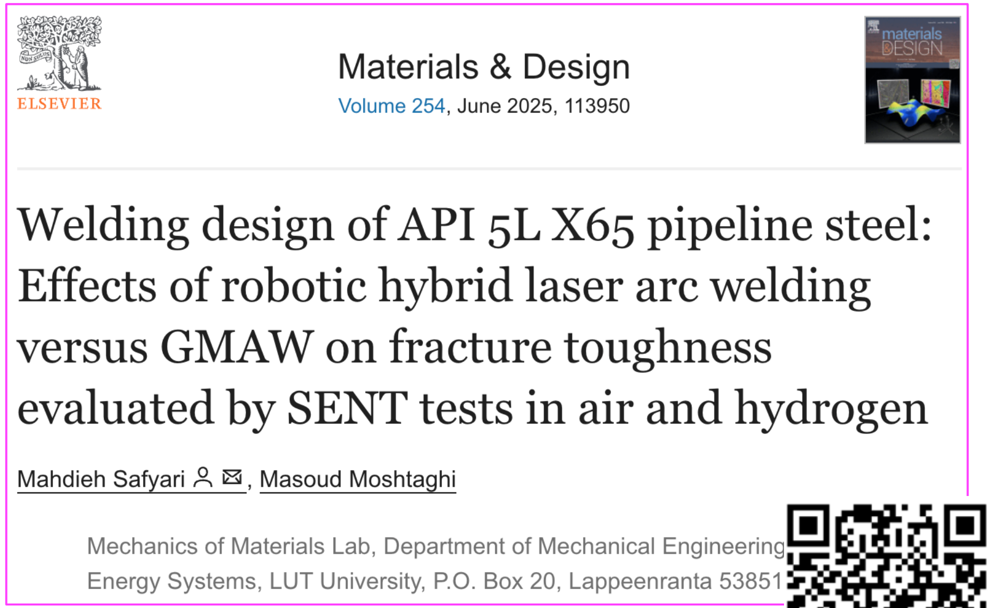
Lower H sensitivity

Formation of low density of AF and HAGBs

Increasing high H concentration at GBs

Higher H sensitivity

## More Information!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Materials & Design

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Welding design of API 5L X65 pipeline steel: Effects of robotic hybrid laser arc welding versus GMAW on fracture toughness evaluated by SENT tests in air and hydrogen

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# Conclusions

For structural integrity and life assessment of H-pipelines:

- It is very important to understand the combined effect of hydrogen, stress, and different microstructures.
- Different service loading conditions should be considered.
- Gaseous charging provides much more realistic material behavior.
- Fracture toughness should be evaluated using multiple methods to capture different features like threshold stress levels and crack initiation behavior over time, predicting component lifetime and energy required for crack propagation.

# Thank you!

Human
Potential is
Unlimited.