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Novel Approaches for Detecting Hydrogen Embrittlement in Pipeline Steels: Microindentation and Alternative Assessment Methods

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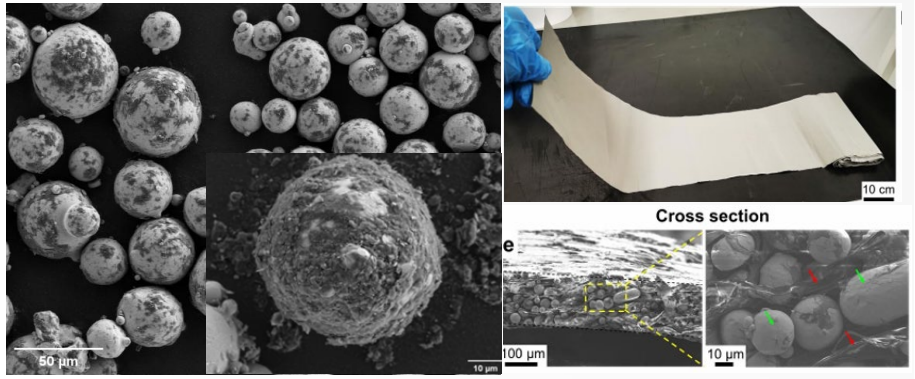
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Science and Technology in Advanced Manufacturing & Materials (STAMM)

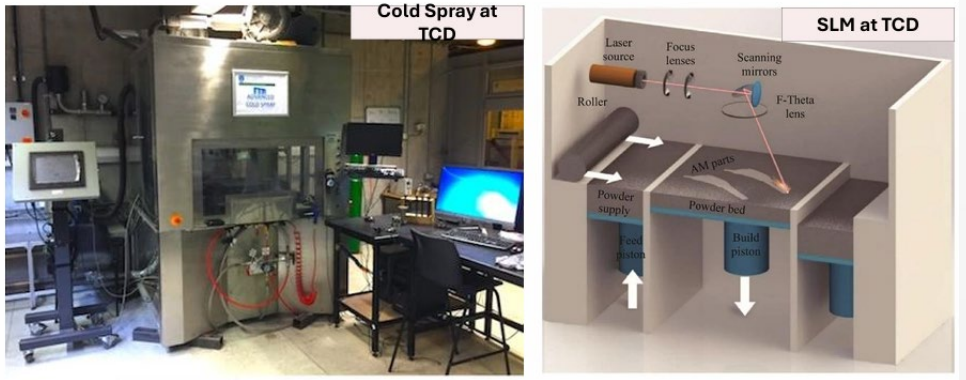
- Led by Professor Rocco Lupoi

Materials Development



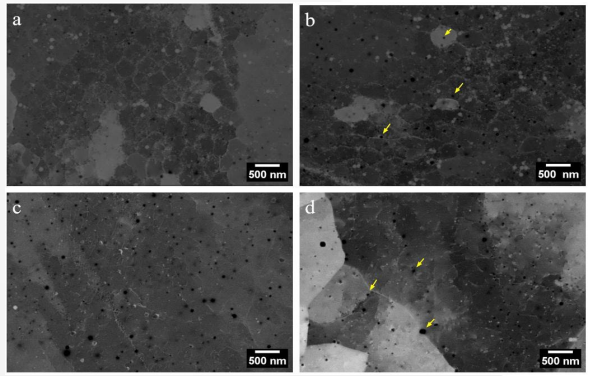
Metal–2D Composites
Powder Metallurgy and MAPS

Advanced Manufacturing



Cold Spray and Laser Powder Bed Fusion

Materials Performance



Microstructure Characterisation Mechanical Testing and Hydrogen Effects

https://stamlab.eu/

stamm

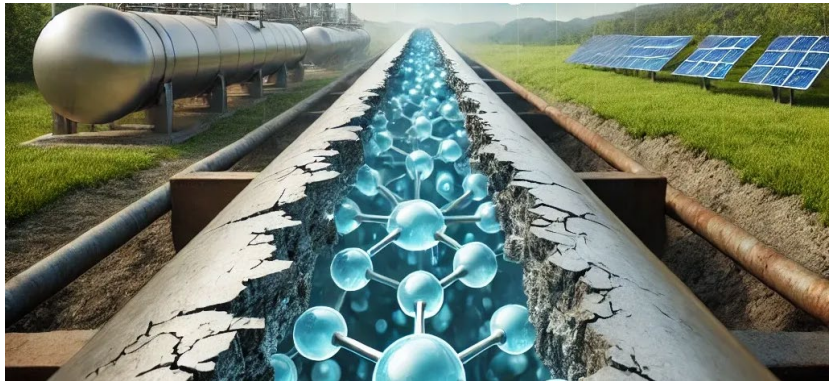
Science and Technology in Advanced Manufacturing & Materials

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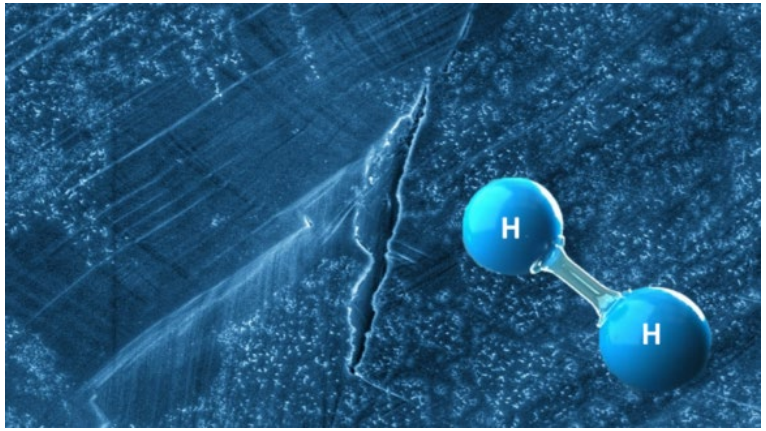
Hydrogen Embrittlement: Challenges for Existing Infrastructure



Why hydrogen?
Support a low carbon economy.



Why existing pipelines?
Infrastructure in place. Potentially lower costs.





The challenge?
Metal embrittlement.
Increased cracking risk.

Dwivedi, Sandeep Kumar, and Manish Vishwakarma. "Hydrogen embrittlement in different materials: A review." *International journal of hydrogen energy* 43.46 (2018): 21603-21616.
Yu, Haiyang, et al. "Hydrogen embrittlement as a conspicuous material challenge—comprehensive review and future directions." *Chemical Reviews* 124.10 (2024): 6271.

Transportation of Hydrogen



Mechanical strength
Withstand internal pressure.

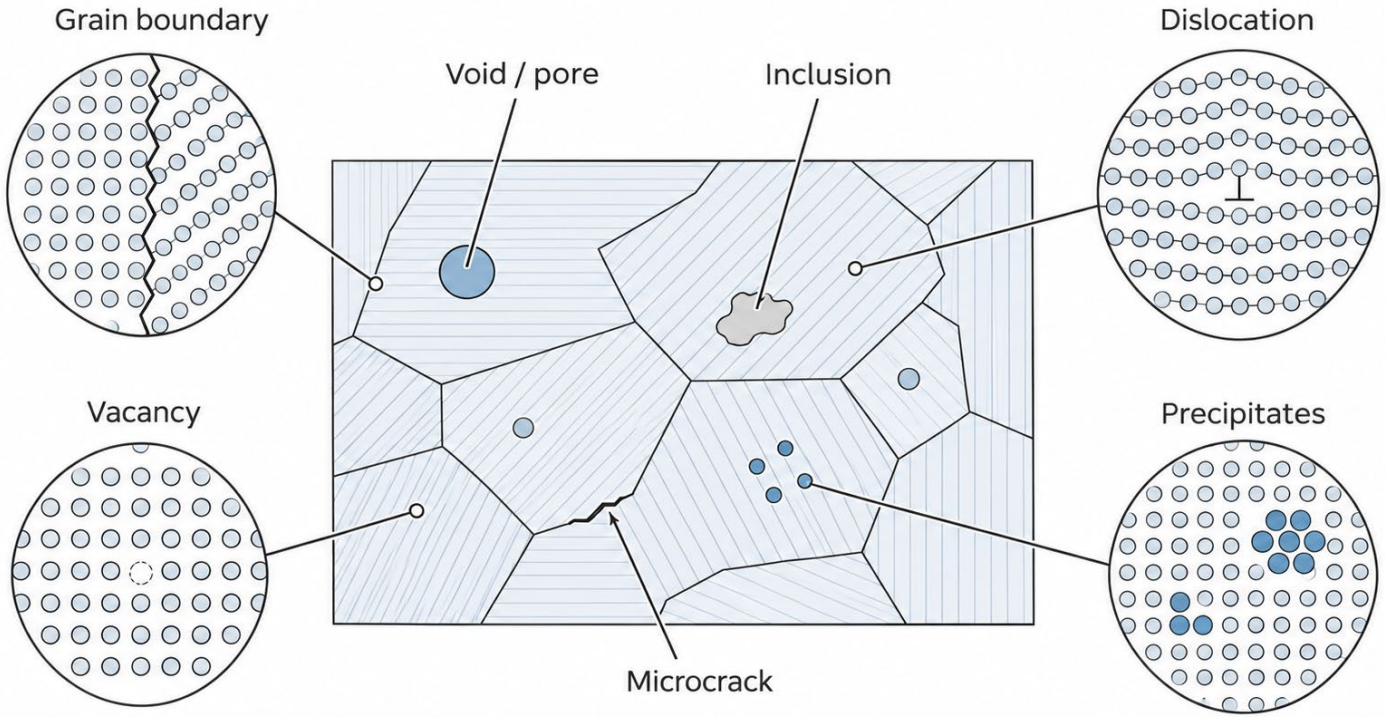
Ductility and toughness
Accommodate deformation. Resist fracture.

Manufacturability
Form and weld long pipe sections.

Hydrogen can compromise these mechanical properties.

Inside a Metal: Microstructure and Defects





Metals contain grains, interfaces and defects.
These features influence deformation and fracture.
Microstructure matters for hydrogen susceptibility.

Research Gap – Our approach

Nanoscale

Research Gap

Macroscale

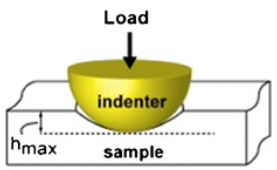
Nanoindentation

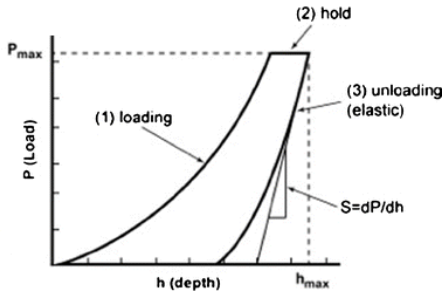
PIP

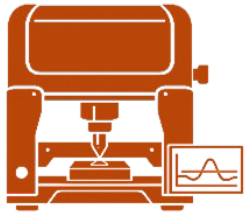
SSRT / FCG

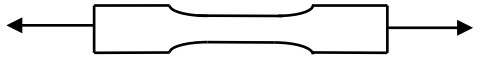
Smaller

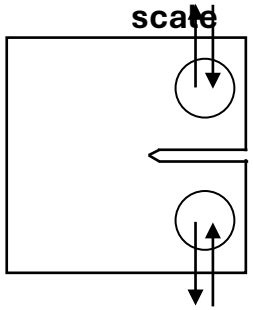
Larger scale











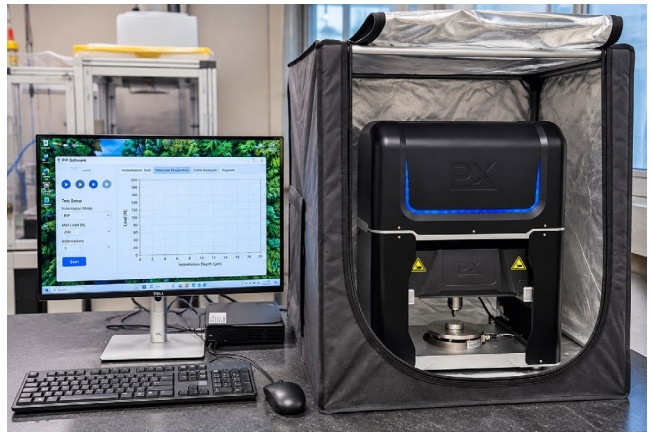
Advantages	Disadvantages
• Local • Mechanistic • Microstructure-sensitive • Small material volume	• Surface artefacts • Poor Bulk relevance • Submicron to micron • Depth: ~0.1–3 μm

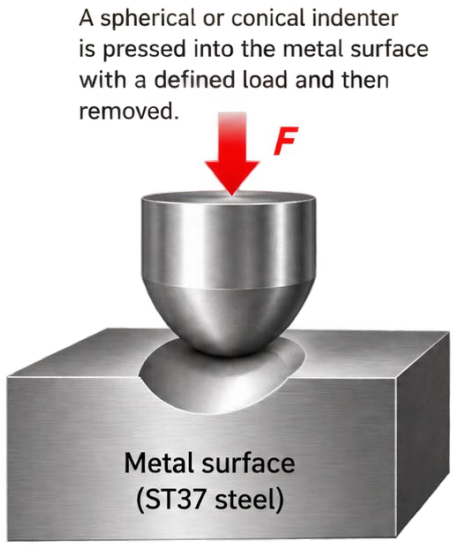
Intermediate Scale
• Profile-based • Small-volume • Submillimetre to millimetre • Diameter: ~0.25–1 mm

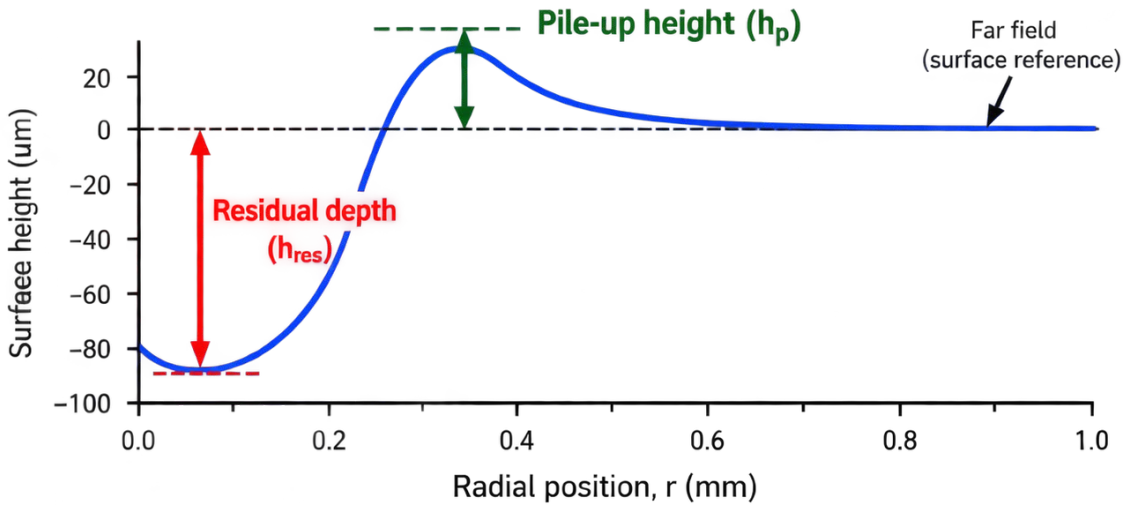
Advantages	Disadvantages
• Simple • Standardised • Comparative screening	• Slow / larger test burden • Millimetre to centimetre

PIP: Probing How Metals Deform

Profilometry based Indentation Plastometry





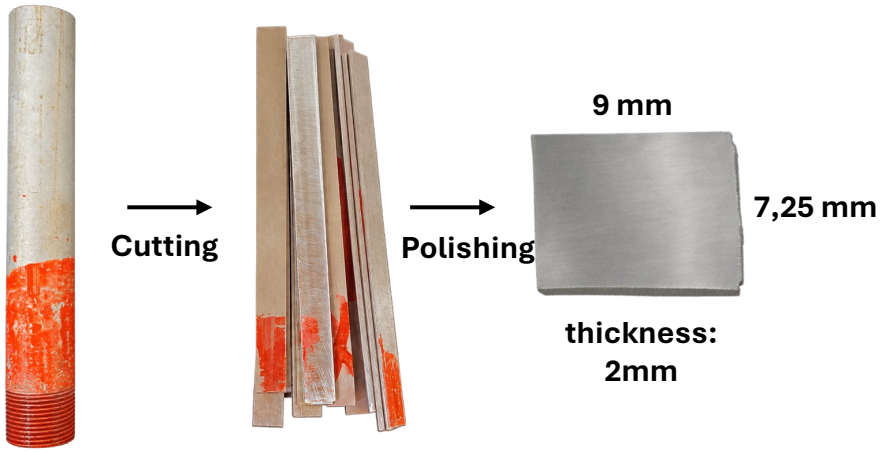


- **Press a spherical indenter**
- **Measure the residual surface profile**
- **Analyse the plastic deformation**

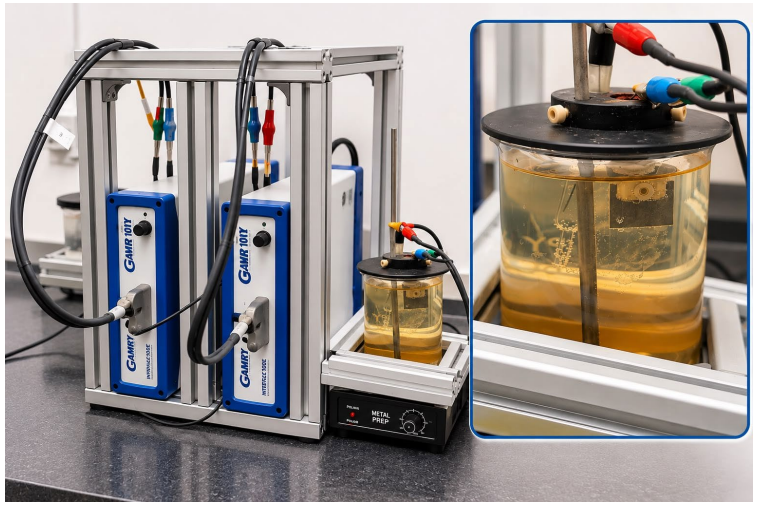
PIP allows us to compare how steel deforms before and after hydrogen charging.

Experimental Setup

Sample Preparation



Electrochemical Charging



- Electrolyte: 3 wt.% NaCl + 0.1 wt.% NH4SCN
- $j = -10\ mA/cm^2$

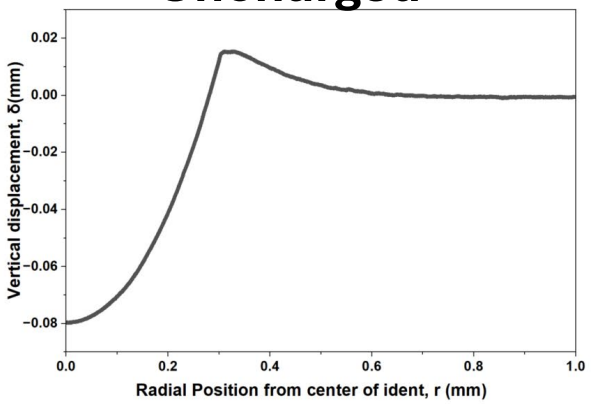
PIP Testing



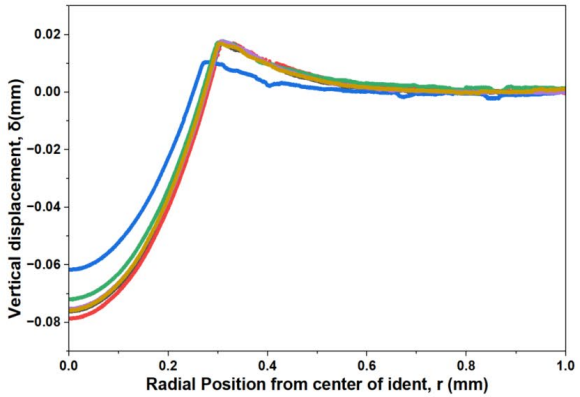
- Plastometrex PLX Benchtop
- Spherical Si3N4 indenter
- Integrated Profilometer
- Residual indent topography for pile-up analysis

ST37 Steel: Assessing Hydrogen Effects

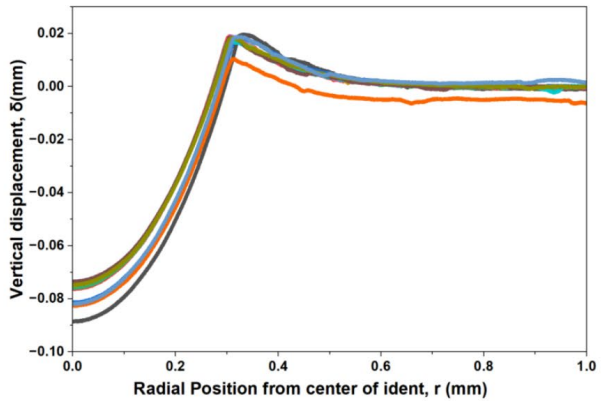
Uncharged



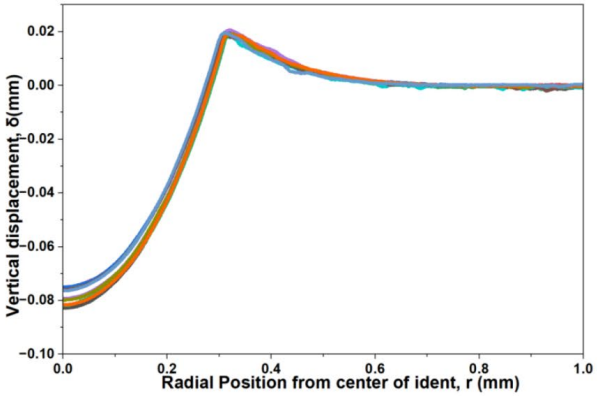
-10 mA/cm²



-20 mA/cm²

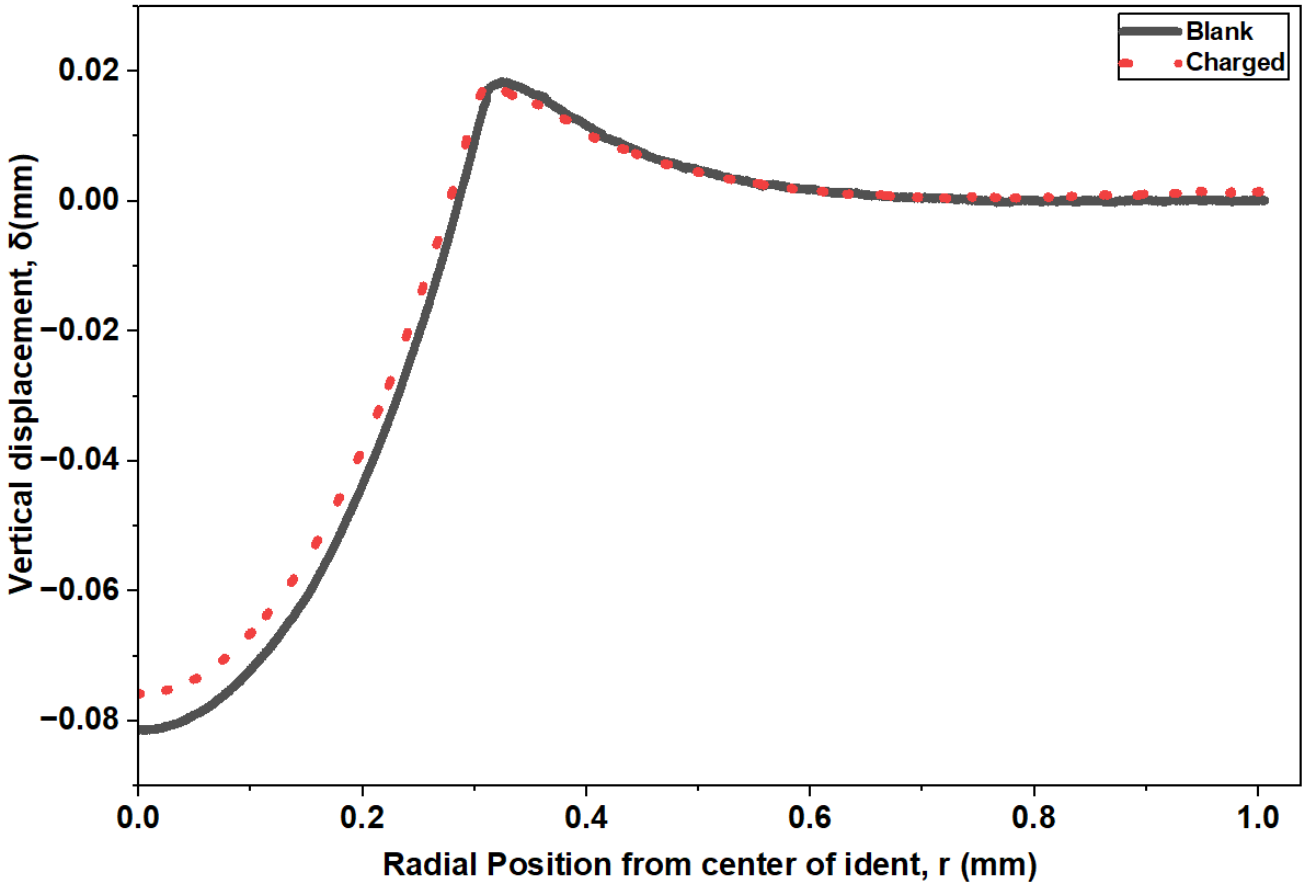


-30 mA/cm²



Samples – Charging Conditions	Residual Depth (µm)	Pile Up height (µm)
Uncharged	79.01 ± 2.05	17.08 ± 5.61
Charged - 10 mA/cm²	73.32 ± 6.06	16.21 ± 2.82
Charged - 20 mA/cm²	78.24 ± 2.97	19.39 ± 0.69
Charged - 30 mA/cm²	72.22 ± 4.01	17.70 ± 0.81

Factors in Hydrogen Testing

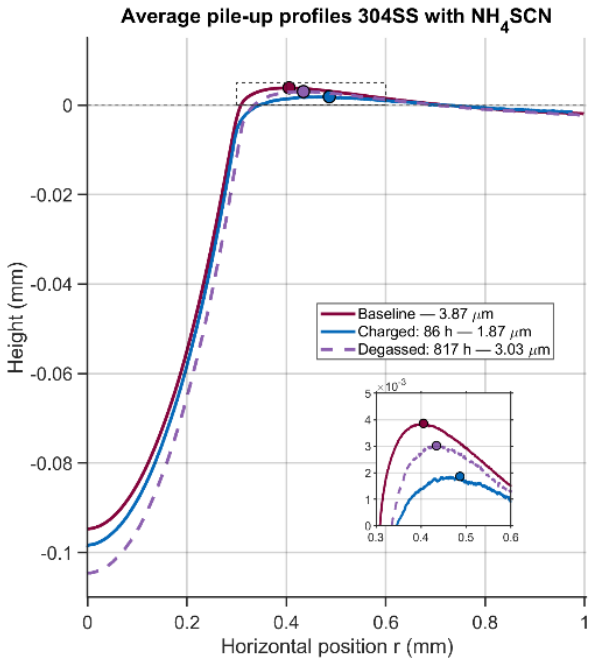


- Several indents per sample.
- **Time between charging and PIP: ~3 min.**
- **Measurement time: 4 min per indentation**
- Last indent: ~60 min.
- Total testing time: ~1 hour.

Consistent timing is essential for meaningful comparisons

Continuous charging: 304SS vs LPBF CoCrFeMnNi

304SS: 86 h @ RT



Baseline	3.99 ± 0.68 μm
Charged (86h)	1.94 ± 0.13 μm
Degassed (817h)	3.09 ± 0.94 μm

$\mathbf{HEI_{peak}(charged) = 51.8\%}$

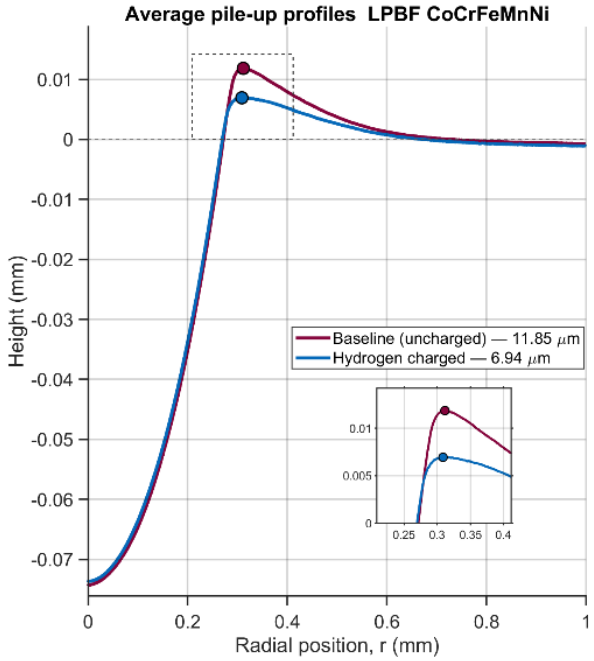
$\mathbf{HEI_{peak}\ (degassed) = 21.7\%}$

Degassing recovery

Partial recovery

Supports hydrogen-reversible effects

LPBF CoCrFeMnNi: 72h @ 85ºC



Baseline	11.93 ± 0.34 μm *
Charged (72h)	7.07 ± 0.55 μm

*** Degassed state not included: limited specimen size**

$\mathbf{HEI_{peak}(charged) = 41.4\%}$

LPBF CoCrFeMnNi: smaller measured response

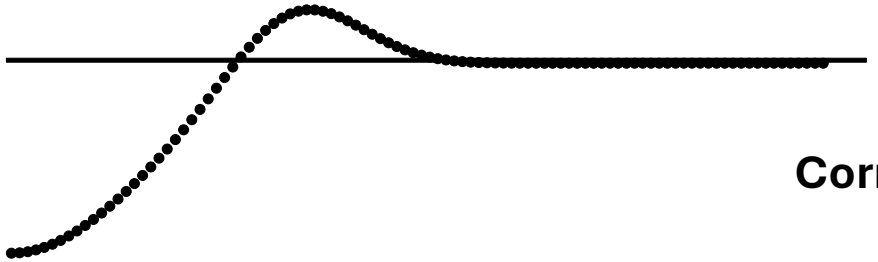
More aggressive thermal conditions

Reduced HEI_{peak}: lower apparent susceptibility

PIP reveals changes in residual deformation after hydrogen charging.

PIP Screening Needs Mechanical Validation

PIP Screening



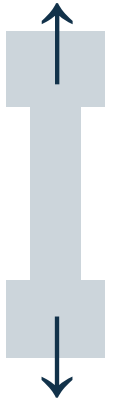
Does the deformation profile change?

Residual depth

Pile up height

Correlation and validation

Mechanical Testing



Does the mechanical performance detoriate?

Ductility

Fracture Resistance.

PIP identifies changes. Mechanical tests establish their significance.

Conclusions and Future Perspectives

PIP offers a promising complementary screening approach.

Hydrogen-charged ST37 samples tend to show shallower residual indents.

304SS and LPBF CoCrFeMnNi show reduced pile-up after hydrogen charging.

304SS shows partial recovery after degassing.

Controlled timing and repeatability are essential.

Simple. Fast. Complementary.

Acknowledgements

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Luke McNally



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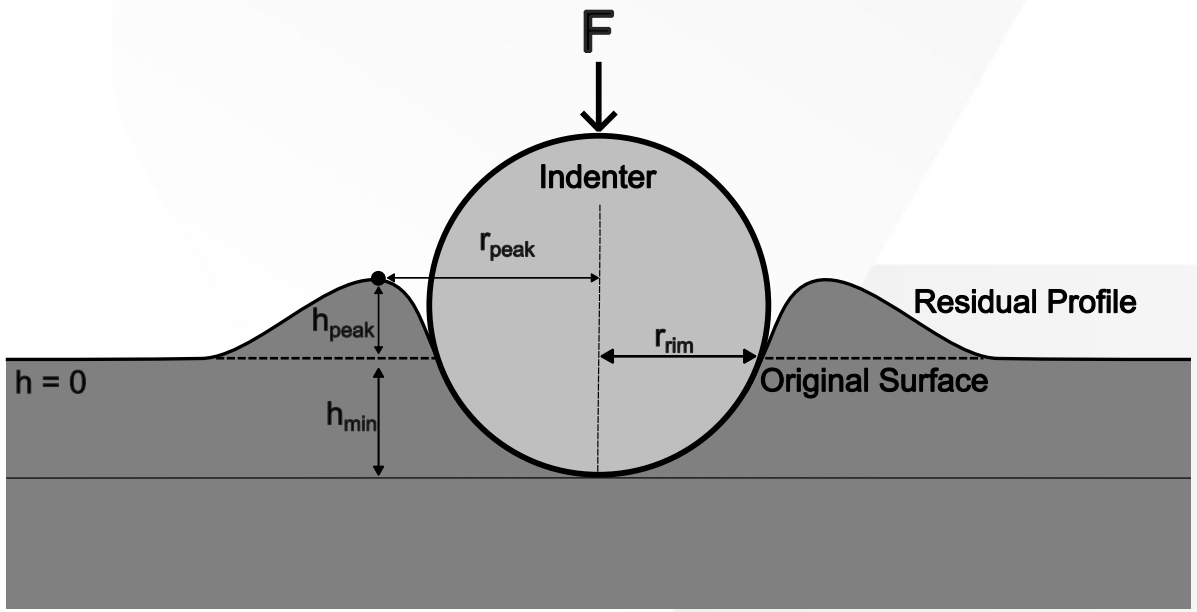
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Residual Profile Descriptions

Descriptor Extraction

- Raw: h_{peak}, h_{min} , r_{peak}, r_{rim}
- Normalized: η, ρ
- HEI from change relative to baseline value



Raw Geometric Descriptors

- h_{peak} : peak pile-up height
- h_{min} : maximum valley depth
- r_{peak} : radial position of peak pile-up
- r_{rim} : radial position of indent rim

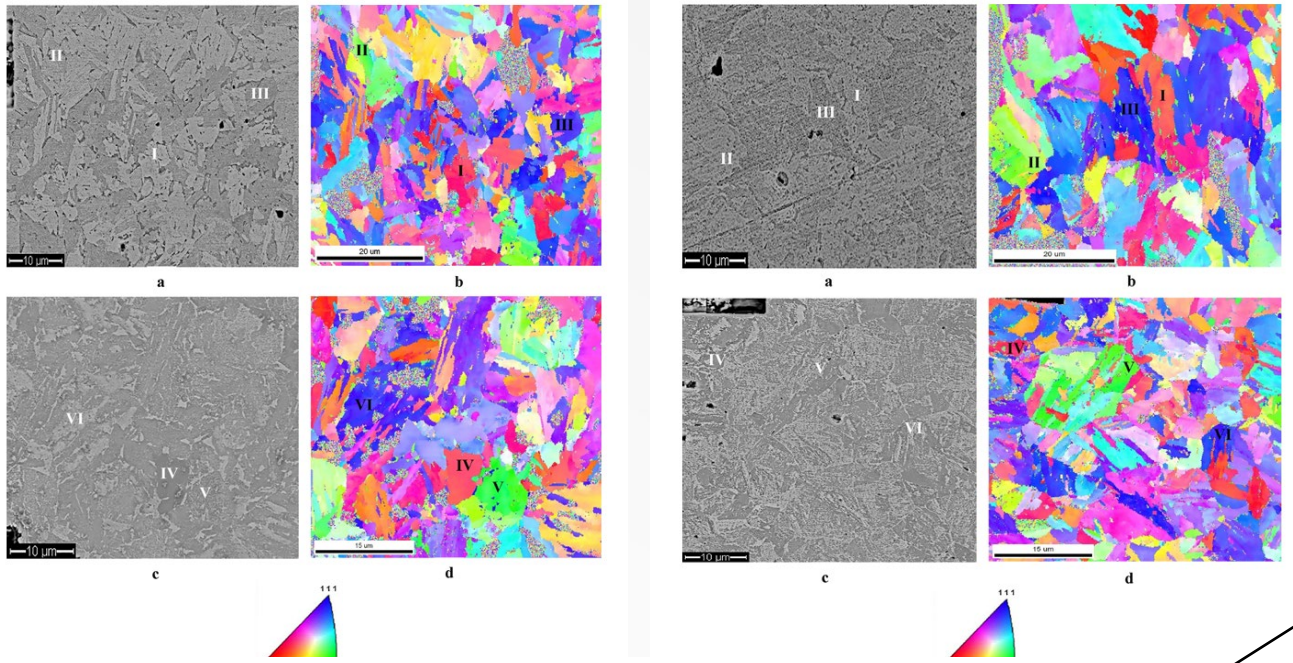
Normalized Descriptors

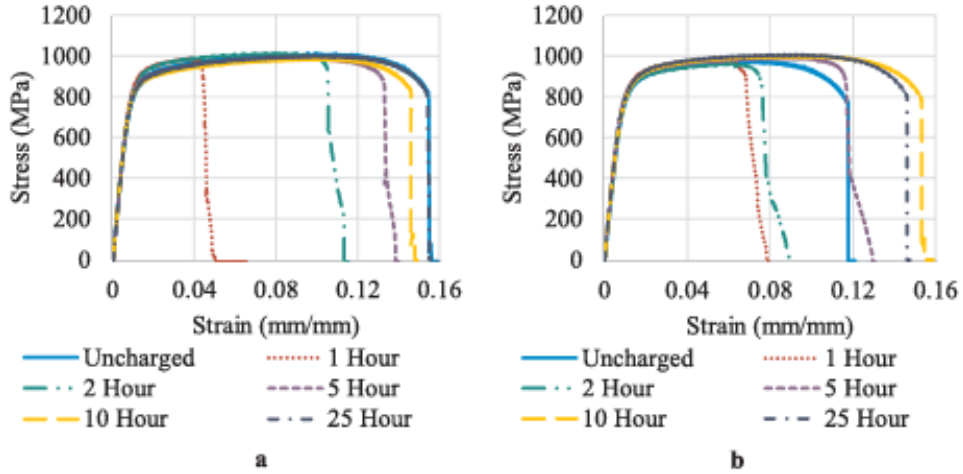
$$\eta = \frac{h_{peak}}{|h_{min}|} \qquad \rho = \frac{r_{peak}}{r_{rim}}$$

Comparative HE metrics

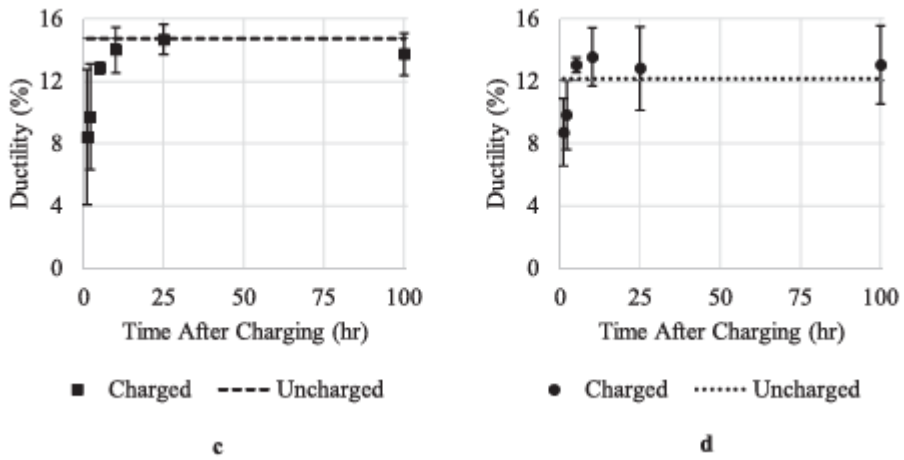
- $HEI = \frac{P_0 - P_{H_2}}{P_0}$
- Primary descriptor in this: h_{peak}

Hydrogen Effects: Recovery after charging





Ductility can recover as diffusible hydrogen escapes.
Recovery depends on the material and charging conditions.
Predicted recovery times: 4.2 h (TBF-980) and 7.2 h (DP-980).



Allen, Q. S., & Nelson, T. W. (2019). Microstructural evaluation of hydrogen embrittlement and successive recovery in advanced high strength steel. *Journal of Materials Processing Technology*, *265*, 12-19.