Evaluation of the tensile properties of X65 pipeline steel in compressed gaseous hydrogen using hollow specimens

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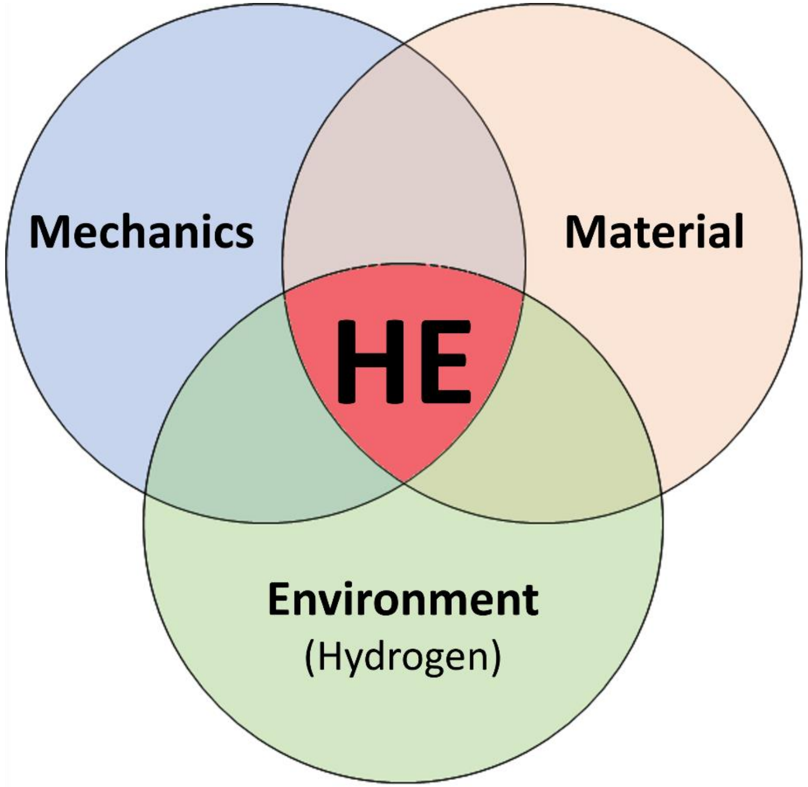
Hydrogen embrittlement

Hydrogen embrittlement is the **loss of materials' mechanical properties** resulting from the ingress of hydrogen atoms into the metal lattice.

HE relies on the **synergistic interaction** of three factors:

- **Environment** → temperature, pressure, hydrogen purity
- **Material** → chemical composition, microstructure, strength
- **Mechanics** → stress concentrations, strain rate

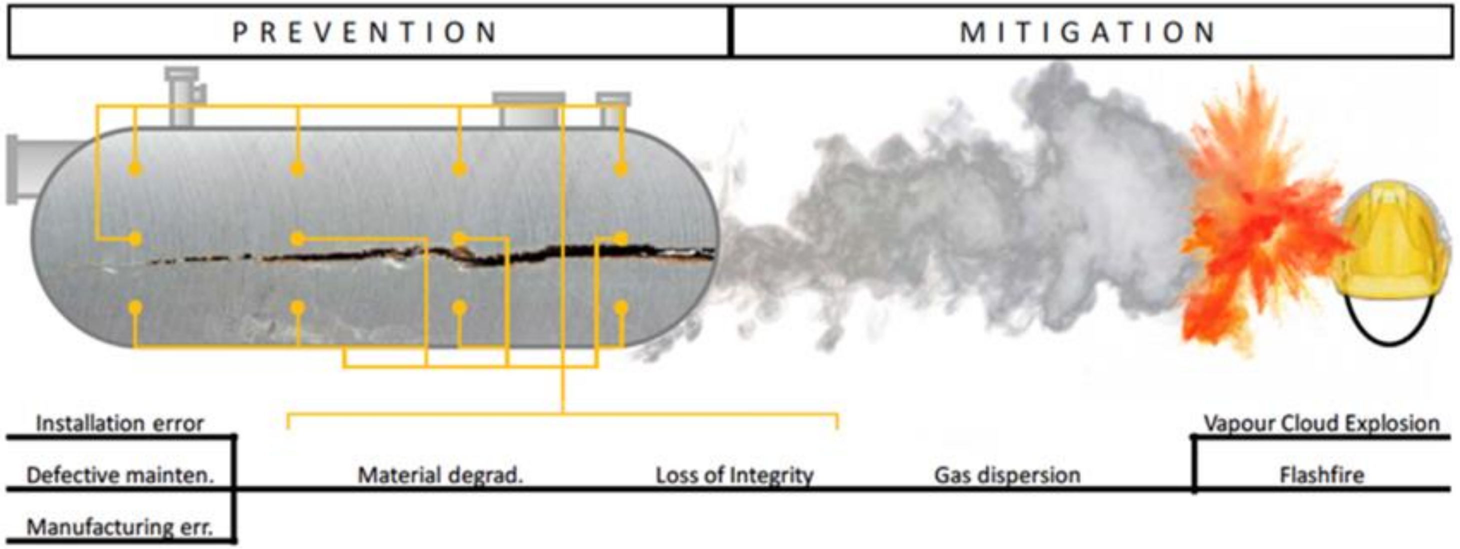


Hydrogen embrittlement

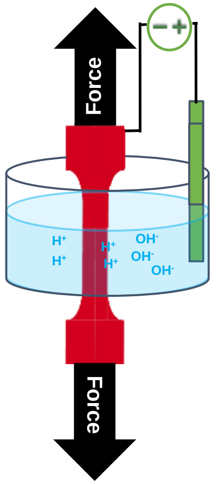
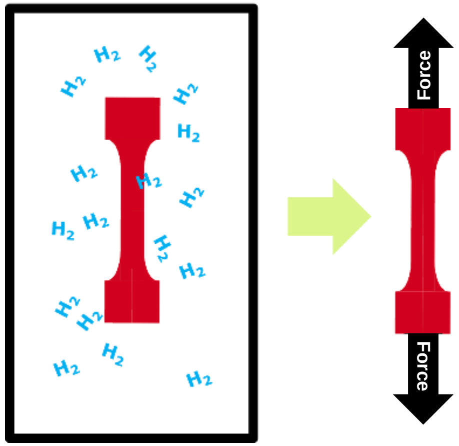
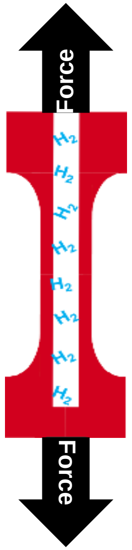
Only a **few engineering standards** consider hydrogen embrittlement in the design, inspection, and maintenance of components.

The lack of a unified regulatory framework results in **over-conservative design criteria** and the use of a **limited variety of materials**.

New standards are required to regulate the design of hydrogen technologies.



Testing techniques in hydrogen

1. Ex-Situ or In-Situ Electrochemical charging	2. Ex-situ precharging with subsequent tensile testing	3. In-situ charging by testing in the autoclave (gaseous hydrogen)	4. In-situ charging by gas volume in the test specimen
			

Testing techniques in hydrogen

Material tested in high-pressure autoclaves:

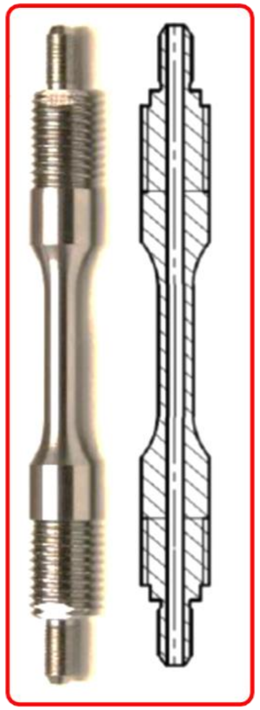
- **Estabilished** method
- Tests performed with **standardized** (solid) specimens
- **High test costs** due to extensive safety regulations
- Complex and **difficult to maintain**
- **Long** overall test **duration**

High Pressure Autoclave [Adapted from MPA Stuttgart]

Testing techniques in hydrogen

Conventional tensile specimen with **inner hole:**

- Hydrogen pressure applied in the inner hole
- **Sample itself** acts as an “autoclave”
- Only static sealings necesarry
- **Simple** to handle and reproduce
- **Shorter** overall test **duration**
- **Lower costs**

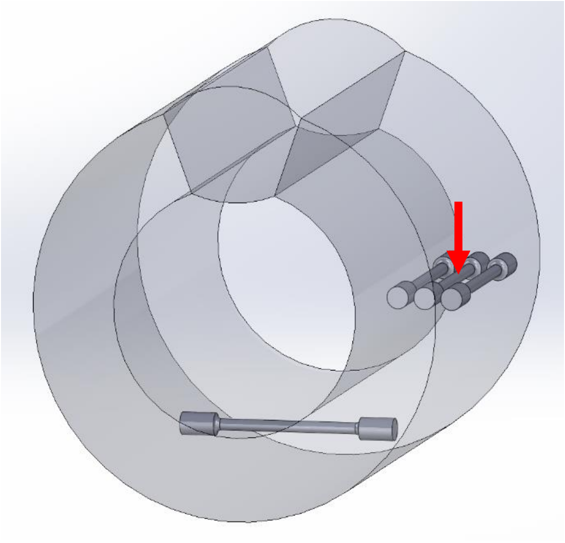


Experimental conditions

The material is a grade **API 5L X65 pipeline steel** manufactured in 1982 by Fukuyama Steel Works and used for natural gas transport subsea.

The material is extracted from the **base metal, pipe mid-thickness, longitudinal direction**.

The chemical composition was measured by **optical emission spectroscopy**.



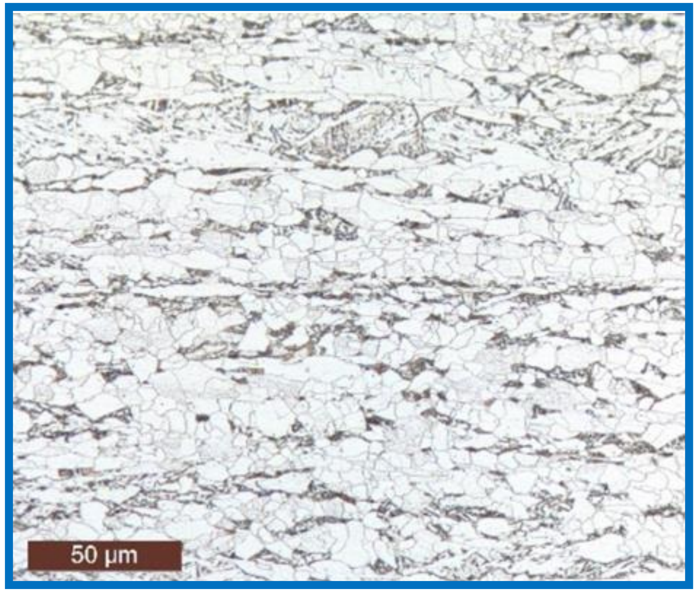
Chemical composition (%wt)

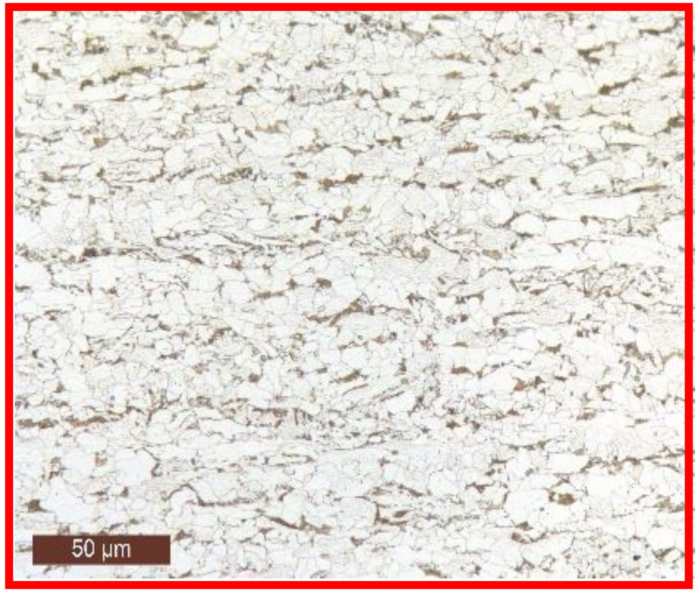
	C	Si	Mn	P	S	Cu	Cr	Ni	Mo	V	Nb	Ti
Nominal	< 0.1	< 0.6	< 1.6	< 0.025	< 0.015	< 0.25	< 0.25	< 0.25	< 0.05	< 0.1	< 0.05	< 0.02
Measured	0.07	0.25	1.53	0.013	< 0.002	<0.01	0.02	0.01	0.01	0.076	0.033	0.009

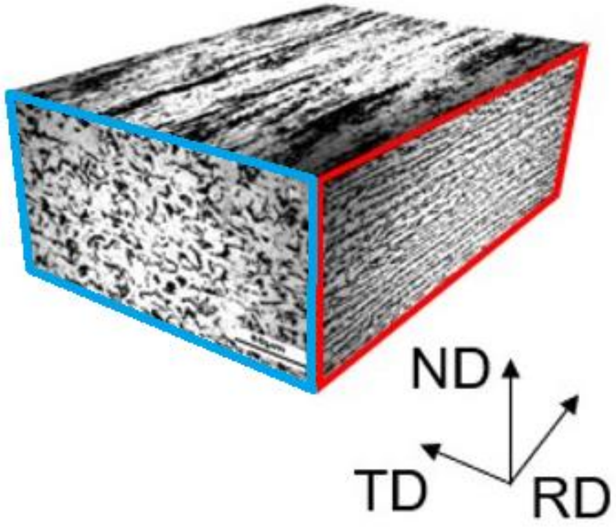
Materials & Methods

The microstructure is composed of **polygonal ferrite** and **pearlite** in **banded appearance**.

There is the presence of **plate-like bainitic bands**, which can be responsible for an **anisotropic behavior** of the mechanical properties.





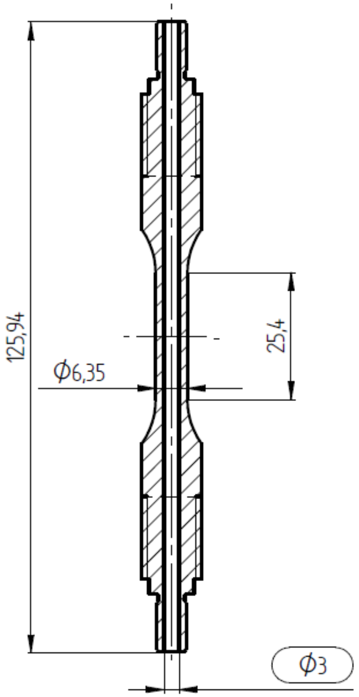


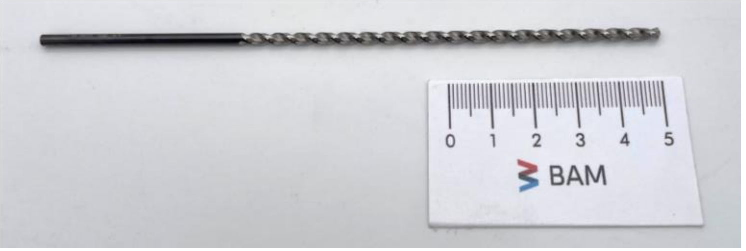
Materials & Methods

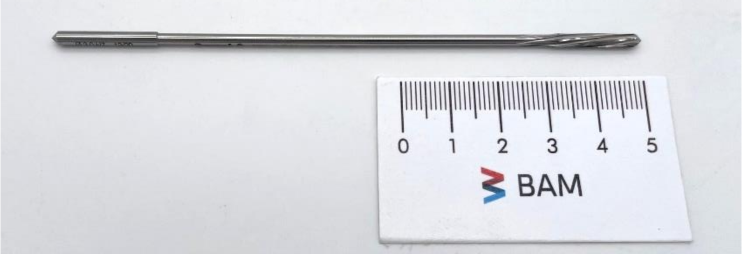
Two manufacturing processes were compared:

- Conventional drilling
  - Drilling wit HSS driller of 3 mm
- Drilling and reaming
  - Drilling of 2.8 mm hole and reaming to diameter of 3 mm

Results in different surface conditions







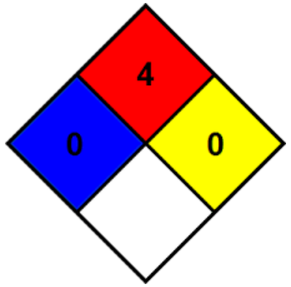
	R_a [µm]	R_z [µm]
Drilled	1.5	8.5
Reamed	0.1	1.4

Materials & Methods

In-situ slow strain rate tensile tests under the following conditions:

Hydrogen purity	99.999%
Nominal maximum oxygen content	2 ppm
Real oxygen content	4.5 ppm
Pressure	6 MPa
Temperature	22 °C
Nominal strain rate	10^{-6} s^{-1}
Displacement rate	$2.54 \cdot 10^{-5}$ µm/s

The hollow specimens are purged six times from 1 MPa to 6 MPa to reach the desired oxygen content.



Results

The hydrogen effect on tensile properties is evaluated through the **embrittlement index** and the **elongation loss**:

$$\mathrm{EI} = \frac{\mathrm{RA_{Ar}} - \mathrm{RA_{H_2}}}{\mathrm{RA_{Ar}}} \cdot 100$$

$$\mathrm{EL} = \frac{\mathrm{El_{Ar}} - \mathrm{El_{H_2}}}{\mathrm{El_{Ar}}} \cdot 100$$

Type	Environment	Average Fracture Area [mm²]	Average EI [%]	Average elongation [mm]	Average elongation loss [%]
Drilled	6 MPa Ar	8.22	-	6.08	-
Reamed	6 MPa Ar	8.1	-	6.065	-
Drilled	6 MPa H_2	12.83	27.76	4.72	21.97
Reamed	6 MPa H_2	10.29	13.37	5.07	16.45

Results

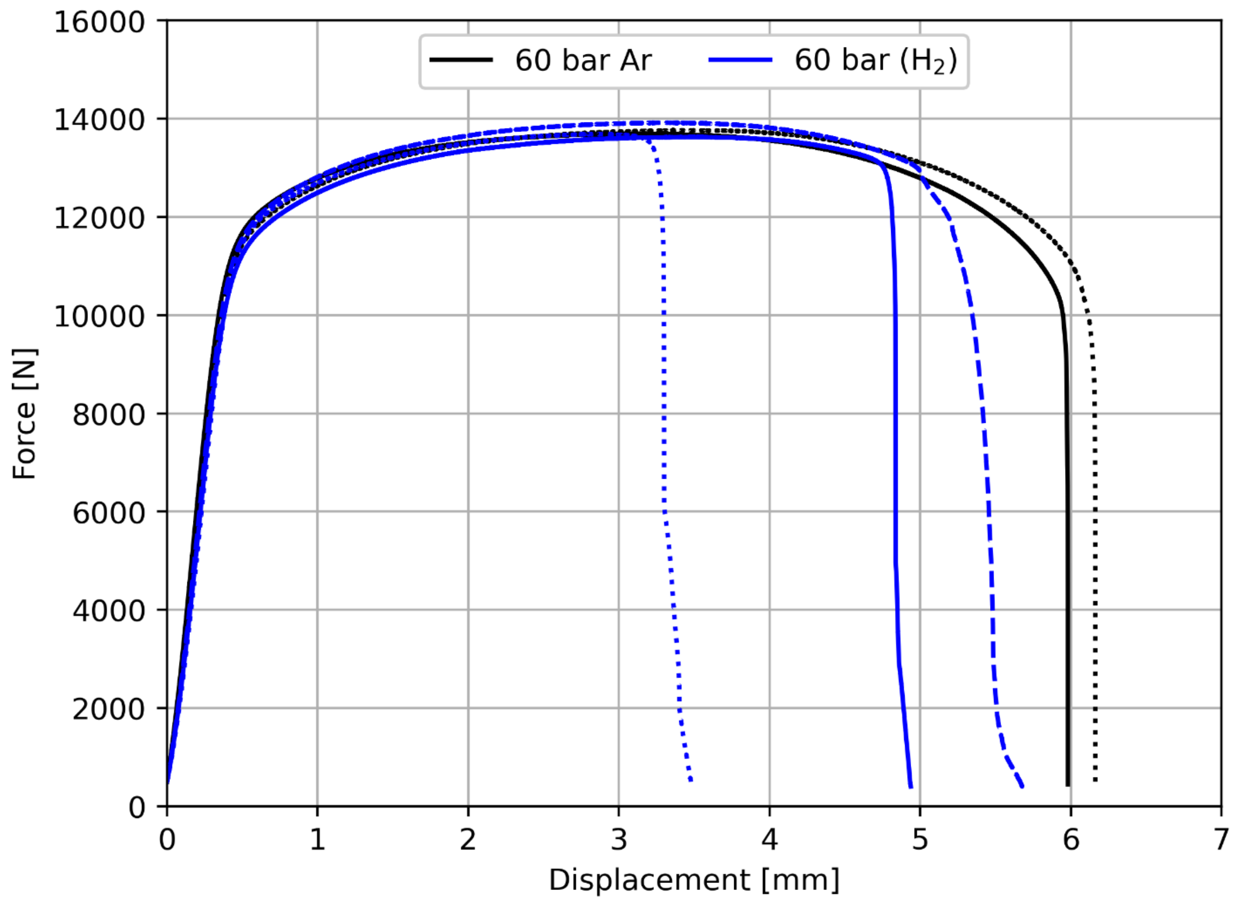
Drilled specimens

The samples tested in **hydrogen** show a **significant loss in elongation.**

The **scattering** is significant in hydrogen compared to argon.

The **inner surface roughness** influence the specimen **susceptibility** to HE.

Surface defects as notches and grooves might act as **crack initiation sites**.



Results

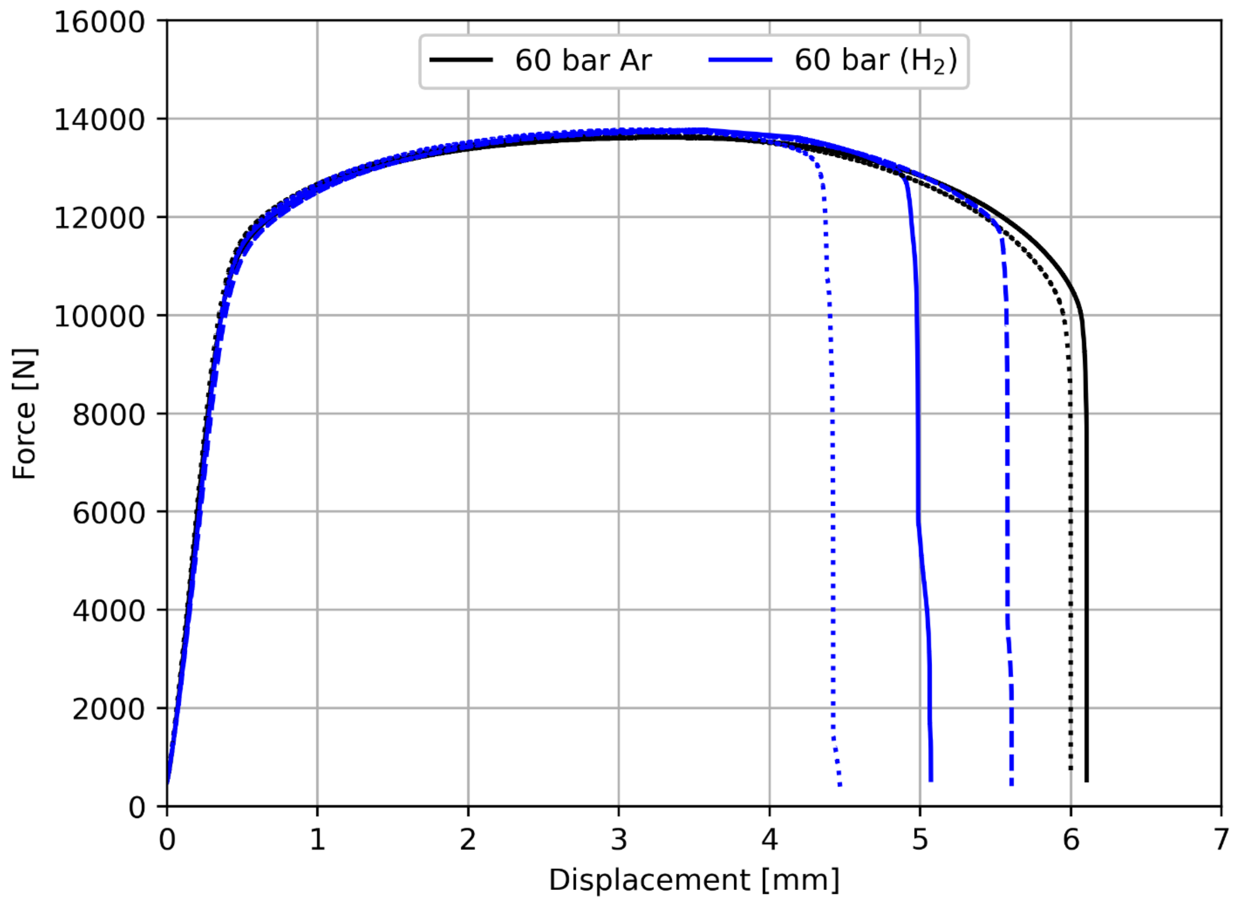
Reamed specimens

The samples tested in **hydrogen** show a **slight loss in elongation.**

The **scattering** in H_2 is lower compared with the drilled hollow specimens, but still significant compared to Ar.

The **lower inner surface roughness** makes these specimens **less susceptible** to HE.

Inner surface roughness affects the HE!



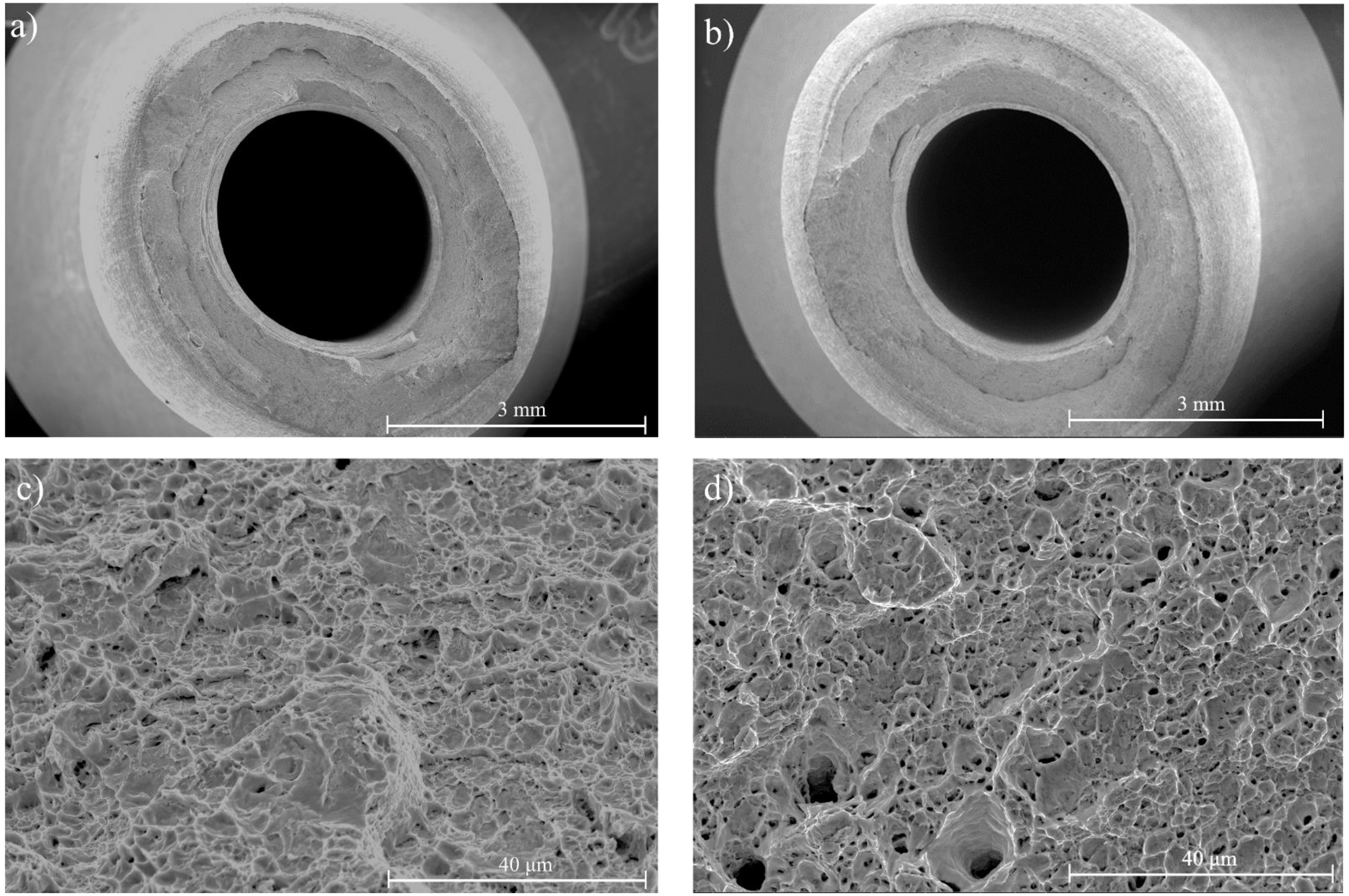
Results

Reference samples

The material tested in Ar shows an **elliptical shape** of the fracture surface due to **anisotropic microstructure.**

Dimples are visible for all specimen tested in Ar, indicating a ductile material behavior.

No differences between the two machining techniques in reference atmosphere!



Drilled Reamed

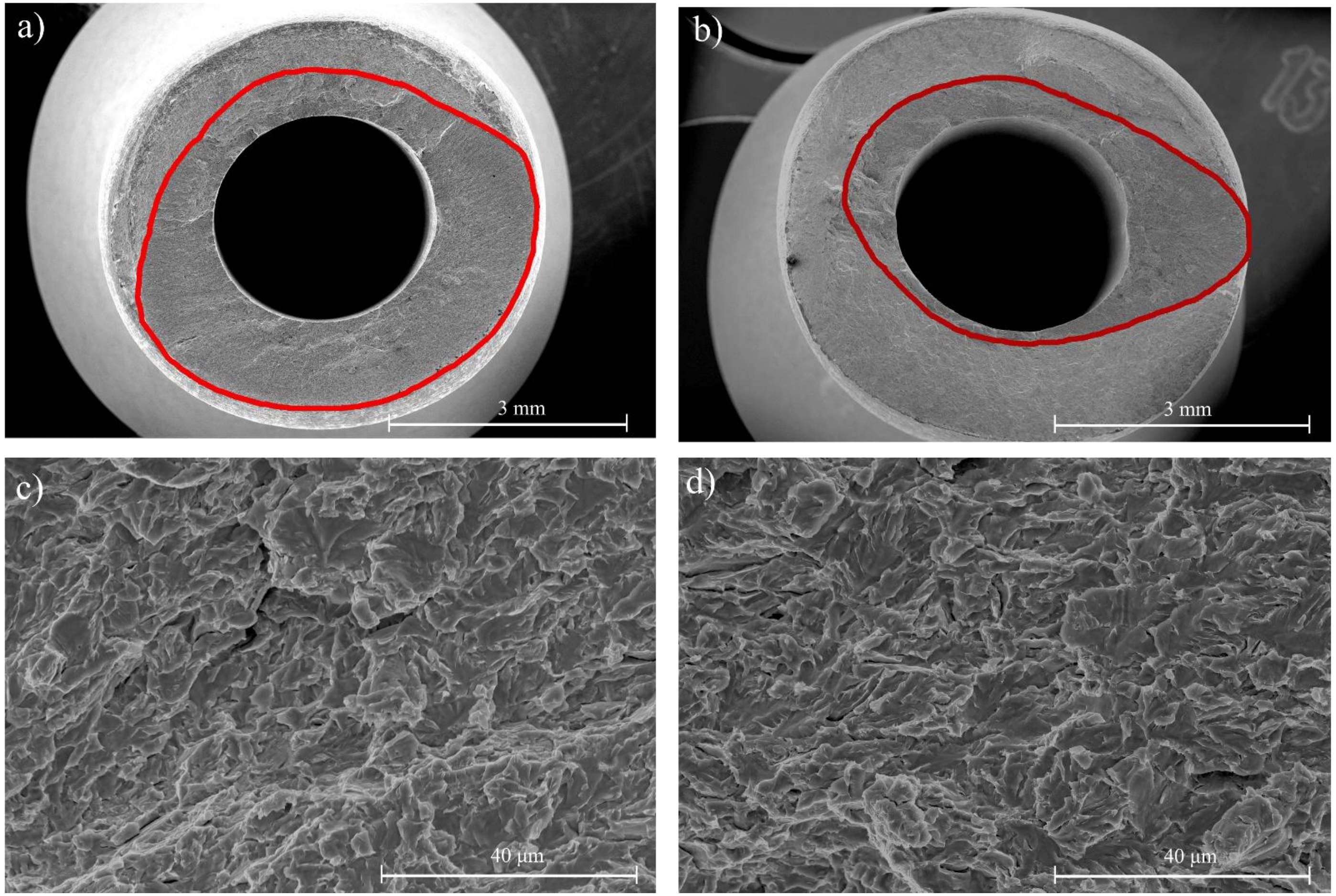
Results

60 bar hydrogen

The specimens tested in H show zones with **quasi-cleavage fracture**, indicating **brittle** material behavior.

Brittle areas are more pronounced for the drilled specimen.

Higher surface roughness leads to higher HE!



Drilled

Reamed

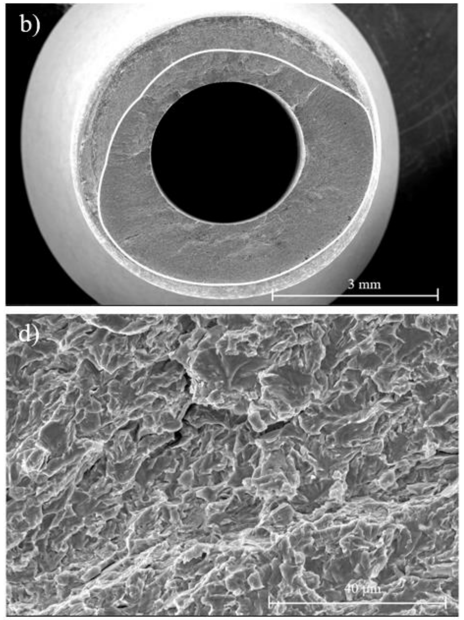
Conclusions

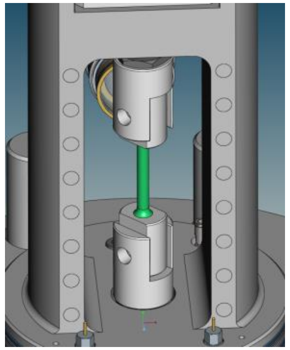
The hollow specimen technique is a **safer, faster, and cheaper solution** to screen the HE susceptibility.

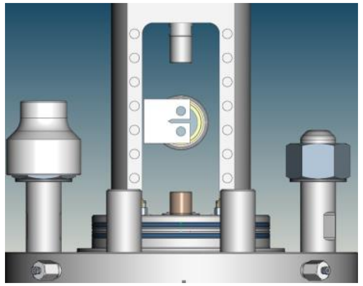
The scope is to **compare** tensile properties obtained from **“traditional” in-situ** pressurized hydrogen gas testing and **hollow specimen** testing.

Special emphasize must be taken on the inner **surface conditions** and sample preparation.

SMART – H Lab is equipped with 100 kN servo-hydraulic machine with autoclave for mechanical testing in hydrogen gas. Tests are ongoing!







Thank you for your attention

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References

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