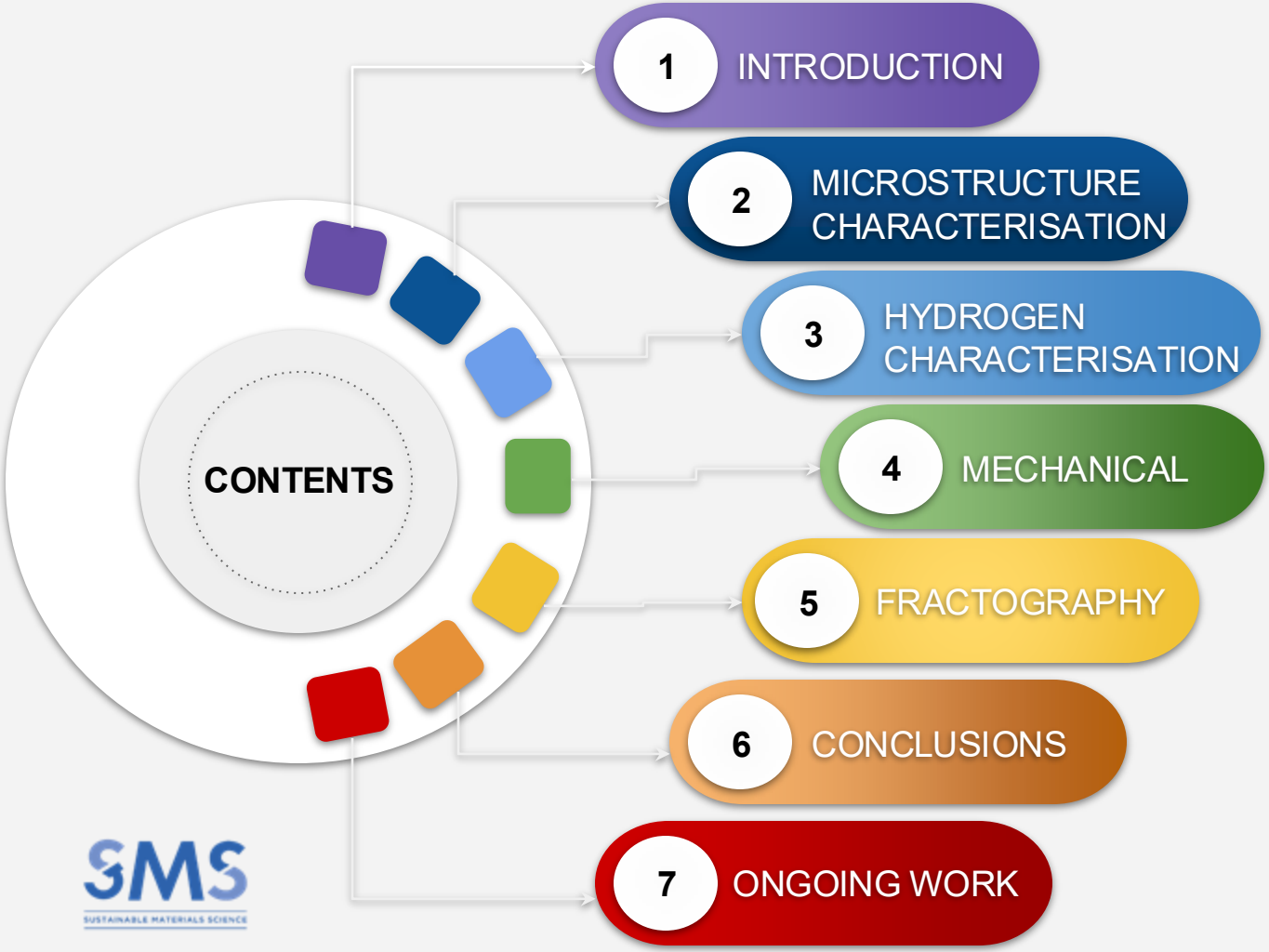
FRACTOGRAPHY OF HYDROGEN-CHARGED X70 PIPELINE STEELS UNDER QUASI-STATIC TENSION

Jubica, Laura De Pue, Lisa Claeys, Wim De Waele, Stijn Hertelé, Tom Depover, Kim Verbeken



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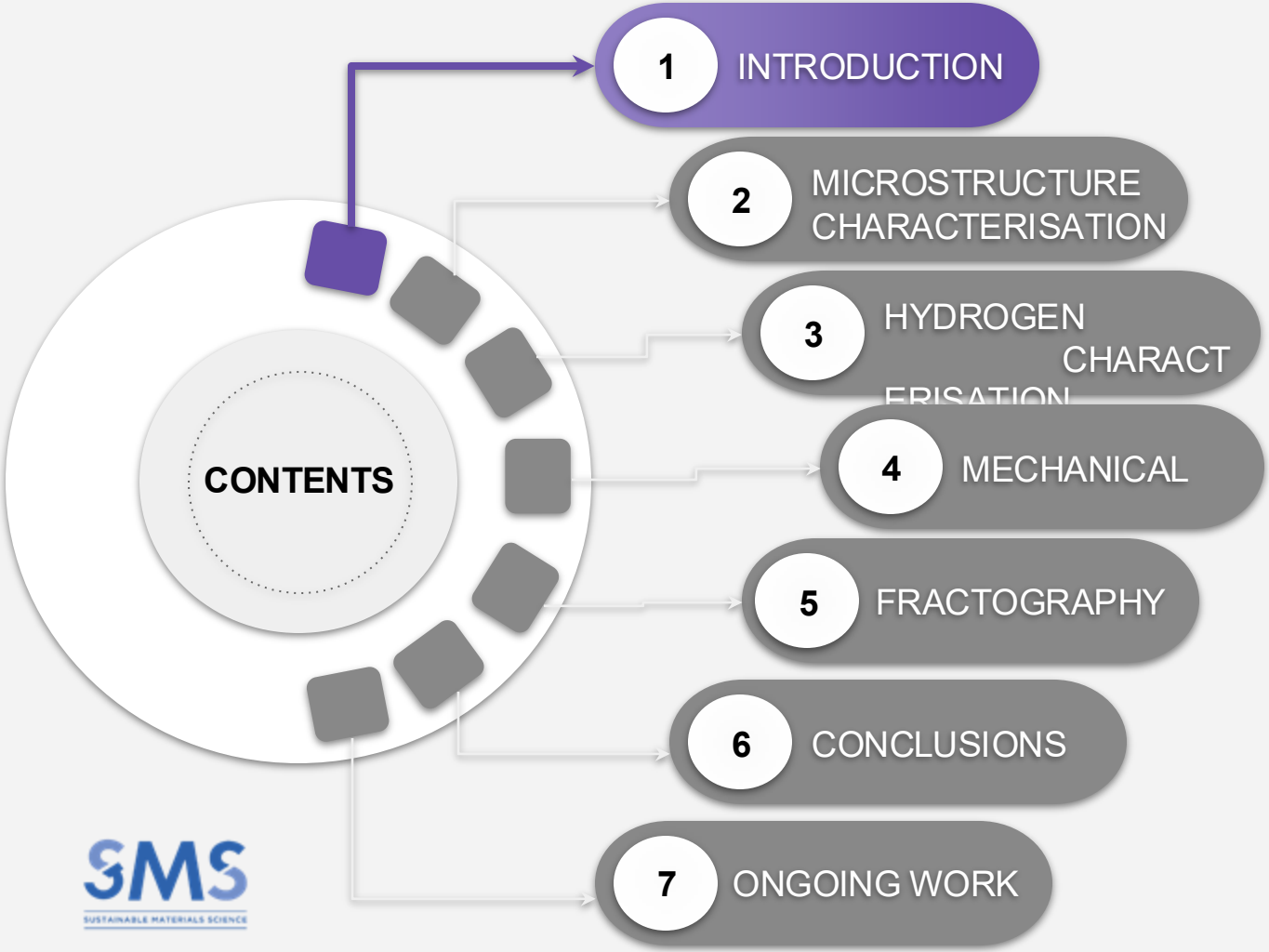

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Energy transition

- ★ European Hydrogen Backbone (EHB) initiative
- ★ Hydrogen infrastructure
- ★ Repurposing pipelines

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Focus area

1 **CHARACTERISATION**
X70 PIPELINE STEELS
WELDS

2 **EXPERIMENTAL**
EX-SITU ELECTROCHEMICAL
QUASI-STATIC TENSILE
TEST

3 **SCREENING METHODOLOGY**

GO/NO-GO PROMPT

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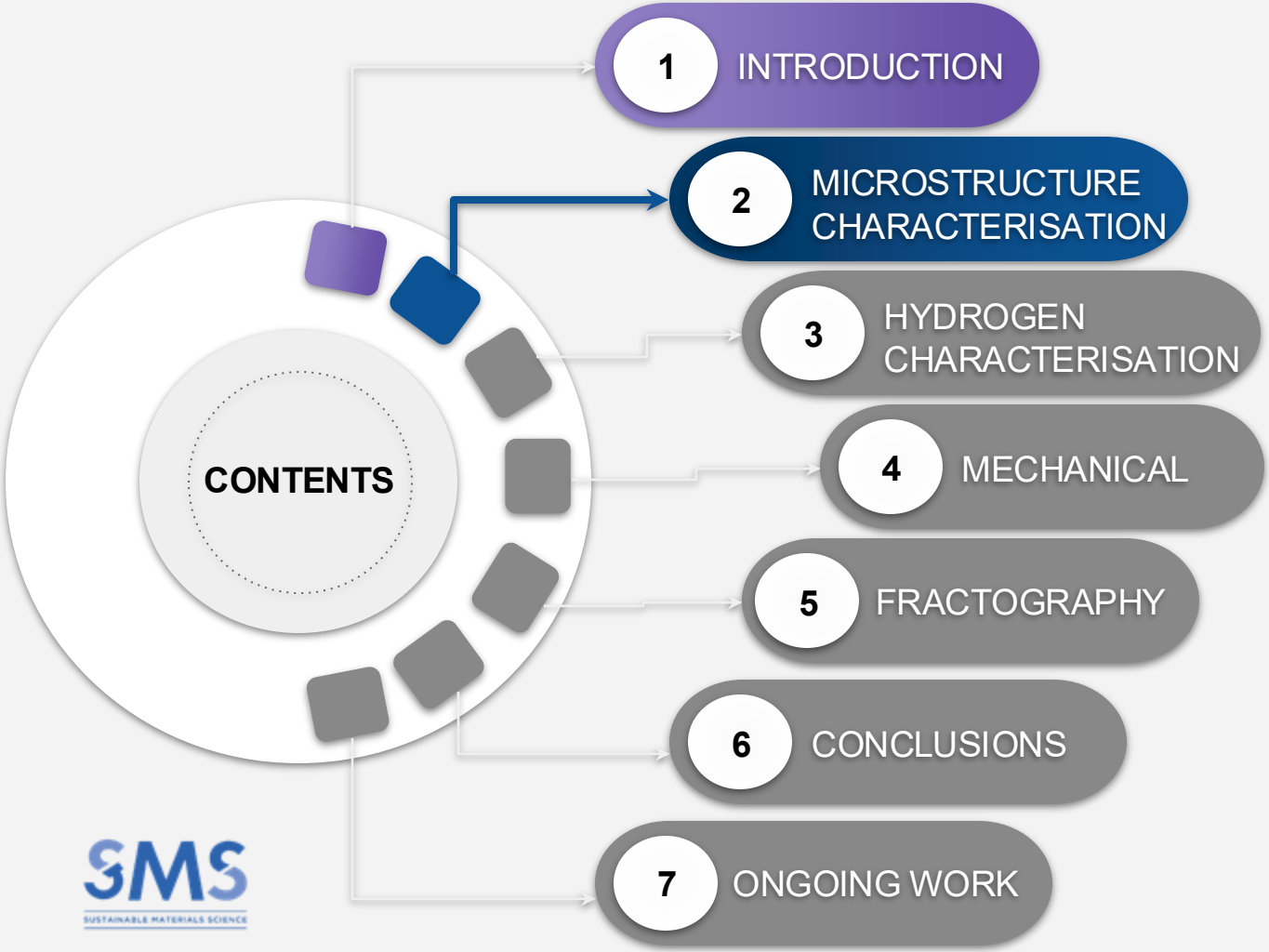

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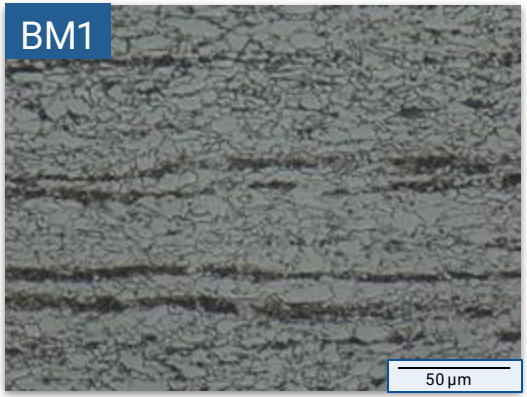
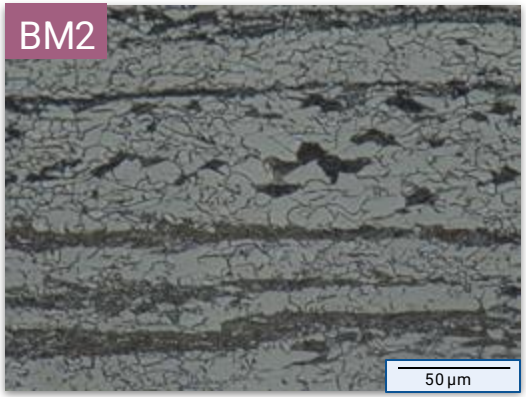
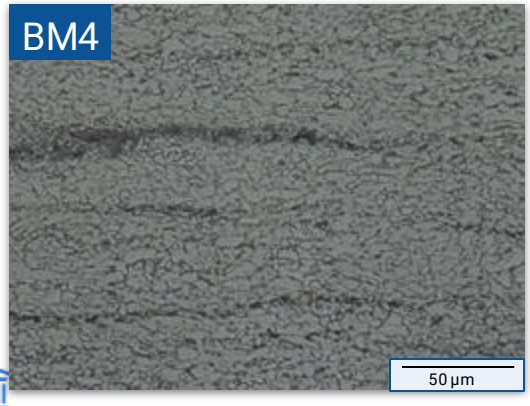
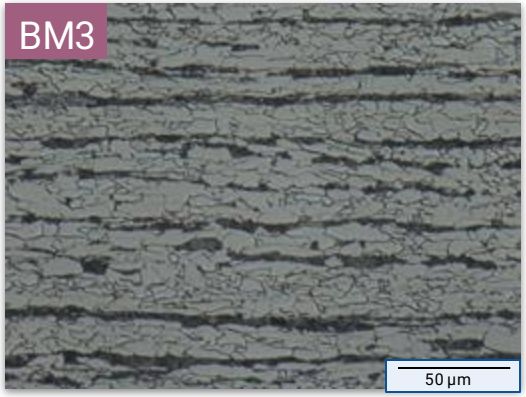
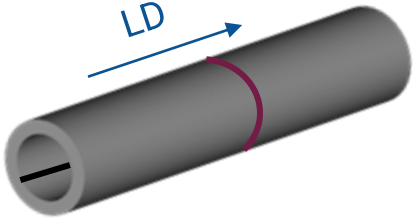
Banded MS – Base Metals

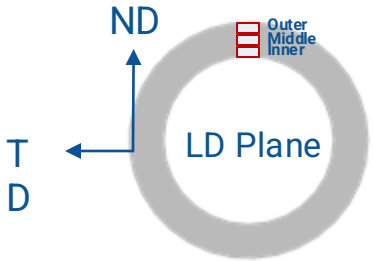


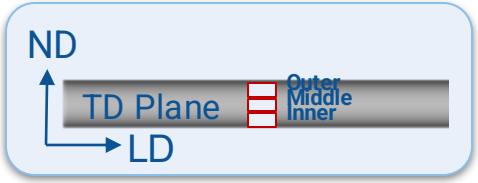












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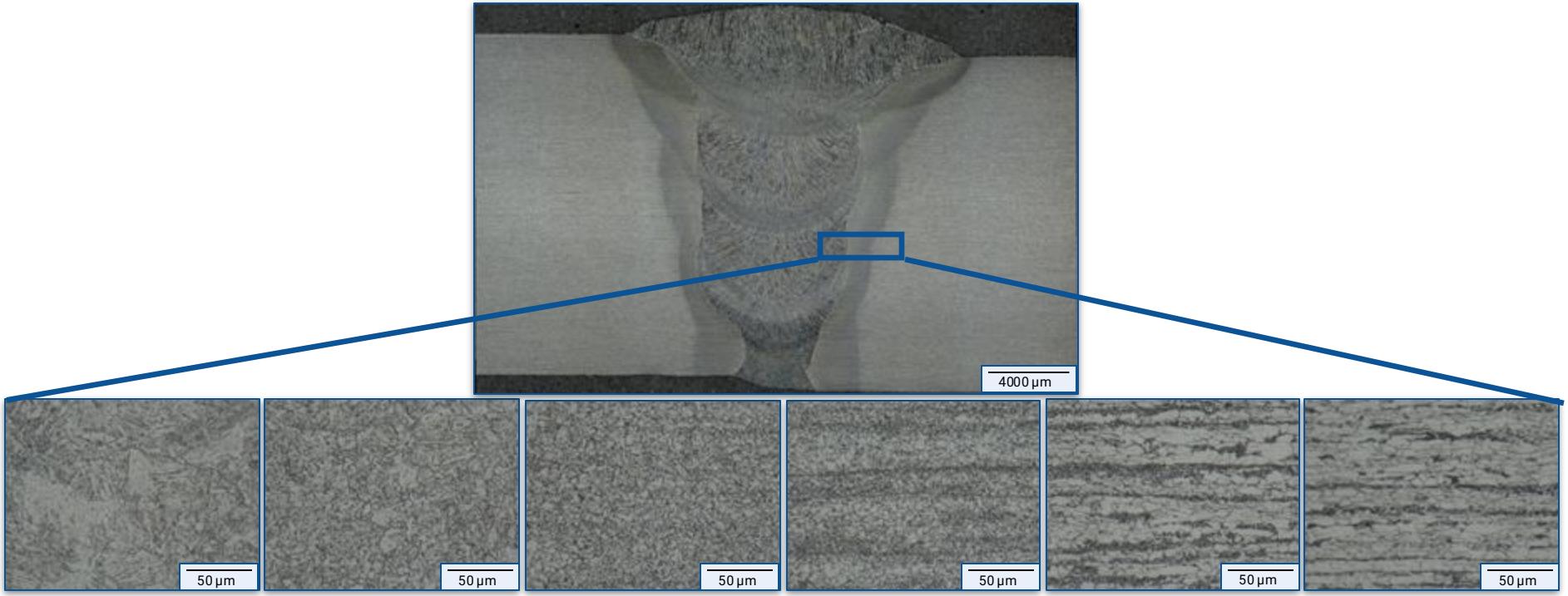
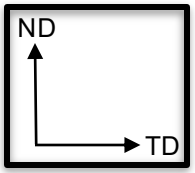
MS Microstructure **BM** Base material

Heterogeneous MS – Girth Weld



Weld metal - Heat affected zone (HAZ) - Base metal



Other microstructural details

- ★ Ferrite grain size rank: BM1 < BM2 also BM4 < BM3
- ★ Pearlite fraction rank: BM1 < BM2 also BM4 < BM3
- ★ Inclusion types : oxides, sulphides, carbides
- ★ Hardness range BM : 205 - 225 HV10
  Hardness range WM : 182 - 241 HV10
- ★ Cold bent no appreciable effect

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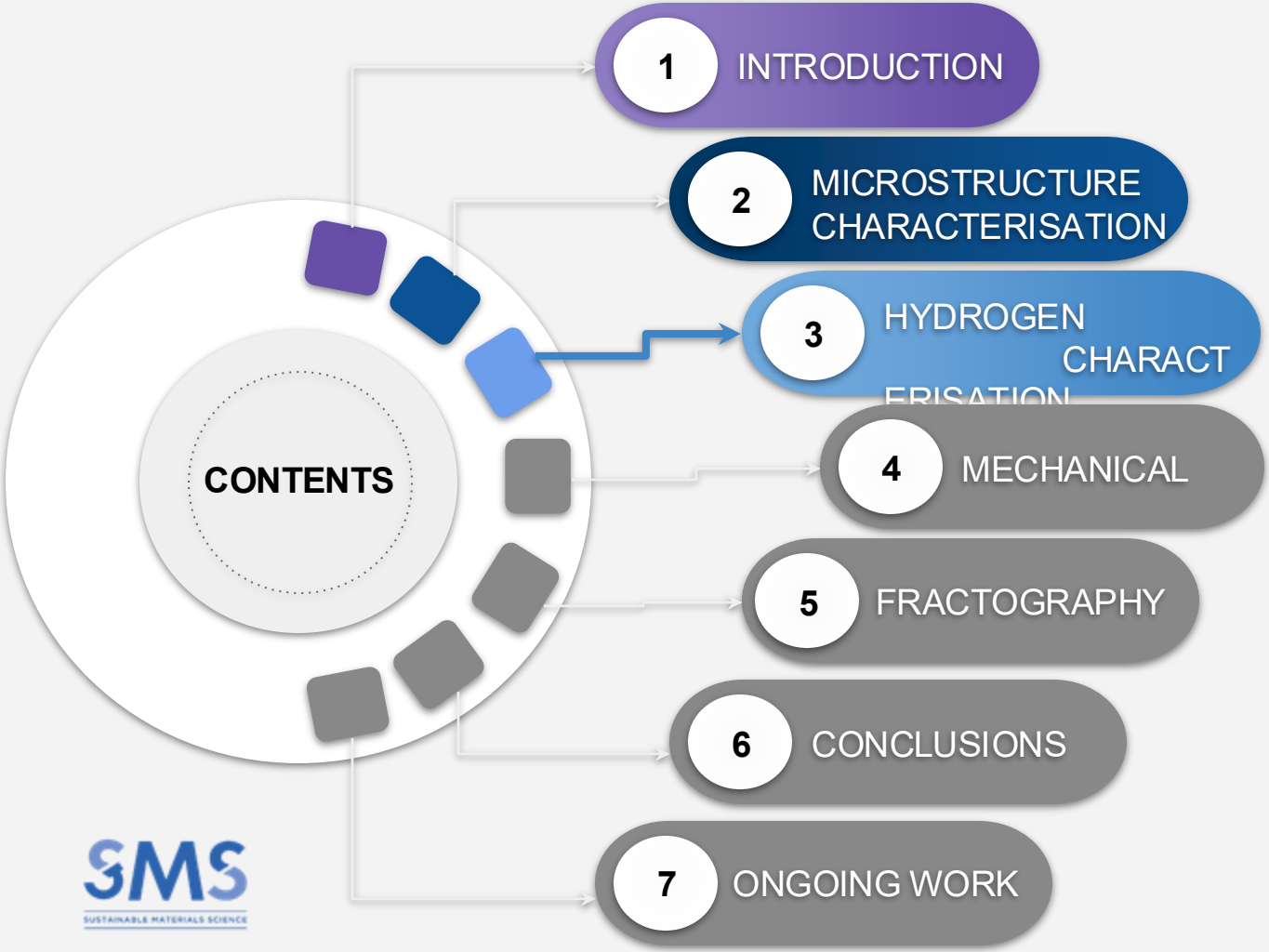
BM Base material **WM** Weld material



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Hydrogen content until saturation

Estimation of hydrogen content until saturation

Material	BM1	BM2	BM3	BM4
H [wppm]	1,19 ± 0,09	1,25 ± 0,15	1,24 ± 0,14	1,63 ± 0,18
Material	LW_WM	LW_HAZ	GW_WM	GW_HAZ
H [wppm]	1,00 ± 0,11	1,41 ± 0,34	0,95 ± 0,14	0,81 ± 0,21

- Charging conditions: 0.8mA /cm^2 in 0.5M H_2SO_4 + 1g/l thiourea
- Sample geometry: 3 x 6 x 8 mm

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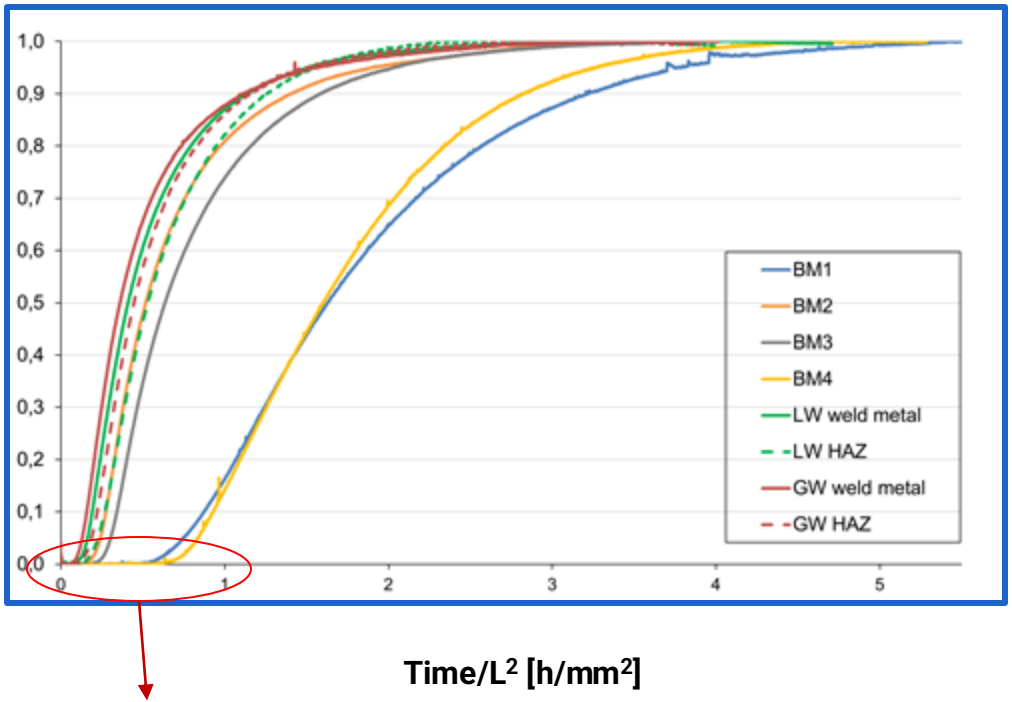
BM Base material
LW_WM Longitudinal weld - weld metal
GW_WM Girth weld - weld metal
LW_HAZ Longitudinal weld - heat affected zone
GW_HAZ Girth weld - heat affected zone

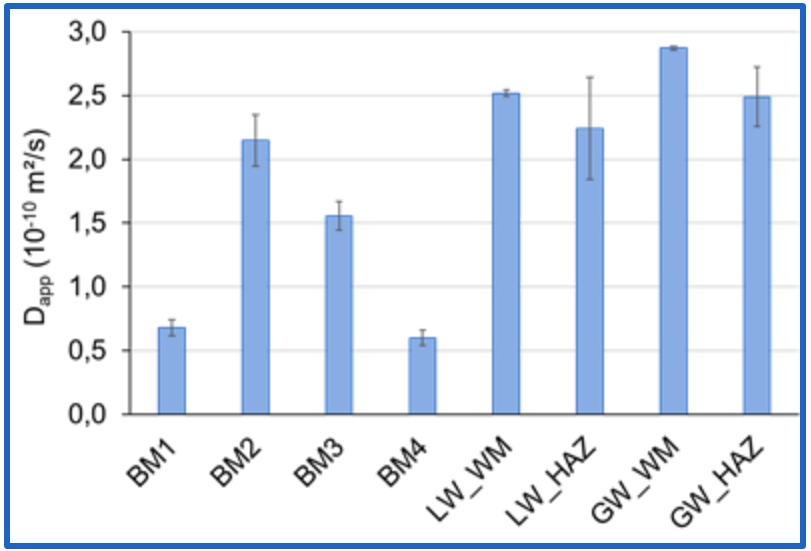
Diffusivity : BM1 = BM4 < BM3 < BM2 < Welds



Breakthrough time

Devanathan-Stachurski permeation method

Hydrogen diffusivity [m²/s]



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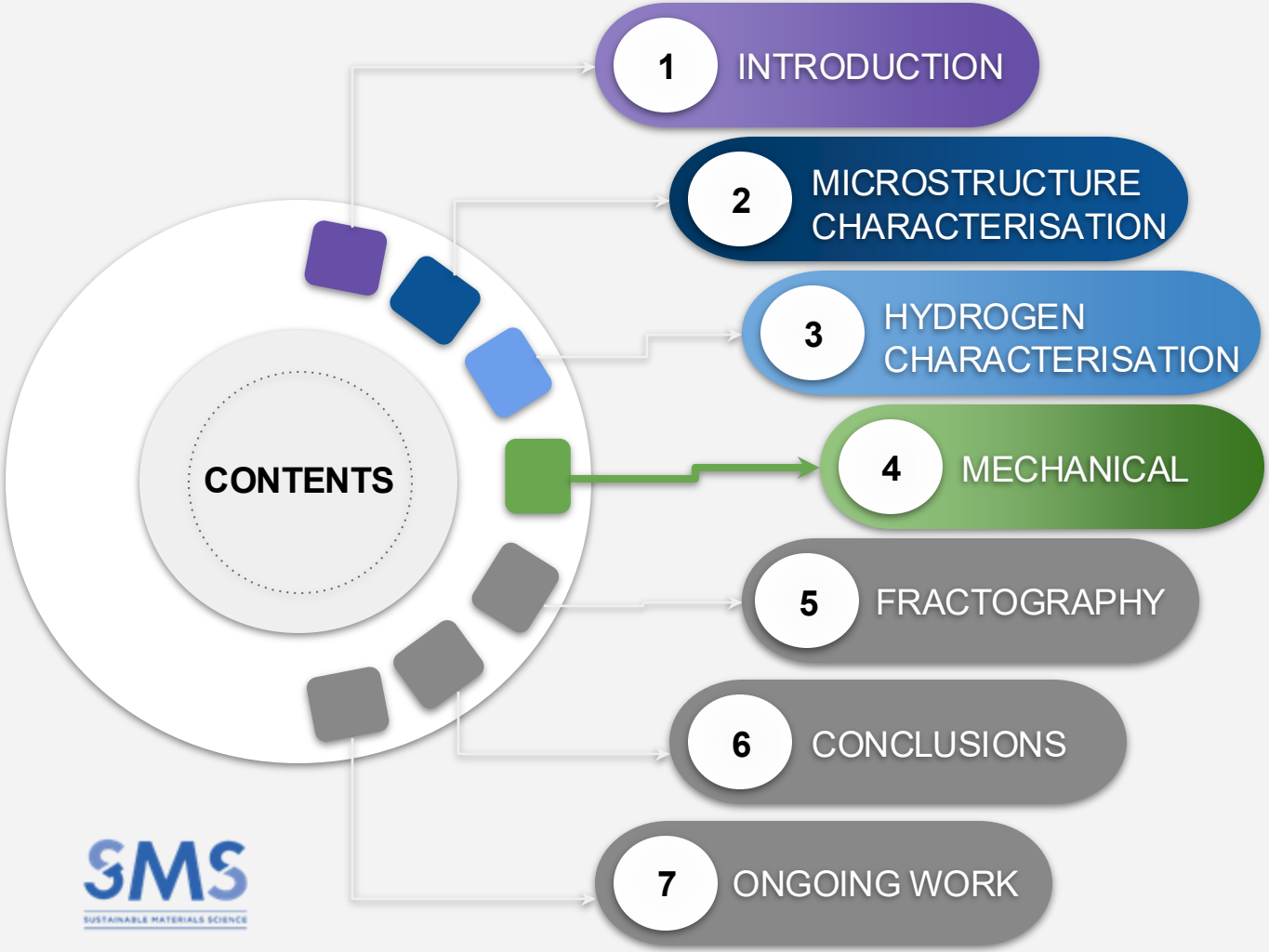

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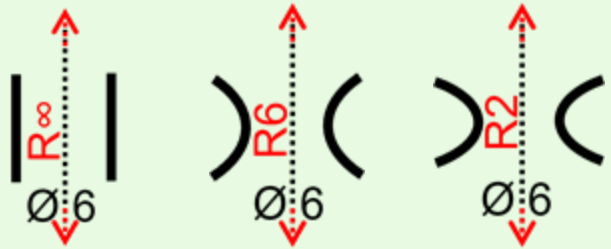
Quasi-static ex-situ tensile test

Different levels of stress triaxiality



Electrochemical hydrogen charging : 18h at 0.8mA/cm^2 in 0.5M H_2SO_4 + 1g/l thiourea

Tensile test : Constant strain rate of 0.00025/s until rupture

Fracture surface

Mechanical characteristics
Hydrogen embrittlement indices
EI = 100%[1-($X_{hydrogen}$/X_{air})]
Force displacement curves

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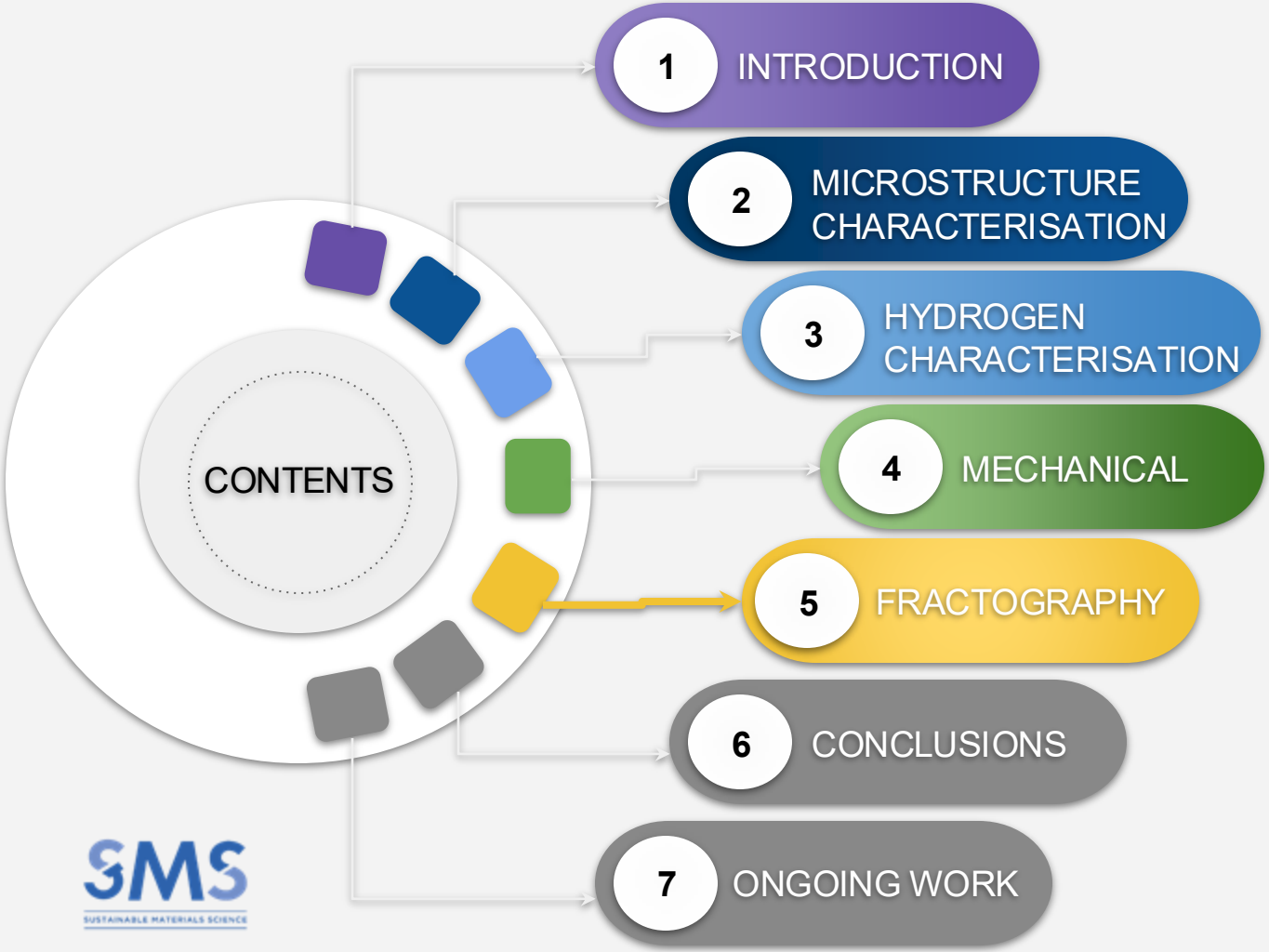

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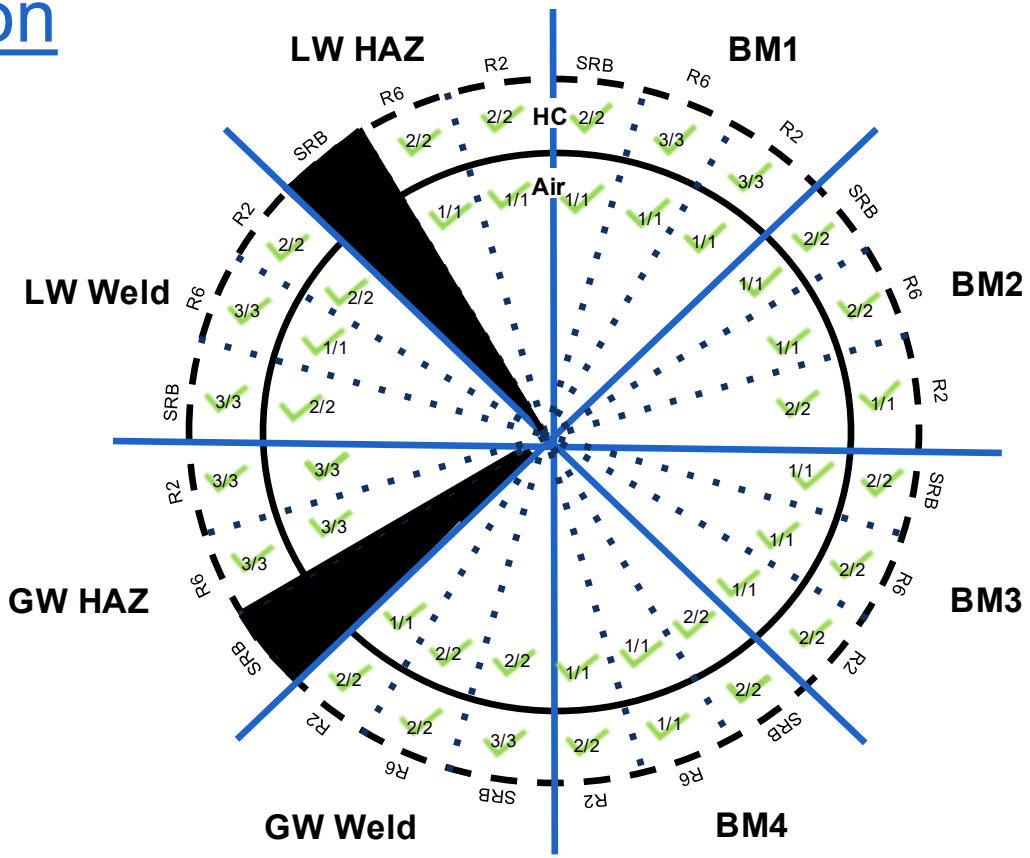
Fractography prioritization

Dart chart

- ➔ 8 sections for materials
- ➔ 3 subsections for stress conditions
- ➔ Inner circle - air tested (reference)
- ➔ Outer circle - hydrogen charged
- ➔ ✓ - number of samples analysed
- ➔ Different features categorized

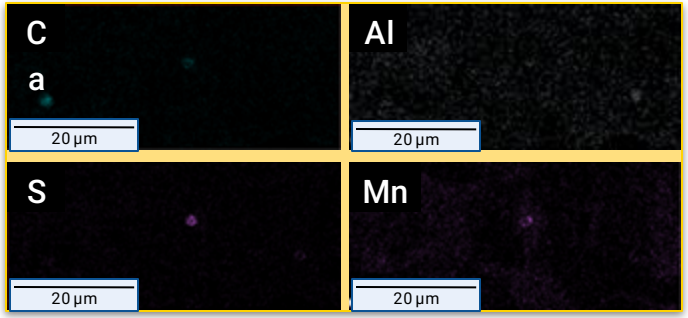


HC Hydrogen charged
SRB Smooth round bar
R6/R2 Radii 6mm/2mm
BM Base material

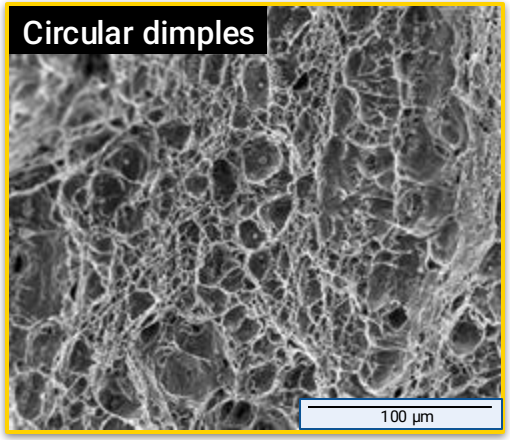
LW_WM Longitudinal weld - weld metal
LW_HAZ Longitudinal weld - heat affected zone
GW_WM Girth weld - weld metal
GW_HAZ Girth weld - heat affected zone

Ductile microvoid coalescence

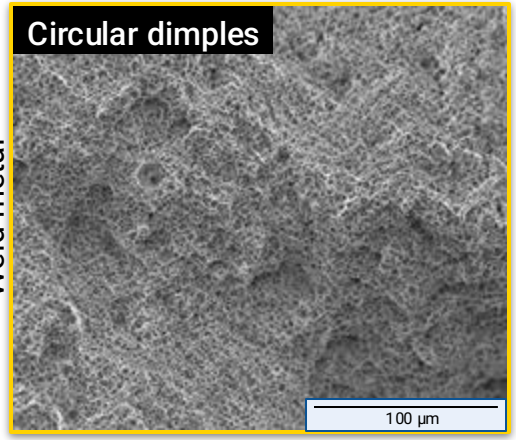
- ➔ Dimples in all materials - air and hydrogen charged
- ➔ Dimple size smaller in weld metal
- ➔ Inclusions as void initiation sites

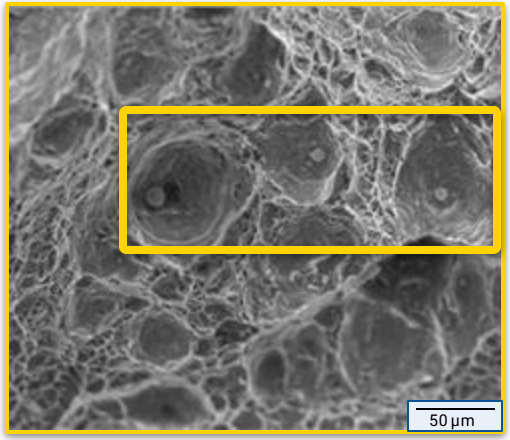


Base material

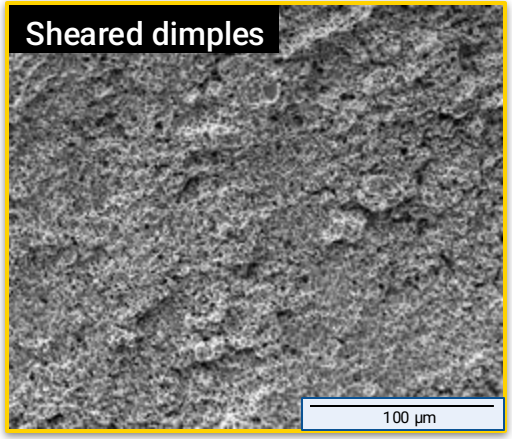


Weld metal

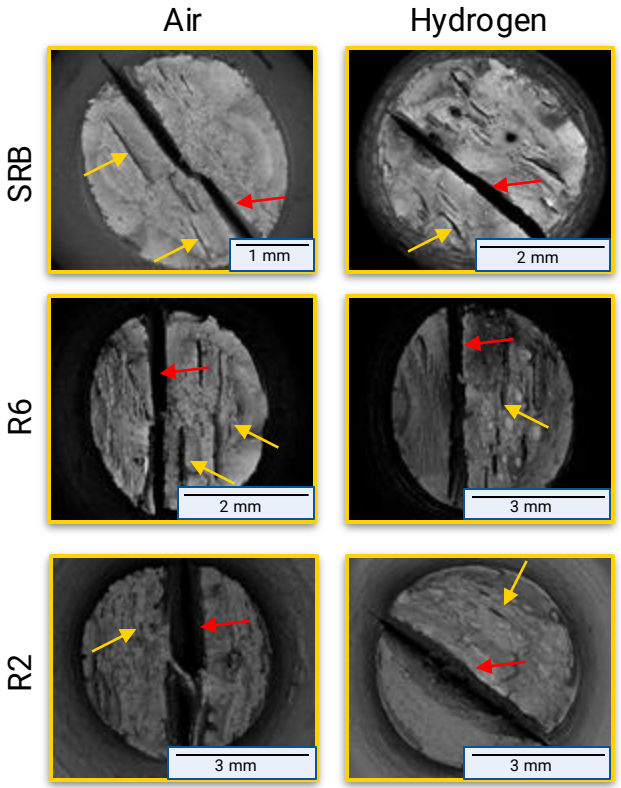




Heat affected zone



Delamination : BM1

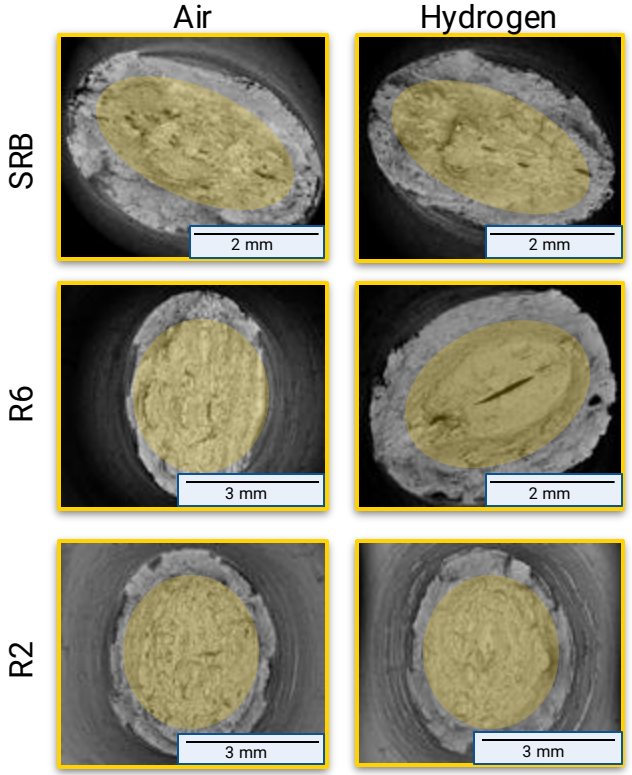


➔ Delamination and Splits/Micro Splits : Separation within a material

➔ Delamination : across the sample

➔ Splits/Micro Splits : distributed in the sample

Micro Splits : BM2



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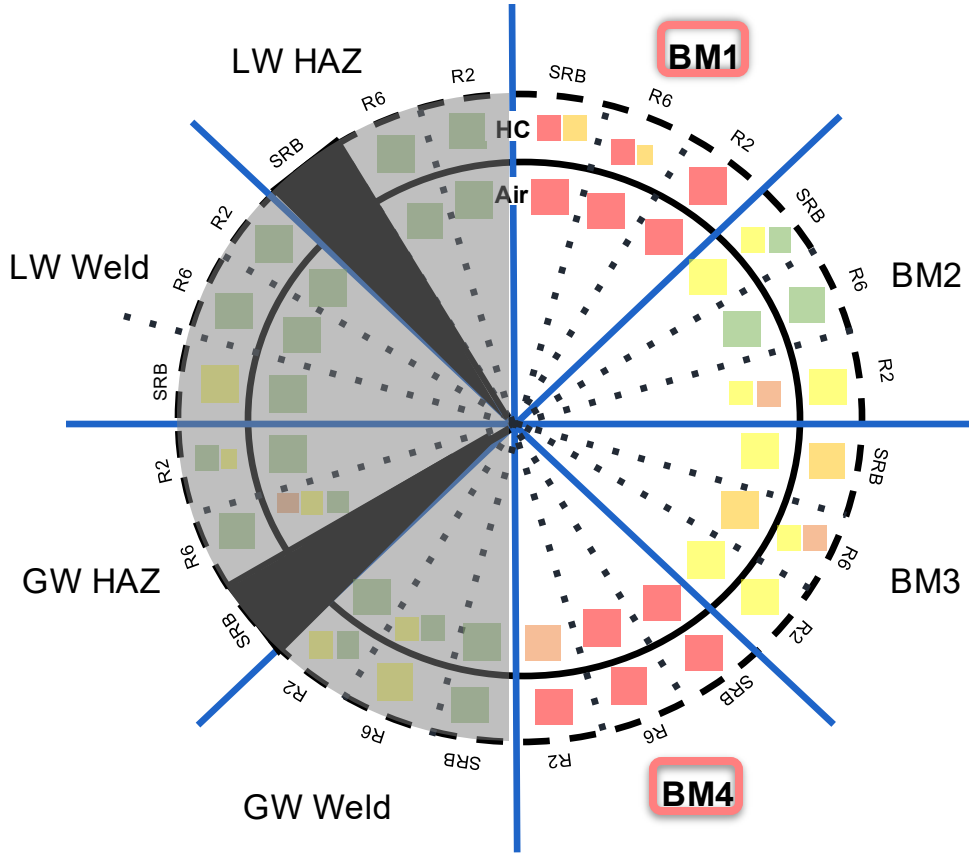
SRB Smooth round bar **R6/R2** Radii 6mm/2mm

Visual Ranking : D&S

Ranking scale

0	no delaminations or splits
1	few splits (<10)
2	many splits (>=10)
3	delaminations
4	delaminations and splits

➔ Air/Hydrogen : BM1 & BM4
BM2 & BM3
Welds

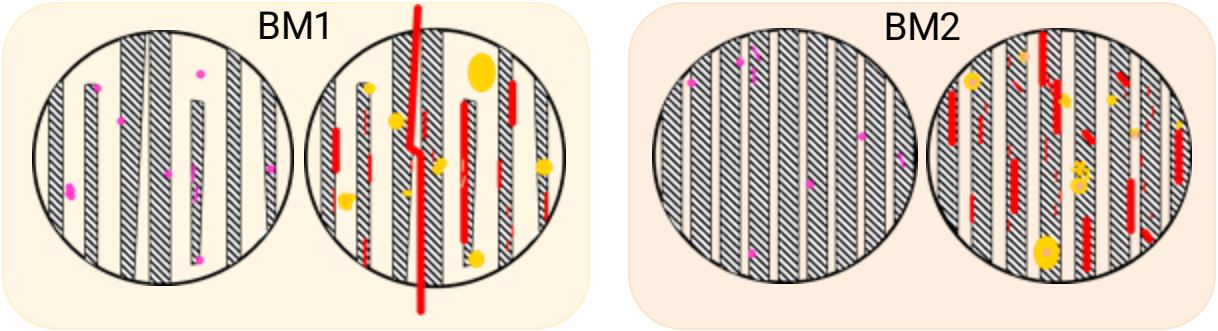


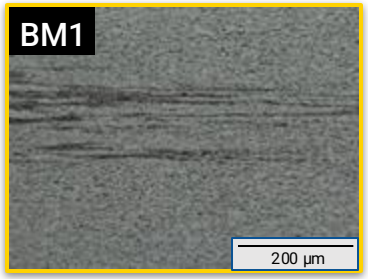
HC Hydrogen charged
SRB Smooth round bar
R6/R2 Radii 6mm/2mm
BM Base material

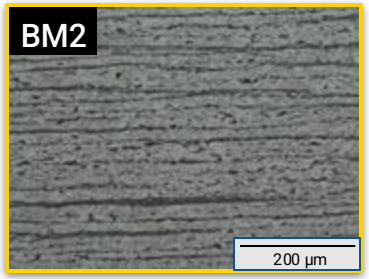
LW_WM Longitudinal weld - weld metal
LW_HAZ Longitudinal weld - heat affected zone
GW_WM Girth weld - weld metal
GW_HAZ Girth weld - heat affected zone

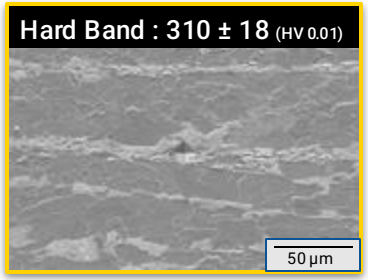
D&S Delamination & (micro)splits

Hard bands lead to delaminations & splits









- ➔ BM1 : dispersed bands ; BM2 : uniformly distributed bands
- ➔ Deformation gradient
- ➔ Stress relaxation
- ➔ Other contributing factors : inclusions

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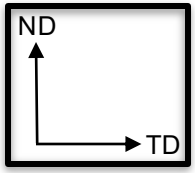
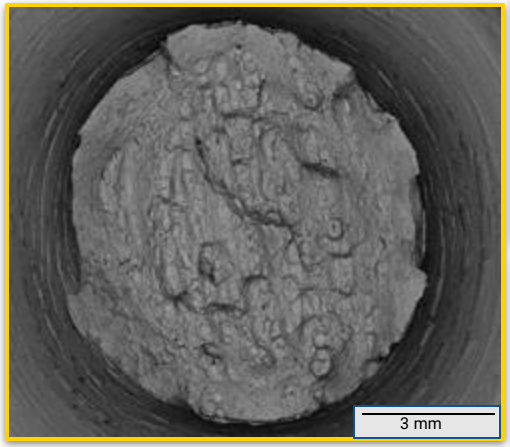
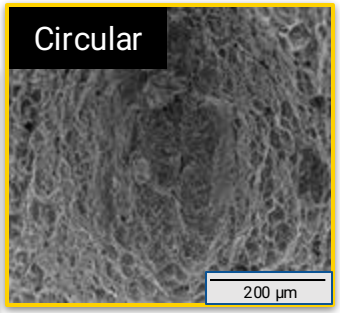
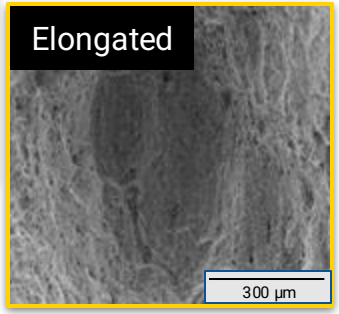
Hard band | Ferrite | Inclusion | Delamination / Splits | Fisheye



Fisheye shape influenced by anisotropy

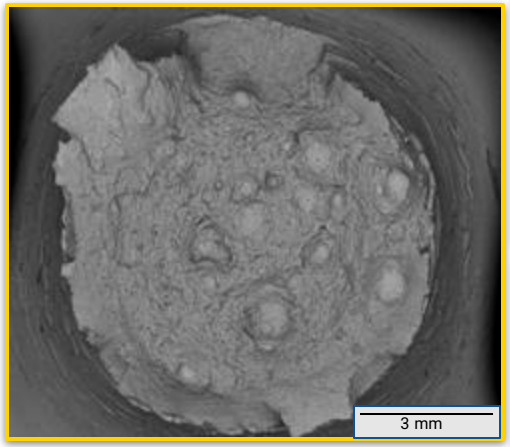


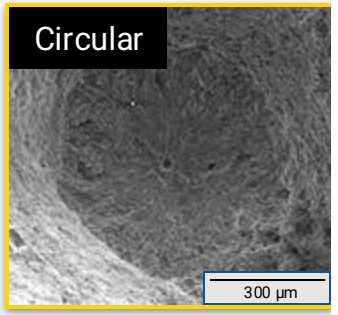


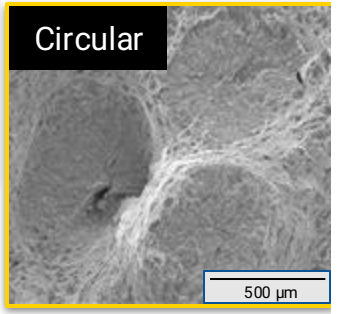


Base metal

- circular + elongated







Weld metal

- mainly circular

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Visual Ranking : Fisheyes & QC areas

Ranking scale

0 no QC areas

1 few small QC areas (<5)

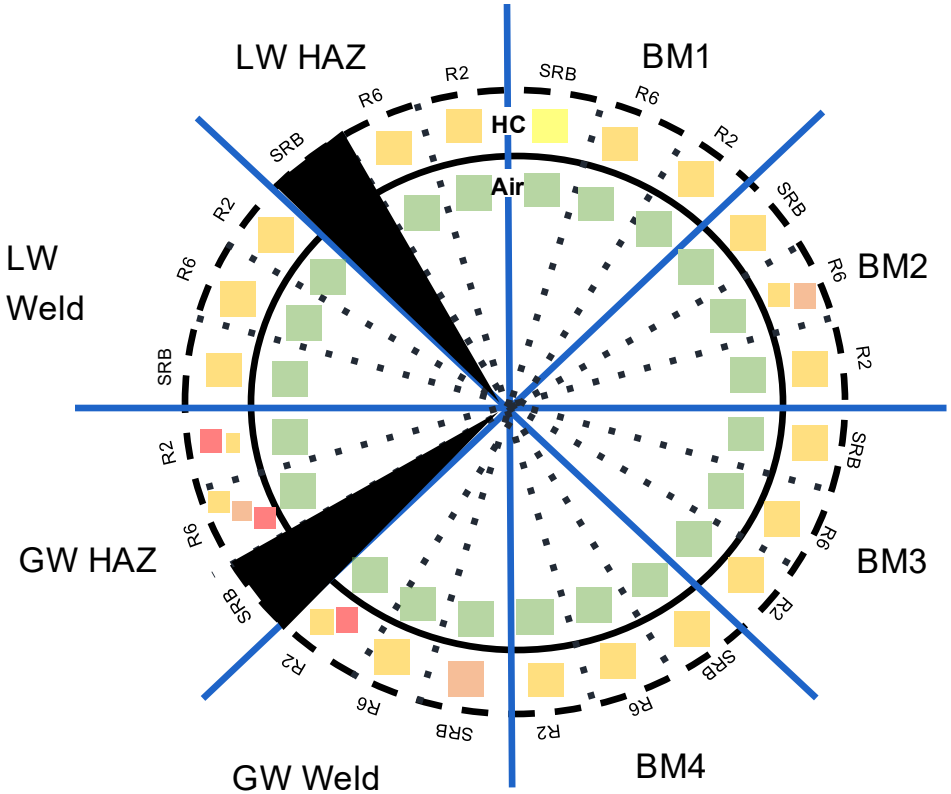
2 many small QC areas (>=5)

3 1 big QC area (diam. > +- 1mm) + small QC areas

4 >1 big QC area + many small QC areas

➔ Air : no fisheyes/ QC areas

➔ Hydrogen : common for all



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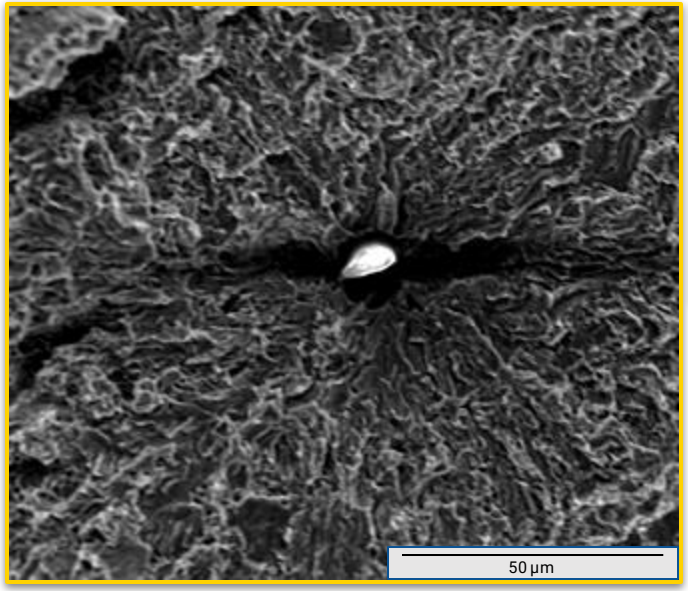
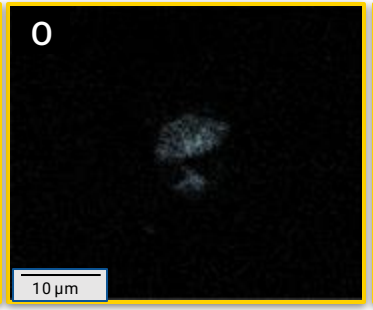
HC Hydrogen charged
SRB Smooth round bar
R6/R2 Radii 6mm/2mm
BM Base material

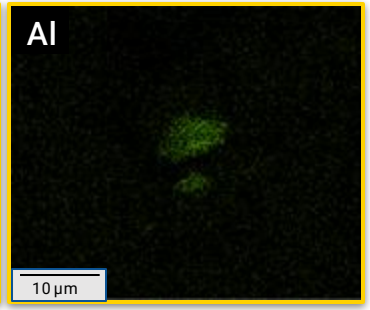
LW_WM Longitudinal weld - weld metal
LW_HAZ Longitudinal weld - heat affected zone
GW_WM Girth weld - weld metal
GW_HAZ Girth weld - heat affected zone

QC Quasi-cleavage

Inclusions cause fisheyes in H charged samples

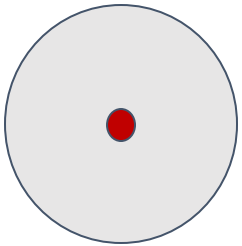






➔ Oxides, carbides, sulphides inclusions distributed in the matrix

➔ Fisheye : Pupil
Iris



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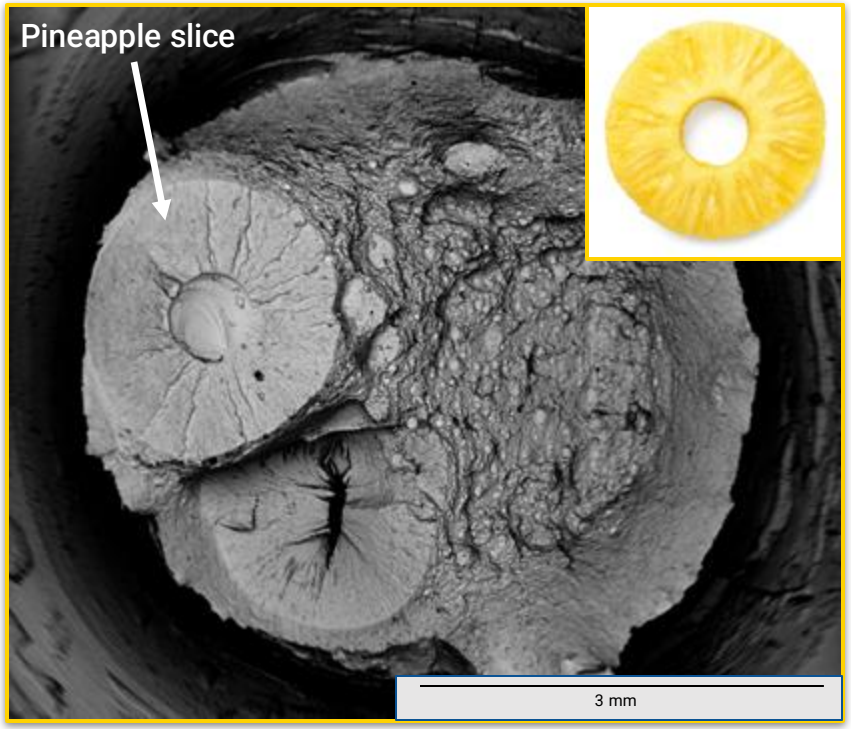
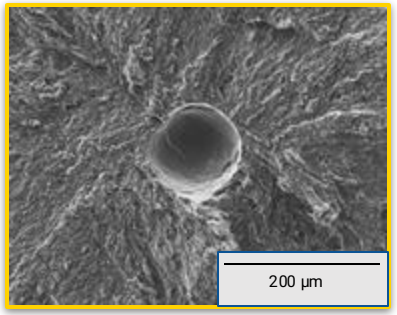
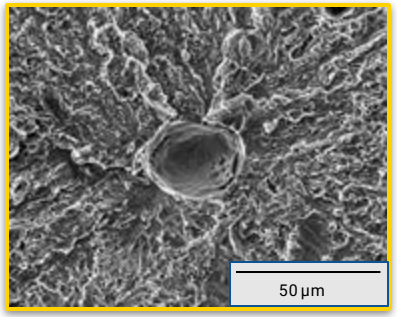
Gas pores form pineapple slice in HAZ

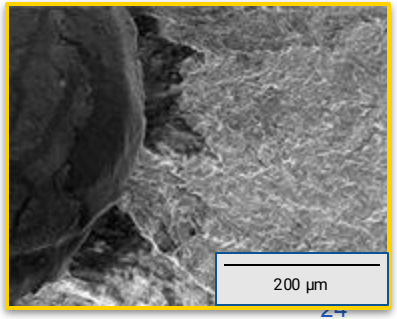
- ➔ Weld flaw on fusion line
- ➔ Bigger initiation spots like gas pores
- ➔ Similar morphology as fisheye
- ➔ Pineapple slice
  = Weld flaw + quasi-cleavage fracture



Girth weld heat affected zone (stress triaxiality radius 2mm)







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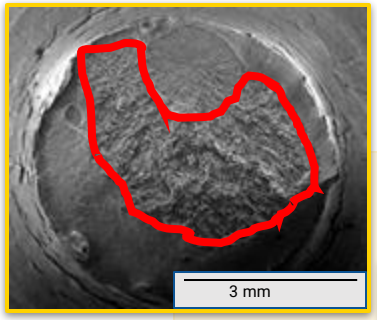
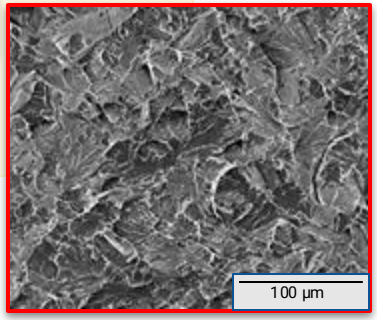
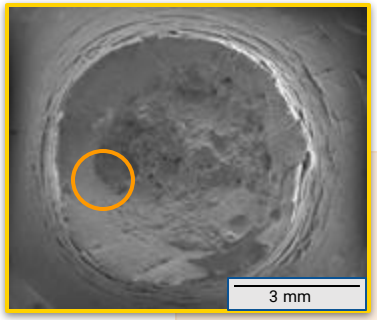
HAZ : Heat affected zone

Brittle faceted fracture in welds

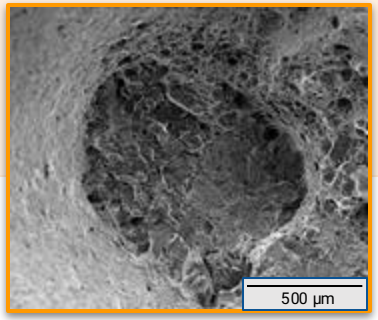


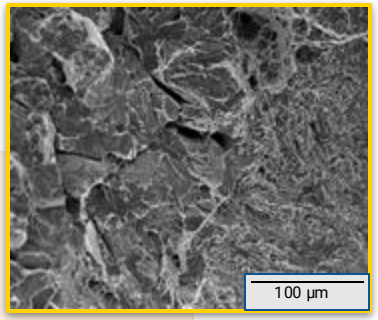
Girth weld





Longitudinal weld





➔ Transgranular/faceted region on fracture surface
➔ Change in fracture mode

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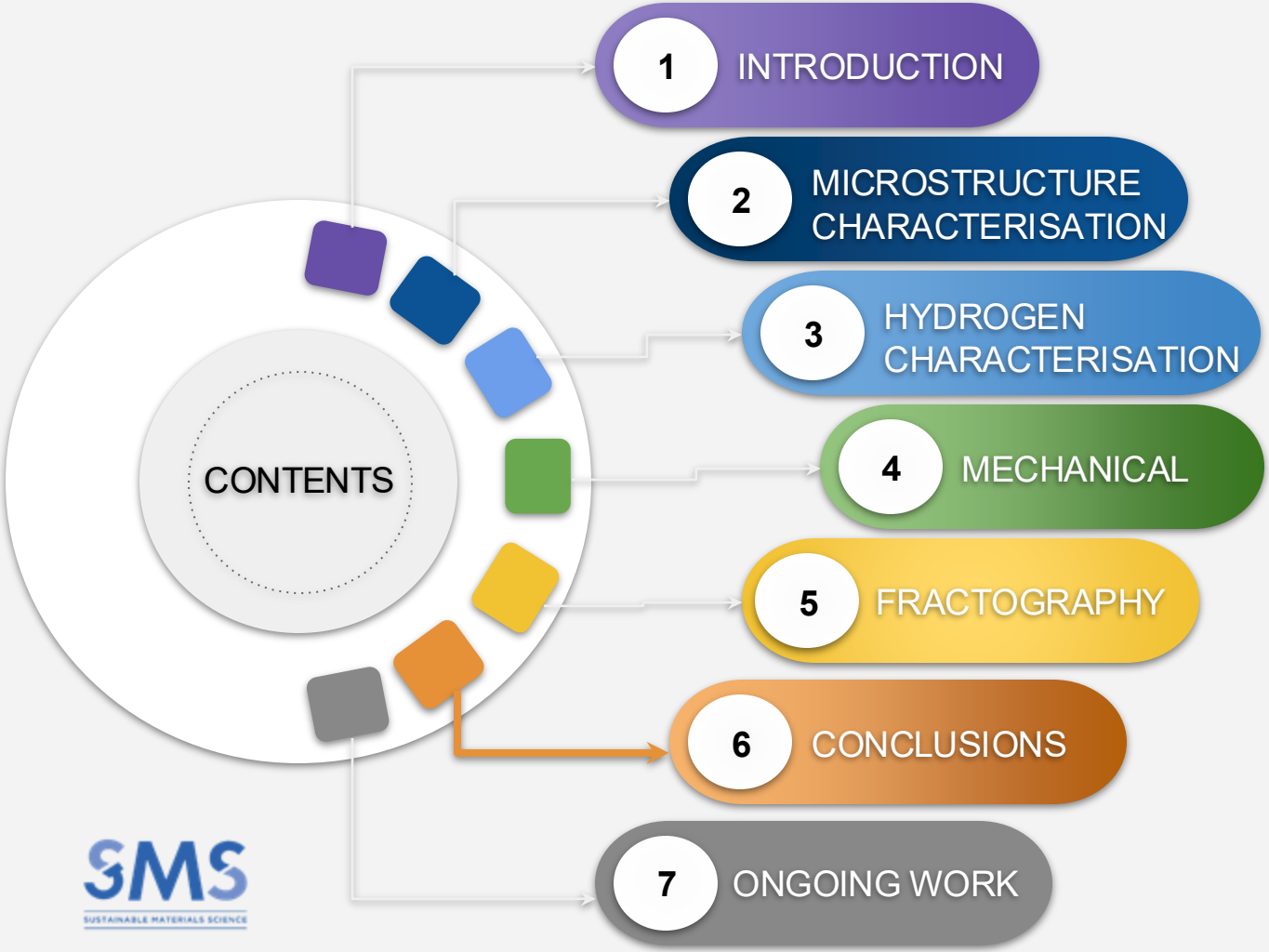

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Learnings from fractography

- ➔ Heterogeneous materials
- ➔ Prominent features : dimples, delamination, splits, fisheyes, quasi-cleavage regions, pineapple slices, faceted areas
- ➔ Sensitive spots : hard bands, inclusions and ferrite - pearlite interfaces, weld flaws

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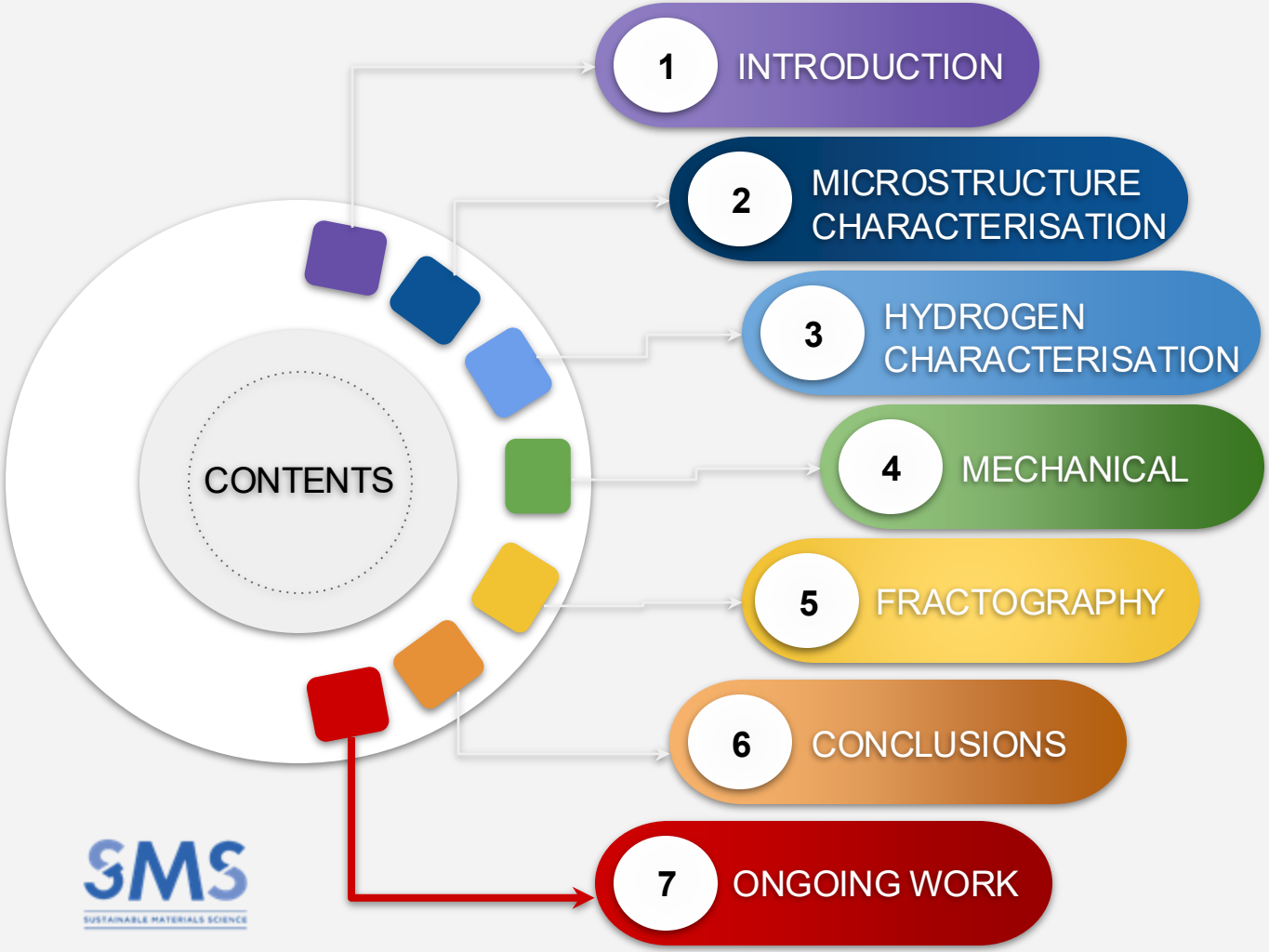

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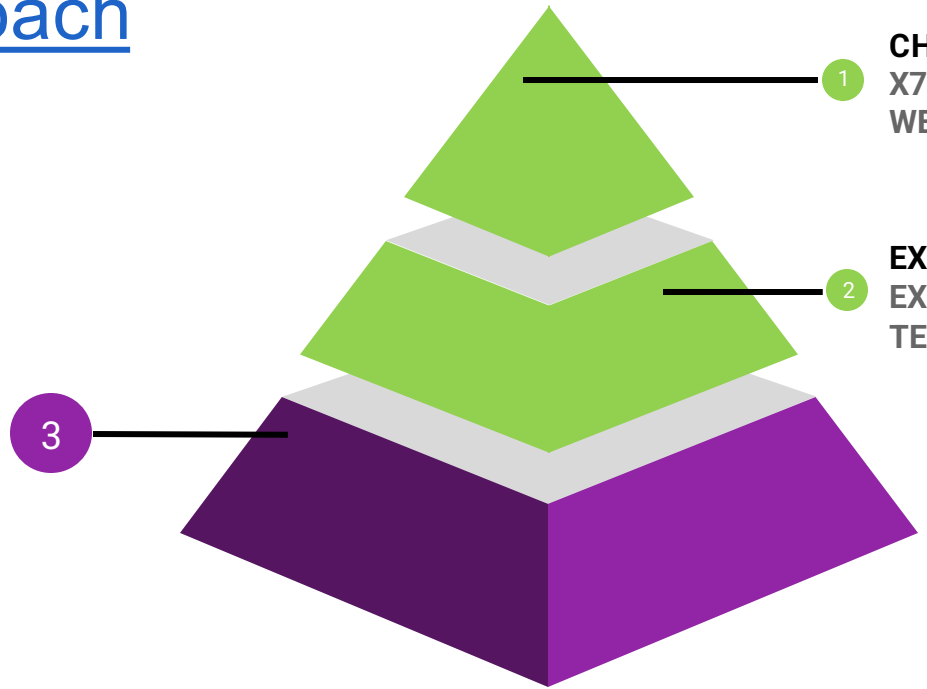
Statistical approach



CHARACTERISATION
X70 PIPELINE STEELS
WELDS

EXPERIMENTAL
EX-SITU ELECTROCHEMICAL TEST

SCREENING METHODOLOGY

MECHANICAL DATA
MICROSTRUCTURE DATA

CORRELATION ANALYSIS

RANK MATERIALS BY PARAMETER-DRIVEN PERFORMANCE

PARAMETERS

Structure: ferrite grain size, pearlite fraction
Properties: diffusivity, hardness, hydrogen content
Performance: hydrogen embrittlement indices

FACULTY OF ENGINEERING AND ARCHITECTURE

Thank you for your attention!

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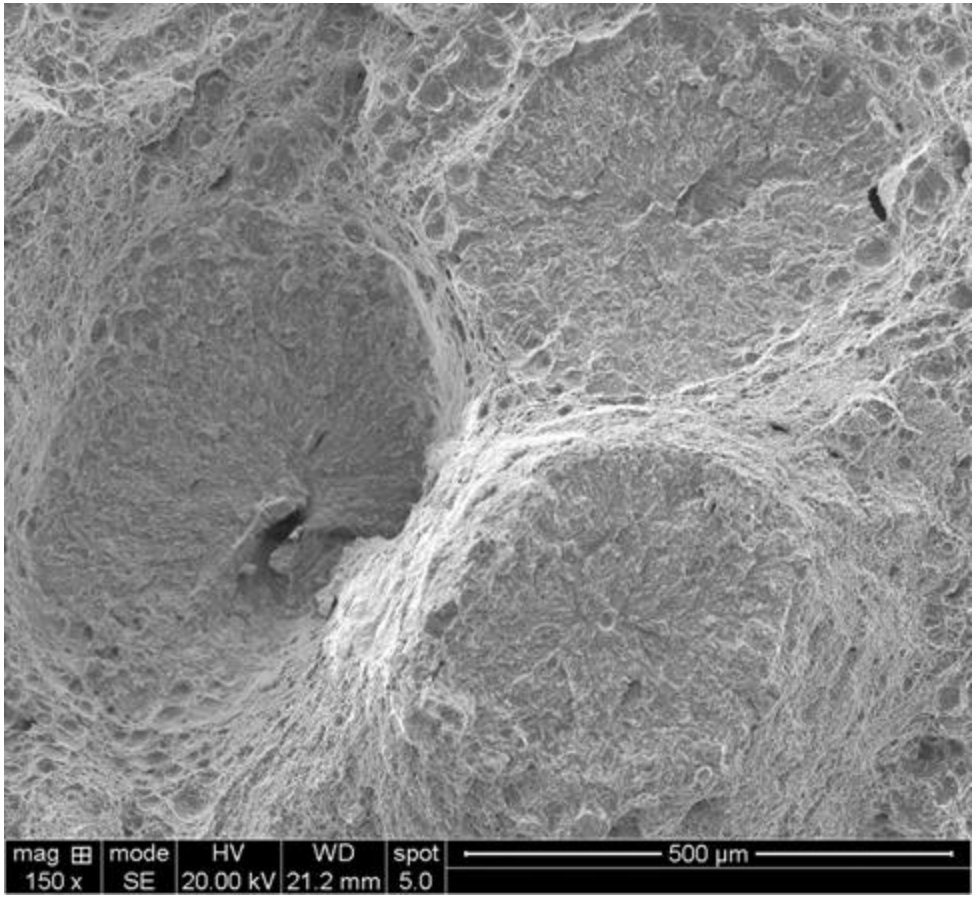
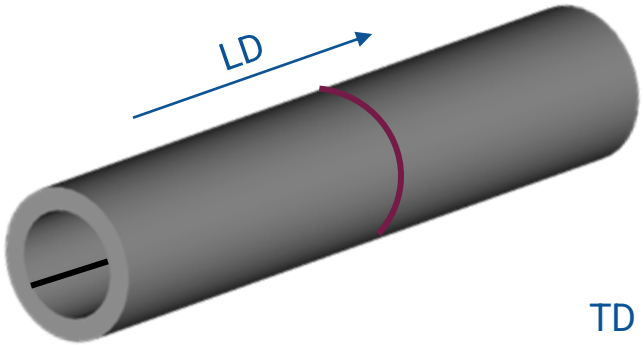
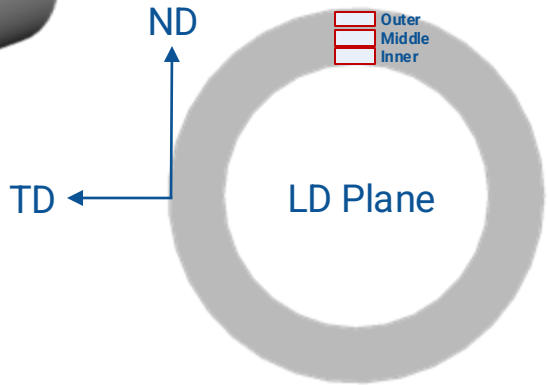
EUROCORR
The European Corrosion Congress

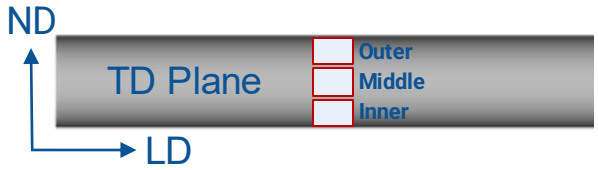
Miscellaneous



Orientation







LD : Longitudinal Direction
Rolling direction

TD : Transverse Direction
Diameter

ND : Normal Direction
Thickness

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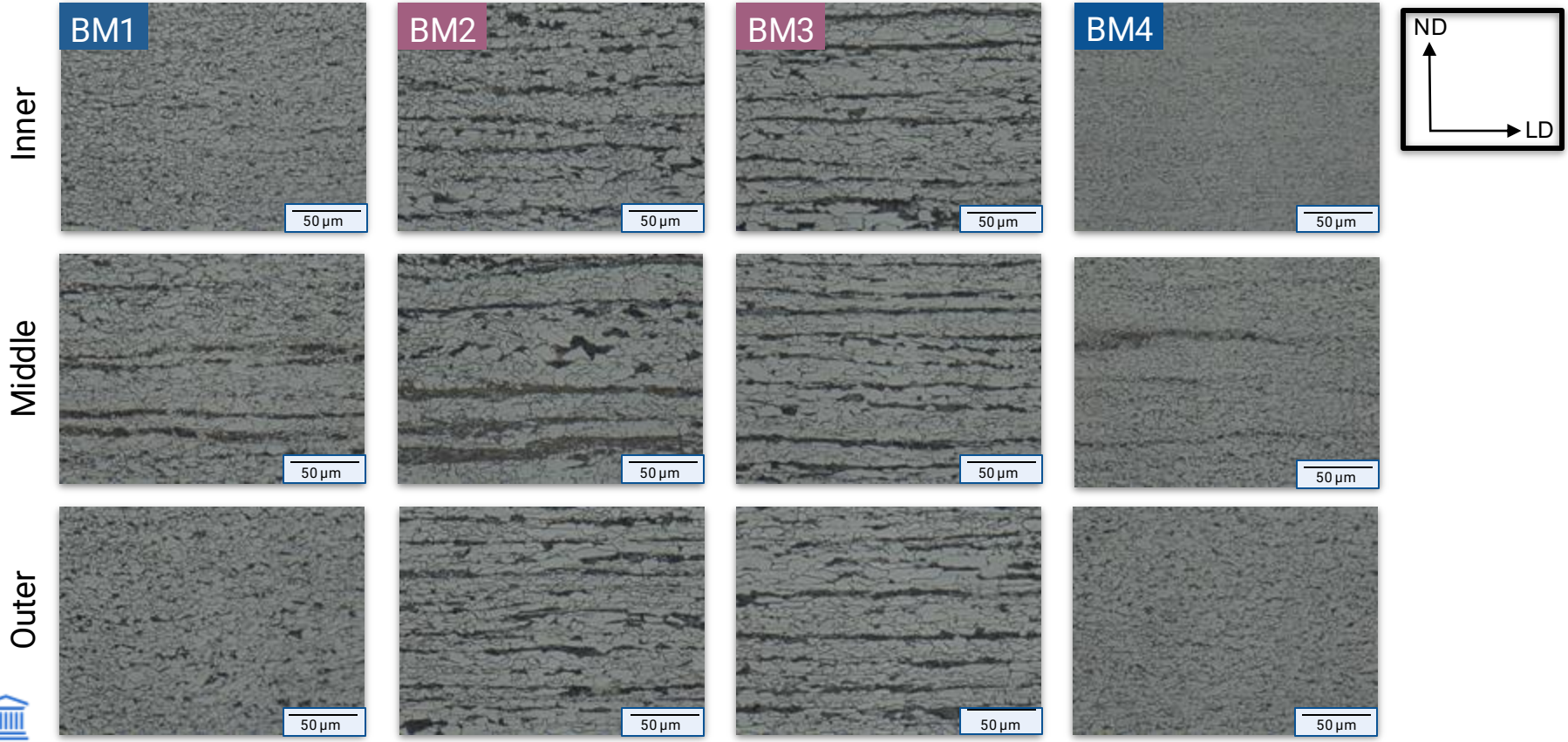
Banded MS – Base Metals



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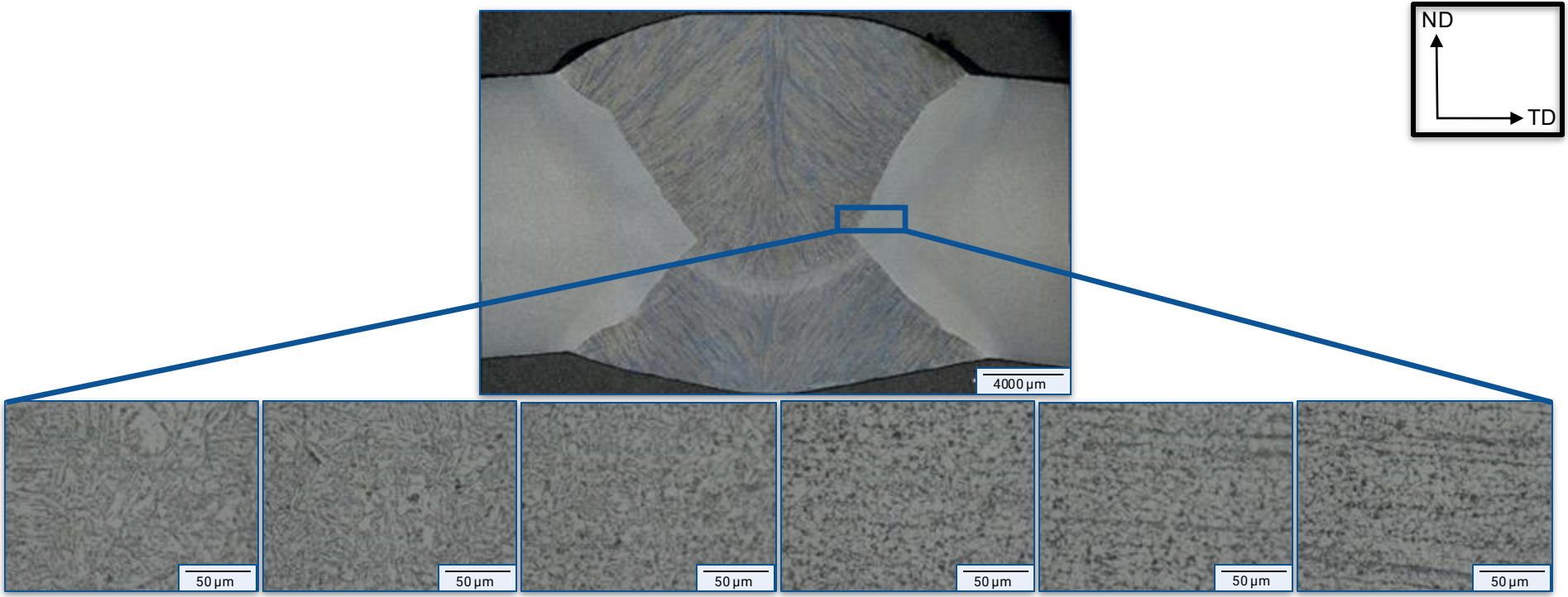
MS Microstructure **BM** Base material

Heterogeneous MS – Longitudinal Weld



Other microstructural details

Ferrite grain size (µm)	BM1	BM2	BM3	BM4
Inner	4,3 ± 0,3	7,1 ± 0,9	5,9 ± 0,3	4,6 ± 0,1
Middle	5,5 ± 0,0	8,3 ± 1,9	7,2 ± 0,3	5,5 ± 0,3
Outer	5,1 ± 0,4	6,2 ± 0,0	5,9 ± 0,3	4,7 ± 0,2

*Pearlite fraction (%)	BM1	BM2	BM3	BM4
Inner	7,3 ± 0,3	20,2 ± 3,5	18,9 ± 0,8	6,5 ± 1,5
Middle	13,3 ± 4,2	22,9 ± 1,7	22,3 ± 4,7	10,4 ± 1,3
Outer	11,8 ± 1,9	20,0 ± 1,0	19,6 ± 1,7	8,1 ± 0,6

*Pearlite fraction (%) can also contain M/A constituents

- ★ Grain size rank:

BM1 < BM2 also BM4 < BM3

- ★ Pearlite fraction rank

BM1 < BM2 also BM4 < BM3

- ★ Inclusion types : oxides, sulphides, carbides
- ★ Hardness range BM :
  Hardness range WM :
- ★ Cold bent no appreciable effect

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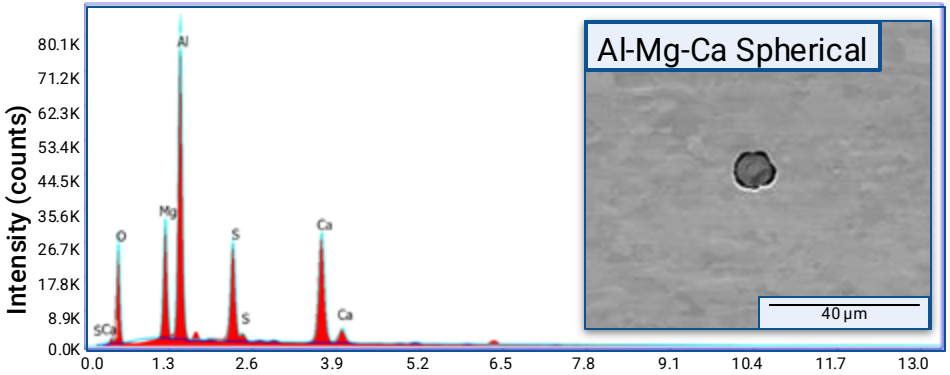
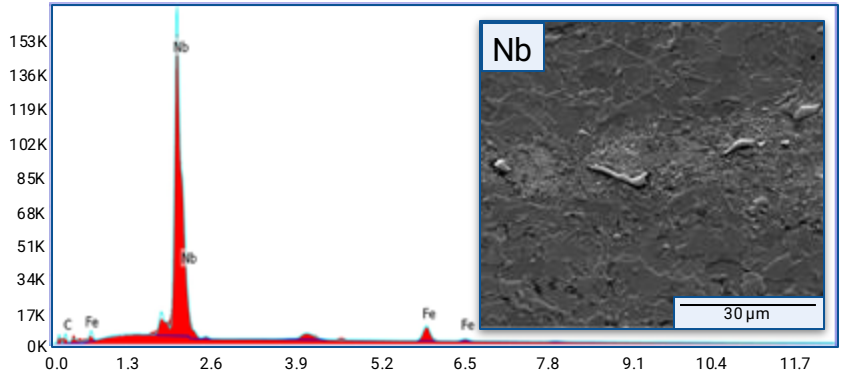
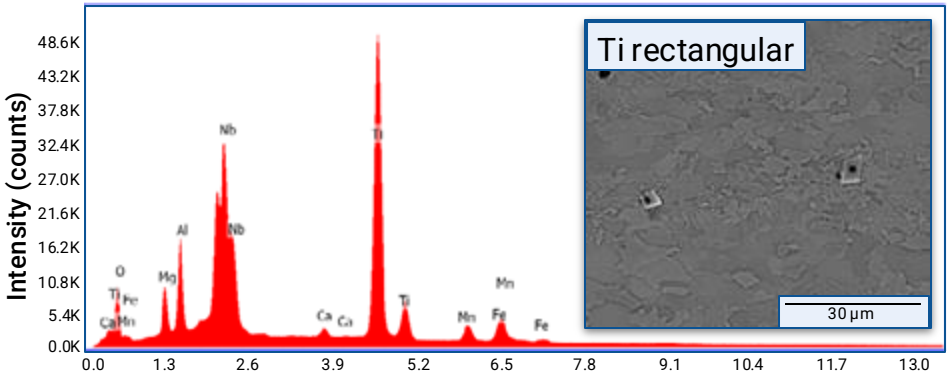
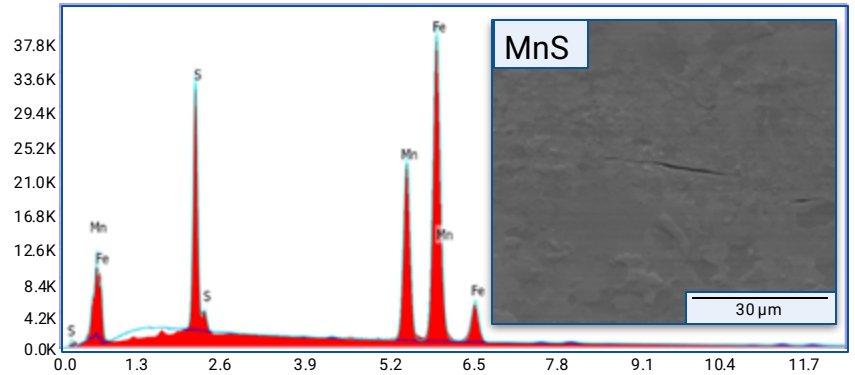
BM Base material **WM** Weld material

Inclusions – Types in Base Metals









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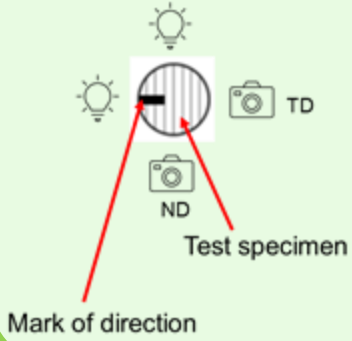
Quasi-static ex-situ tensile test

Different levels of stress triaxiality

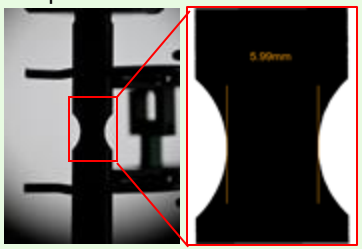
Electrochemical hydrogen charging
: 18h at 0.8mA/cm^2 in 0.5M H_2SO_4
+ 1g/l thiourea

Tensile test : Constant strain rate of 0.00025/s until rupture



Elongation

Optical Diameter Reduction



Fracture surface

Mechanical characteristics
Hydrogen embrittlement indices
EI = 100%[1-($X_{hydrogen}$/X_{air})]
Force displacement curves

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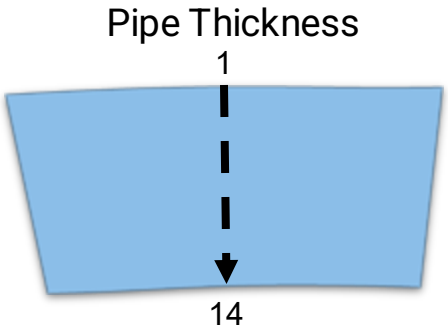
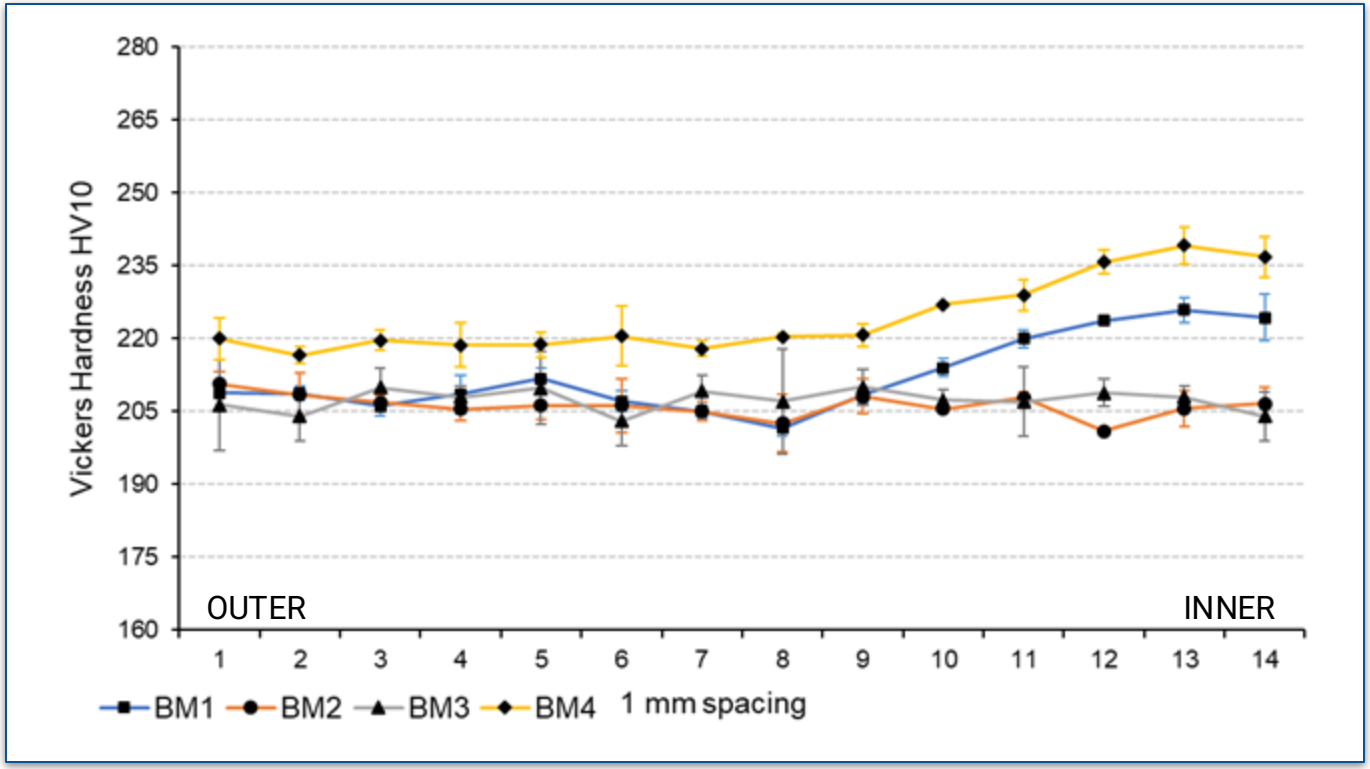
Hardness - slight variation in Base Metals

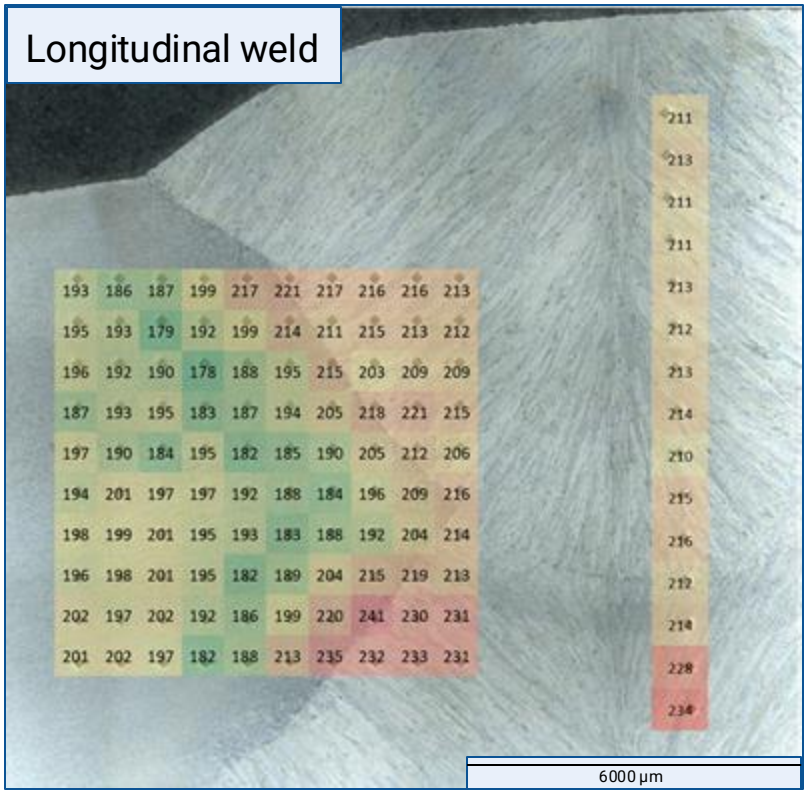




*Vickers Hardness 10 Kgf

Hardness map - Welds

Longitudinal weld



Girth weld

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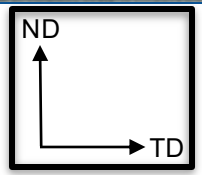
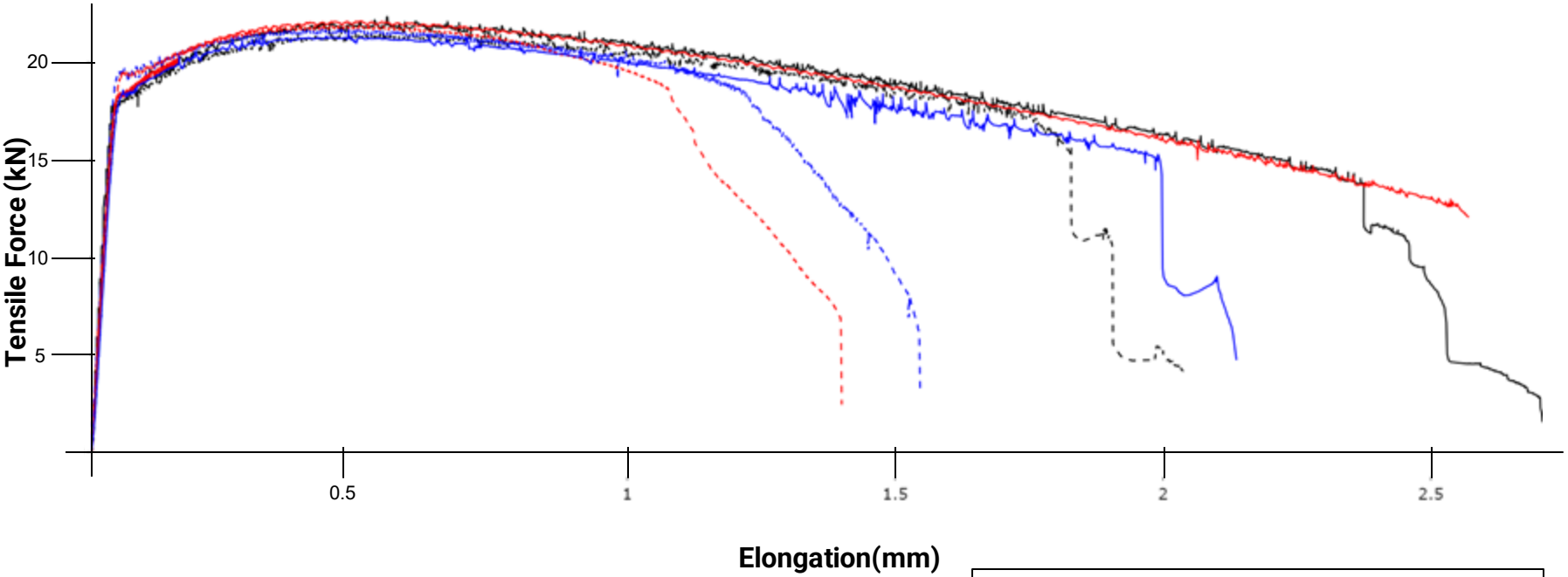
*Vickers Hardness 10 Kgf



Selection of samples for fractography



	BM1	Heat affected zone	Weld
Air	———	———	———
Hydrogen	- - -	- - -	- - -

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Chemistry

	C	N	Al	Si	P	S	Ca	Ti
BM1	0.089	0.006	0.035	0.430	0.016	0.002	0.003	0.004
BM2/BMA	0.117	0.003	0.036	0.312	0.020	0.002	0.002	0.013
BM3	0.118	0.003	0.035	0.314	0.021	0.002	0.002	0.013
BM4	0.086	0.005	0.036	0.429	0.015	0.002	0.004	0.004
BMB	0.107	0.008	0.041	0.293	0.021	0.002	0.008	0.002
LW	0.090	0.005	0.020	0.320	0.019	0.003	0.001	0.009
GW	0.080	0.002	0.004	0.556	0.008	0.006	0.001	0.032
GWB	0.112	0.024	0.003	0.282	0.020	0.009	0.001	0.014
	V	Cr	Mn	Ni	Cu	Nb	Mo	Fe
BM1	0.090	0.036	1.701	0.050	0.029	0.049	0.005	97.412
BM2/BMA	0.004	0.112	1.527	0.047	0.033	0.042	0.020	97.683
BM3	0.004	0.113	1.546	0.047	0.034	0.044	0.020	97.647
BM4	0.091	0.036	1.684	0.050	0.029	0.047	0.005	97.431
BMB	0.010	0.022	1.606	0.037	0.019	0.076	0.243	97.465
LW	0.005	0.029	1.573	0.046	0.038	0.026	0.185	97.528
GW	0.002	0.021	1.318	0.705	0.073	0.009	0.007	97.084
GWB	0.014	0.014	0.791	0.030	0.030	0.016	0.387	98.144

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