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DE LA RECHERCHE À L'INDUSTRIE

Experimental characterization and modeling of intergranular fracture of austenitic stainless steels in PWR environment

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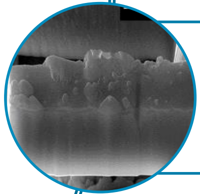
Outline

1. Stress Corrosion Cracking (SCC) issue in PWR environment

2. Study methodology



3. Grain boundary strength of oxidized Si enriched steel

4. Conclusions and perspectives

1. SCC issue in PWR environment
Context

Pressurized water reactor conditions (PWR): Water (1000ppm B, 2ppm Li) at 300°C, 155bars + neutrons irradiation

Irradiation Assisted Stress Corrosion Cracking (IASCC) of austenitic stainless steel constitutive of the internal parts of the reactor

Figure 1: Reactor assembly

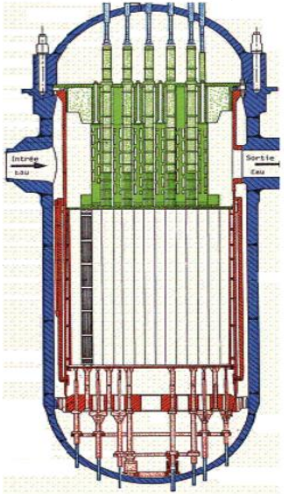
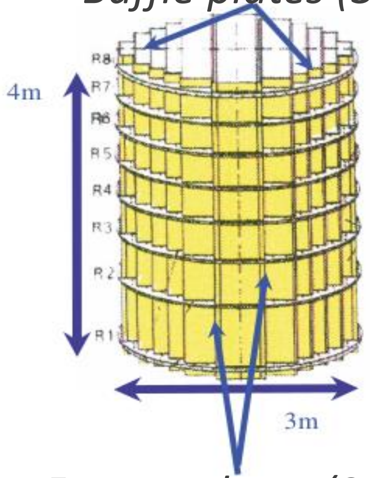


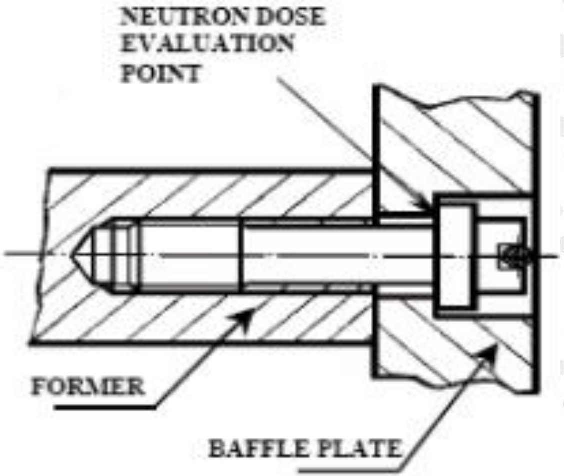
Figure 2: Reactor internal parts

Baffle plates (304L)



Former plates (304L)

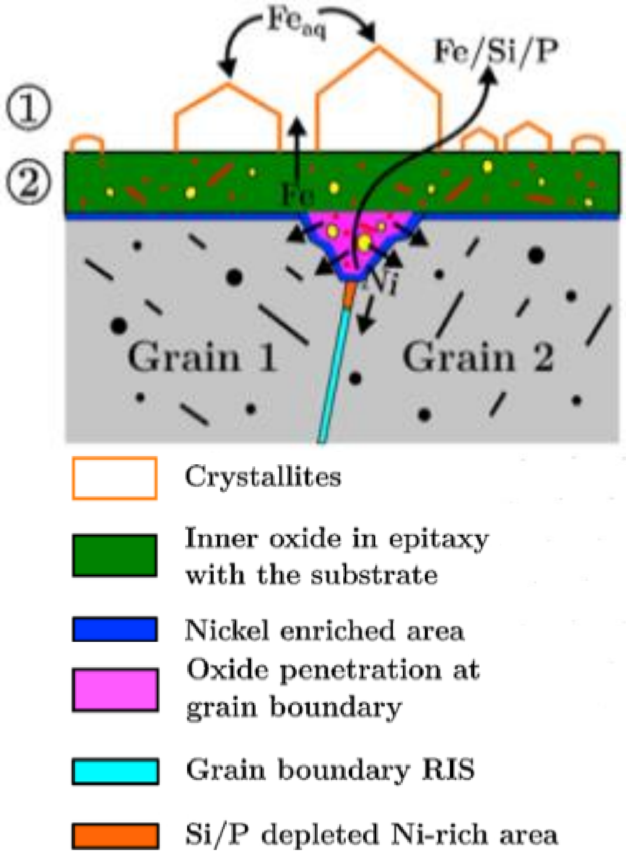
Figure 3: Baffle to former bolt (316L)



B. Tanguy. Corrosion sous contrainte assistée par l’irradiation des aciers inoxydables austénitiques (IASCC). Revue de Métallurgie, 108(1) :39–46, 2011.

1. SCC issue in PWR environment

Oxidation at grain boundary



Austenitic stainless steel corrosion in PWR environment

➡ Duplex oxide with 2 layers

Deeper penetration of oxide at GB:

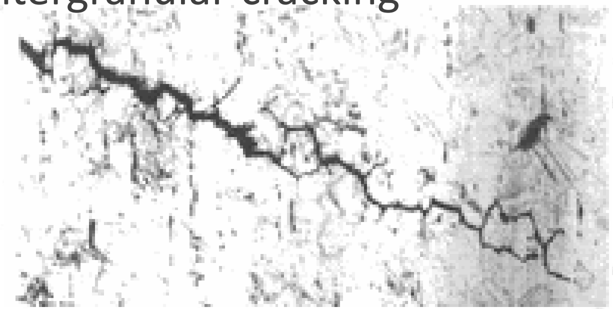
- GBs act as diffusion short cuts
- Irradiation ➡ Radiation induced segregation (RIS) at GB ➡ change of chemistry

➡ Increase IGSCC susceptibility of irradiated austenitic stainless steels

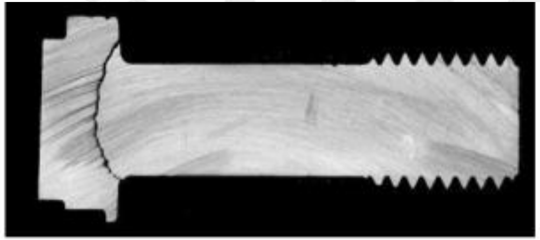
Boisson, M., Legras, L., Andrieu, E., & Laffont, L. (2019). Role of irradiation and irradiation defects on the oxidation first stages of a 316L austenitic stainless steel. *Corrosion Science*, *161*, 108194.
T. J. Griesbach, G. J. Licina, P. C. Riccardella, J. Rashid, and R. E. Nickell. A probabilistic approach to baffle bolt IASCC predictions. 2012.

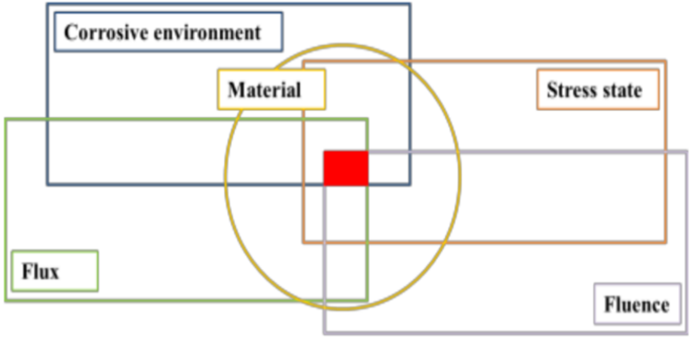
1. SCC issue in PWR environment
Oxidation outcome

Intergranular cracking



Bolts failure





IASCC phenomena

Mostly intergranular cracking

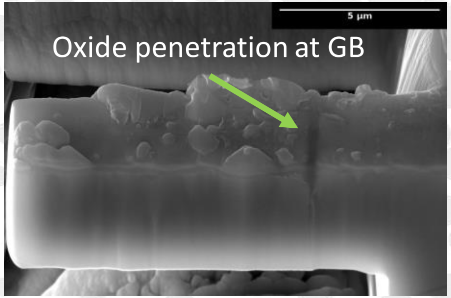
Need to assess GB strength to evaluate IGSCC susceptibility

Boisson, M., Legras, L., Andrieu, E., & Laffont, L. (2019). Role of irradiation and irradiation defects on the oxidation first stages of a 316L austenitic stainless steel. *Corrosion Science*, *161*, 108194.
T. J. Griesbach, G. J. Licina, P. C. Riccardella, J. Rashid, and R. E. Nickell. A probabilistic approach to baffle bolt IASCC predictions. 2012.

Determination of the crack initiation stress of oxidized grain boundary

Definition of an experimental protocol:

- FIB cross-section polishing
- EBSD mapping on cross-section to locate GB
- FIB milling procedure of bi-crystalline micro-beams
- Bending test of micro-beams to access the critical load at failure

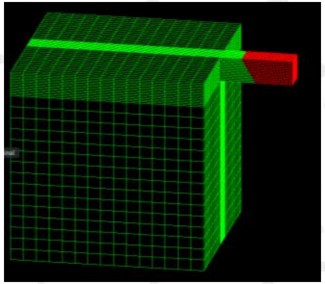


Presentation focus

Application to model alloy

Application to commercial alloy

FE simulation of micro-bending tests

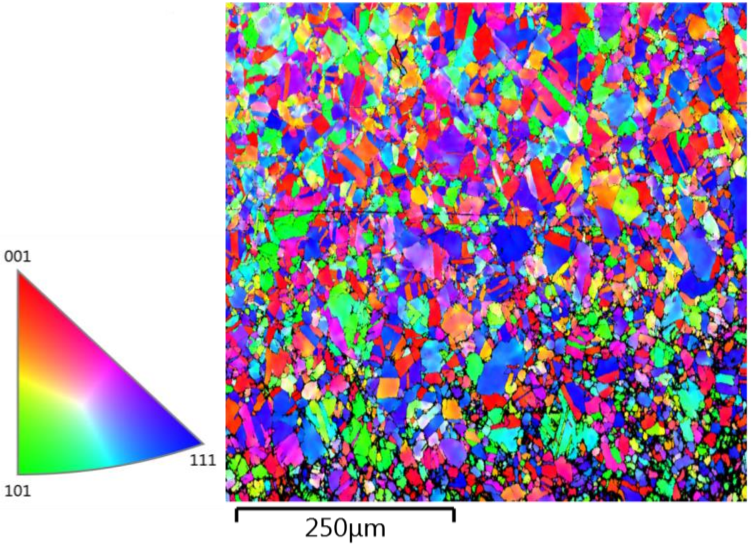


Extract the GB critical stress at failure

Main Si enriched steel chemical elements (model alloy):

Fe	Cr	Ni	Si
50% wt.	12% wt.	26% wt.	3% wt.

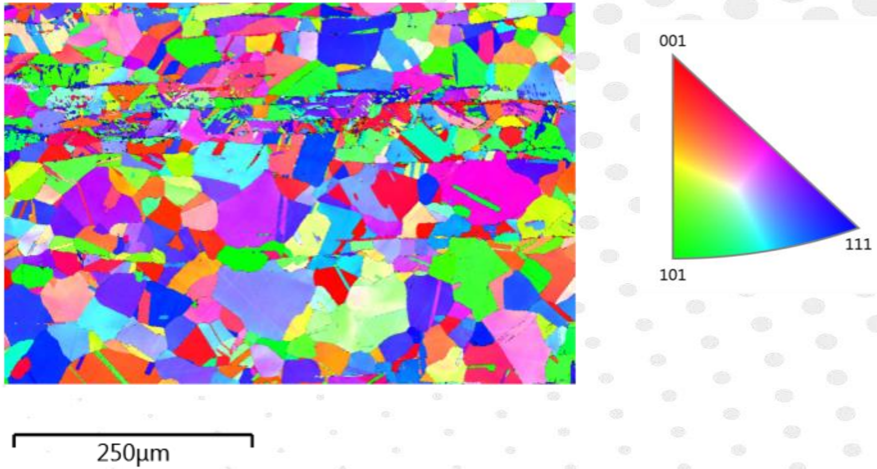
Mean grain size: 10µm



Main 304L steel chemical elements (commercial alloy):

Fe	Cr	Ni	Mn	Si
70% wt.	18% wt.	8% wt.	1.6% wt.	0.4% wt.

Mean grain size: 21µm



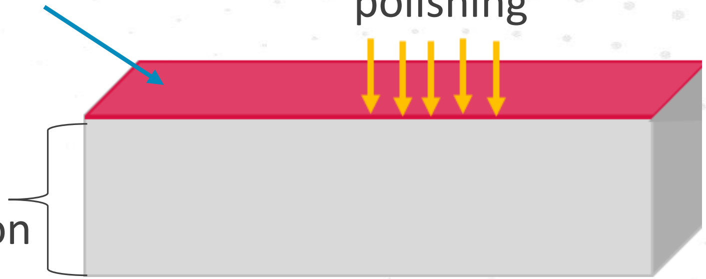
2. Study methodology
FIB cross-section polishing

18x2x2mm bar oxidized in PWR environment

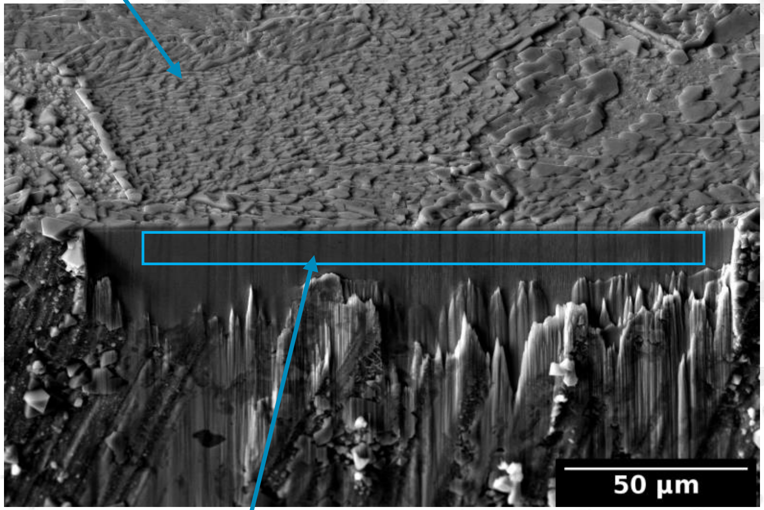
Oxidized surface of interest

FIB Ga^{+} ions polishing

Bar cross-section



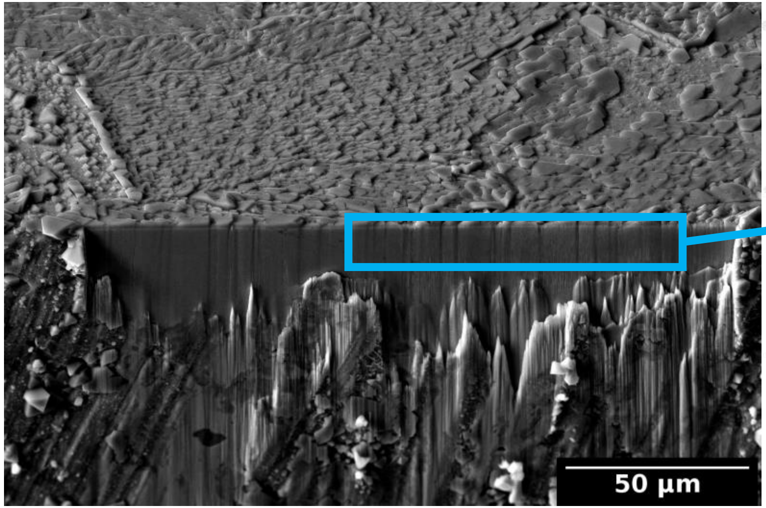
Oxidized surface of interest



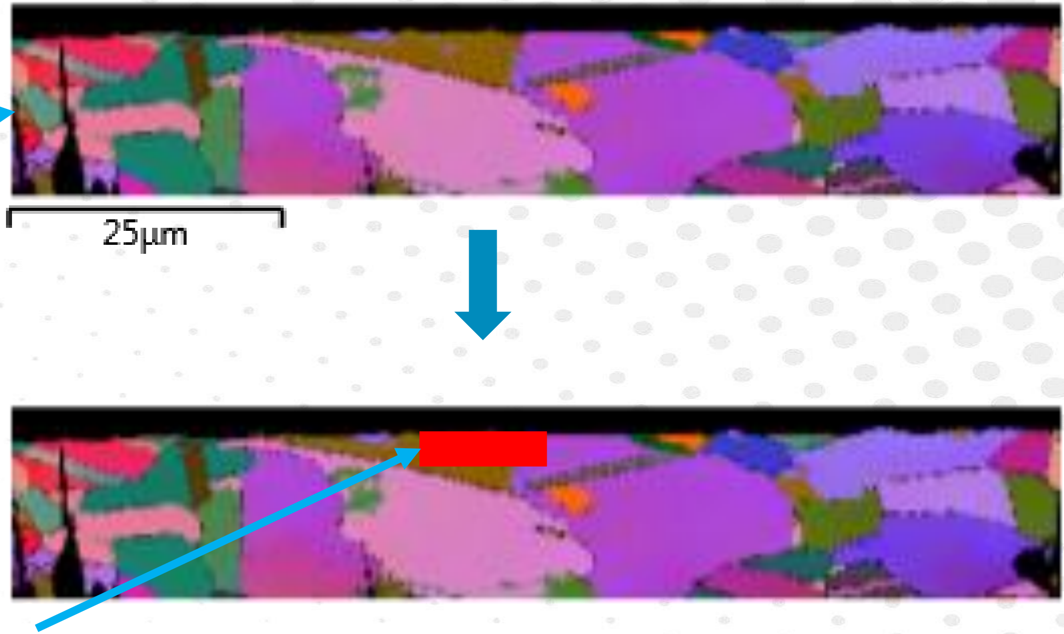
Cross-section polished region for EBSD mapping

2. Study methodology
EBSD mapping of the polished cross-section

Polished cross-section



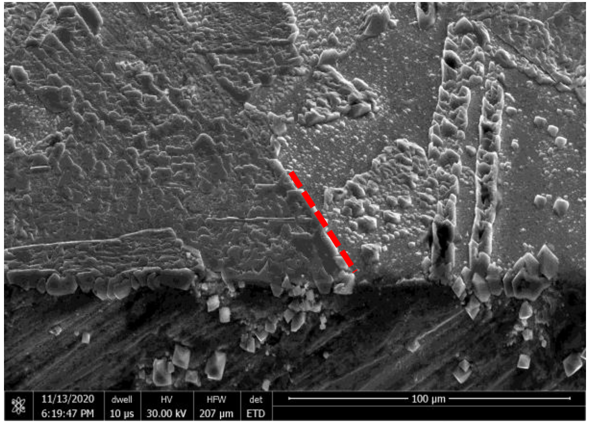
EBSD mapping to locate GB



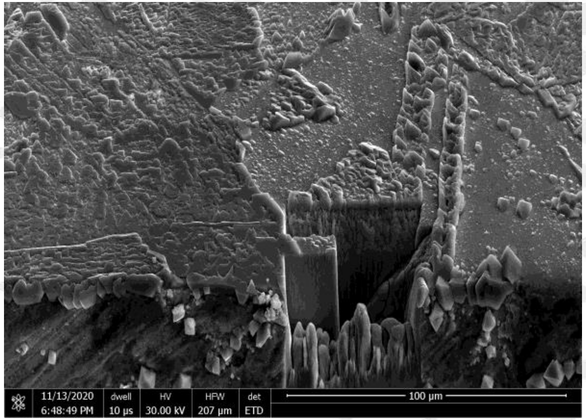
Location of the bi-crystalline micro-beam

2. Study methodology
Milling of 16x5x4 µm micro-beam using SEM-FIB

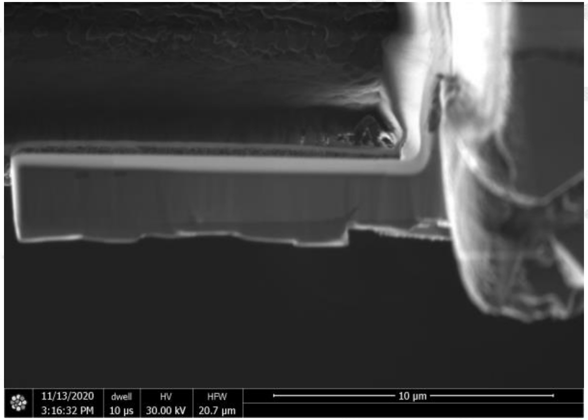
CEMES



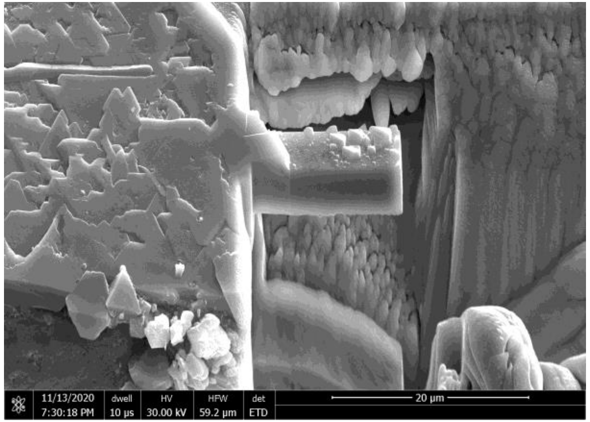
Milling current
21nA
9nA



Milling current
21nA
9nA
2nA
1nA



Milling current
2nA
1nA

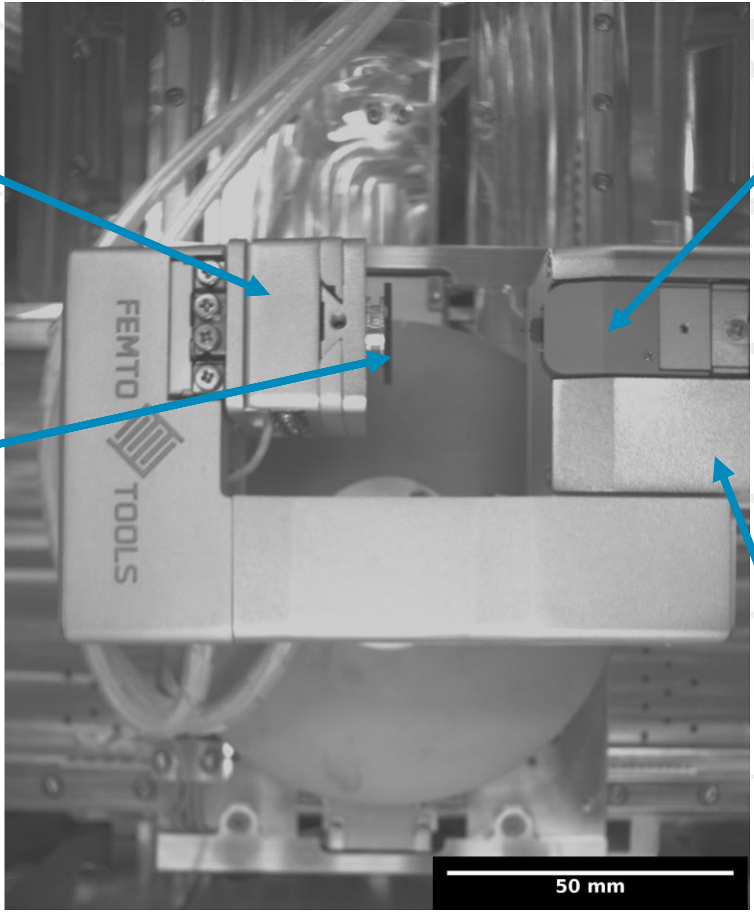


2. Study methodology

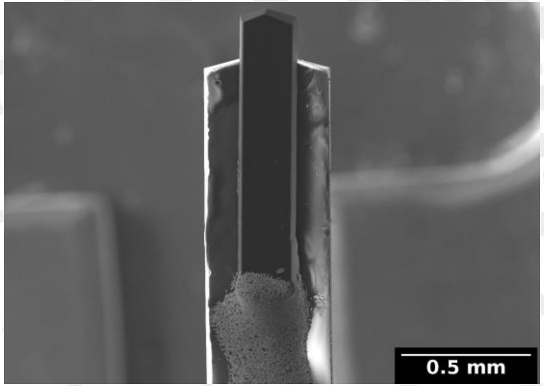
Experimental setup

Two axis positioning platform

Sample



Sensing probe (0.5mN to 200mN) terminated by the Berkovich diamond tip

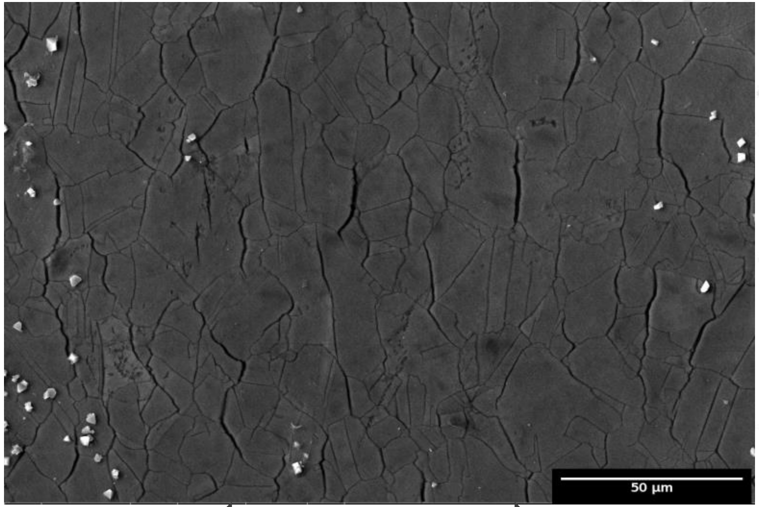


One axis positioning linear piezo scanner

3. Grain boundary strength of Si enriched steel

Oxidation and IGSCC susceptibility

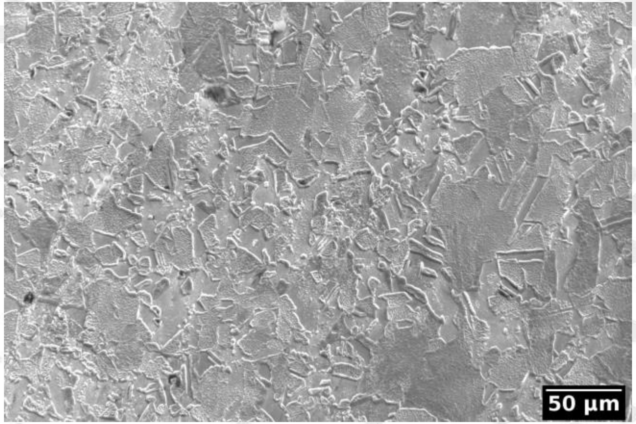
Slow Strain Rate Tensile Test of 40x2x2µm in PWR simulated environment (4% strain, strain rate $5.10^{-8}s^{-1}$, 300h, σ^{max} 490MPa)



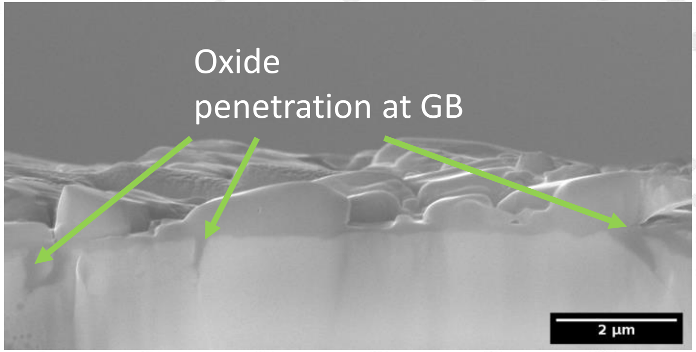
Loading direction

Model alloy susceptibility to IGSCC confirmed

3700h oxidation of 18x2x2µm sample (340°C, 1000ppm B, 2ppm Li water)

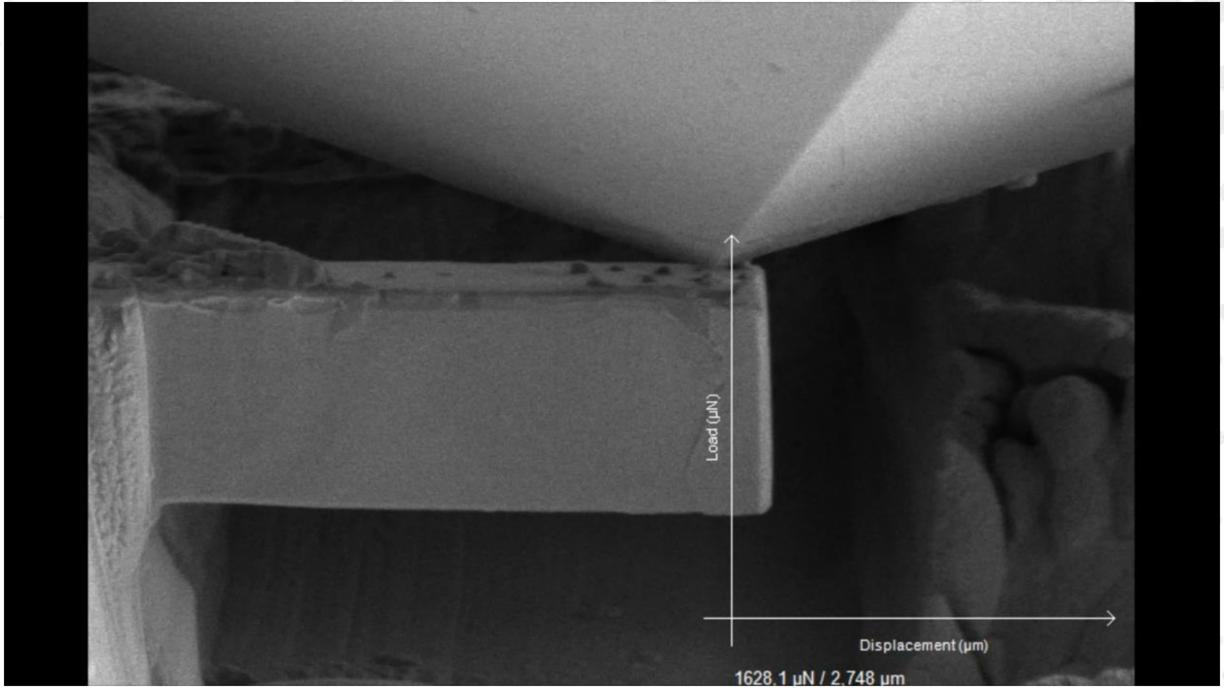


Oxide layer: 2µm
Oxide penetration at GB: up to 1.7µm



3. Grain boundary strength of Si enriched steel
Micro-bending test

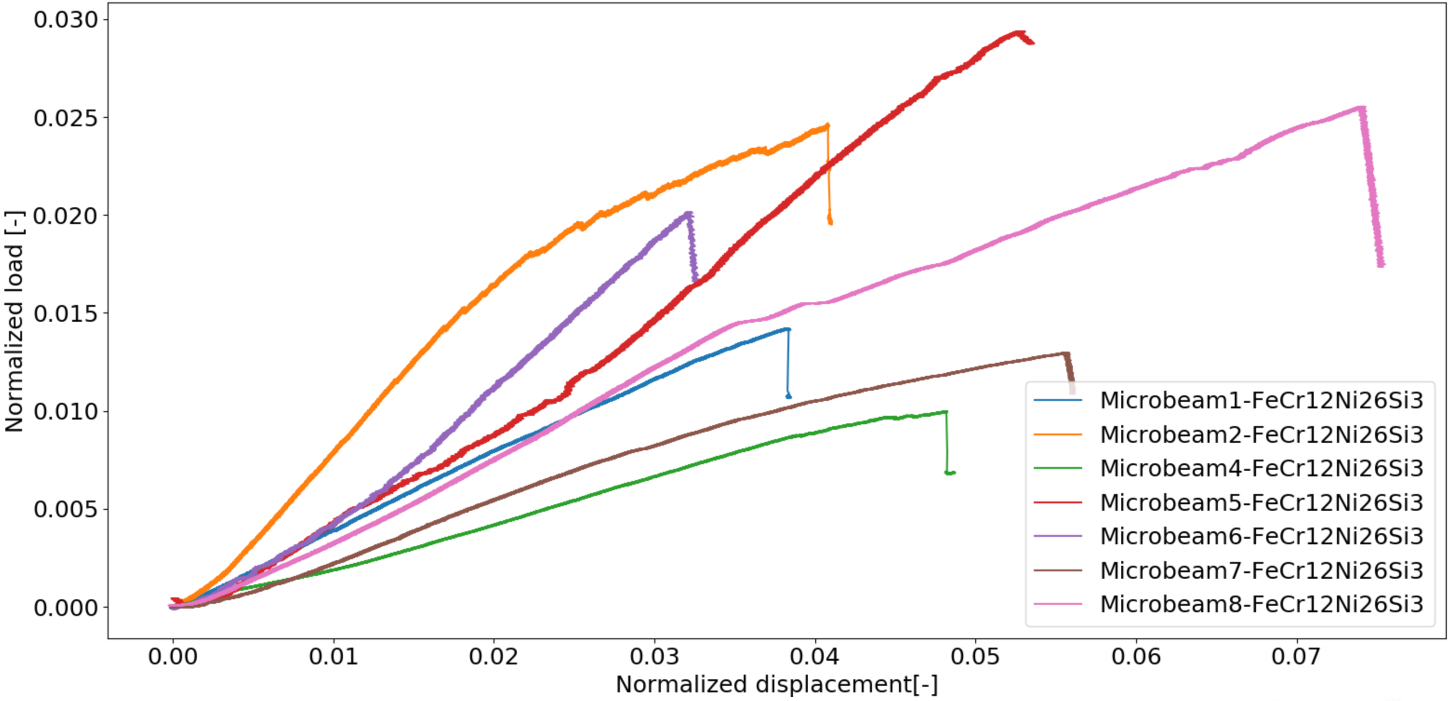
SEM *in-situ* testing of a 16x5x4 µm micro-beam at 20nm/s at room temperature



3. Grain boundary strength of Si enriched steel

Experimental campaign

8 micro-bending tests on Si enriched austenitic steel



Maximum beam deflection equation

$$d = \frac{PL^3}{6EI}$$

d: beam deflection
P: load
L: beam length
E: Young modulus
I : moment of inertia

Plotted displacement: $\frac{d}{L}$

Plotted load: $\frac{PL^2}{6EI}$

➡ All tested grain boundaries show fragile fracture

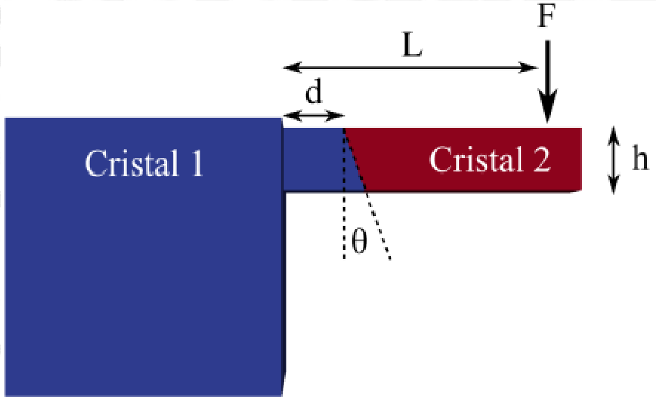
3. Grain boundary strength of Si enriched steel

Normal GB stress determination

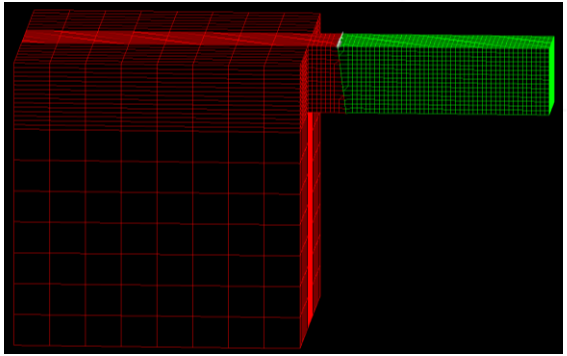
- Estimation of maximum GB stresses using beam theory equations:

$$\sigma_{nn}^{max} = \sigma_{yy}^{max} \cos^2 \theta = 6\frac{F(L-d)}{bh^2}\cos^2 \theta$$

$$\tau^{max} = \sigma_{xy}^{max} \cos\theta \sin\theta = 6\frac{F(L-d)}{bh^2}\cos\theta \sin\theta$$



- 3D FE simulations of beam elastic deformation



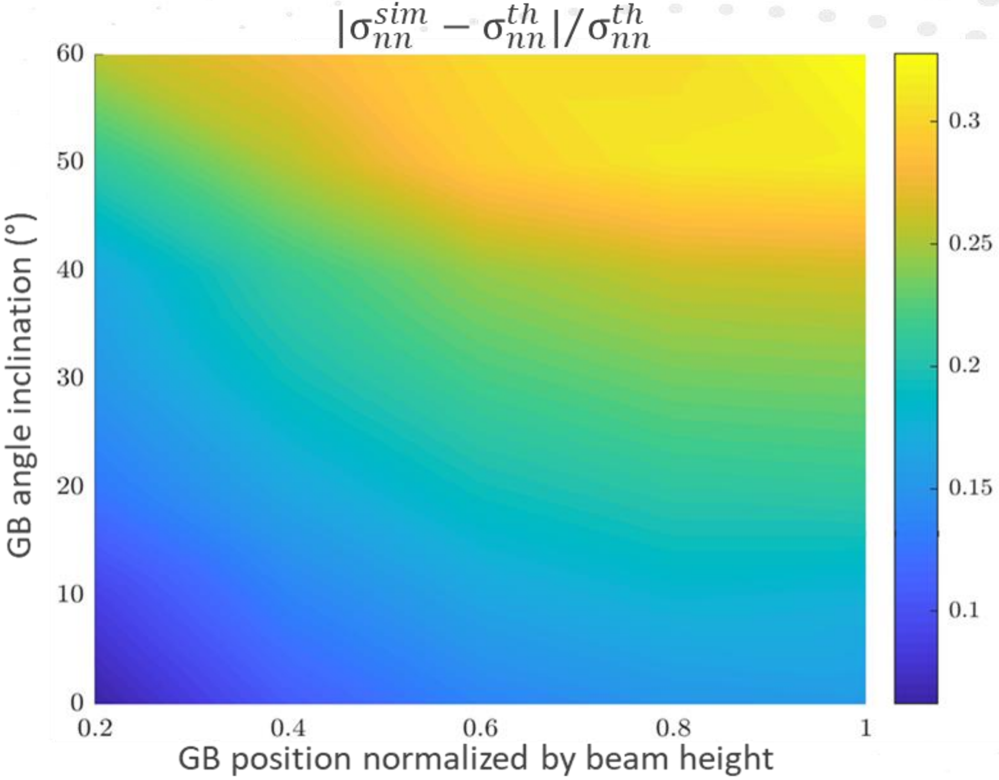
More parameters taken into account

- Crystals orientations
- Indenter tip size and position along beam width

➡ closer to experimental conditions

3. Grain boundary strength of Si enriched steel
Normal GB stress determination

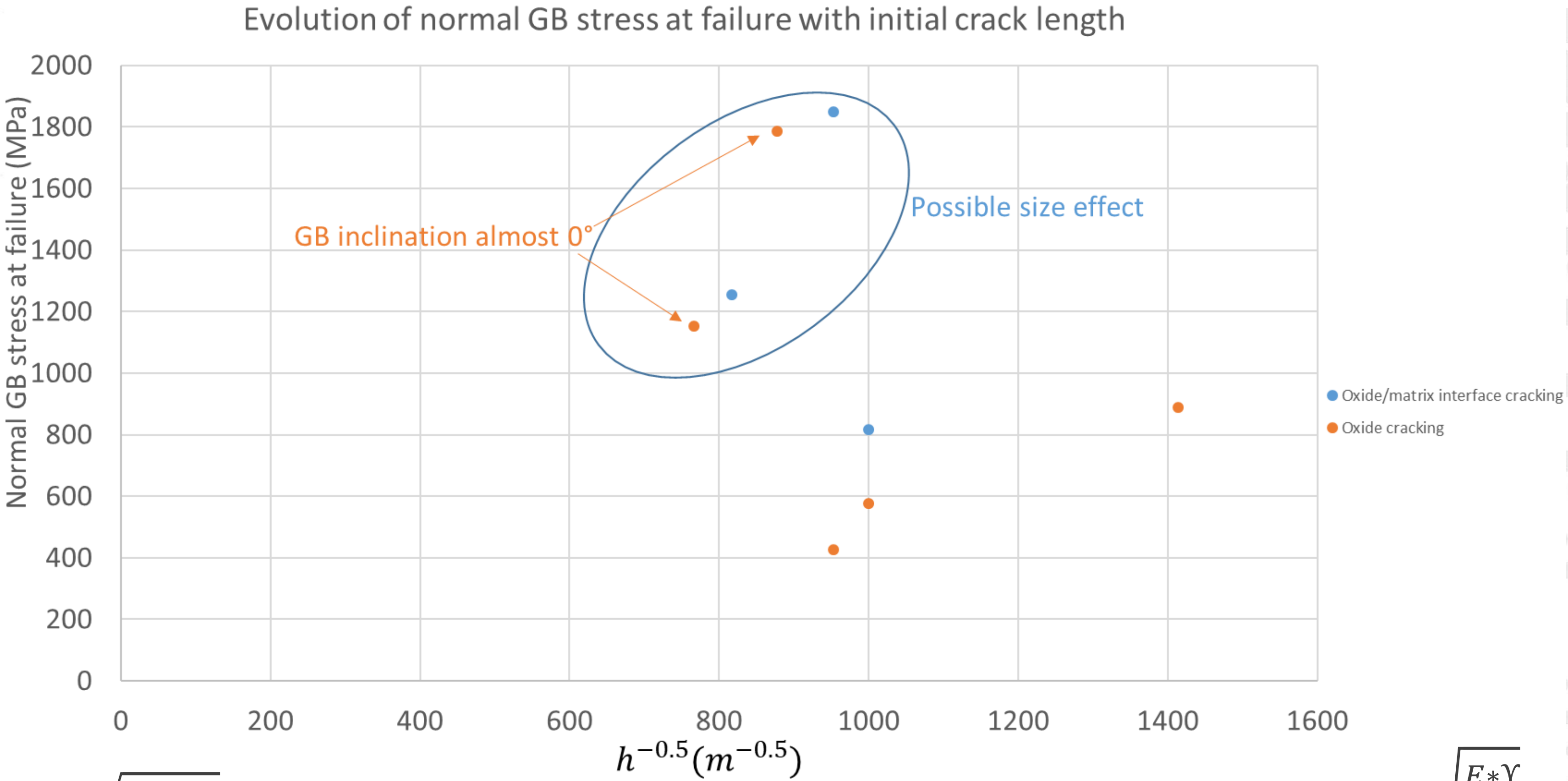
Evolution of theoretical/simulated GB stress ratio with GB position and GB inclination angle



Low ratio between theoretical and simulation GB stresses ➡ GB stress from beam theory equation close the simulation GB stress

➡ Beam theory equation can be rely on to estimate the normal GB stress

3. Grain boundary strength of Si enriched steel
Normal GB stress determination



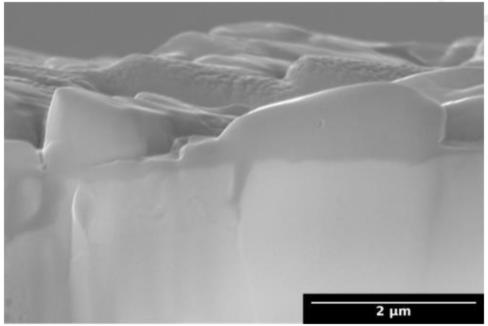
slop =$\sqrt{E * \Upsilon}$: access to the rupture energy according to Griffith model $\sigma_{rupt} \propto \sqrt{\frac{E * \Upsilon}{h}}$

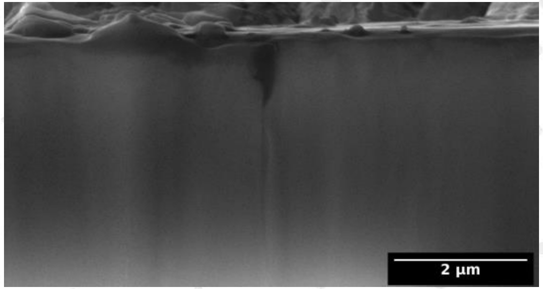
3. Grain boundary strength of Si enriched steel
Discussion of the results

Global trend: higher initial crack length seems to lead to lower normal GB stress at failure

BUT scattering in experimental data:

- Crack occurs either in matrix/oxide interface or inside the oxide (different mechanical properties)
- Oxide thickness on top of GB may increase GB stress at failure → need to be removed





- Different crystalline orientations
- Size effect on mechanical properties (beams heights from 2µm to 6µm)

4. Conclusions and perspectives

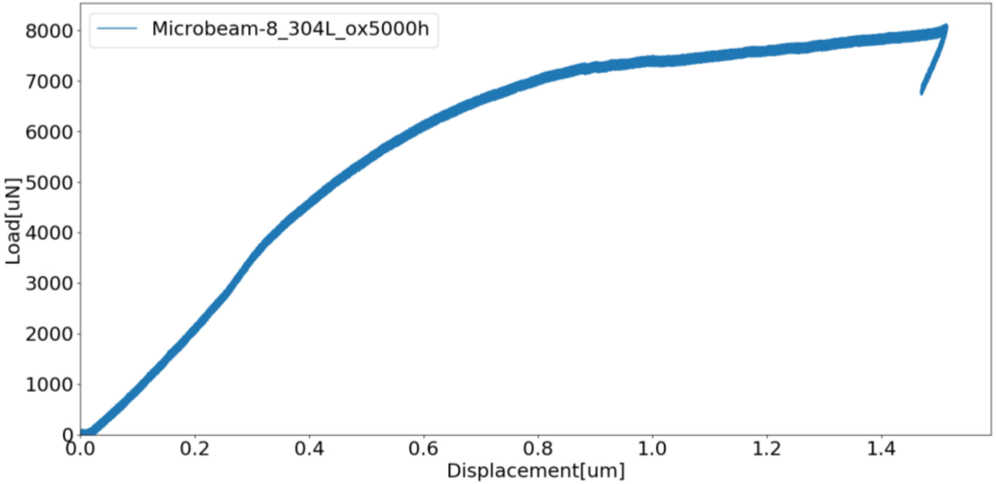
Conclusions:

- Development of an experimental protocol to study intergranular cracking and quantify stress at fracture of oxidized GB in PWR environment
    - Model alloy susceptible to IGSCC
    - Micro-bending of oxidized GB
    - GB stress at failure determination based on combination between analytical equations and FE simulation
- Si enriched austenitic steel shows intergranular cracking after 3700h oxidation in PWR environment
- First results seem to show a decrease of the normal GB stress at failure with the initial crack length but scattering in data calls for further experimentations

Perspectives:

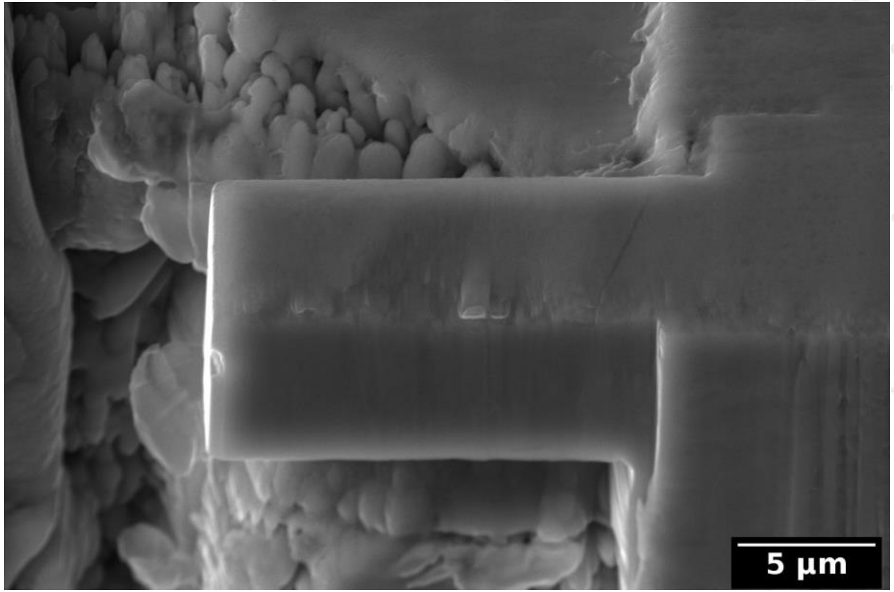
- Application of the experimental protocol to commercial alloy 304L
    - Micro-bending tests on 304L with further oxidation
    - Micro-bending tests on oxidized 304L irradiated with proton at 1dpa
- Improve the modeling experiments to extract GB the stress at failure

SEM *in-situ* testing of a 304L 16x6x7 µm micro-beam at 20nm/s at room temperature

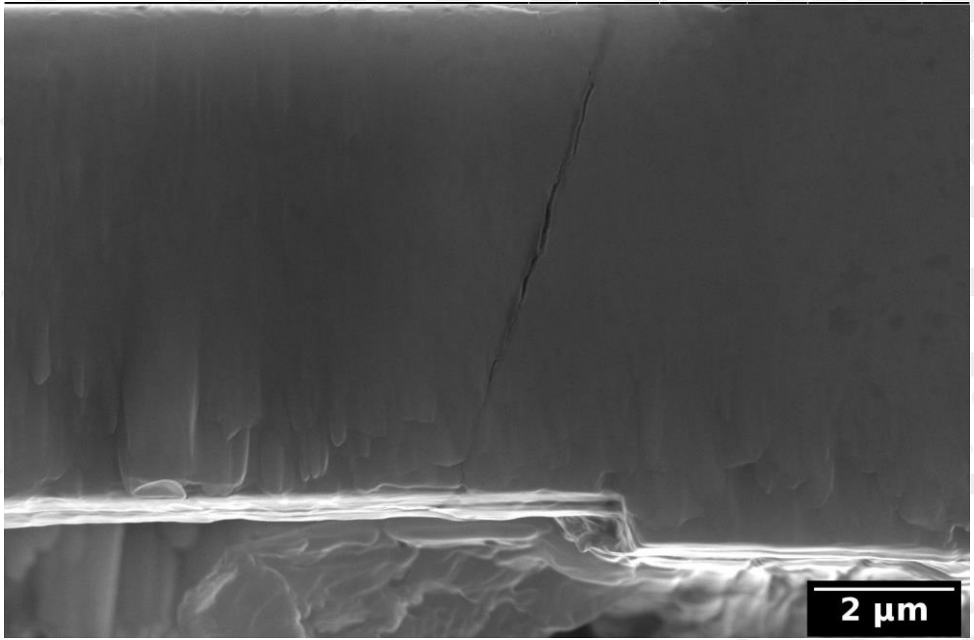


First results on commercial alloy 304L

Before testing



After testing



Micro-cracking of an oxidized GB observed on one test (out of 11) ➡ low tendency to GB cracking

➡ Need to test more sensible GB (irradiated and oxidized)

Thank you for your attention!

Questions?