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Stress corrosion cracking of Ni-base alloys in PWR primary water

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DE LA RECHERCHE À L'INDUSTRIE

cea den

STRESS CORROSION CRACKING OF NI-BASE ALLOYS IN PWR PRIMARY WATER

Catherine GUERRE

CEA and MINES ParisTech : E. Chaumon, P. Laghoutaris

CEA : O. Raquet

Mines ParisTech: J. Crépin, C. Duhamel, M. Sennour, F. Gaslain, H.T. Le

Ecole Polytechnique: E. Héripé

CNRS: J. Chêne

AREVA : P. Combrade, P. Scott (retired)

EDF : Th. Couvant , F. Vaillant (retired)

IRSN : M. le Calvar, I de Curières

**Den-Service de la Corrosion et du Comportement des Matériaux dans leur
Environnement (SCCME), CEA, Université Paris-Saclay, F-91191, Gif-sur-Yvette, France**

cea den Nickel base alloys

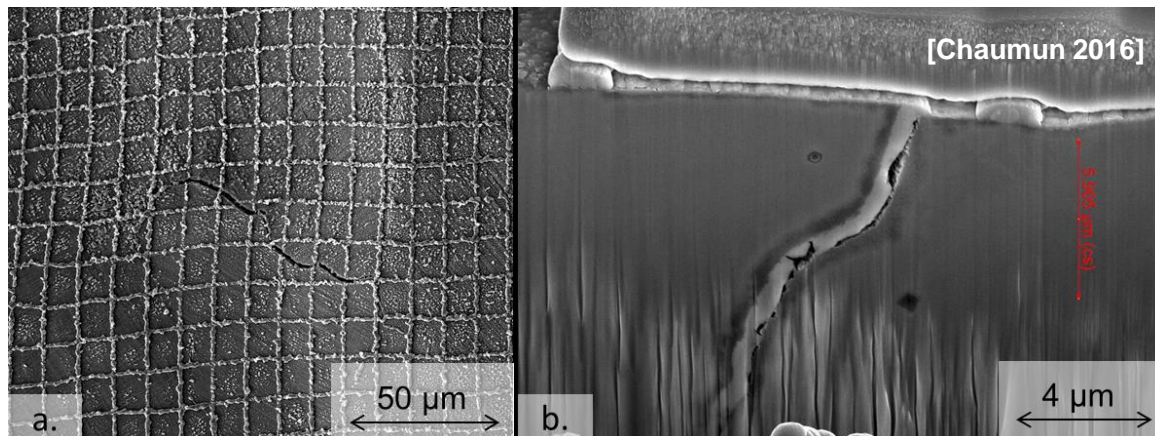
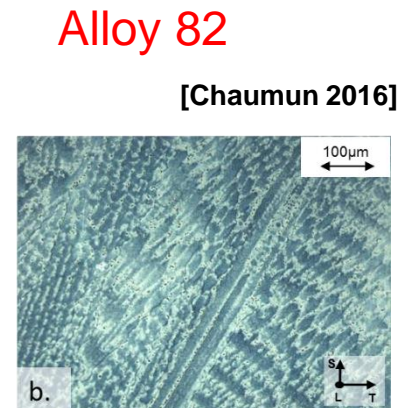
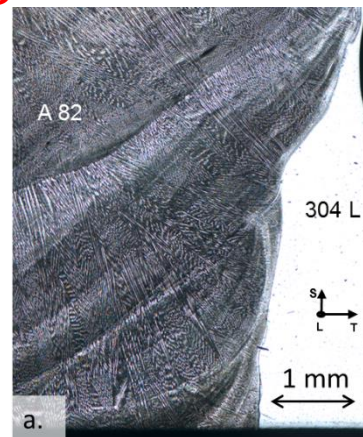
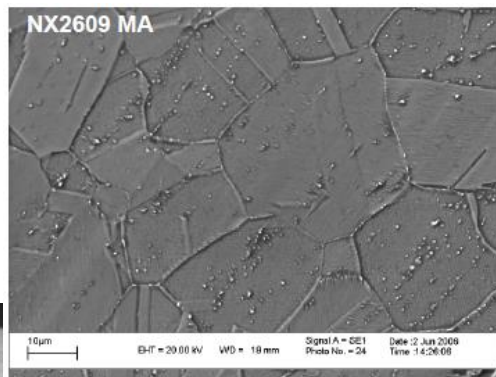
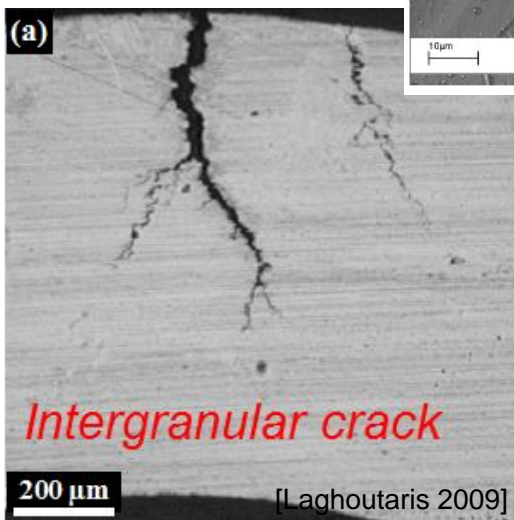
- Alloy 600 MA, Alloy 600 TT (Ni-16Cr-9Fe) and its weld metals (**Alloy 82** and Alloy 182)
- Alloy 690 TT (Ni-30Cr-10Fe) and its weld metals (Alloy 52 and Alloy 152)



Less susceptible to SCC

intergranular and oxidized cracks

Alloy 600 MA



Alloy 600, U-bend 1200 hr @ 325°C

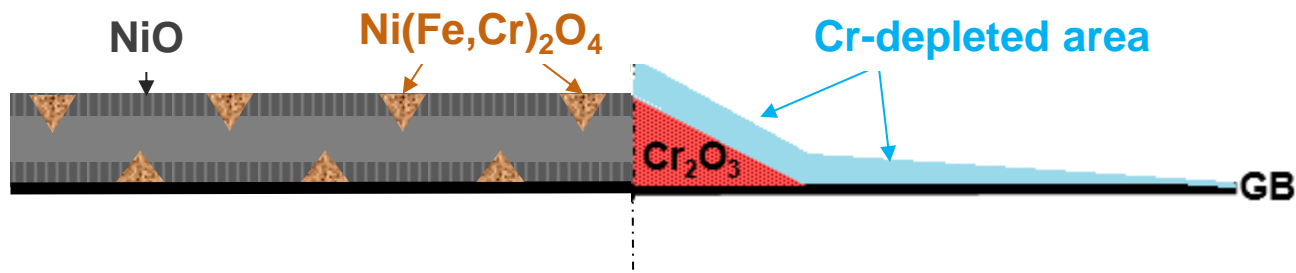
Alloy 82, U-bend, 2500 hr @ 400°C

Since the first laboratory SCC crack for **Alloy 600** in high temperature water (Coriou 1959), many laboratory studies have been performed :

- Parametric studies : effect of hydrogen, temperature, microstructure, chemistry, ...
- Time to initiation and crack growth rate
- More recently : local characterization of SCC crack tips

thanks to the development of high-resolution characterization techniques such as analytical transmission electron microscopy (ATEM) or atom probe tomography as well as site-specific sample preparation with focused ion beam (FIB)

Primary crack : the deepest propagating crack
(from Laghoutaris 2009 and Sennour et al. 2009)



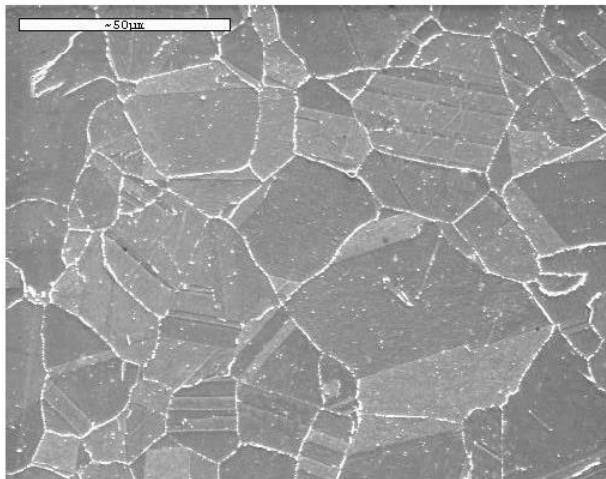
Towards a **SCC mechanism for nickel alloys** (to confirm the low susceptibility of Alloy 690)

CEA/EDF program on SCC initiation of A690 in PWR primary water

Experimental conditions of the French R&D program (EDF and CEA)

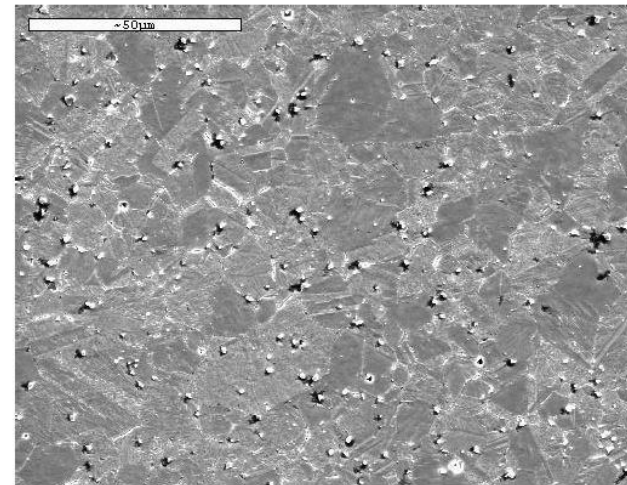
- 21 industrial tubes (high MA temperature (1040-1080°C, generally H₂) + TT 700°C) :
intergranular carbides
- 19 experimental tubes, (MA (980°C < T < 1040°C, NH₃ or H₂) + TT 700°C)
 - some with intergranular carbides
 - others with inter + intra carbides, among them **WE092** and **WE094** with a 2 steps HT

WF771



Attaque brome/méthanol – observation MEB

WE092 (MA 1040°C – 1070°C)



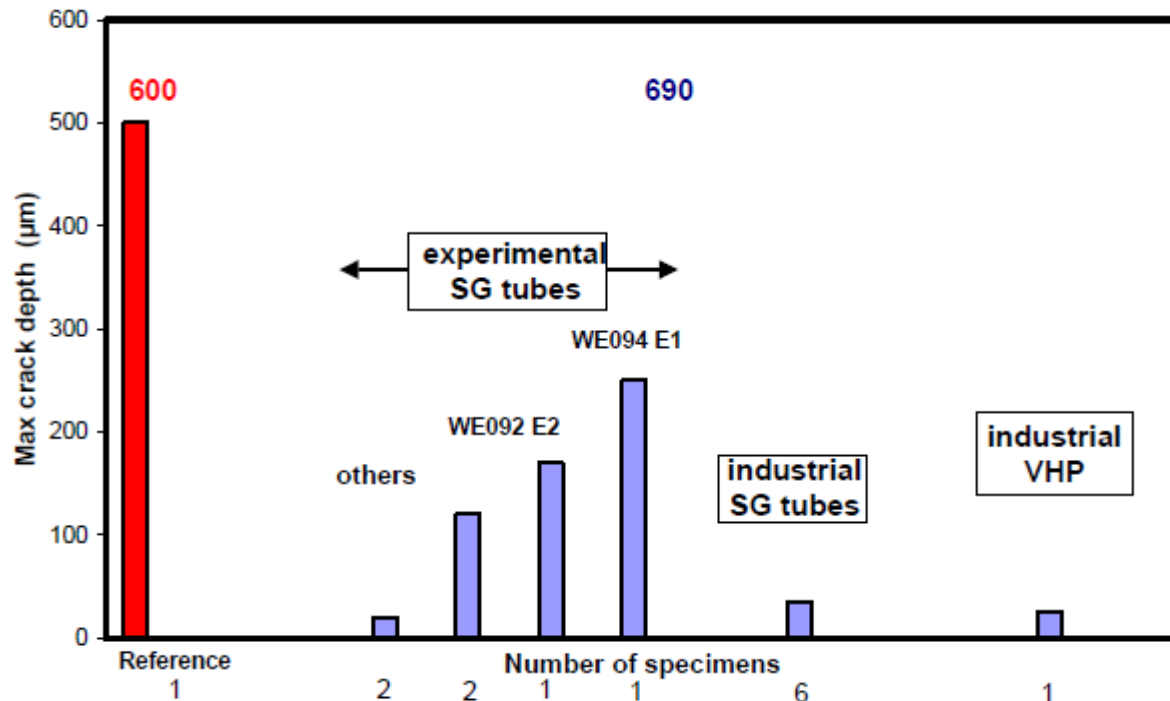
Attaque brome/méthanol – observation MEB

cea den SCC initiation of Alloy 690

CERTS in simulated primary water at 360°C ($5 \cdot 10^{-8} \text{ s}^{-1}$).

It was concluded :

- Not significant SCC susceptibility on most of the tested materials (CERTS specimen)
- Except for experimental heats with unusual microstructure (intragranular carbides) : IGSCC



Conclusion (as presented International PWSCC of Alloy 600 Conference and Exhibit Show (MRP-154) 2005)

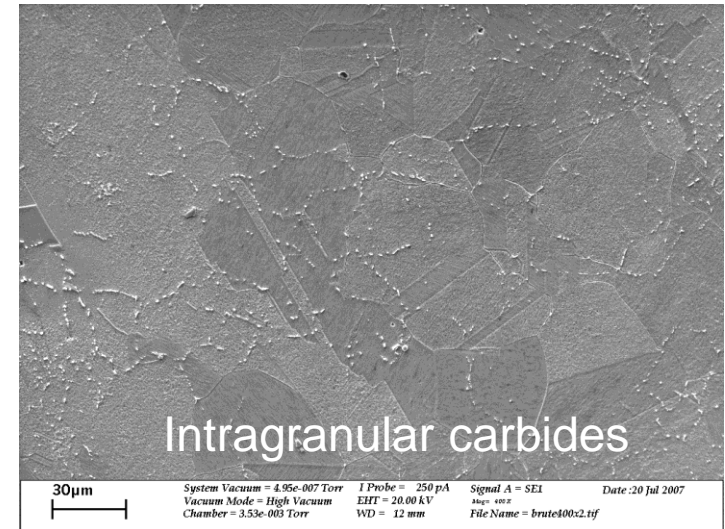
- All of the many the laboratory tests that have been performed have demonstrated a very high resistance to SCC of Alloy 690 in PWR primary water.
- In a few cases, a limited susceptibility to PWSCC has been observed in laboratory tests for Alloy 690 with a microstructure characterized by intragranular carbide precipitation (and low grain boundary coverage with carbides) when subjected to extremely severe loadings.
- For these cases, an approach based on the strain rate damage model was developed. Based on this model, no significant cracking would be expected in roll transitions of SG tubes during the lifetime of PWR plants.
- Moreover, the results show that no SCC is expected for industrial products having the specified intergranular carbide microstructure.

Alloy 600 with model microstructure :
influence of intergranular carbides precipitation on SCC
initiation (GB oxidation)

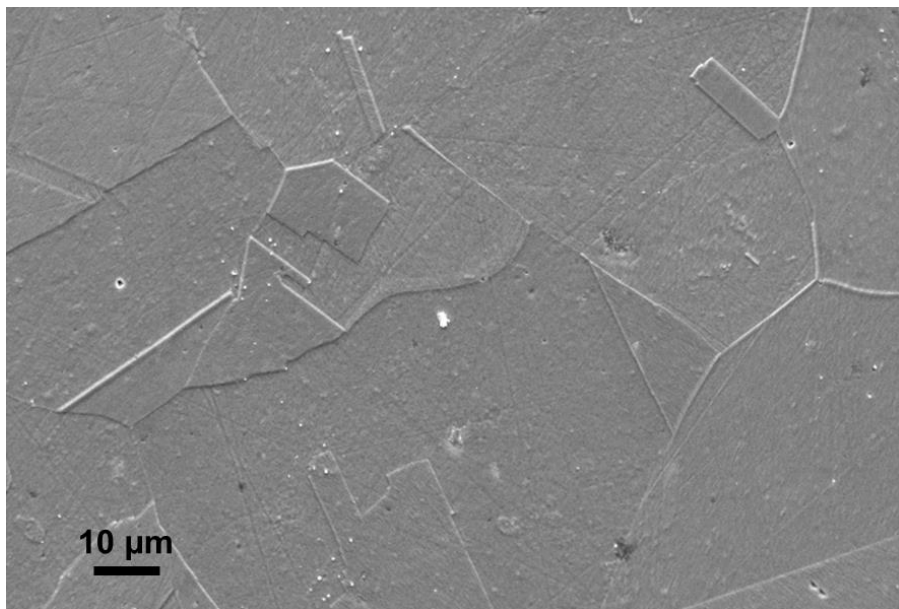
Model microstructure alloys

- Alloy 600 heat WF675
(very susceptible to SCC in PW)
- Heat treatment in vacuum :
 - Solution annealed + water quenching
-> no carbide
 - Solution annealed + air cooling
-> low intergranular carbide precipitation
 - Solution annealed + water cooling + 16 hr at 700 °C
-> high intergranular carbide precipitation
- No CW or CW up to 50 % (250 Hv to app. 320 Hv)

WF675

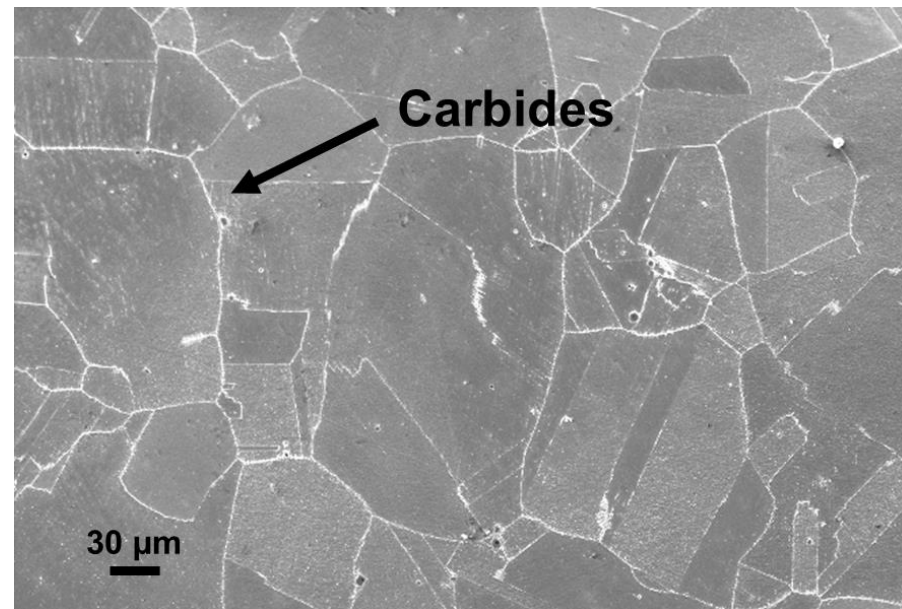


Sample SA "Solution Annealed"
No carbide



1050 °C for 1 h + water quenching

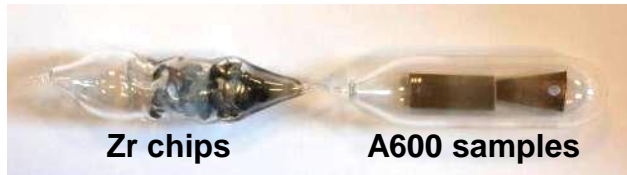
High IG precipitation



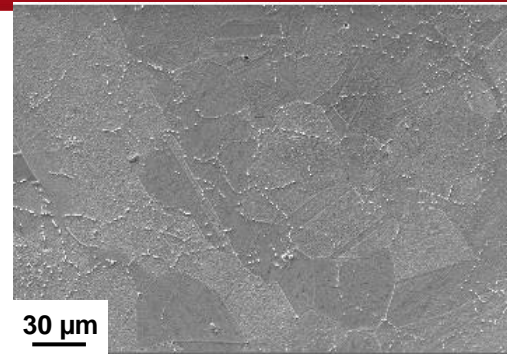
1050 °C for 1 h + water quenching
+ tempering at 700 °C for 16 h

cea den Alloy 600 with model microstructures

Model microstructure



Alloy 600



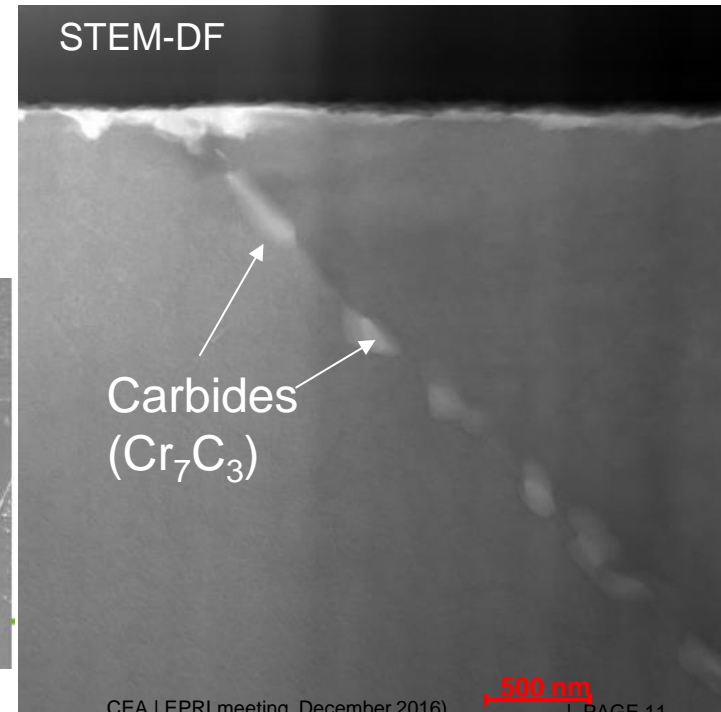
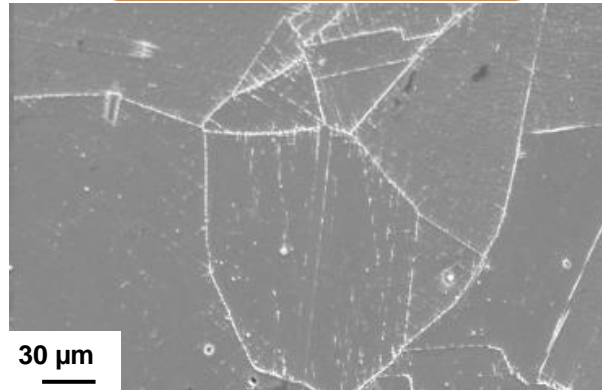
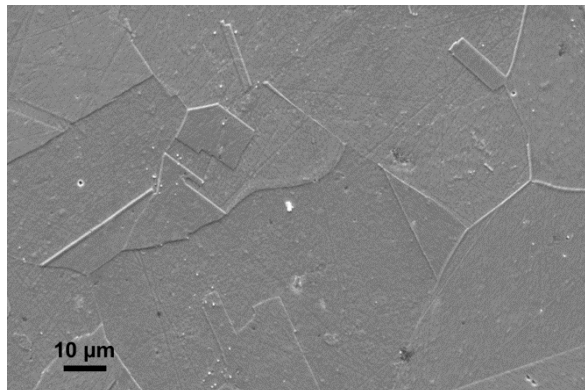
Heat WF675
Susceptible to SCC

1050°C – 1h
water quenching

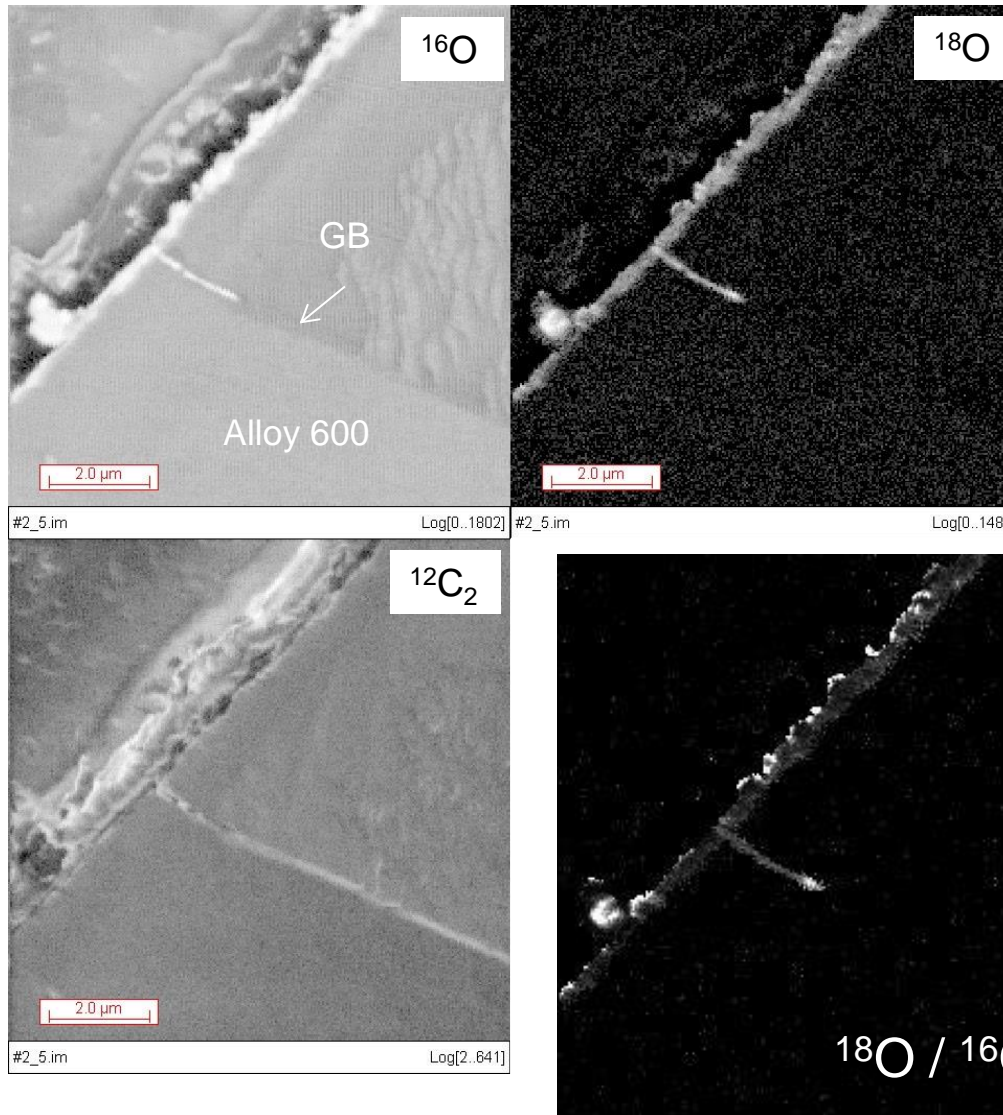
ageing
700°C – 16h

Strong intergranular
carbide precipitation

Solution annealed (SA)



Solution – annealed (no IG carbide)



Nano-SIMS analysis on intergranular attack formed during exposure in water.

Exposure conditions :

- 1340 hr in nominal primary water at 325°C
- 67 hr in water containing tracers (^{18}O) at 325°C

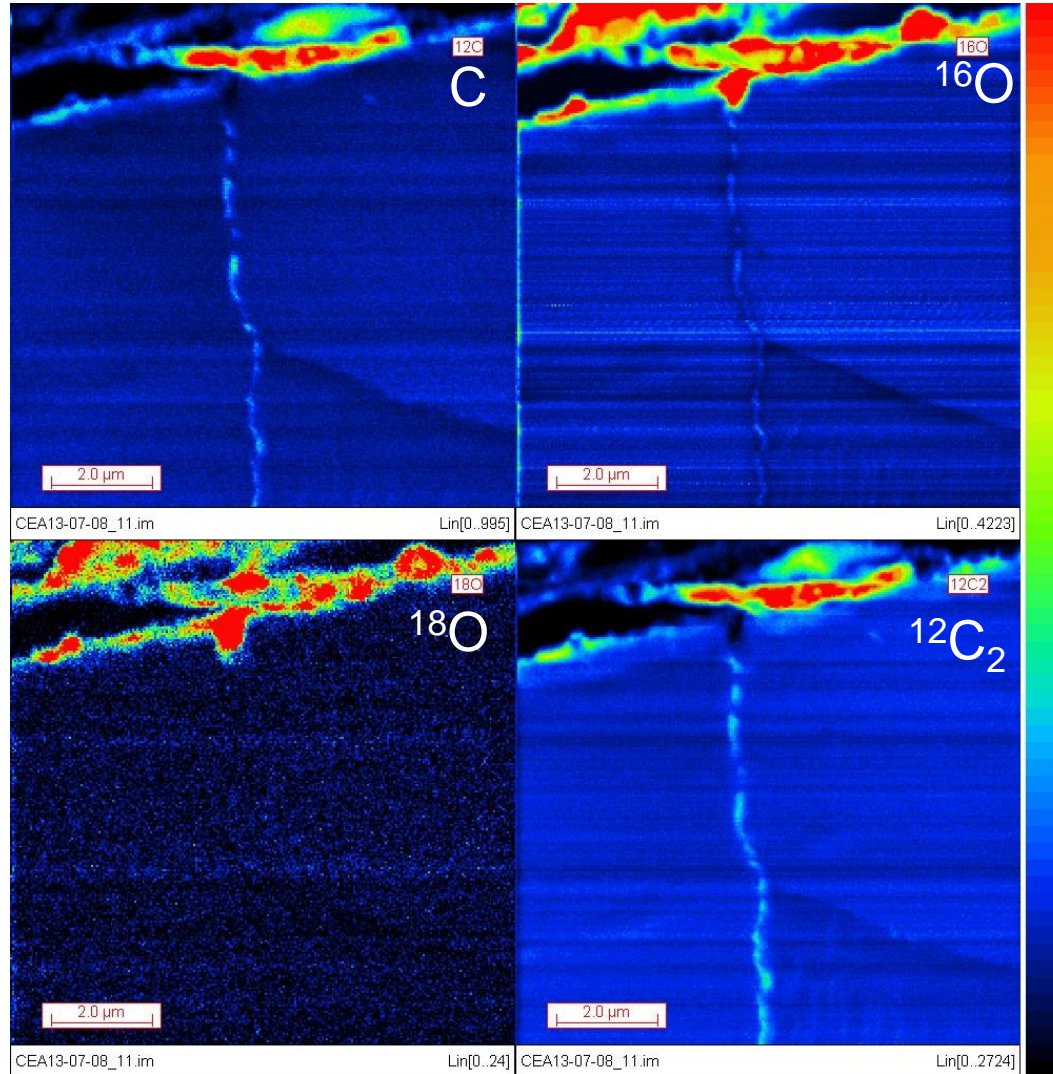
-> ^{18}O is located at the tip of the IG penetration and in the outer part of the oxide surface layer

-> ^{18}O transport during 67 hr up to the tip of the oxidized grain boundary

-> long and thin IGA

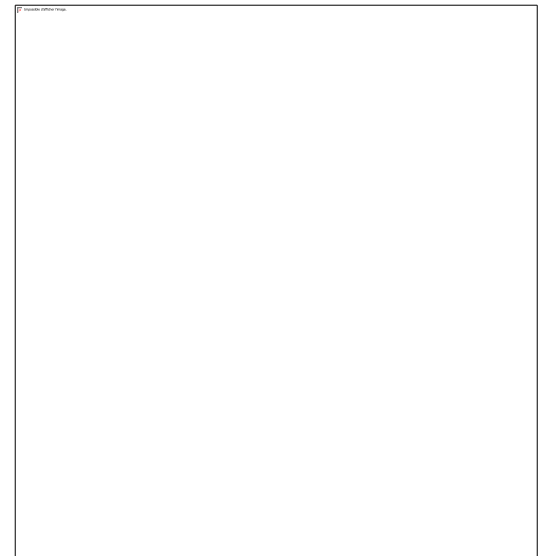
*Nano-SIMS analysis were performed by Nathalie Vallé
Public research centre Gabriel Lippmann, Material
analysis department*

Alloy with IG carbide precipitation

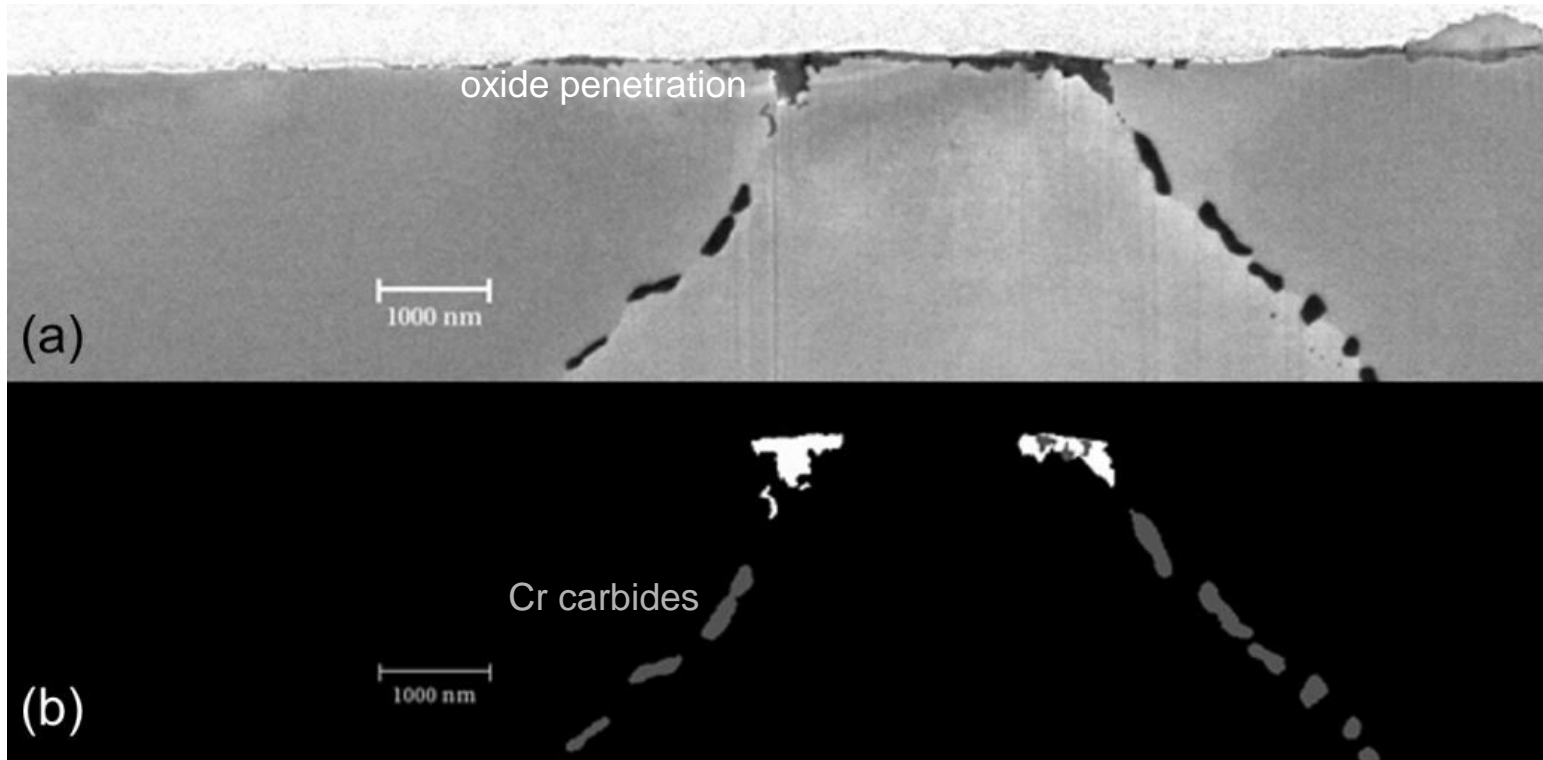


Nano-SIMS analysis
Same exposure conditions.

-> large and short IGA



Alloy with IG carbide precipitation

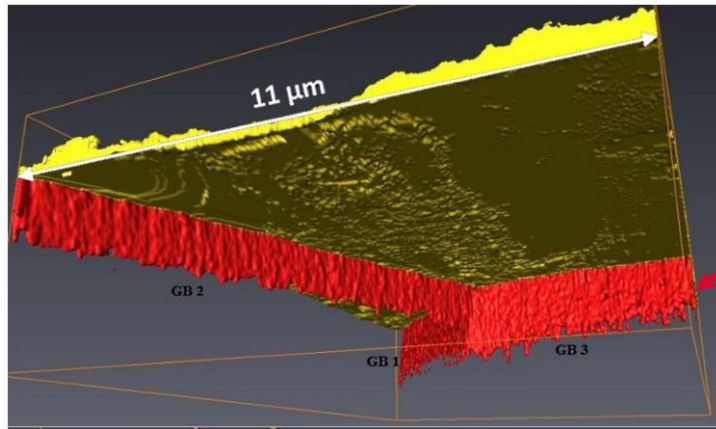


SEM image : large and thin IG oxidation

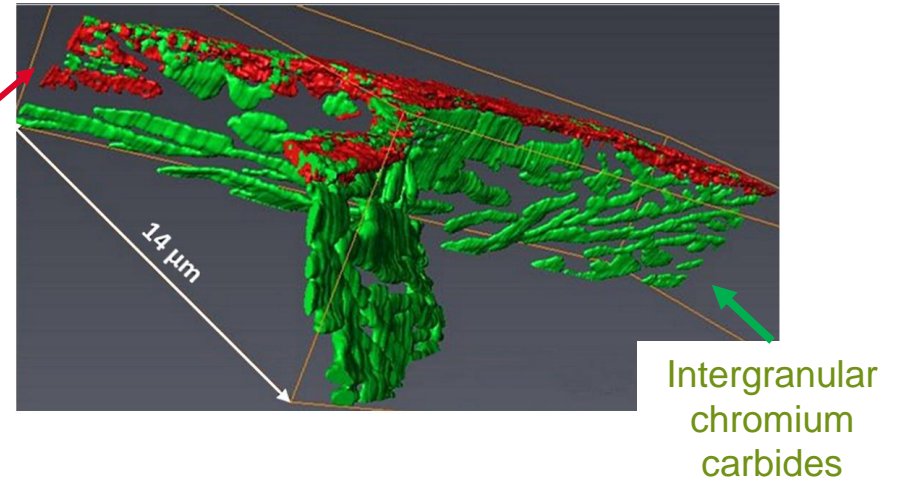
[Laghoutaris 2009]

[Gaslain et al. 2016]

No IG carbide

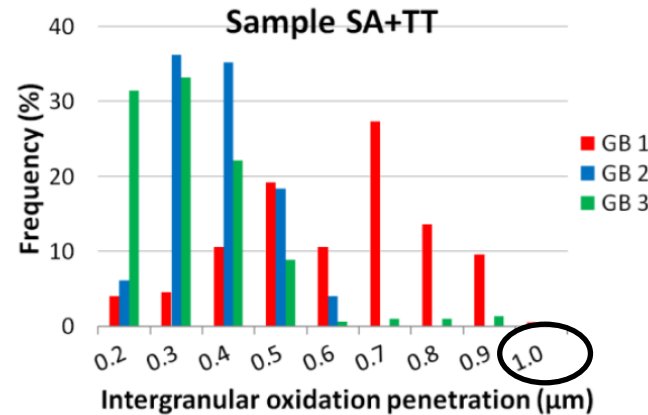
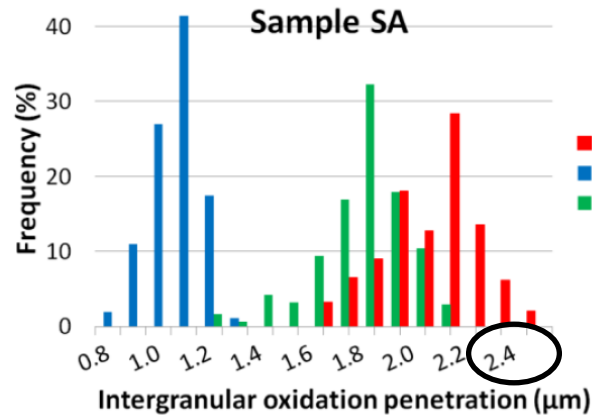


Alloy with IG precipitation



oxide

Intergranular chromium carbides



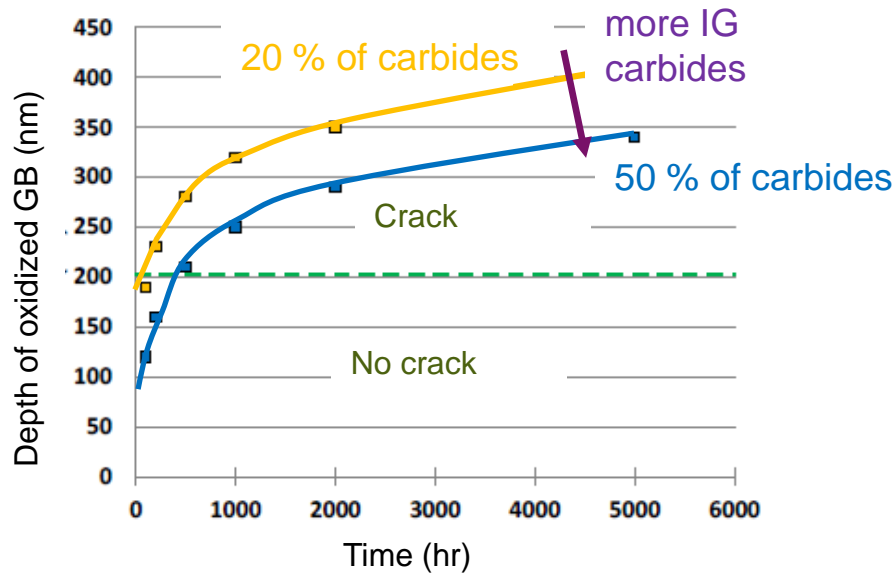
FIB/SEM image : the oxidation is deeper in the case of the alloy without any IG carbide

[Laghoutaris 2009]

[Gaslain et al. 2016]

Correlation between grain boundary oxidation and IG oxidation for A182

M. Wehbi EDF and MINES ParisTech (Env. Deg. 2015)

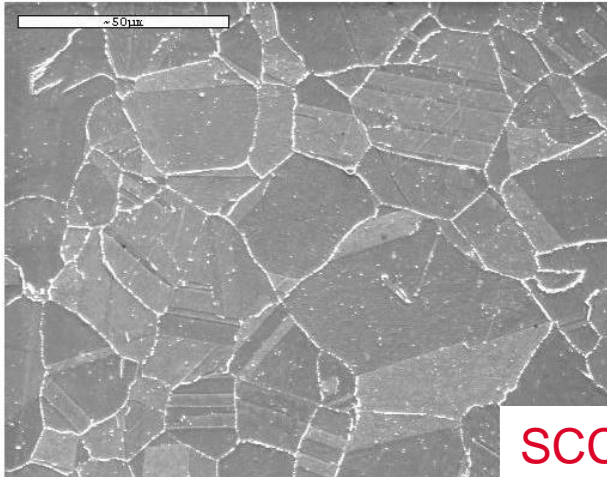


-> Critical intergranular oxidation : crack initiation

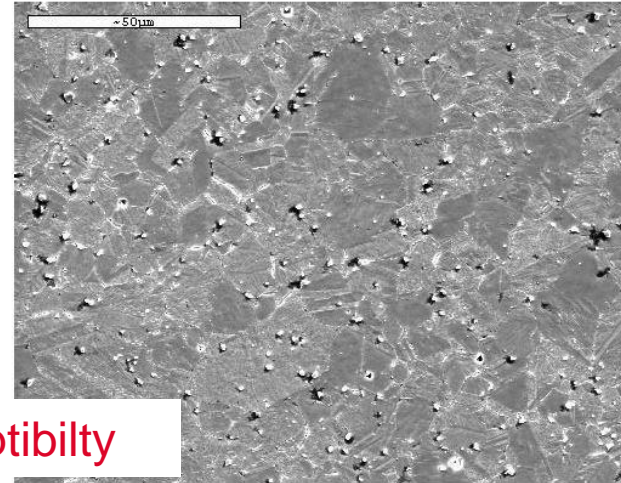
Cinetic law of intergranular oxidation for two grain boundary coverages (GBC) for a Ni weld at 320°C (from M. Wehbi, PhD thesis, Mines ParisTech, 2014)

A690 (industrial and experimental tubes)

WF771



WE092 (MA 1040°C – 1070°C)



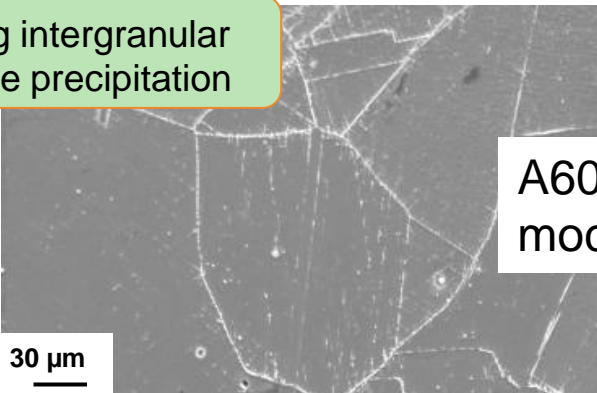
SCC susceptibility



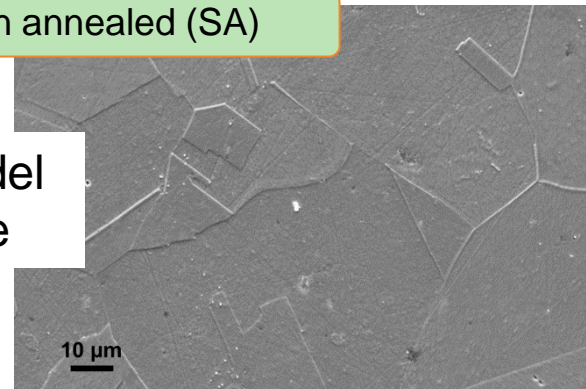
IG precipitation



Strong intergranular carbide precipitation



Solution annealed (SA)

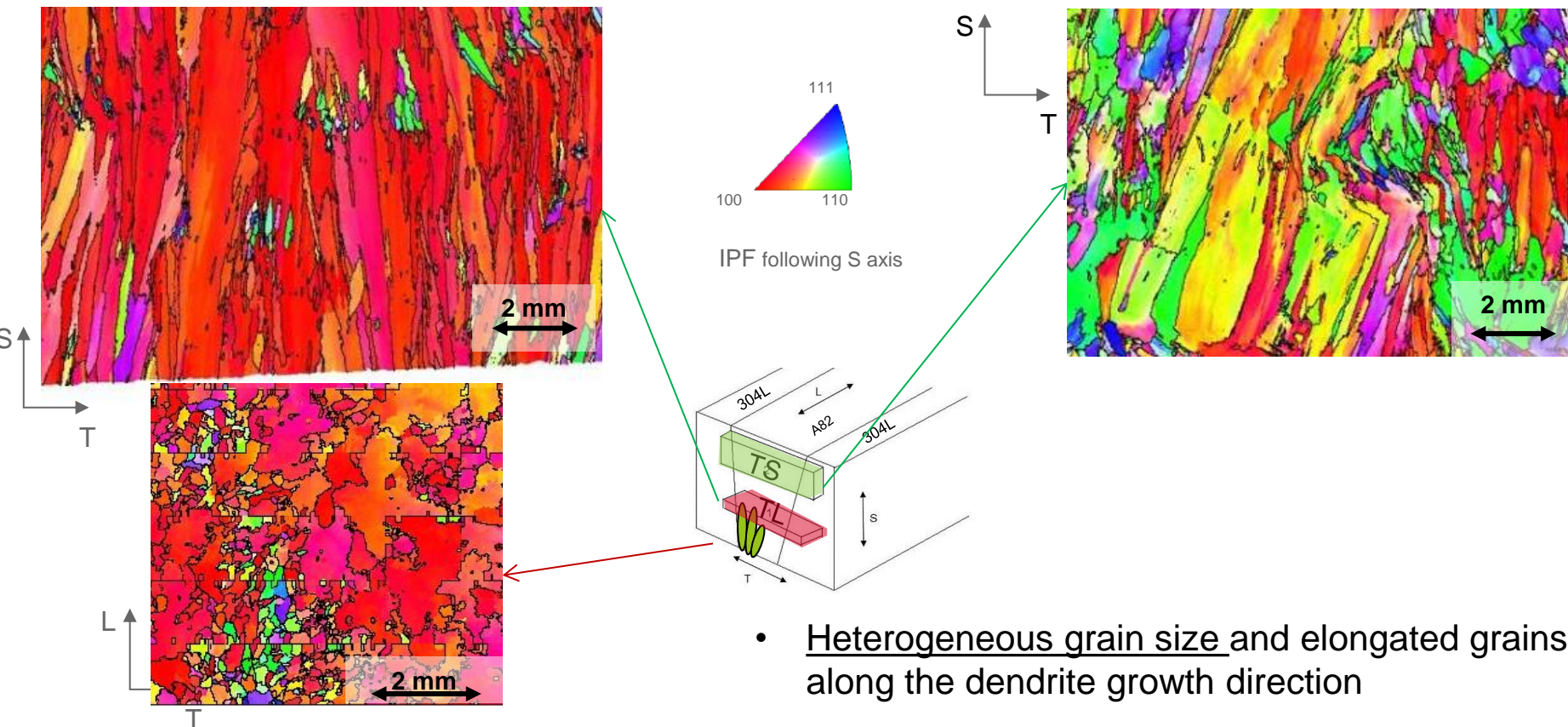


A600 with model microstructure

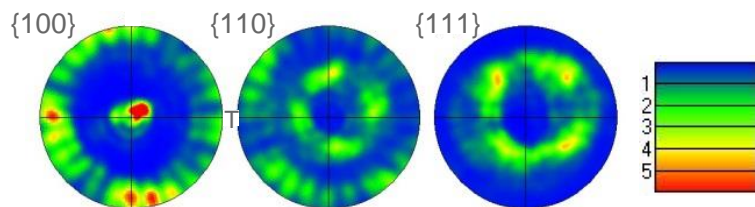
Nickel weld (Alloy 82) and SCC

Weld A as-welded

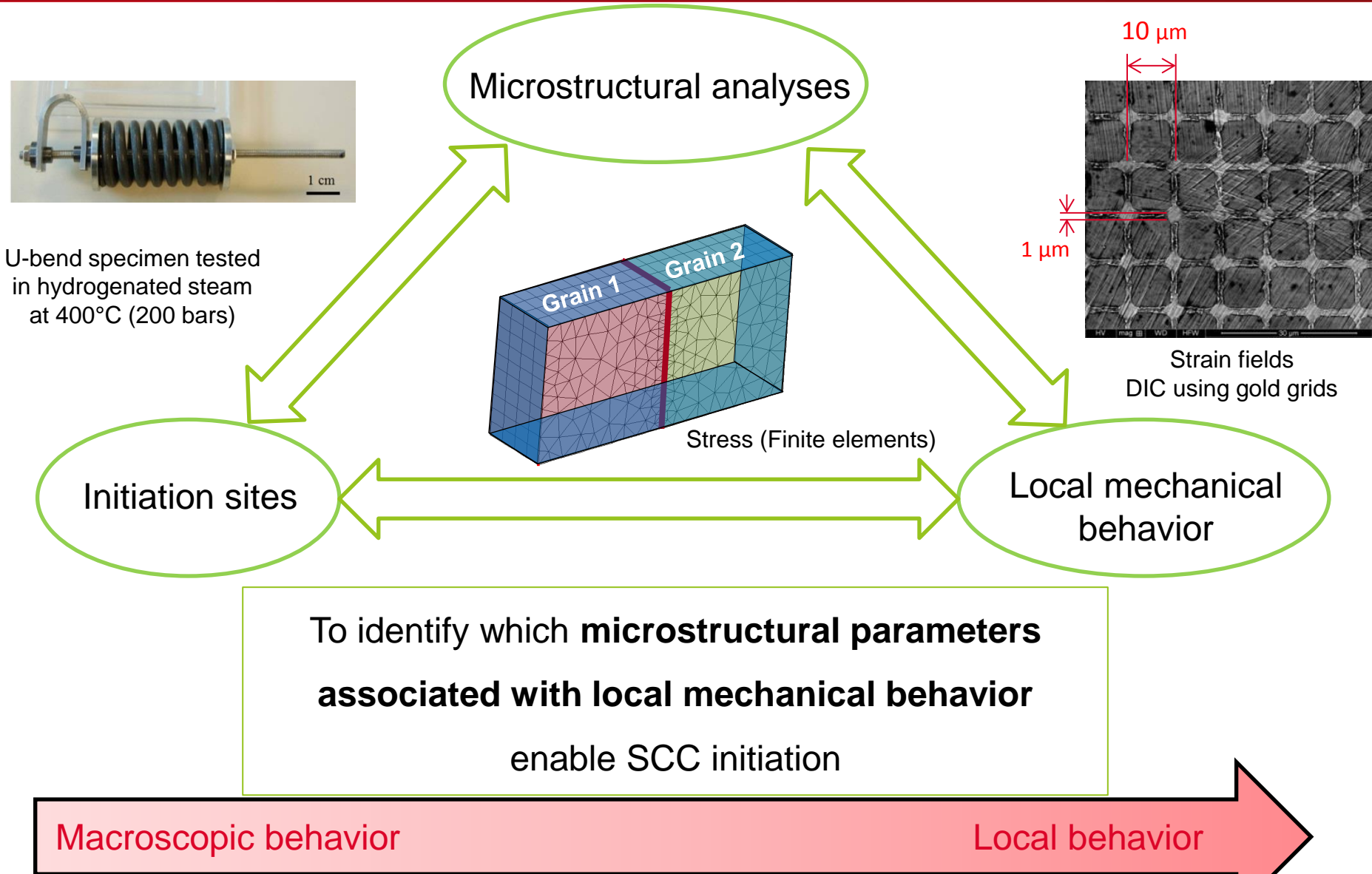
Weld B as welded



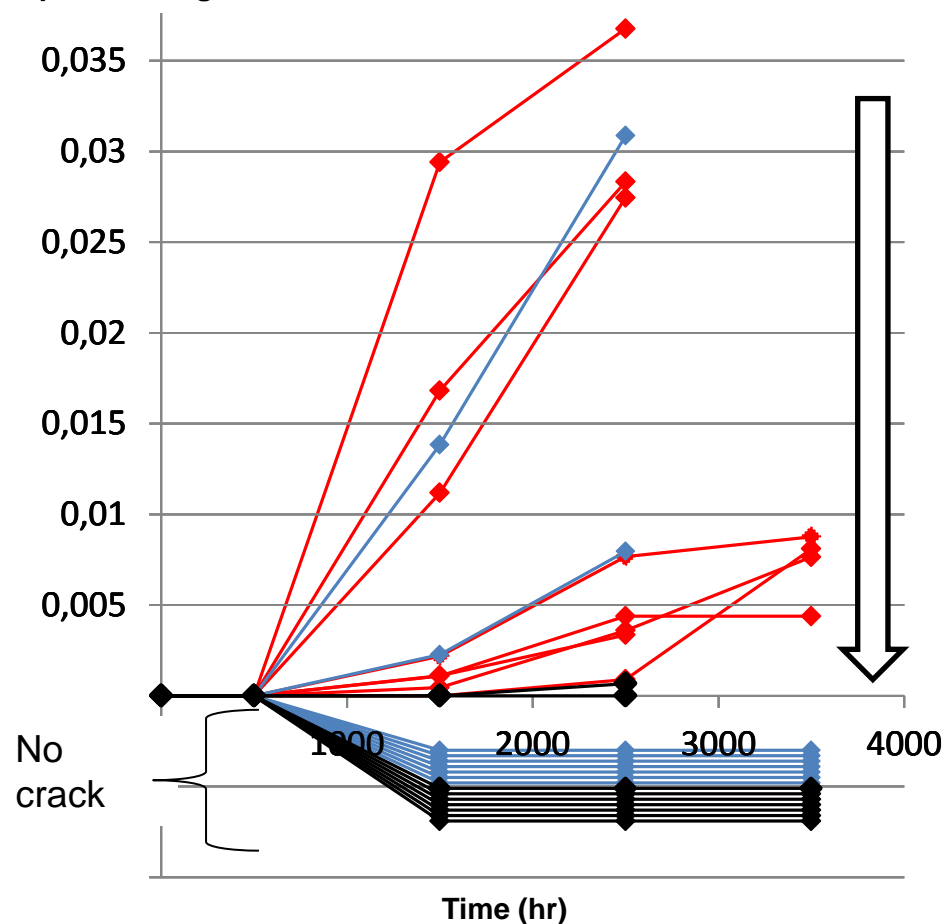
- Heterogeneous grain size and elongated grains along the dendrite growth direction
- Morphology and texture depends on the weld (on the welding process)
- Representative elementary volume close to 1 cm³



Approach



Number of cracks / number of perpendicular grain boundaries



— Alloy B/AW : A82 18%Cr, FCAW, as-welded
 — Alloy A/AW : A82 19%Cr, GTAW, as-welded
 — Alloy A/HT : A82 19%Cr, GTAW, heat-treated

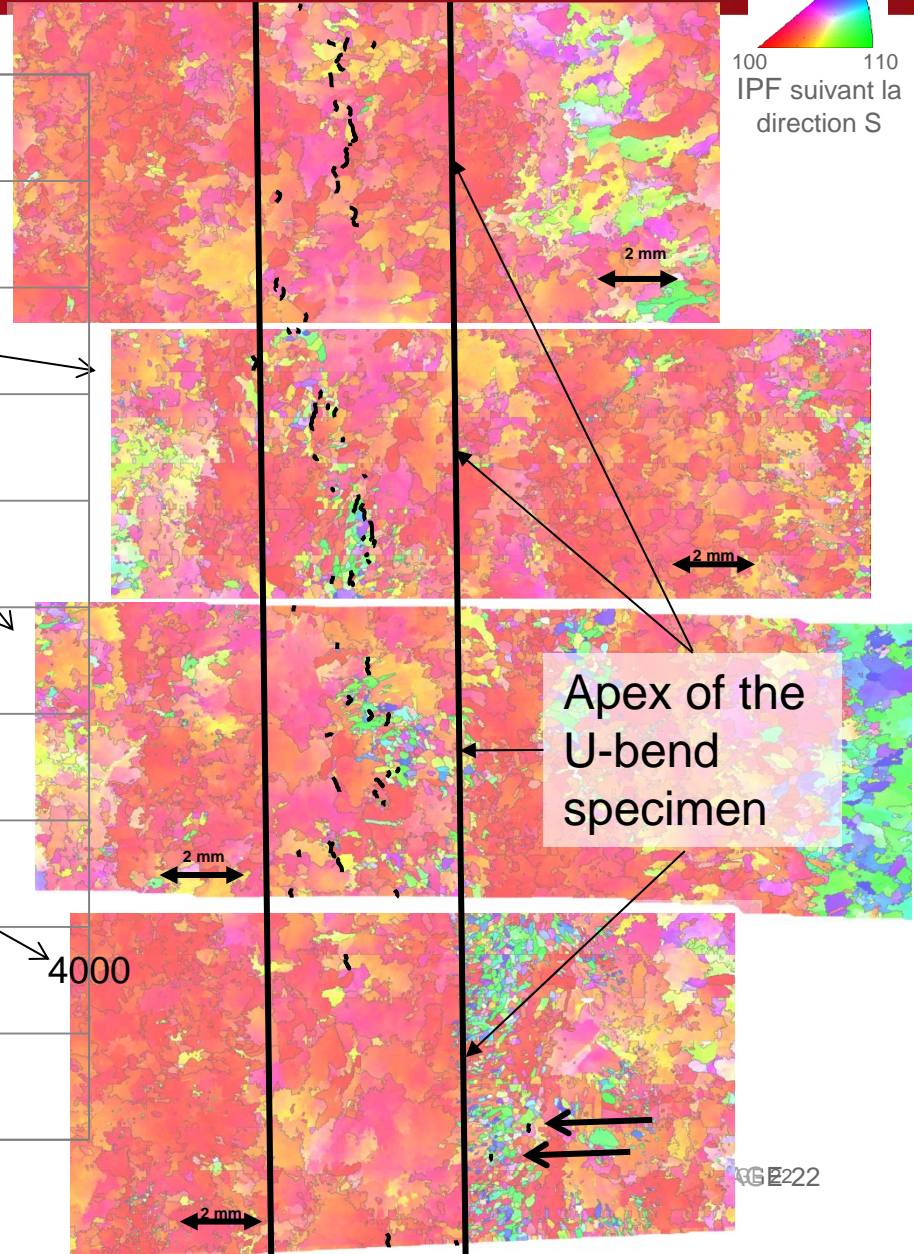
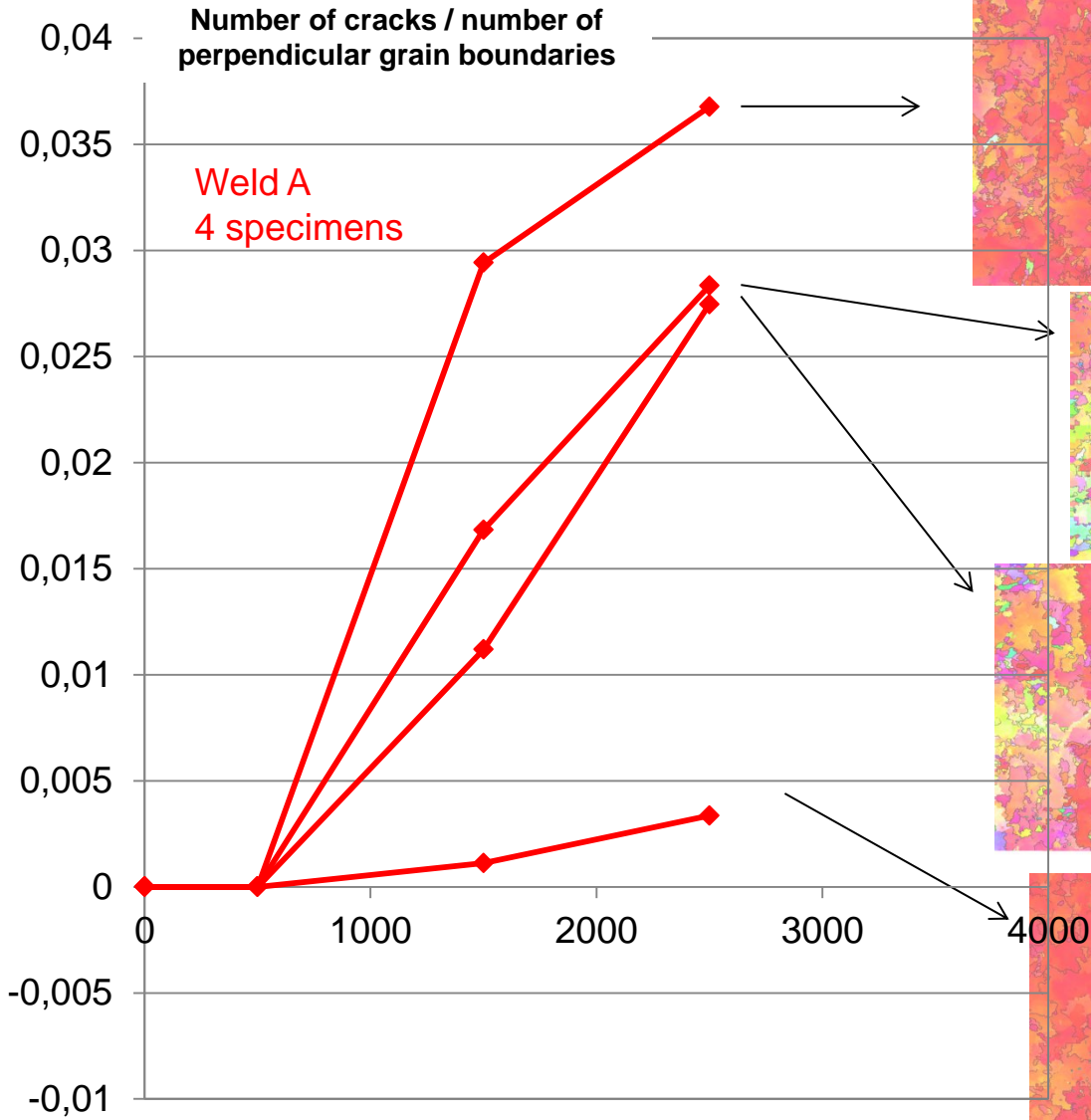
U-bends specimen tested in hydrogenated steam at 400°C (same mechanism as in PWR primary water but faster initiation)

- **Weld B/As Welded** is less susceptible to SCC than **weld A/As-Welded**

- **Weld B/Heat Treated** is less susceptible than **weld B/As-welded**

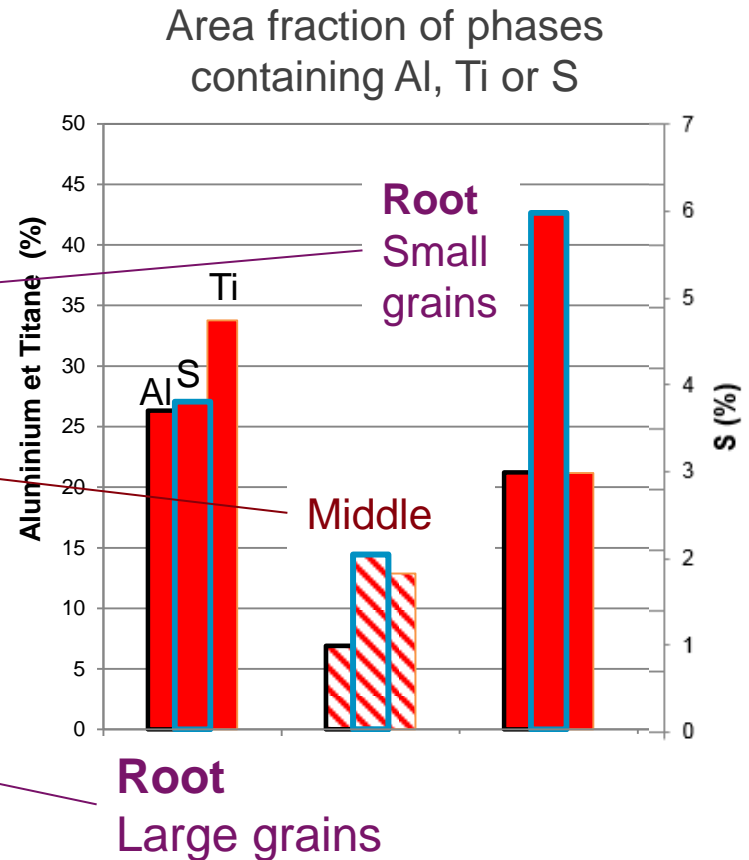
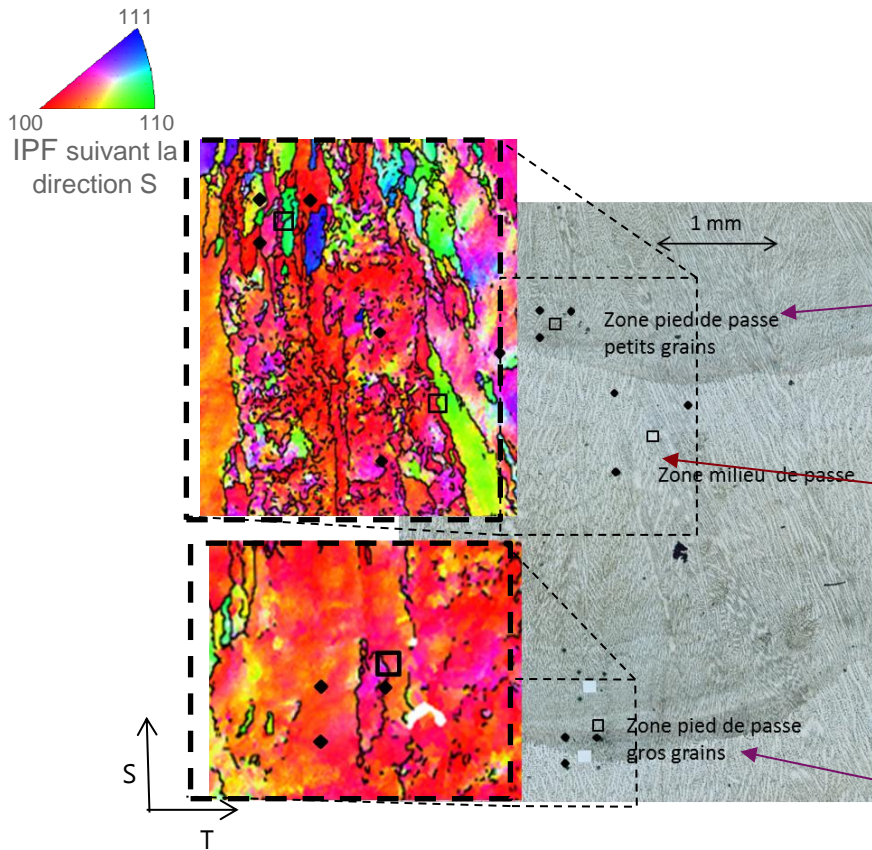
- “weld to weld” variability
- beneficial effect of the heat treatment assumed to be due to the formation of intergranular chromium carbides [Sennour2013]
- but some scattering for the same weld + specimen size versus representative elementary volume

Initiation tests : scattering



SIMS analysis at different locations in weld passes (weld A):

at the root of the weld pass (with small grains or large grains) and in the middle of the pass



More impurities at the roots of the weld passes (small grains or large grains).

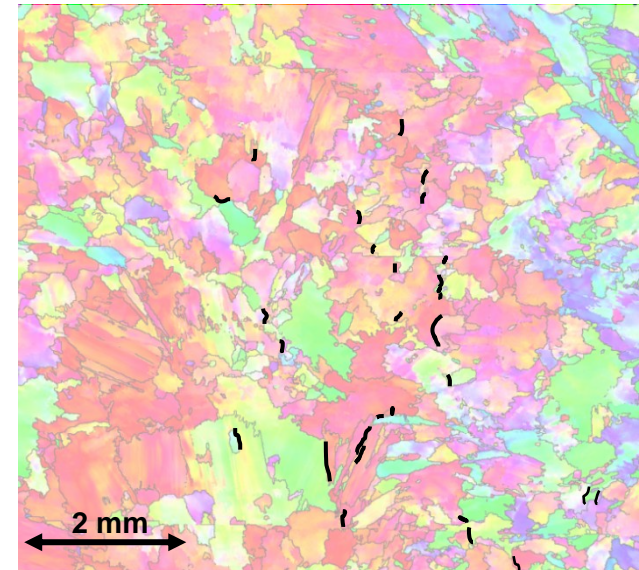
Depending of the welding process, the chemical composition or the thermal treatment

- more impurities can be found in some places in the welds (roots of the welds passes for instance)
- chromium carbides can precipitate in the grain boundaries
 - modify the grain boundary cohesion energy

But for the same chemistry and / or precipitation, not all the grain boundaries crack.

What about the mechanical fields ?

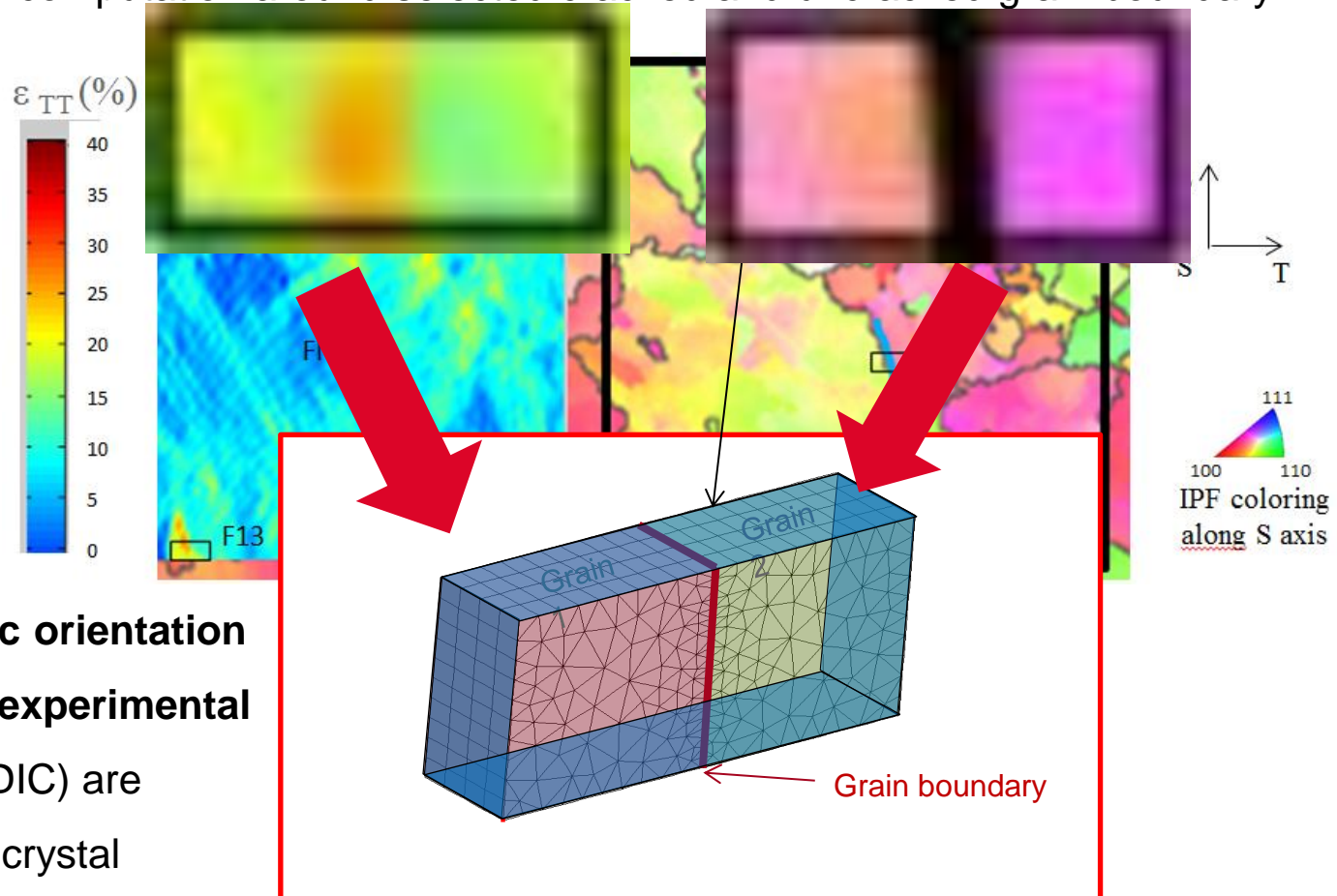
- Strain is not a sufficient parameter to model the SCC initiation behaviour [Chaumon et al., 2015] [Chaumon, 2016]
 - **Stress (finite elements analysis)**



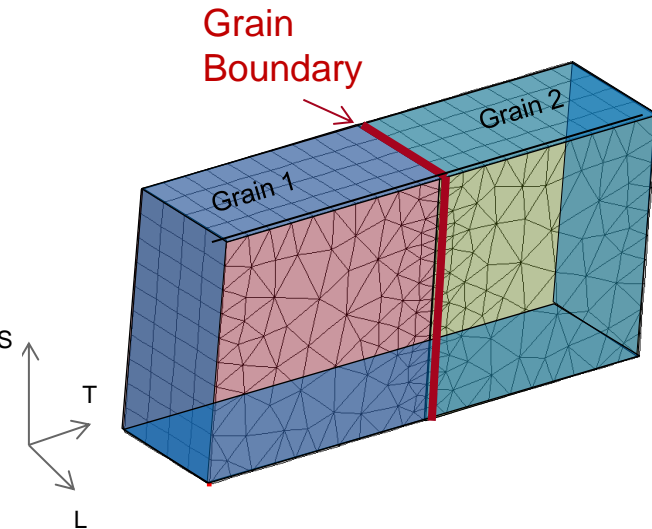


Finite elements computations

→ Finite elements computation around selected cracked and uncracked grain boundary



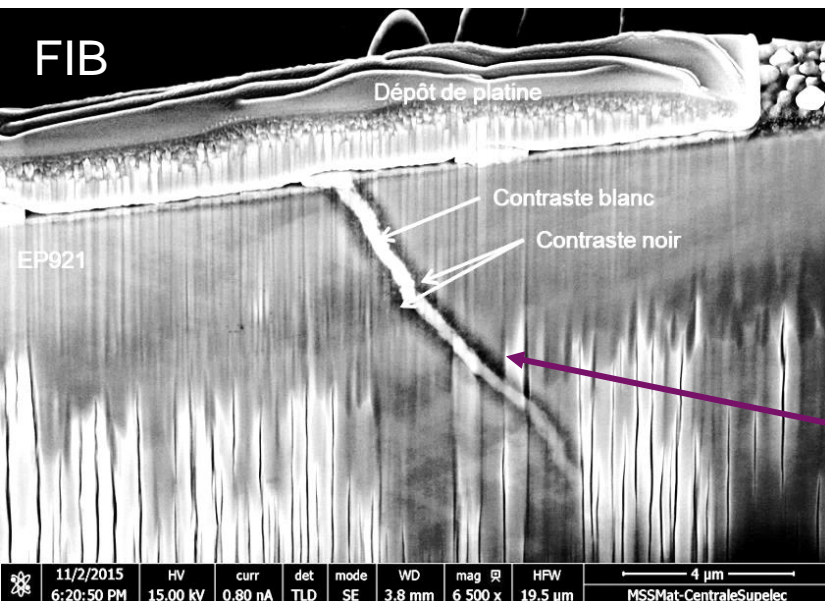
- **Crystallographic orientation** (EBSD) and the **experimental displacement** (DIC) are applied to the bi-crystal system.



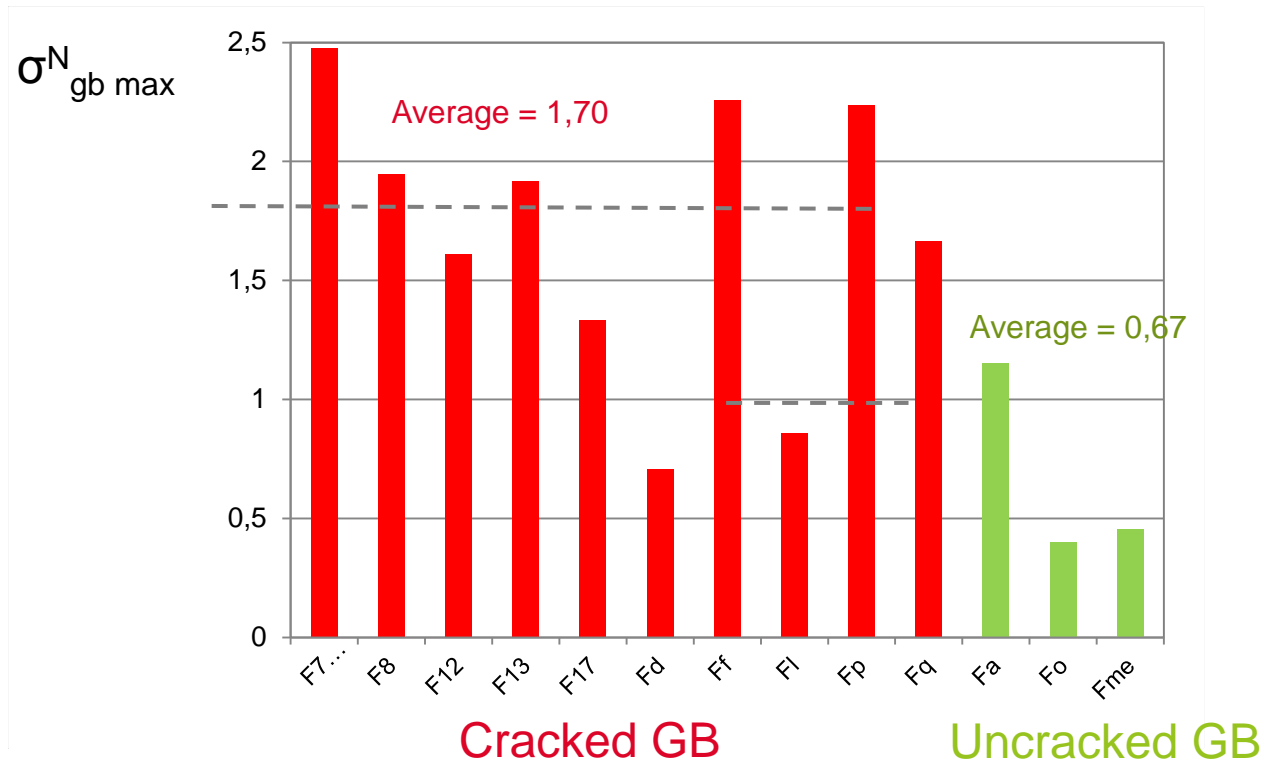
For selected cracked and uncracked grain boundaries (as observed by FIB)

Results :

- σ_{gb}^N = Maximum normal stress σ_{gb} normalized by the average normal stress of all computations



Crack initiation



→ The average of the normalized maximum normal stress is higher for the cracked GB than for the uncracked ones -> towards **an opening stress criteria**.

Macroscopic behavior

Alloy 82 is susceptible to SCC initiation in hydrogenated steam at 400°C.

Its susceptibility depends on the welding process, chemical composition and thermal treatment

- depending of the chemical composition, the heat treatment can induce intergranular chromium carbides formation that are beneficial.

The susceptibility depends on the location in the weld passes

- the roots of the weld passes can contain more impurities (correlation with the weld process and with the chemical composition).

The susceptibility depends on the GB binding energy which depends on the GB chemistry, on the strain discrepancy [Wehbi2014], on the precipitation, ...

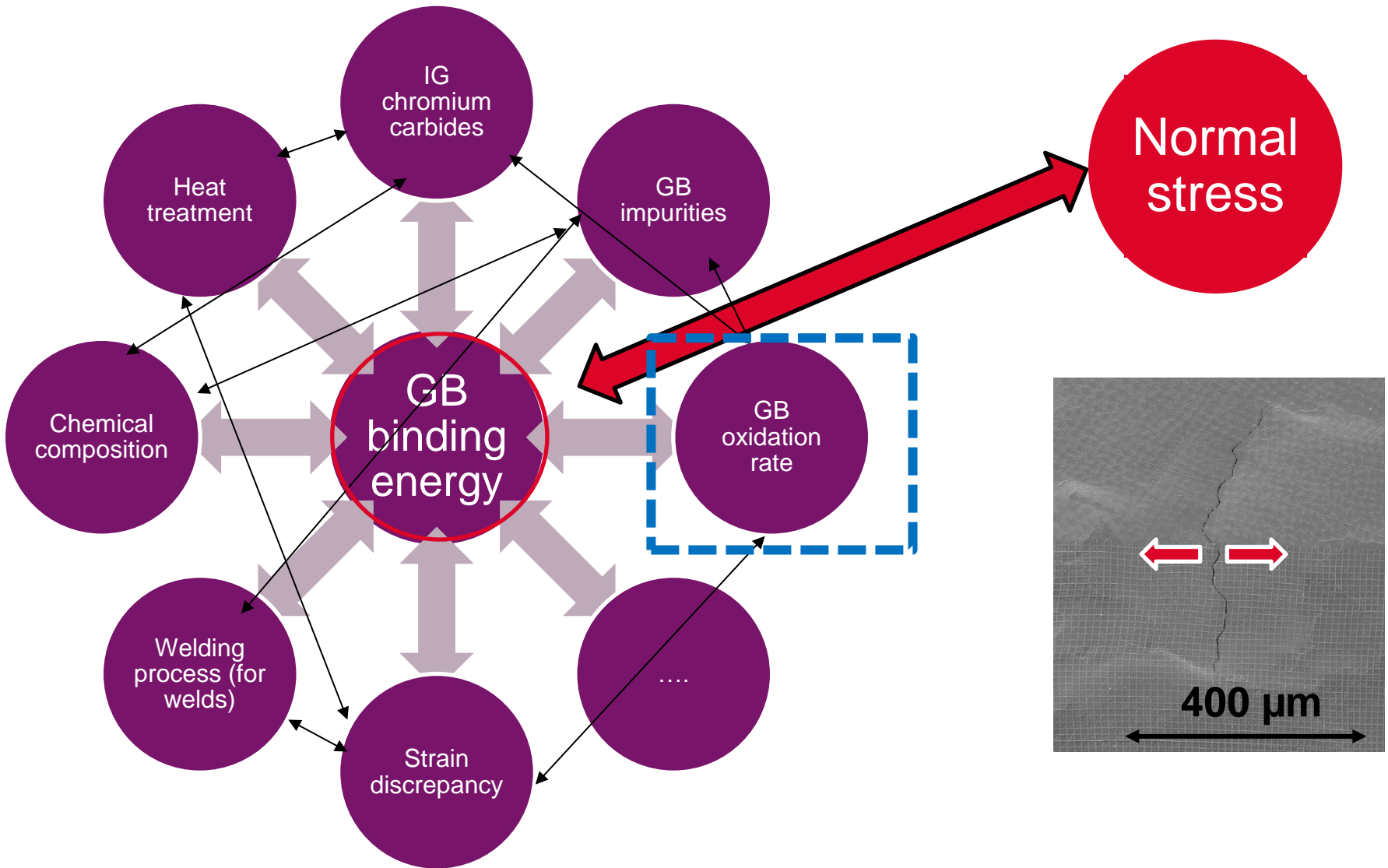
The susceptibility can not be explained by only one parameter
→ **BUT a coupling of parameters**

Tend to a **initiation criterion** =

mechanical behavior (maximal normal stress and deformation discrepancy) +
chemical parameter (intergranular oxide, grain boundary cohesion energy...)

Local behavior

SCC of nickal alloy : conclusions



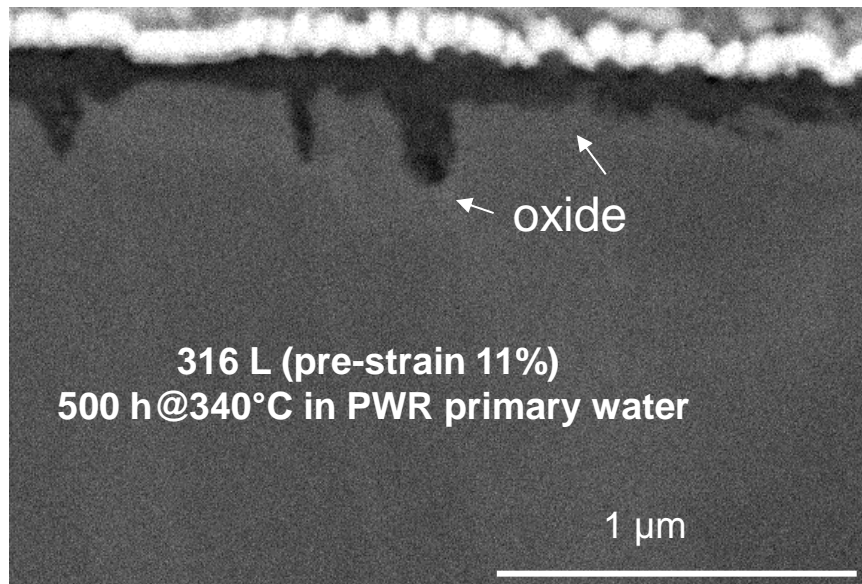
To improve the modelling, the work in progress deals with :

- A better understanding of the **oxidation of the grain boundaries** (with or without intergranular carbides known to be of great importance for the SCC behavior, depleted grain boundary)
- As **strain** is not sufficient, **stress maps** must be known (finite element analysis is therefore needed).

The local applied stress must be compared to the binding energy of the grain boundary.

In the frame of the extension of the operating life of the PWR in France, a better understanding of the SCC mechanism for Alloy 600 will permit :

- **to confirm the good behavior of Alloy 690 and its weld metals**
- **to model the SCC behavior of the remaining components in Alloy 600 (and its weld metals)**



Work in progress in
collaboration with IRSN

[Chaumon 2016] PhD thesis, Mines ParisTech, 2016

[Gaslain et al. 2016] F O M Gaslain, H T Le, C Duhamel, C Guerre and P Laghoutaris, The role of intergranular chromium carbides on intergranular oxidation of nickel based alloys in pressurized water reactors primary water, EMAS 2015 Workshop IOP Publishing IOP Conf. Series: Materials Science and Engineering 109 (2016) 012004 doi:10.1088/1757-899X/109/1/012004

[Laghoutaris 2009], PhD thesis, Mines ParisTech, 2009

[Sennour et al. 2009] Advanced TEM characterization of stress corrosion cracking of Alloy 600 in pressurized water reactor primary water environment. M. Sennour, P. Laghoutaris, C. Guerre and R. Molins 2009 J. Nucl. Mater. 393 254-266

[Sennour et al. 2013] M. Sennour, E. Chaumon, b, J. Crépin, C. Duhamel, F. Gaslain, C. Guerre, I. de Curières, TEM investigations on the effect of chromium content and of stress relief treatment on precipitation in Alloy 82, Journal of Nuclear Materials, Volume 442, Issues 1–3, November 2013, Pages 262–269

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des matériaux dans leur environnement
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