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

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DE LA RECHERCHE À L'INDUSTRIE

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

Catherine GUERRE

CEA and MINES ParisTech : E. Chaumun, P. Laghoutaris
CEA : O. Raquet
Mines ParisTech: J. Crépin, C. Duhamel, M. Sennour, F. Gaslain, H.T. Le
Ecole Polytechnique: E. Héripré
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

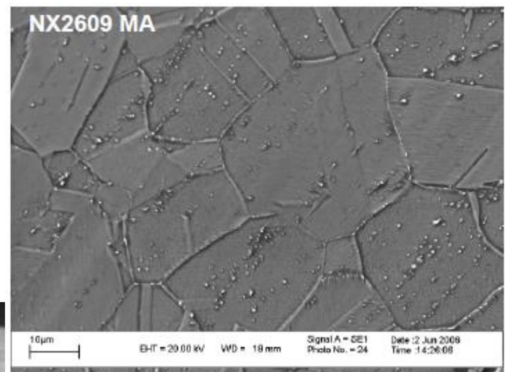
Nickel base alloys

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

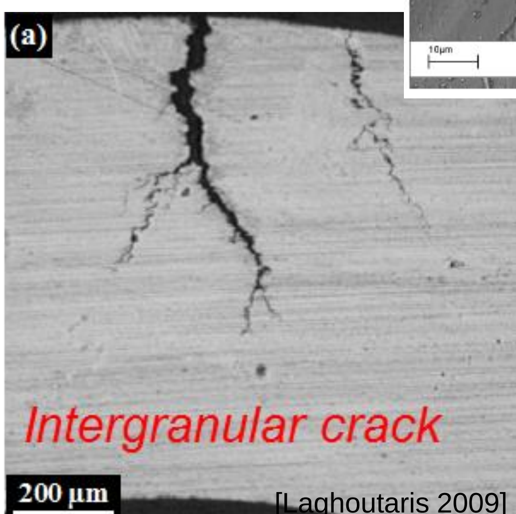
- **Alloy 690 TT** (Ni-30Cr-10Fe) and its weld metals (Alloy 52 and Alloy 152)

Less susceptible to SCC

<u>intergranular and oxidized cracks</u>



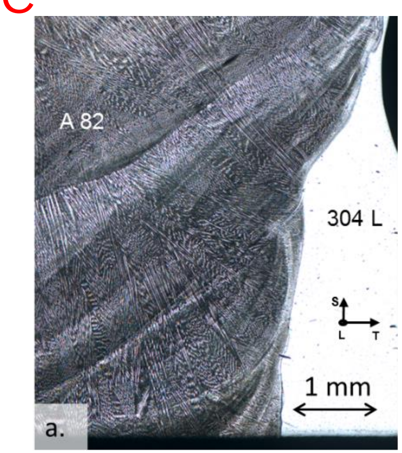
Alloy 600 MA

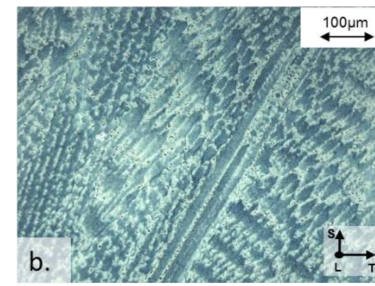


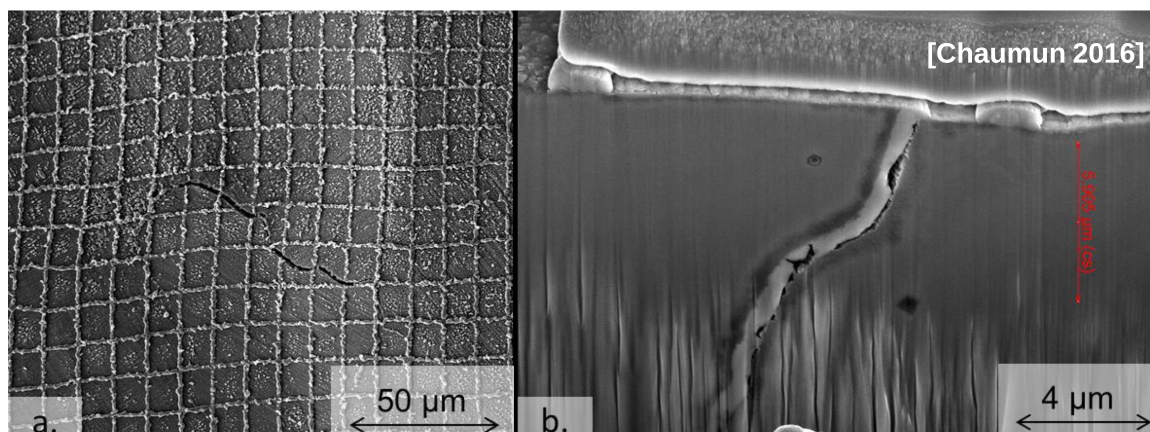
Alloy 600, U-bend 1200 hr @ 325°C

Alloy 82

[Chaumun 2016]







Alloy 82, U-bend, 2500 hr @ 400°C

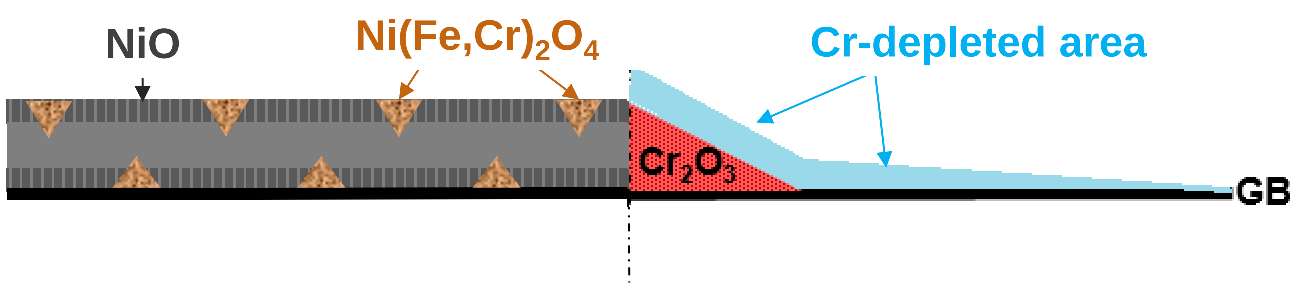
Introduction

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)

DE LA RECHERCHE À L'INDUSTRIE

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

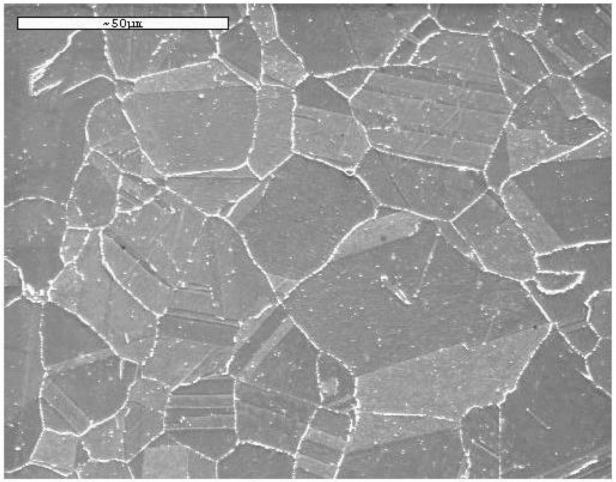
SCC initiation of Alloy 690

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

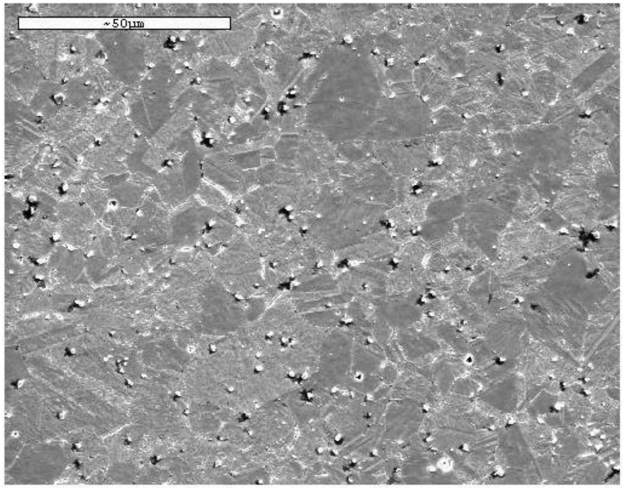
- 19 experimental tubes, (MA (980°C<T<1040°C, NH3 or H2) + TT 700°C)
  - some with intergranular carbides
  - others with inter + intra carbides, among them **WE092** and **WE094** with a 2 steps HT

WF771



Attaque brome/méthanol – observation MEB

WE092 (MA 1040°C – 1070°C)



Attaque brome/méthanol – observation MEB

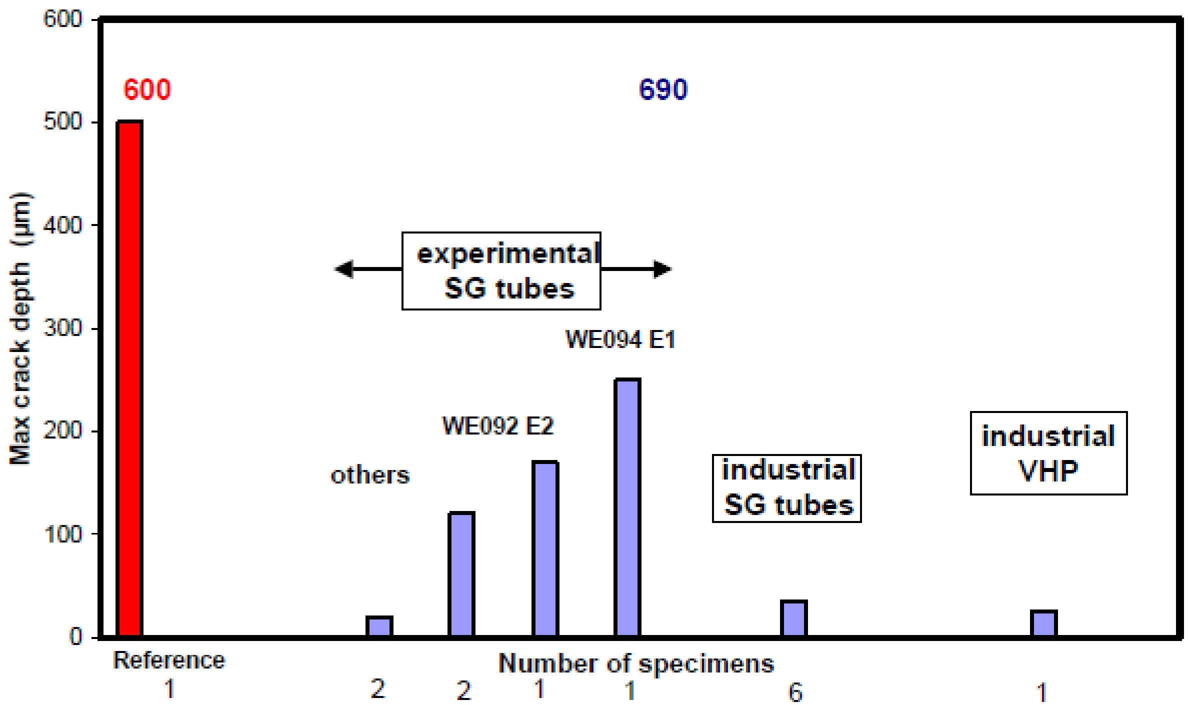
F. Vaillant et al., published in 2005 International PWSCC of Alloy 600 Conference and Exhibit Show (MRP-154)

SCC initiation of Alloy 690

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

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



SCC initiation of Alloy 690 : conclusion

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 inititiation (GB oxidation)

Alloy 600 with model microstructures

Model microstructure alloys

- Alloy 600 heat WF675

(very susceptible to SCC in PW)

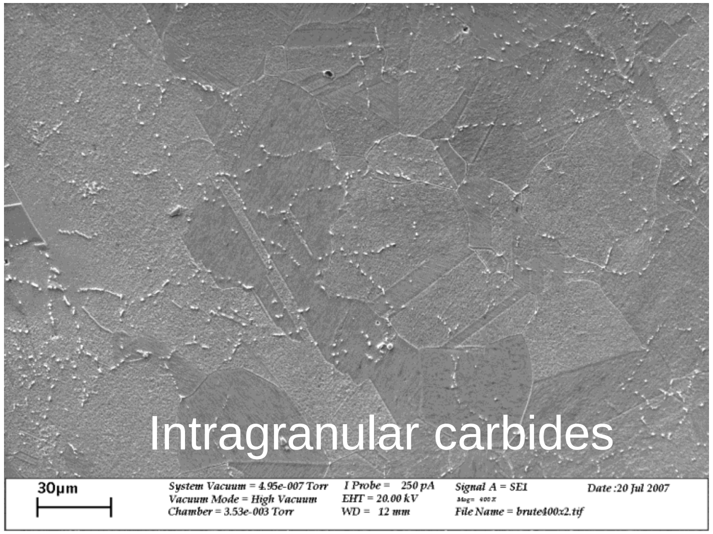
- Heat treatment in vacuum :
  - Solution annealed + water quenching

    -> no carbide
  - Solution annealed + air cooling

    -> low intergranualar carbide precipitation
  - Solution annealed + water coolling + 16 hr at 700 °C

    -> high intergranular carbide precipitation

WF675

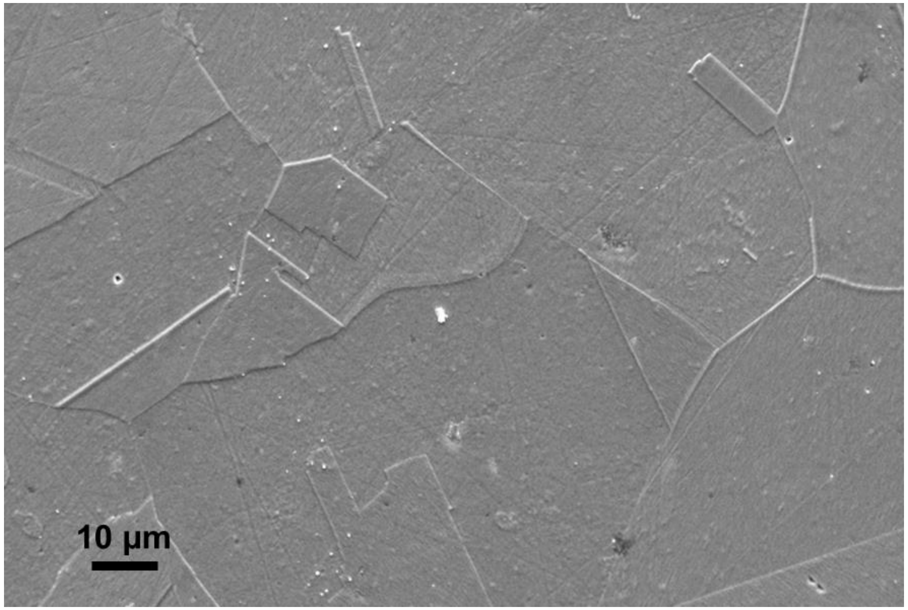


- No CW or CW up to 50 % (250 Hv to app. 320 Hv)

P. Laghoutaris, thesis

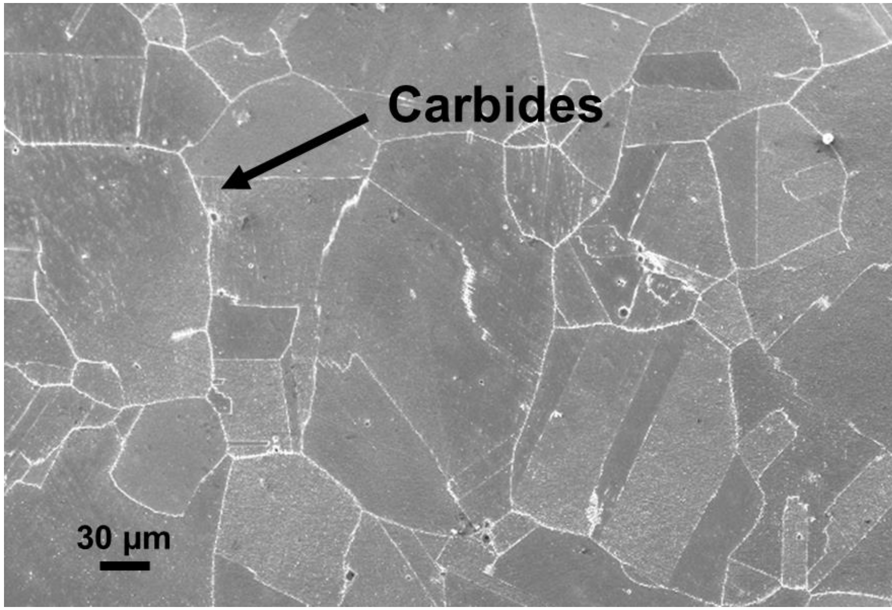
Alloy 600 with model microstructures

Sample SA "Solution Annealed"
No carbide



1050 ºC for 1 h + water quenching

High IG precipitation

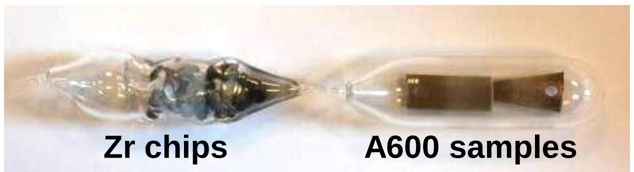


1050 ºC for 1 h + water quenching
+ tempering at 700 ºC for 16 h

Alloy 600 with model microstructures

Model microstructure

Alloy 600

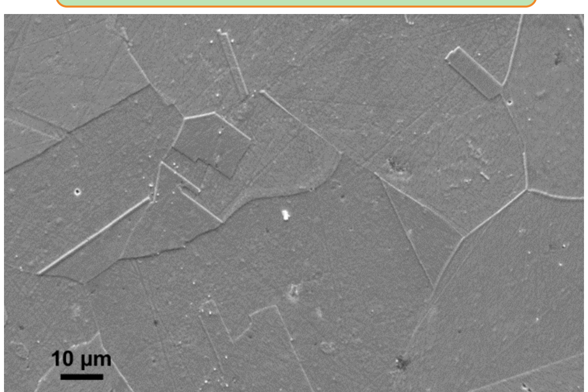


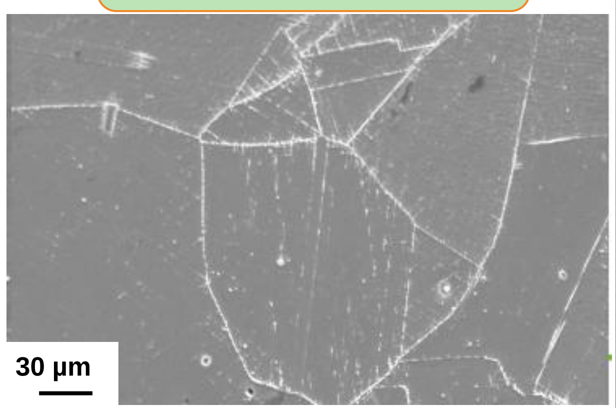
1050°C – 1h
water quenching

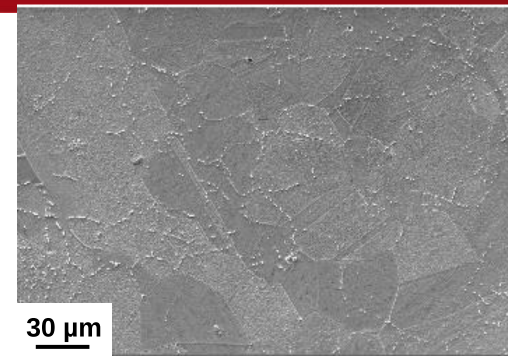
ageing
700°C – 16h

Solution annealed (SA)

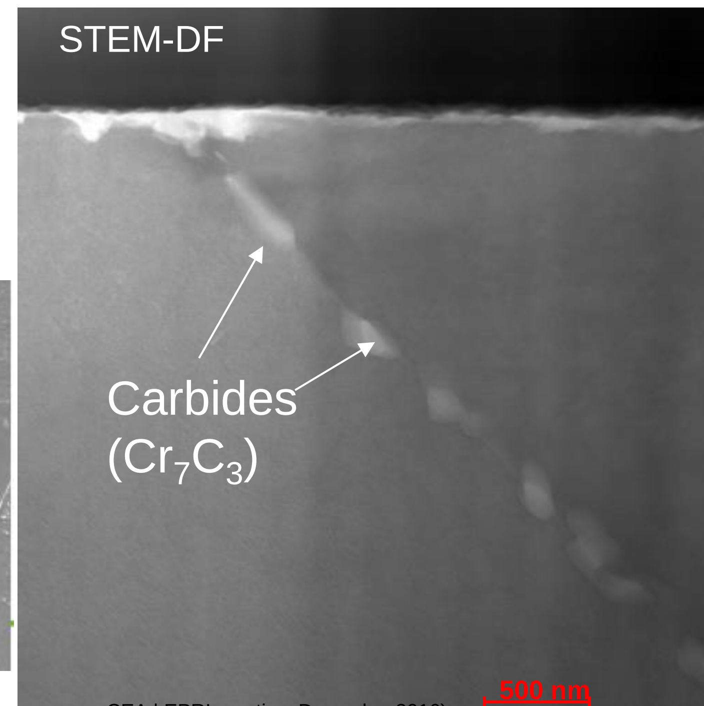
Strong intergranular carbide precipitation







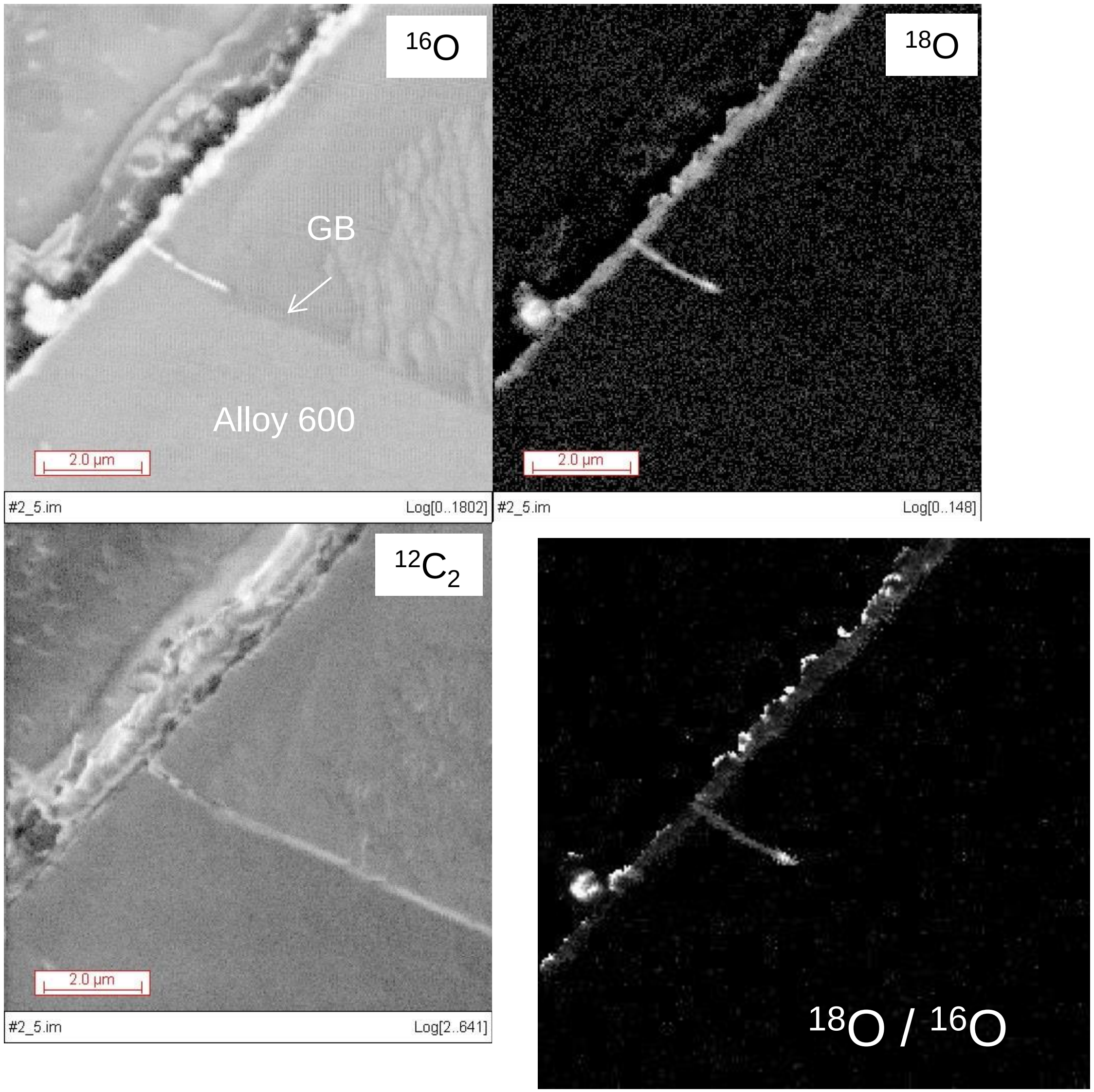
Heat WF675
Susceptible to SCC



P. Laghoutaris, thesis

Alloy 600 with model microstructures

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

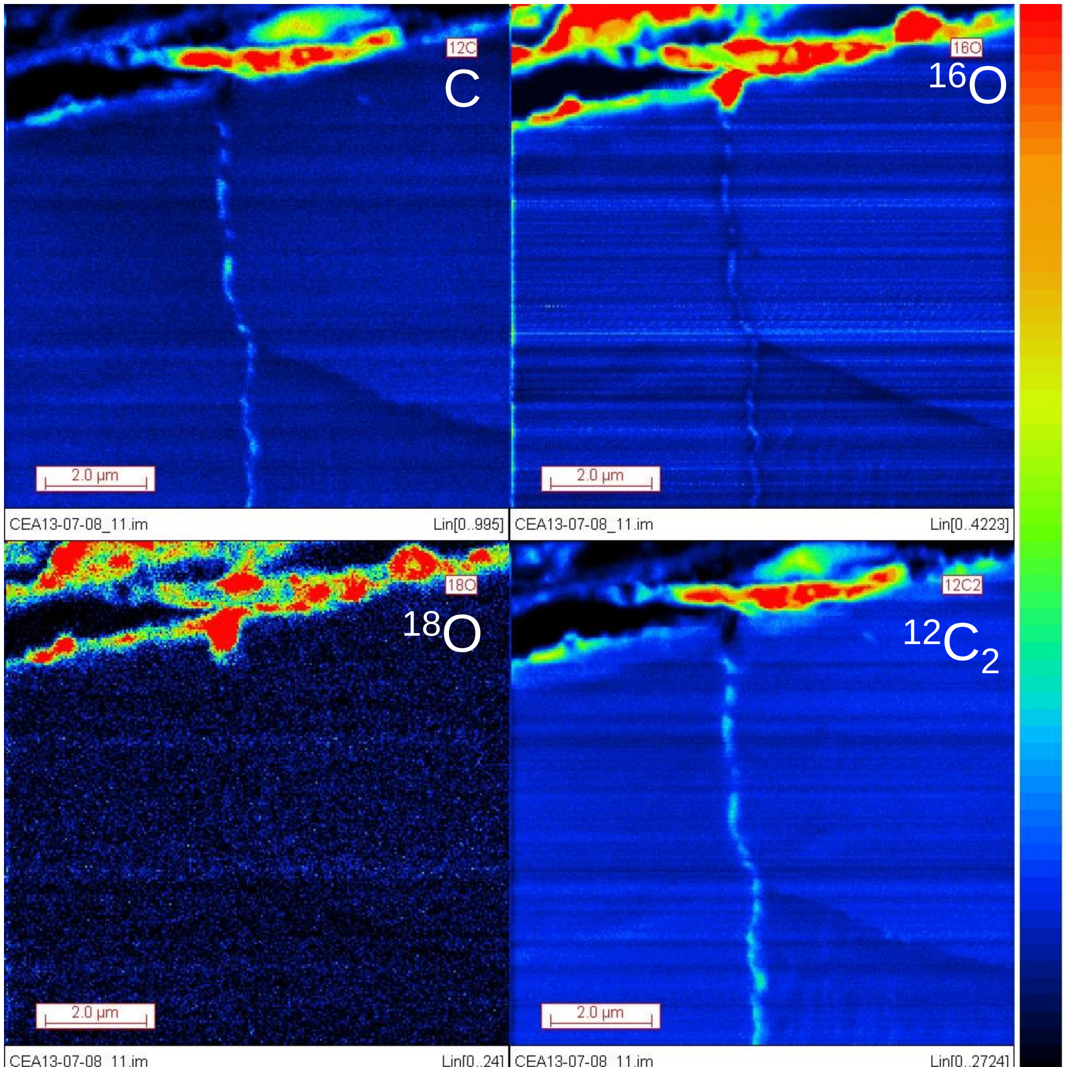
-> ^{18}O transport during 67 hr up to the tip of the oxidized grain boundary

-> long and thin IGA

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

Alloy 600 with model microstructures

Alloy with IG carbide precipitation

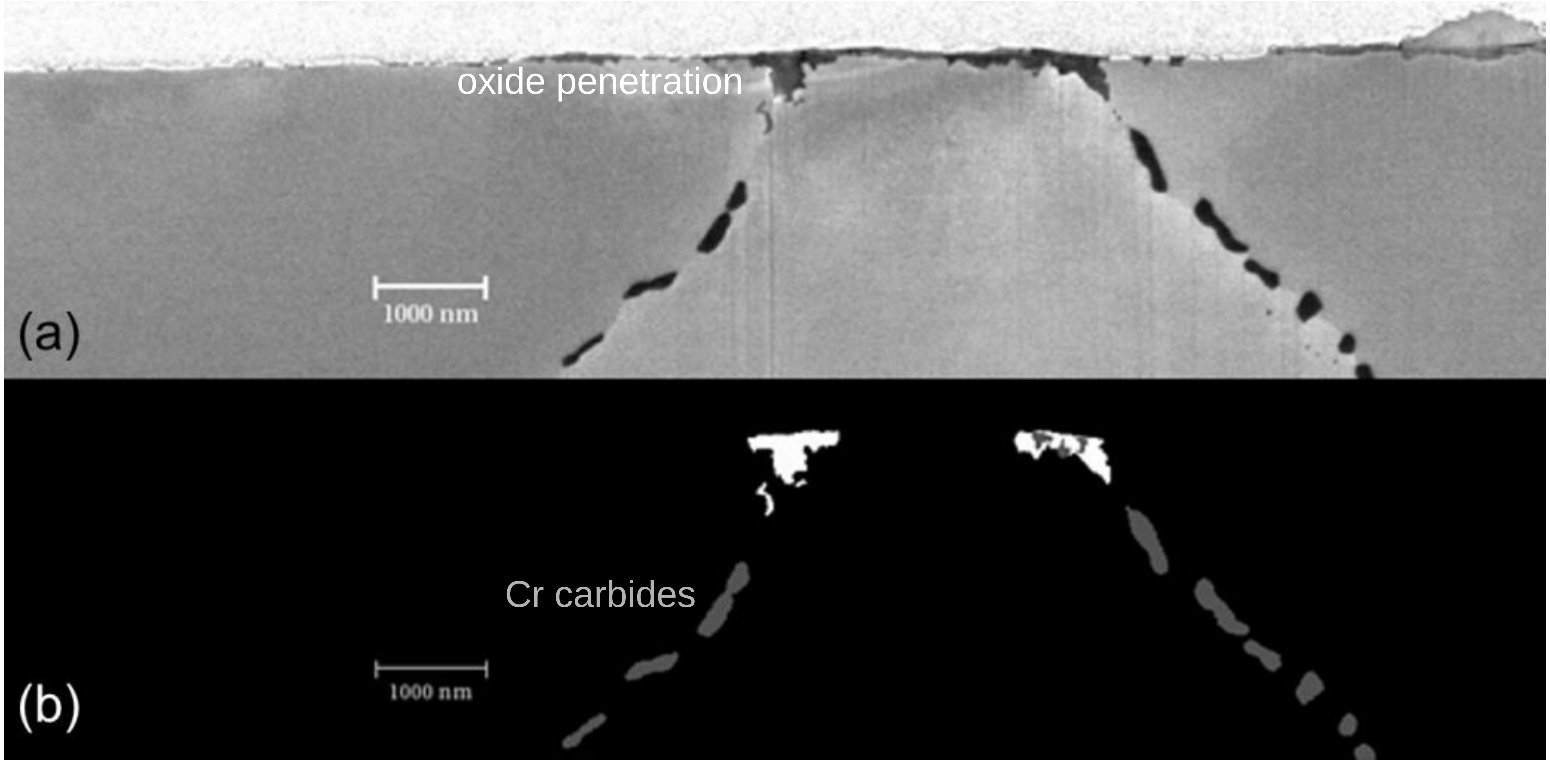


Nano-SIMS analysis
Same exposure conditions.

-> large and short IGA

Alloy 600 with model microstructures

Alloy with IG carbide precipitation



SEM image : large and thin IG oxidation

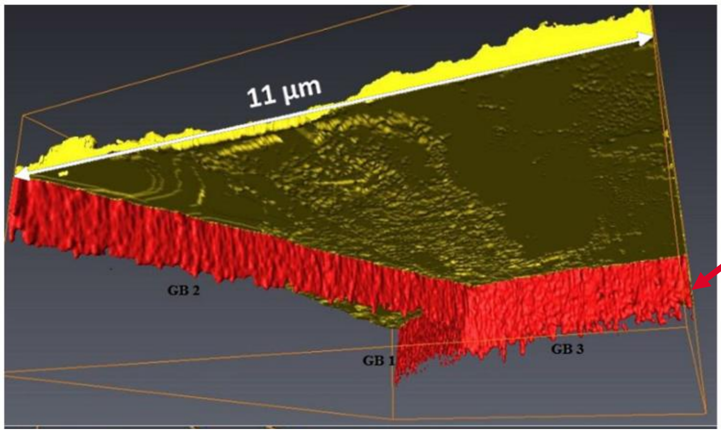
[Laghoutaris 2009]

[Gaslain et al. 2016]

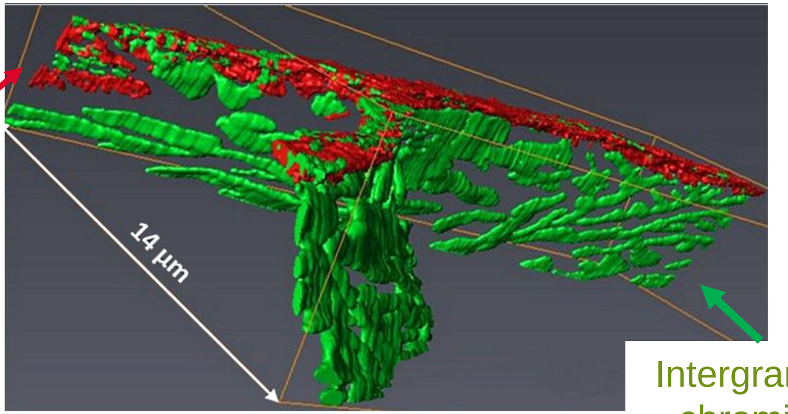
Alloy 600 with model microstructures ….

No IG carbide

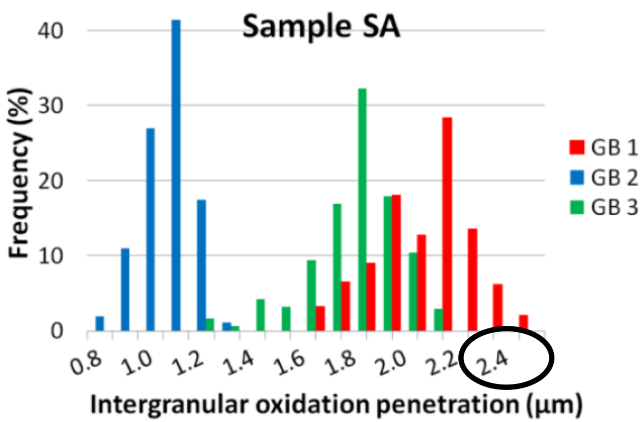
Alloy with IG precipitation

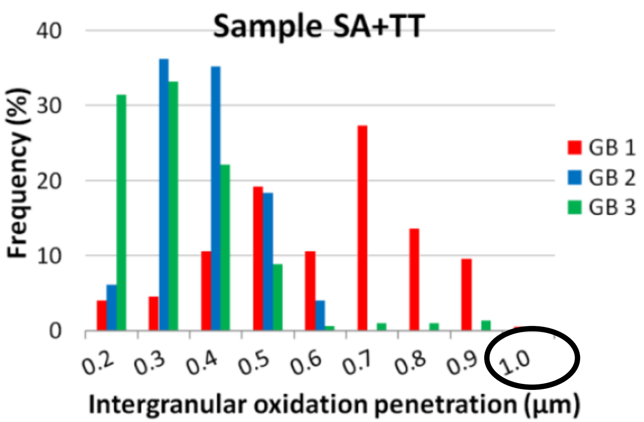


oxide



Intergranular chromium carbides





[Laghoutaris 2009]

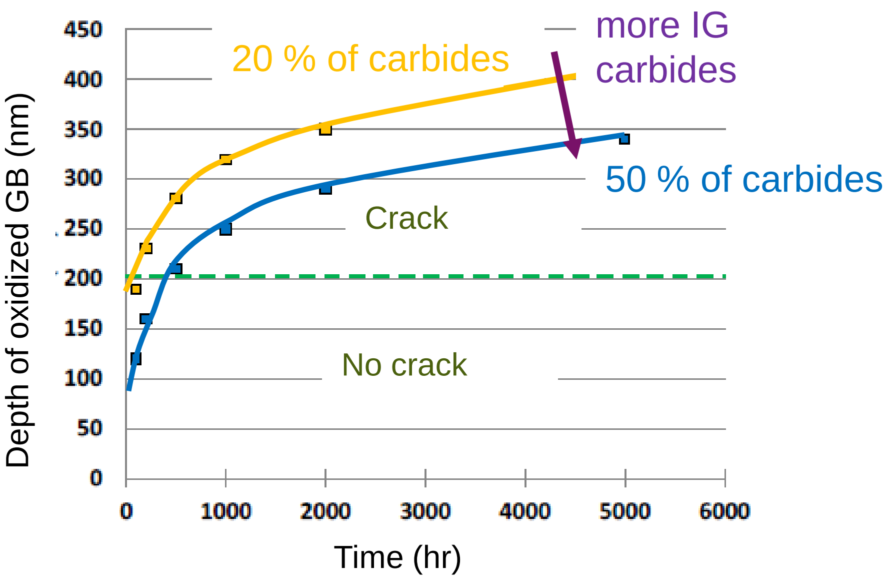
[Gaslain et al. 2016]

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

Alloy 182 : IG precipitation and oxidation

Correlation between grain boundary oxidation and IG oxidation <u>for A182</u>

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



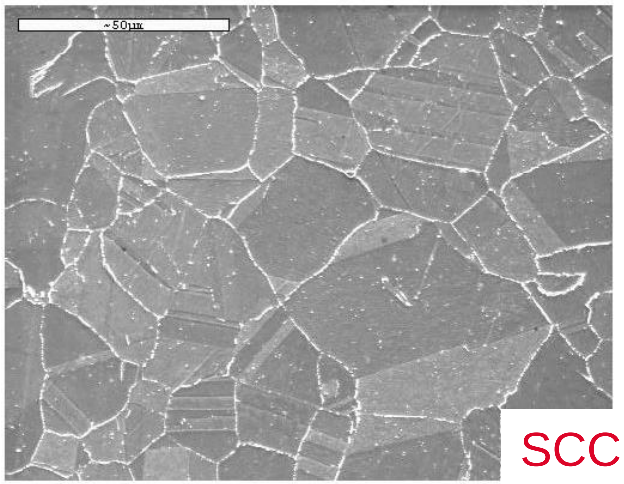
-> Critical intergranular oxidation : crack initiation

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)

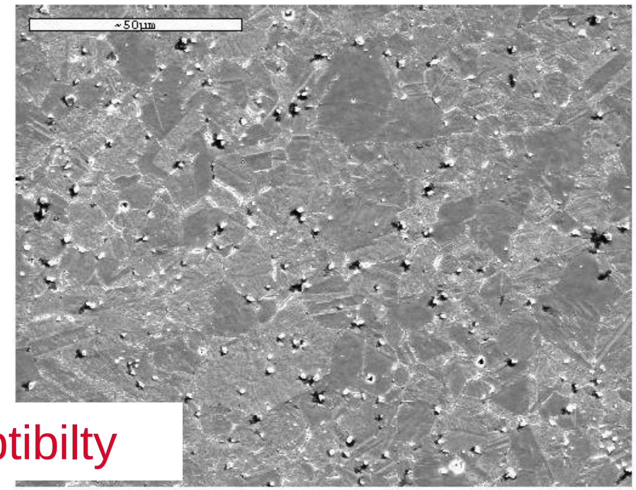
A690 microstructure

A690 (industrial and experimental tubes)

WF771



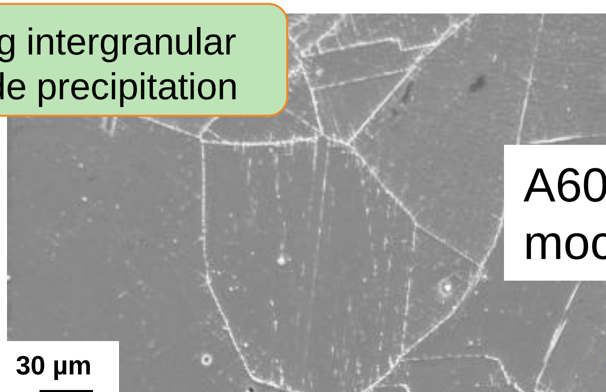
WE092 (MA 1040°C – 1070°C)



SCC susceptibilty

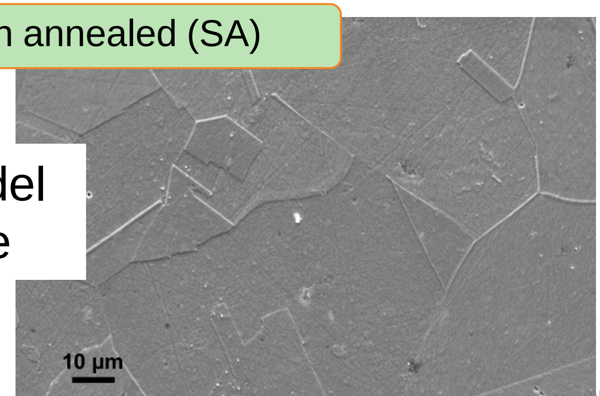
IG precipitation

Strong intergranular carbide precipitation



A600 with model mocrostructure

Solution annealed (SA)

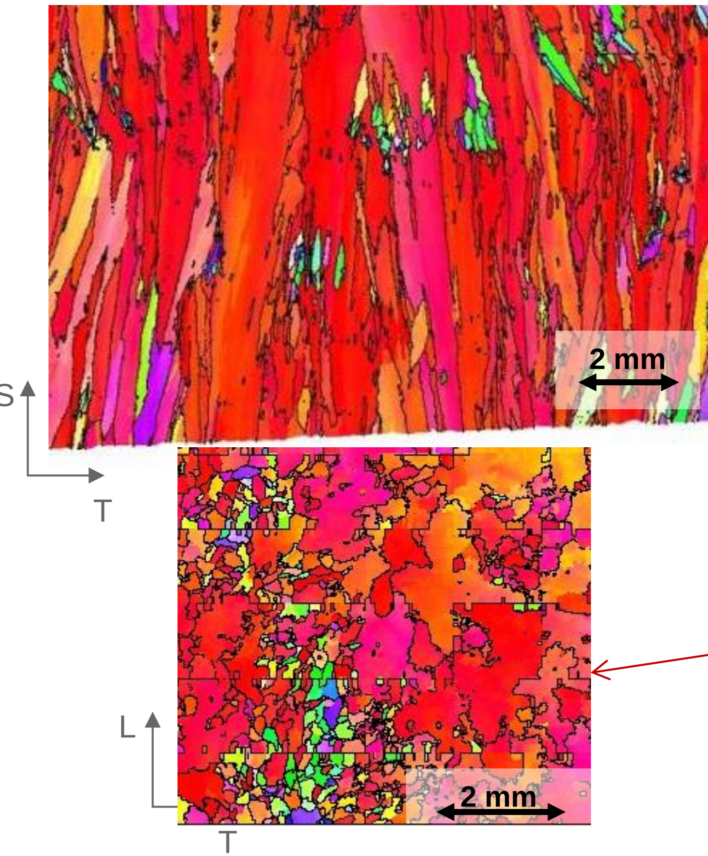


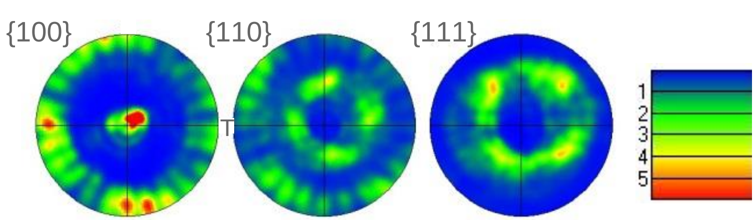
Nickel weld (Alloy 82) and SCC

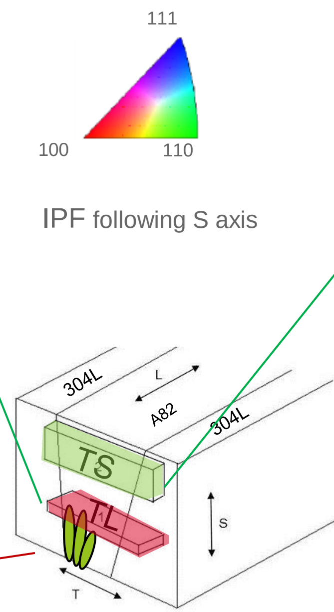
Microstructure of nickel base weld metal

[Chaumun 2016]

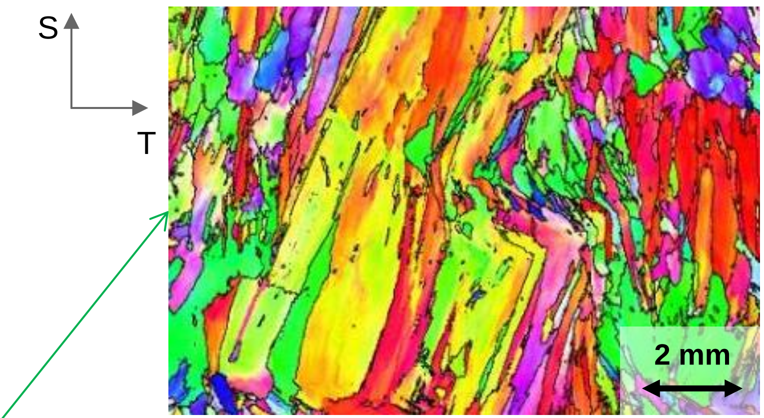
Weld A as-welded







Weld B as welded

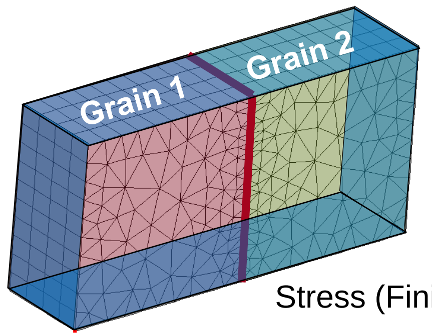


- Heterogeneous grain size and elongated grains along the dendrite growth direction
- Morphology and texture depends on the weld (on the welding process)
- Representative elementary volume close to 1 cm^3

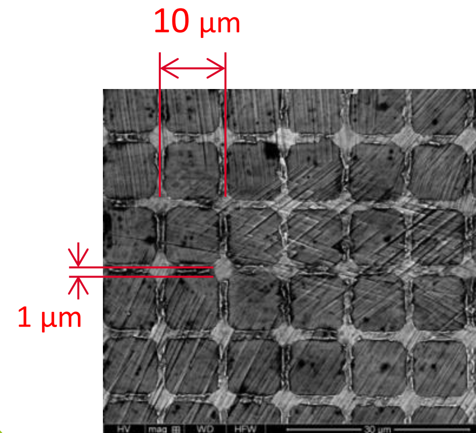
Approach

U-bend specimen tested in hydrogenated steam at 400°C (200 bars)

Microstructural analyses



Stress (Finite elements)



Strain fields
DIC using gold grids

Initiation sites

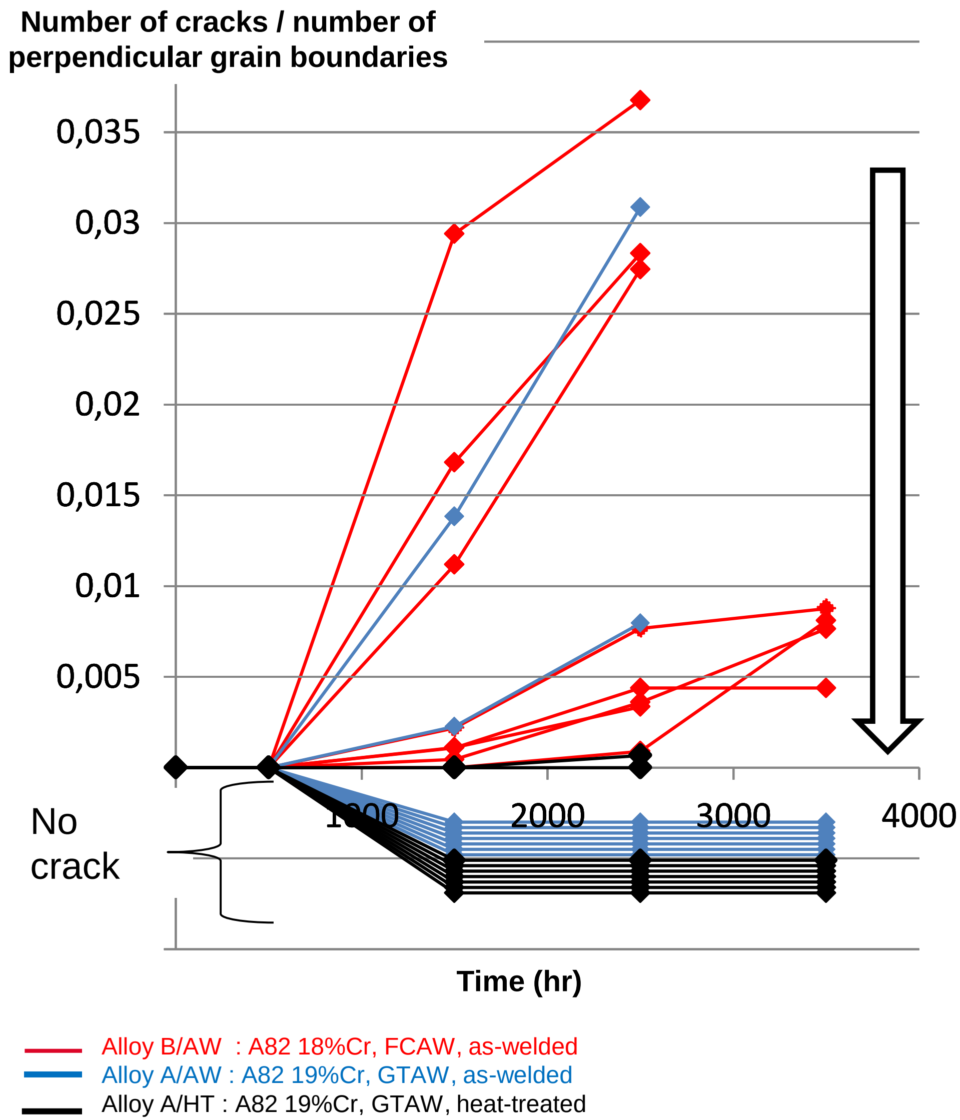
Local mechanical behavior

To identify which **microstructural parameters associated with local mechanical behavior** enable SCC initiation

Macroscopic behavior → Local behavior

Initiation test : macroscopic results

[Chaumun 2016]



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

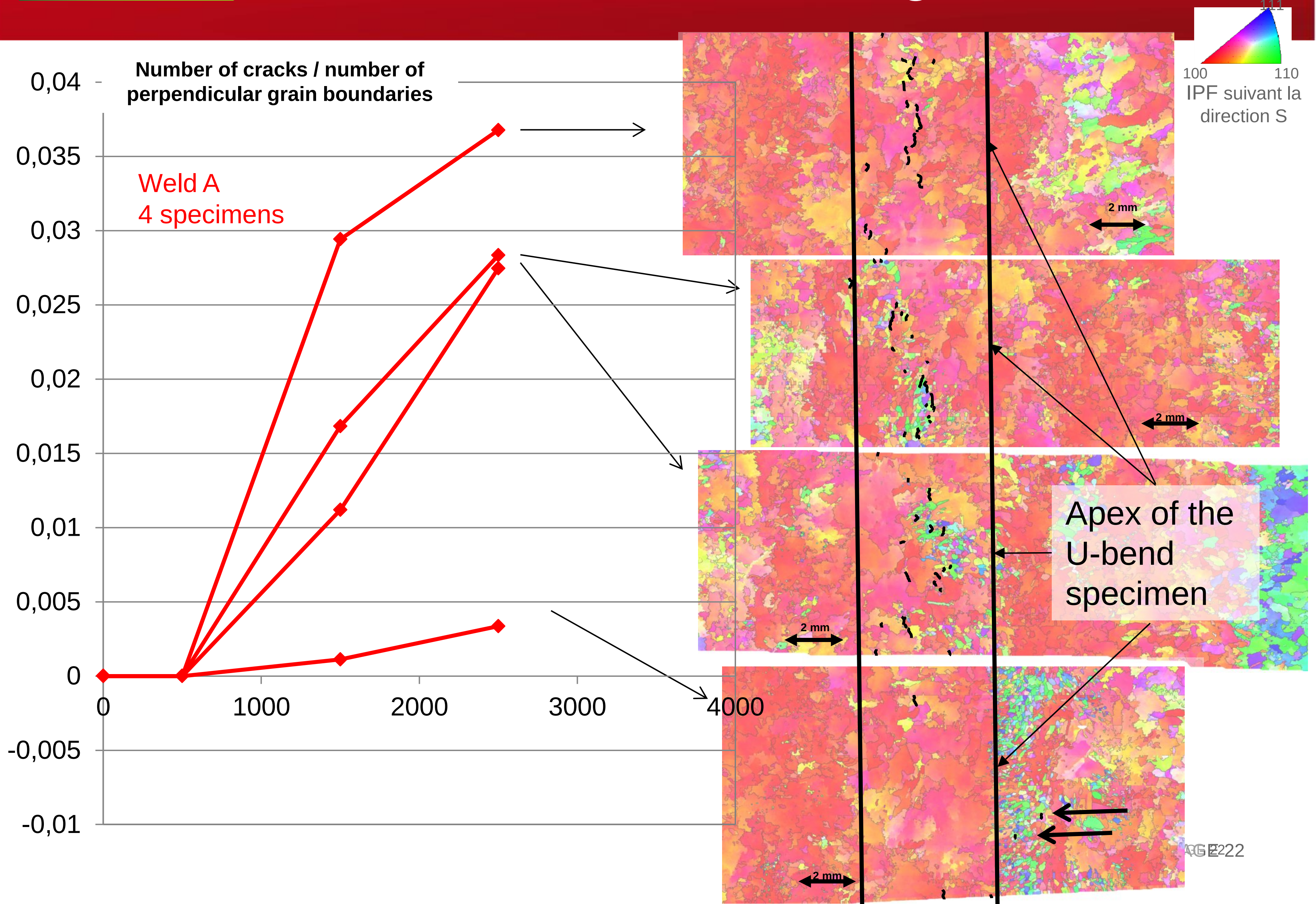
➢ beneficial effect of the heat treatment assumed to be due to the formation of intergranular chromium carbides [Sennour2013]

➢ but some scattering for the same weld
+ specimen size versus representative elementary volume

[Sennour2013] M. Sennour et al. JNM, 2013

Initiation tests : scattering

[Chaumun 2016]

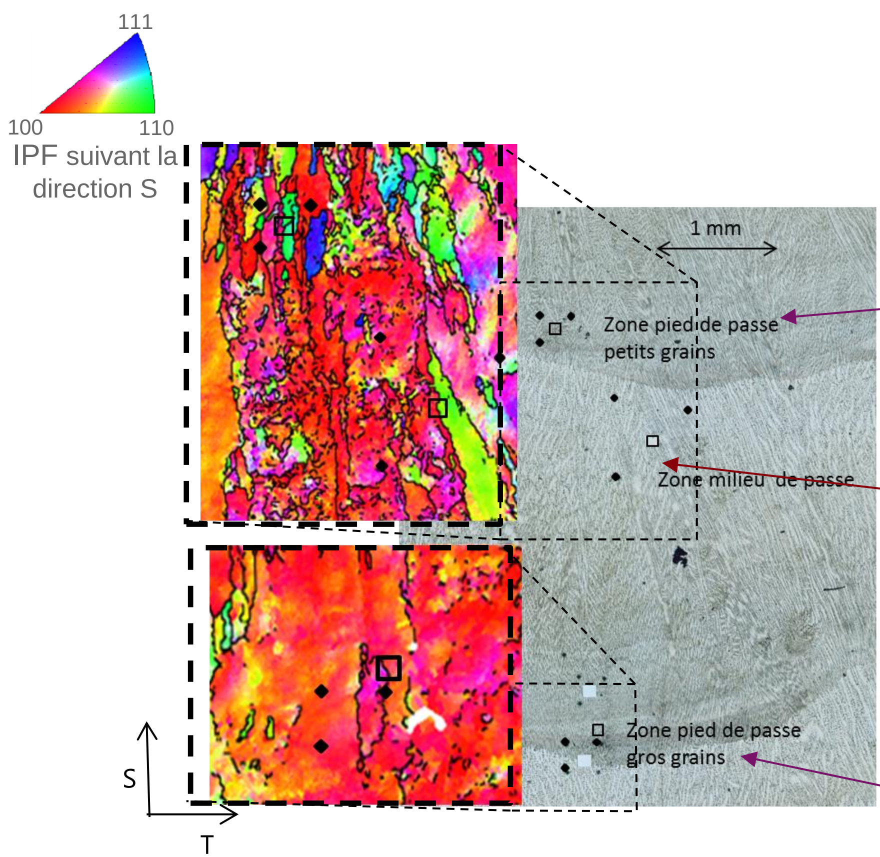


Chemical heterogeneities in the weld passes

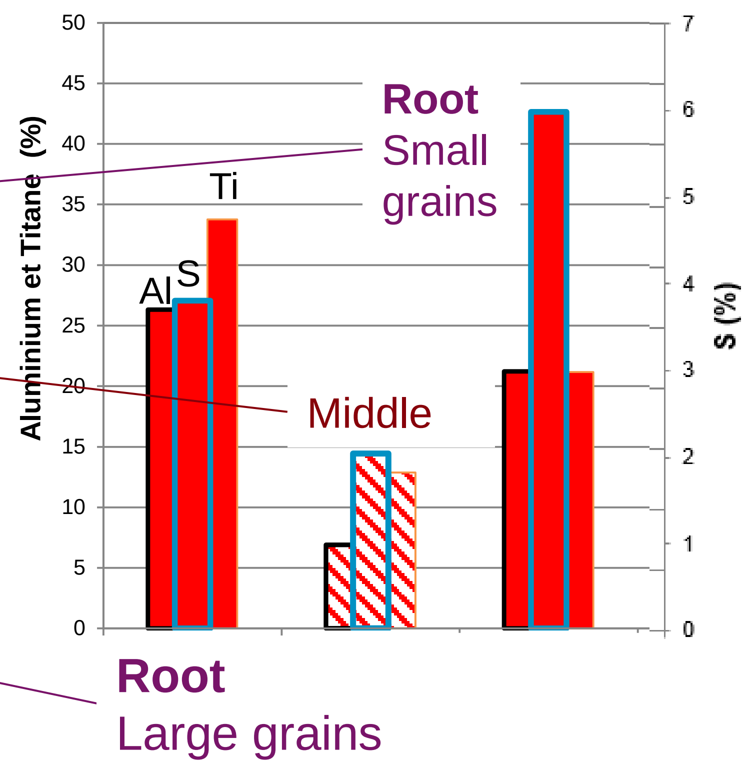
[Chaumun 2016]

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



Area fraction of phases containing Al, Ti or S



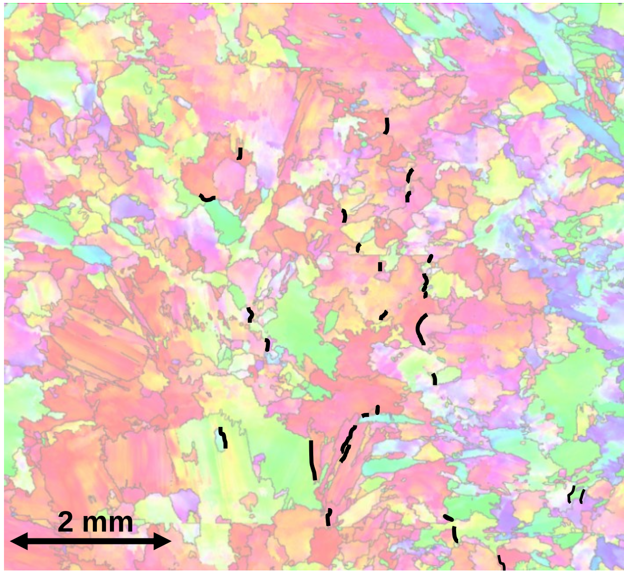
More impurities at the roots of the weld passes (small grains or large grains).

SCC initiation and microstructure

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 [Chaumun et al., 2015] [Chaumun, 2016]

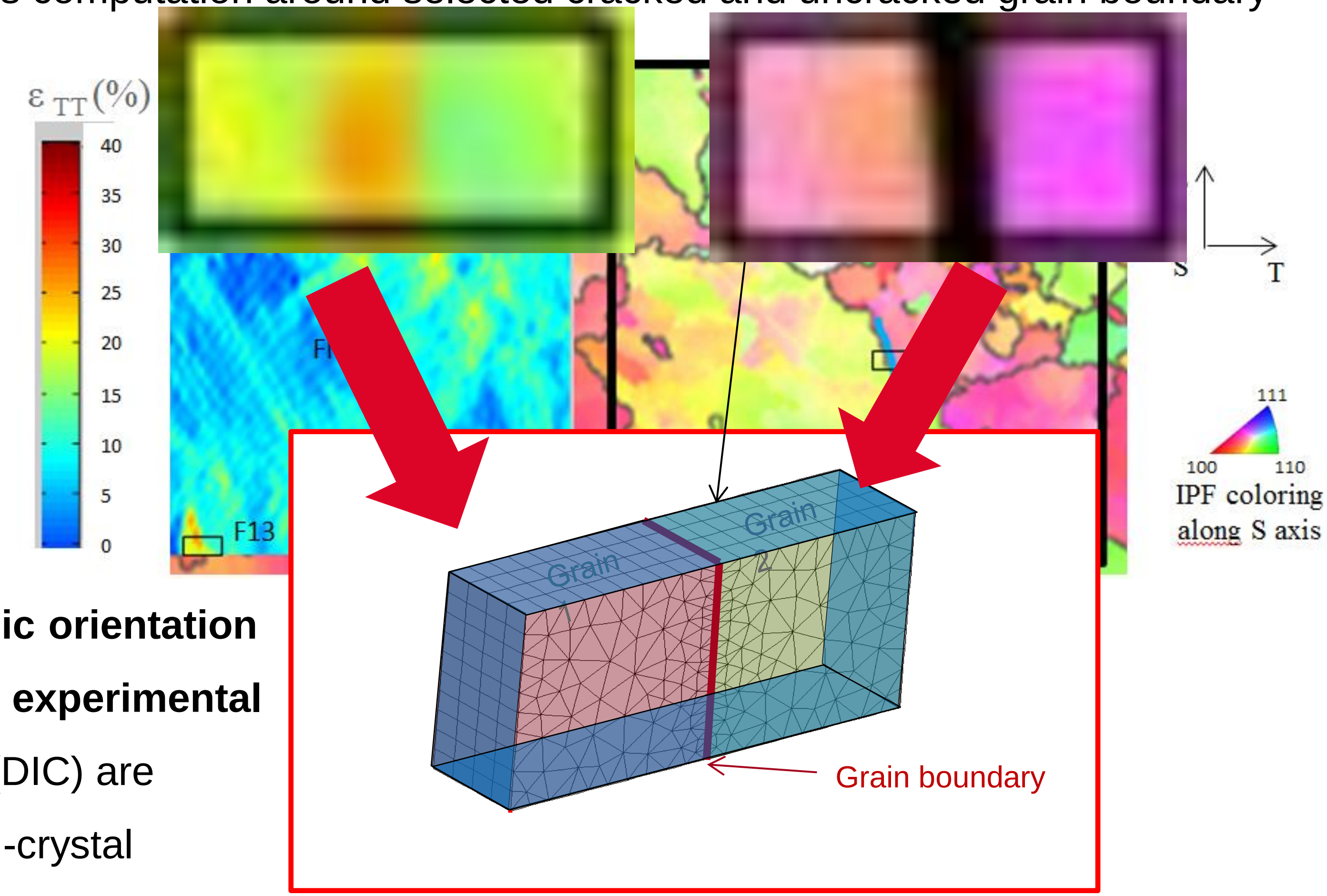
  - **Stress (finite elements analysis)**

SCC initiation and stress

[Chaumun 2016]

Finite elements computations

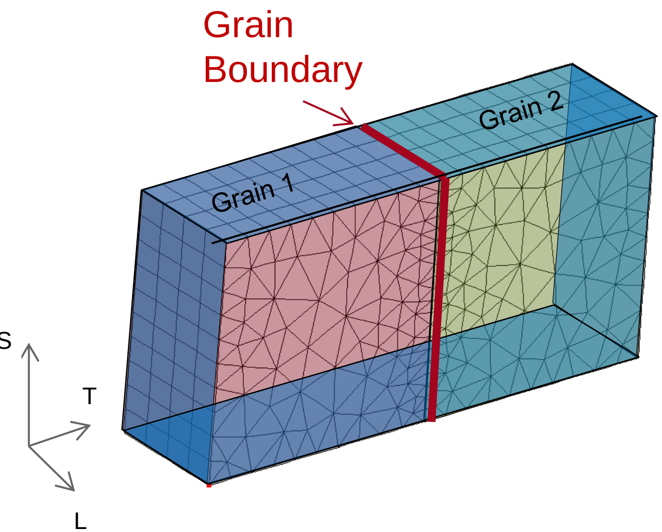
→ Finite elements computation around selected cracked and uncracked grain boundary

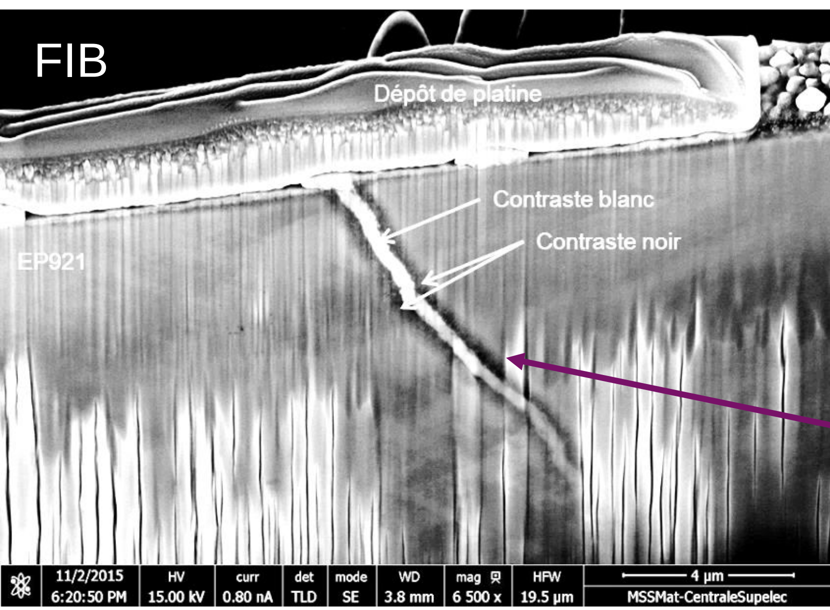


- **Crystallographic orientation** (EBSD) and the **experimental displacement** (DIC) are applied to the bi-crystal system.

SCC initiation and stress

[Chaumun 2016]





Crack initiation

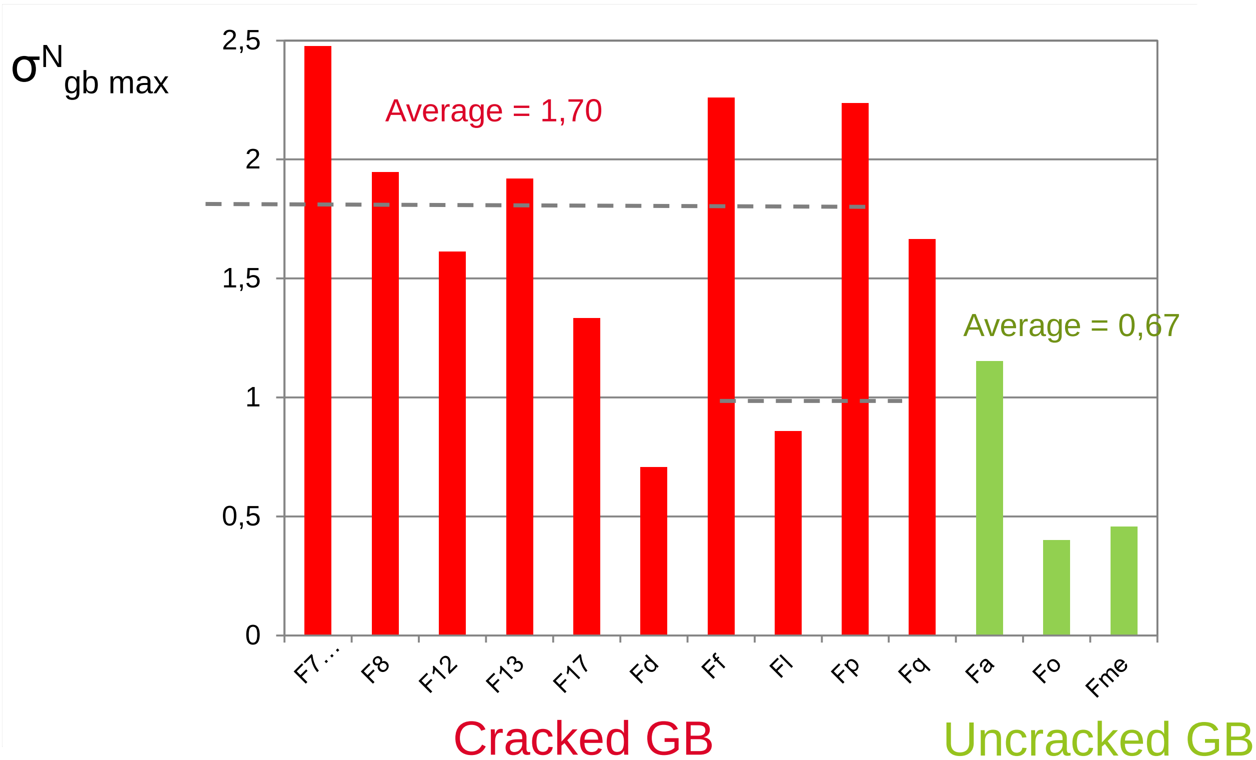
For selected cracked and uncracked grain boundaries (as observed by FIB)

Results :

- $\boldsymbol{\sigma}^{N}_{gb}$ = Maximum normal stress $\boldsymbol{\sigma}_{gb}$ normalized by the average normal stress of all computations

SCC initiation and stress

[Chaumun 2016]



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

SCC of Alloy 82 : conclusions

Macroscopic behavior → Local 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

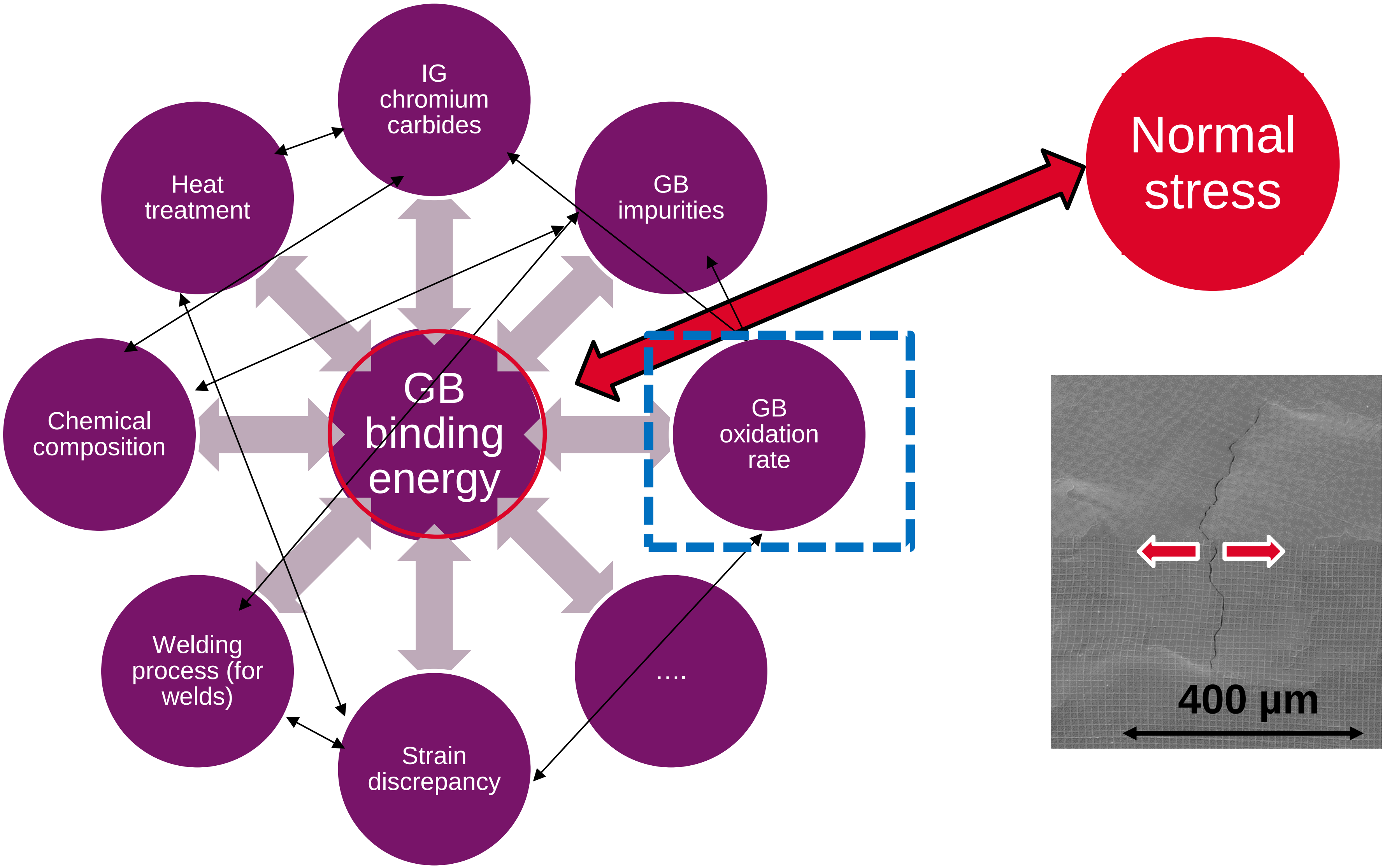
- the roots of the weld passes can contain more impurities (correlation with the weld process and with the chemical composition).

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…)

SCC of nickal alloy : conclusions



Conclusion and future work …

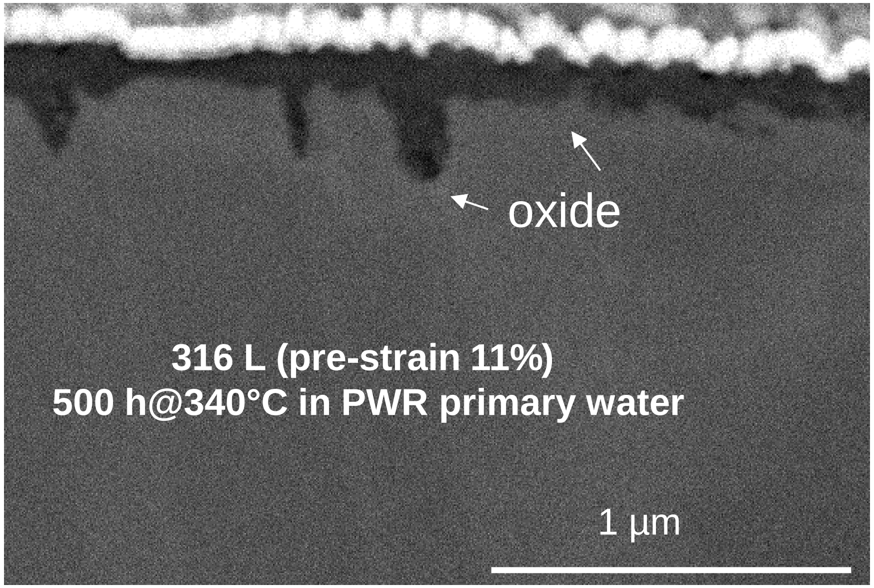
To improve the modelling, the work in progress deals with :

- 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

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Direction de l’énergie nucléaire
Département de physico-chimie
Service de la corrosion et du comportement des matériaux dans leur environnement
Laboratoire d’étude de la corrosion aqueuse

Catherine GUERRE : catherine.guerre@cea.fr
Commissariat à l’énergie atomique et aux énergies alternatives
Centre de Saclay | 91191 Gif-sur-Yvette Cedex
T. +33 (0)1 69 08 16 26 | F. +33 (0)1 69 08 15 86

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