Interactions Oxidation – Mechanical behavior

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Outline

- Scientific and experimental background
- Basic mechanisms affecting the mechanical behavior

Some illustrations

- Effect of oxidation on the global mechanical behavior:

Composite effect (FeCrAl alloys)

Coupling oxidation and mechanical behavior

(nickel based alloys and superalloys)

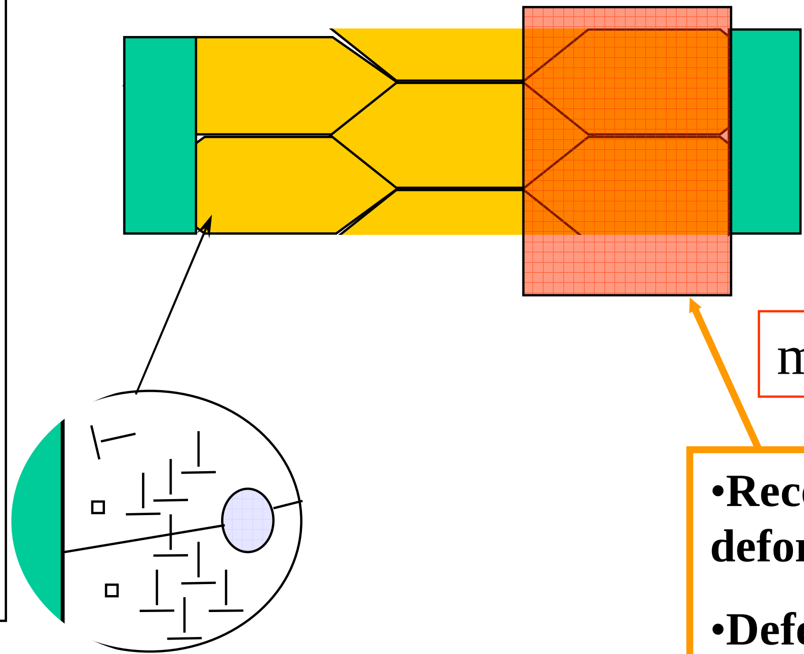
- Effect of oxidation on the local mechanical behavior

Crack initiation and propagation (nickel based superalloys)

Perspectives and challenges

Several ranges of interactions

- Oxygen, nitrogen enrichment
- Internal oxidation
- Precipitation or solutioning
- Defect injection
- Cavities nucleation
- Chemical composition evolution



< 1µm

Short range

Composite effect

mean range

(100 µm)

- **Recovery of deformation structures**
- **Defect injection**
- **recristallisation**

Study and observation of interactions

- Dedicated experiments

Time consuming and sometimes risky

- Tests on smooth specimens

Study of the interactions at a global scale

Does the specimen remain a representative volume element or not?

Tests on cracked or notched specimens

Study of the interactions at a local scale.

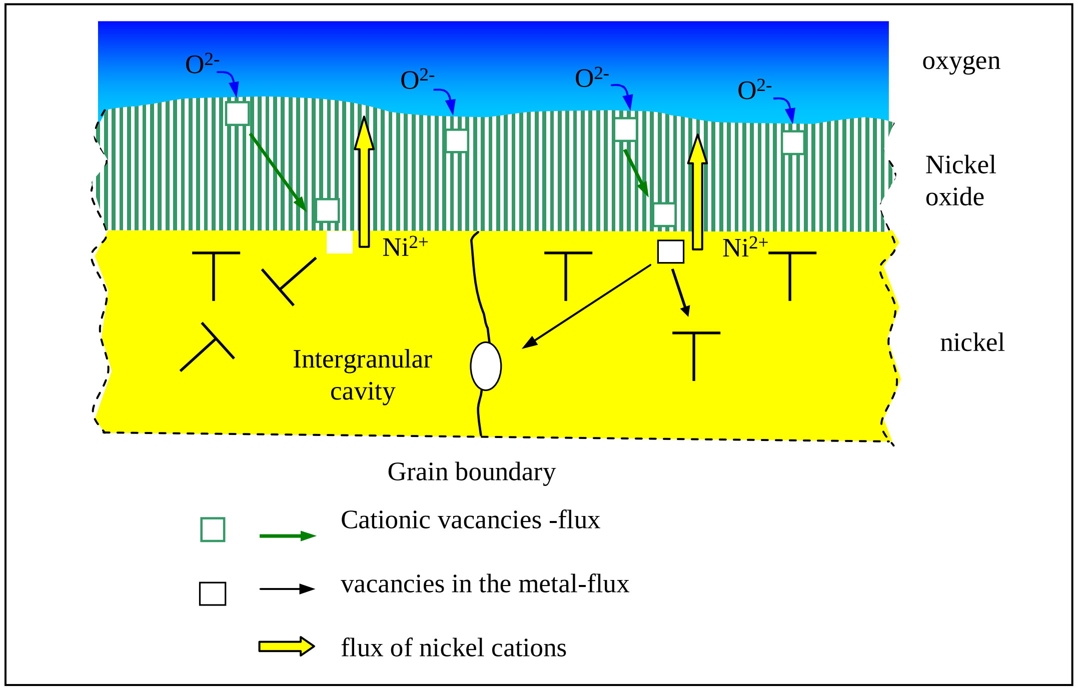
Basic mechanisms involved in the interactions

Several illustrations:

- Interface reactions
- Interface mobility
- Creep of Mg, Ni
- Vacancies injection
- Chemical composition evolution
- Intergranular oxidation

Interface reactions

Cationic growth of nickel oxide



$$O_{ads} \rightarrow O_O^x + V_{Ni}^{''} + 2\dot{h}$$

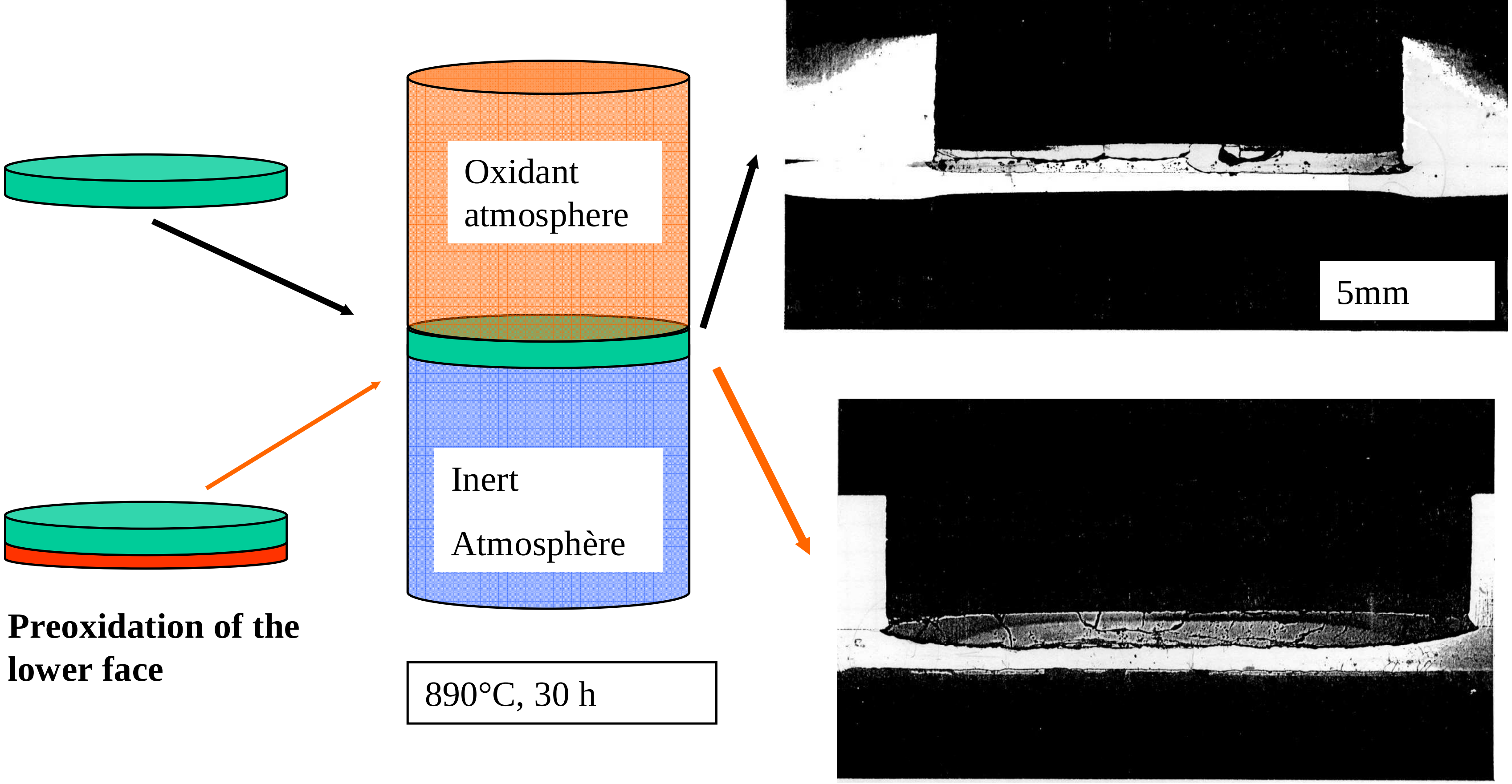
$$\underline{Ni}_{Ni} + 2\dot{h} + V_{Ni}^{''} \rightarrow Ni_{Ni}^x + \underline{V}_{Ni}$$

Interface metal-oxide is free to move → interface acts as a perfect vacancies sink

Interface locked → vacancies injection

Effect of interface pinning on the oxidation of iron disks.
(R.Francis and D.G.Lees, Mat Science Eng, A120(1989))

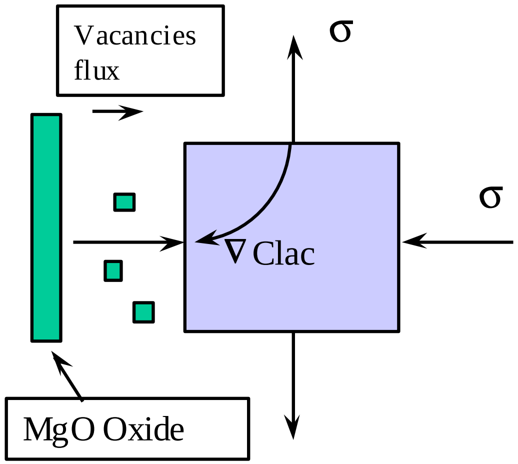
Disk geometry:
ϕ=35mm;δ=1.7mm

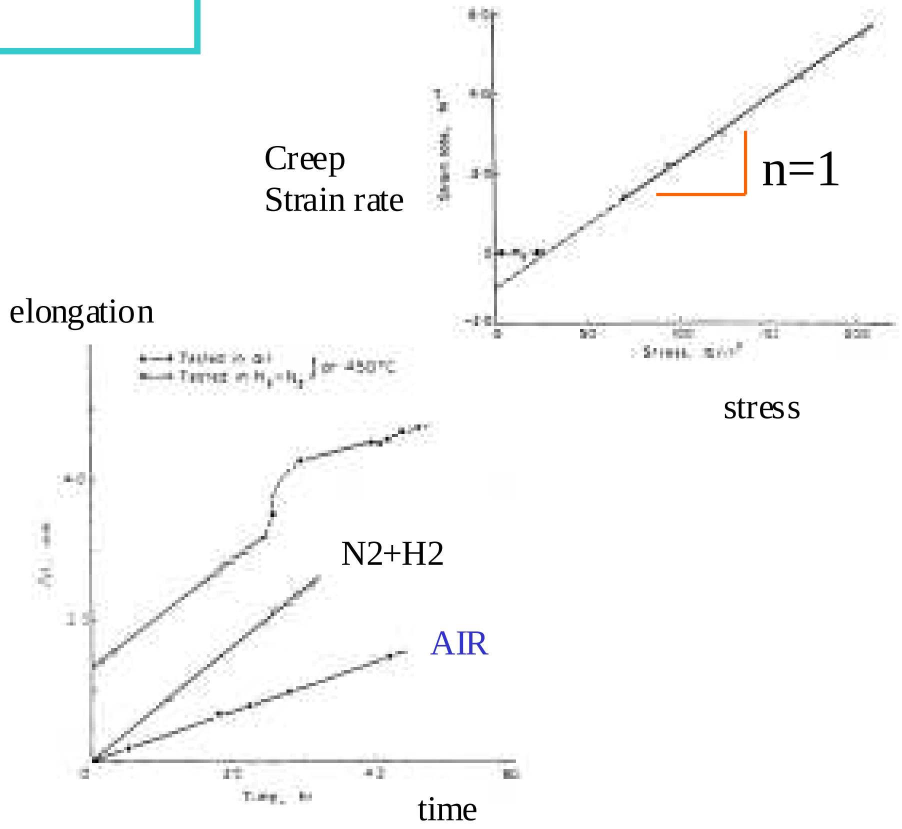


Vacancies injection

Creep tests on magnesium (Hales et al 1969)

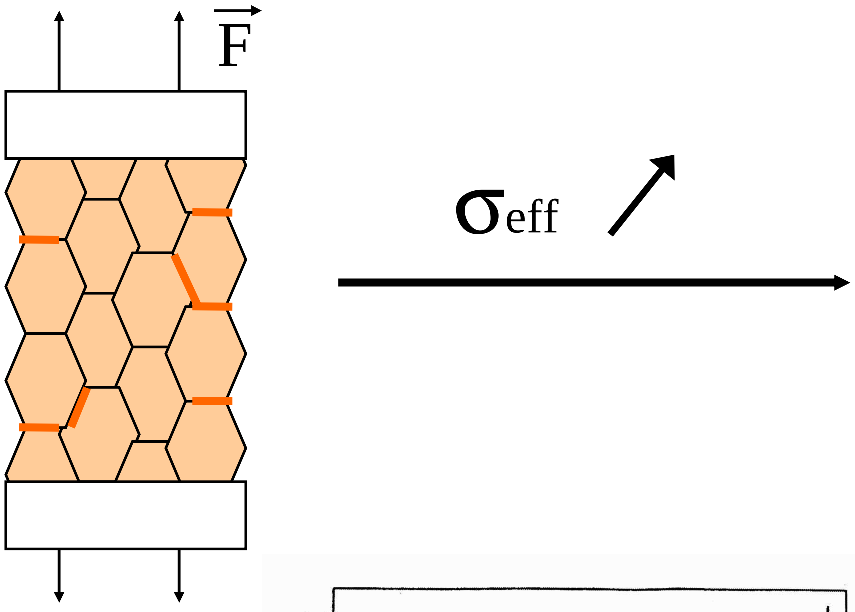
Stress and temperature domain corresponding to Nabarro-Herring creep mechanism



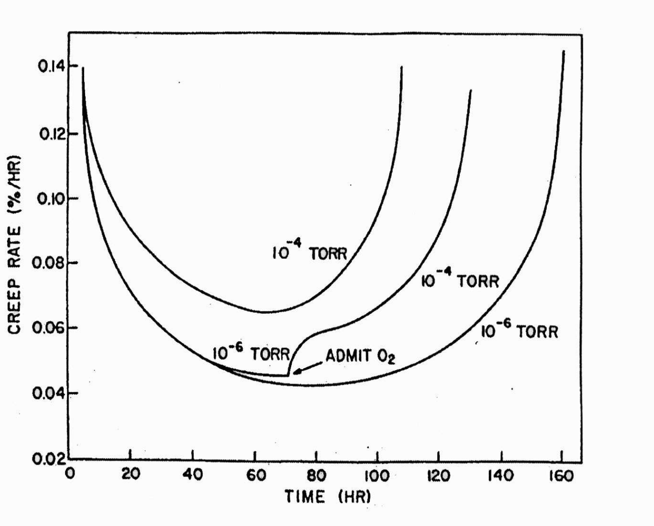


Nabarro-Herring creep regime is slowed down due to vacancies injection

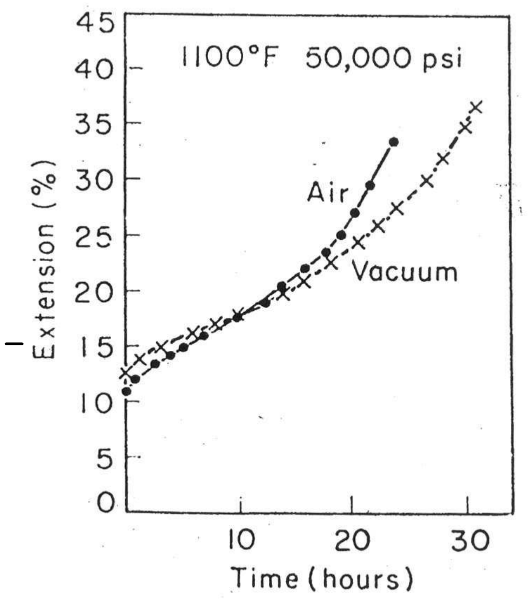
Coupling oxidation and creep strain rate



- Creep strain rate is increased
- History effect (to be proved)
- Decreasing the elastic modulus (to be proved)
- Decreasing deformation at fracture



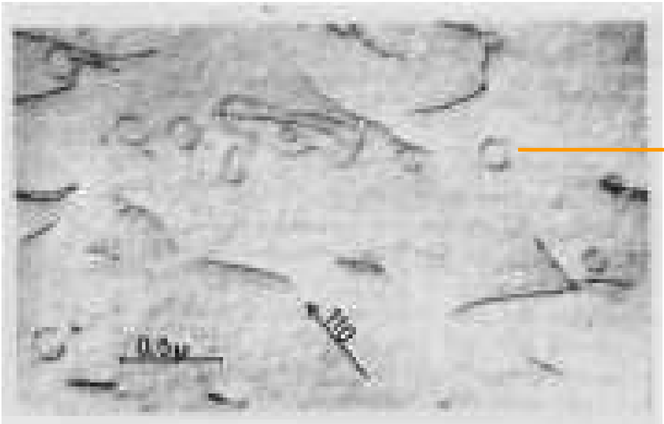
Stegman 1969

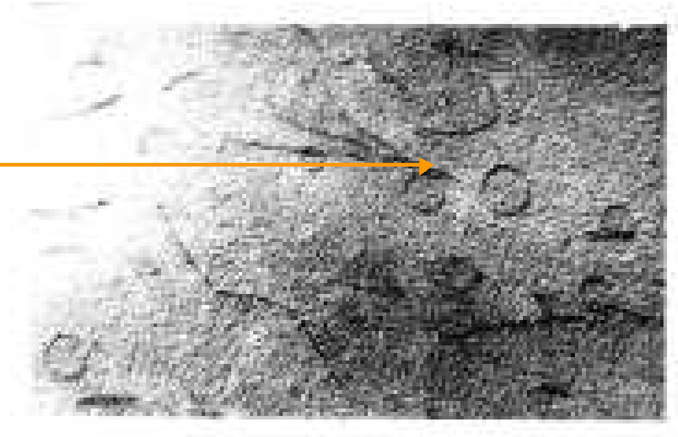


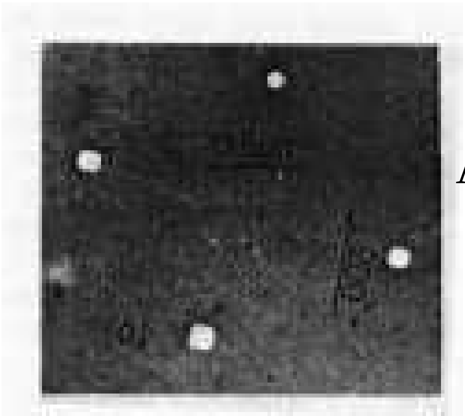
Vacancies injection

Oxidation-induced defects in NiAl Fraser et al (1973) Phil. Mag.

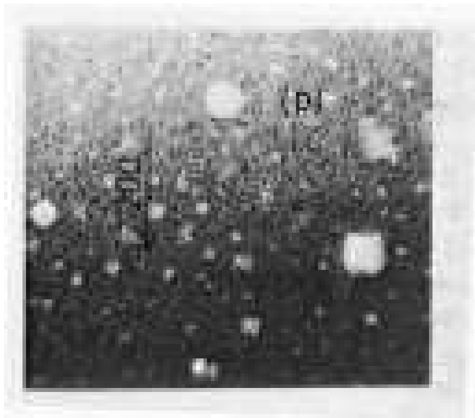
In situ TEM study of annealing of voids and vacancy loops







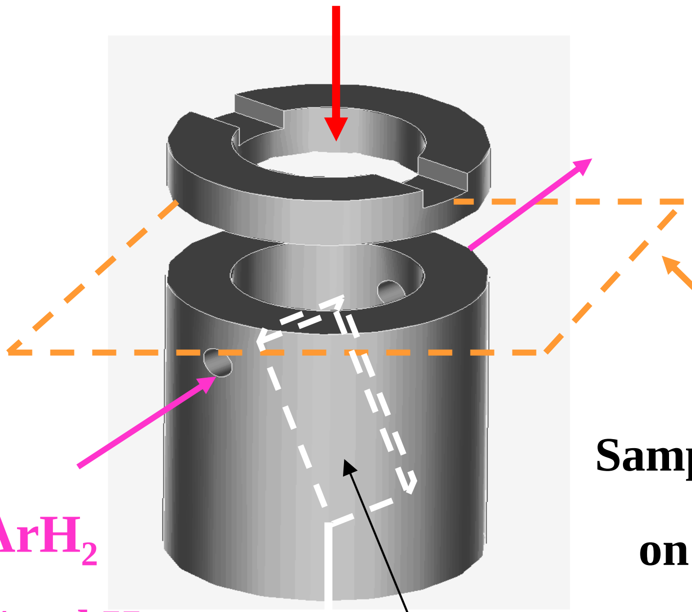
Après 1h à 920°C



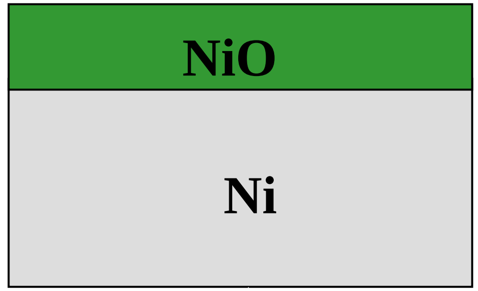
Vacancies injection study: an experimental device

S.Perusin PhD thesis

Air



1000°C

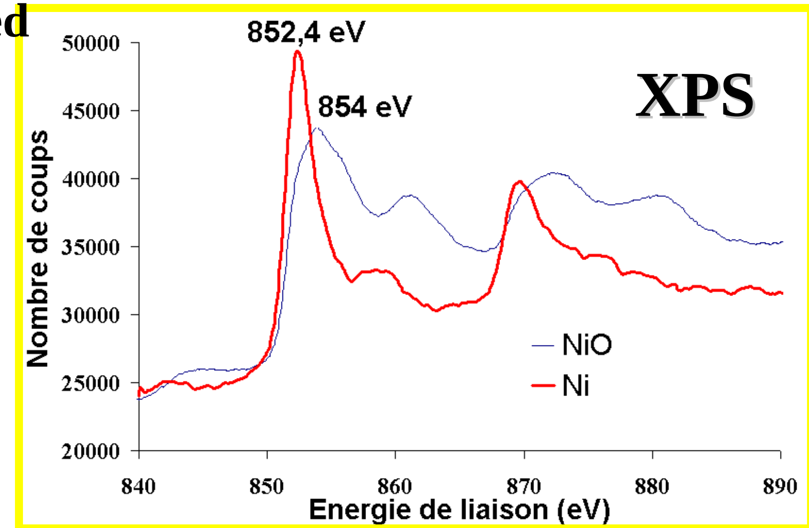


ArH_2

5% vol H_2

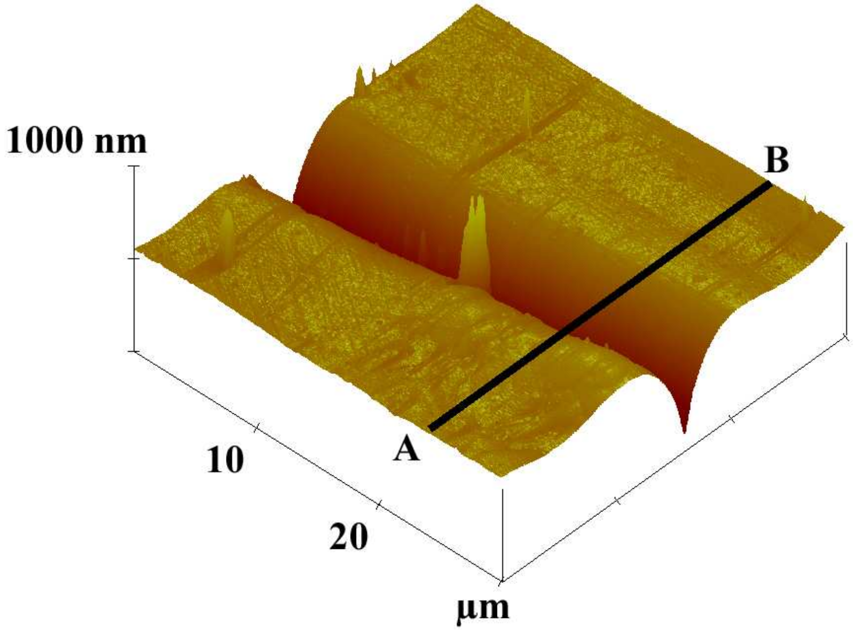
Sample to be oxidised

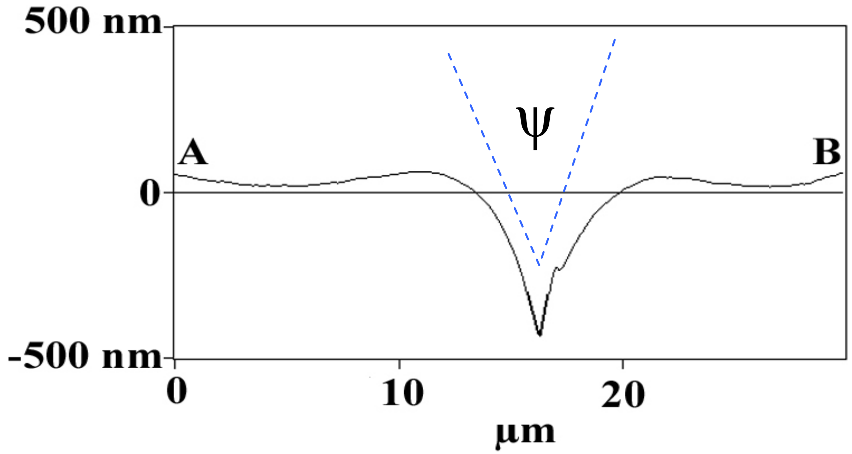
on the upper face



Reference sample:
only thermal grooving

Characterisation of the grooves (AFM)



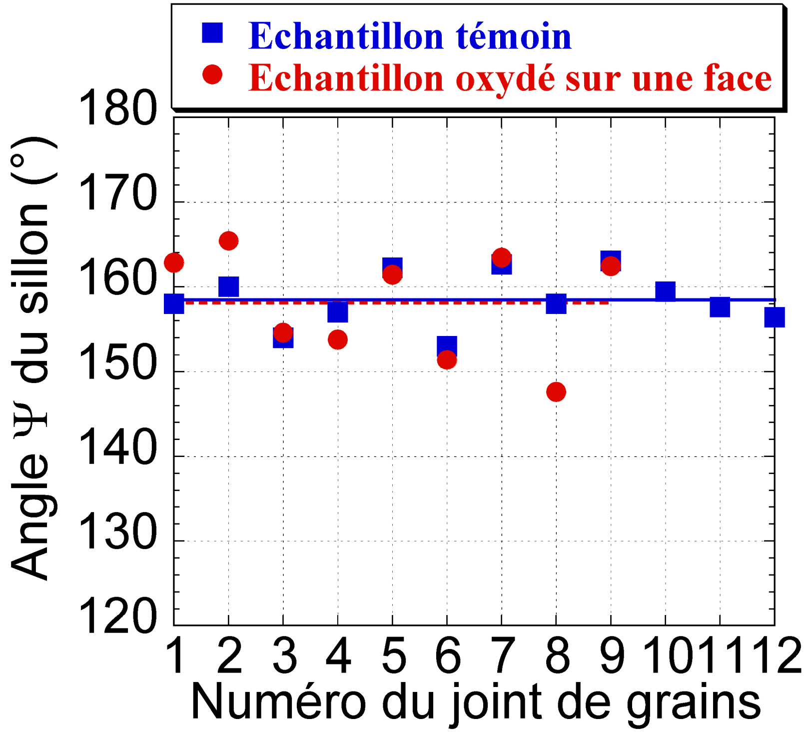


Study of the thermal grooves

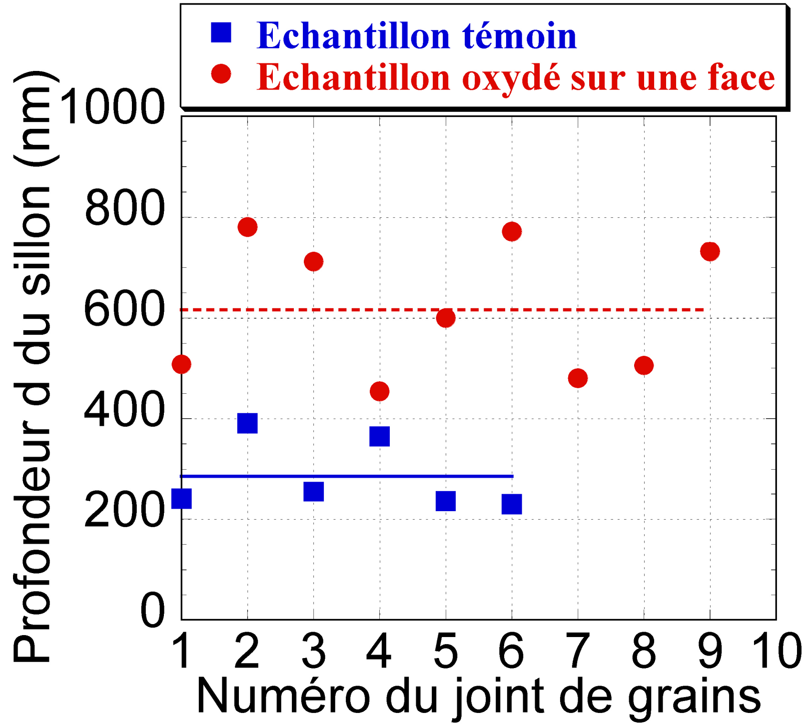
Thin plate (e=125µm)

1000°C/15h

Equilibrium angle (Ψ)



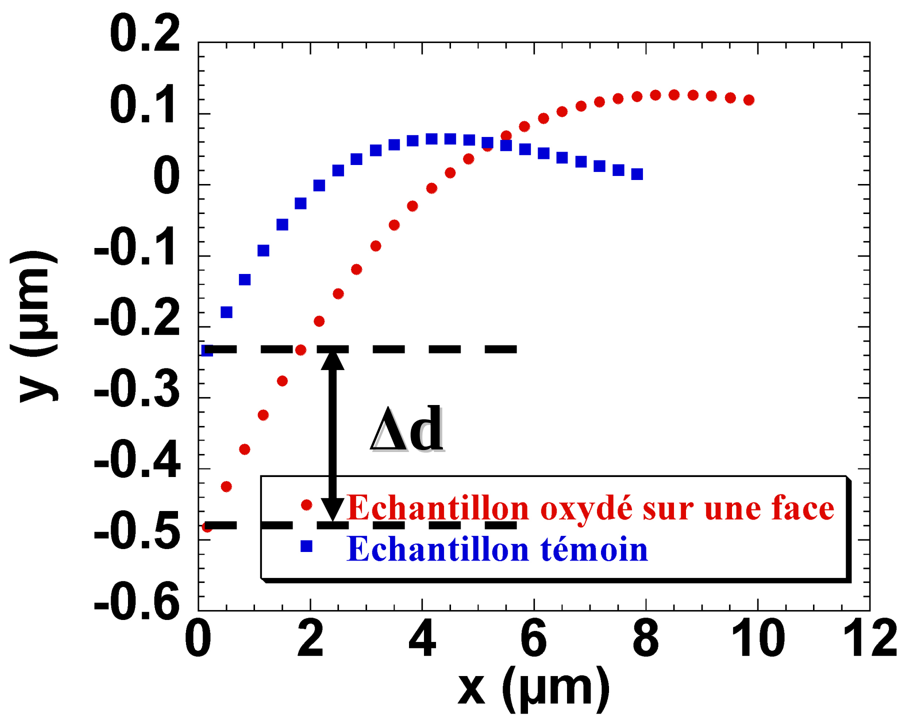
Depth of the groove (d)



$\Psi_{moyen} = \Psi_{moyen} = 158°$

$d_{moyen} >> d_{moyen}$

Study of the grooves geometry

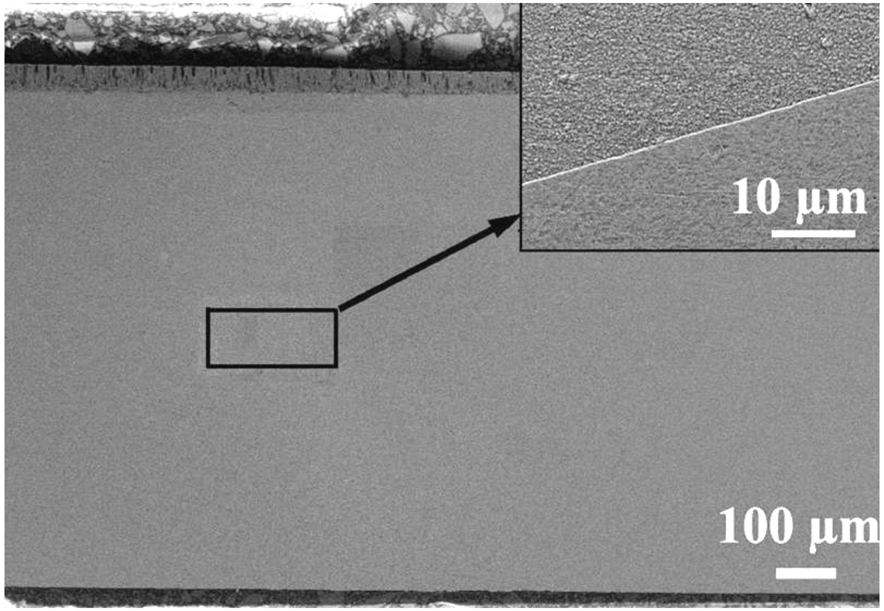


To explain Δd measured, 0.1% of the total number of metallic vacancies theoritically produced by interface reactions are necessary

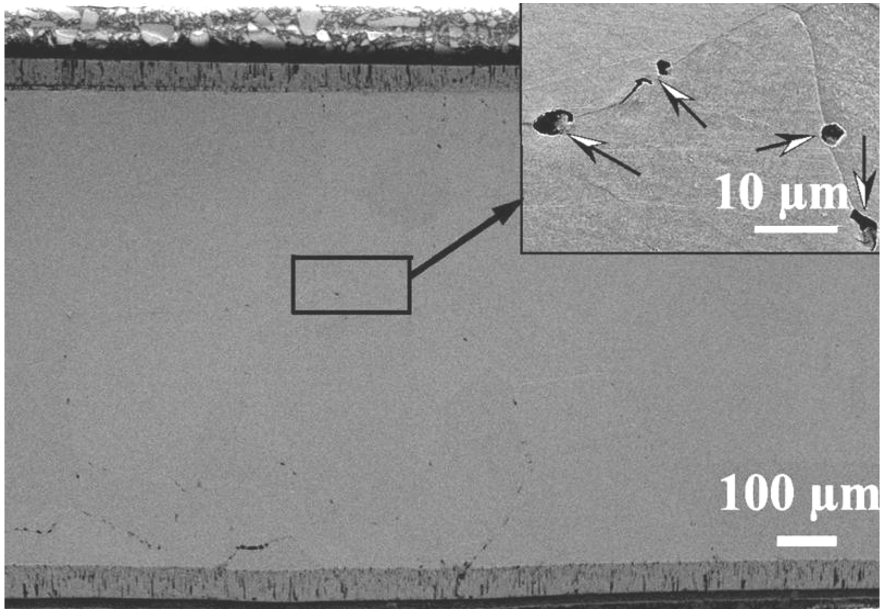
Grain boundaries act as diffusion paths and vacancies sinks?

Plate (e=1mm)
1000°C/48h

Oxidation 1 side



Oxidation two sides



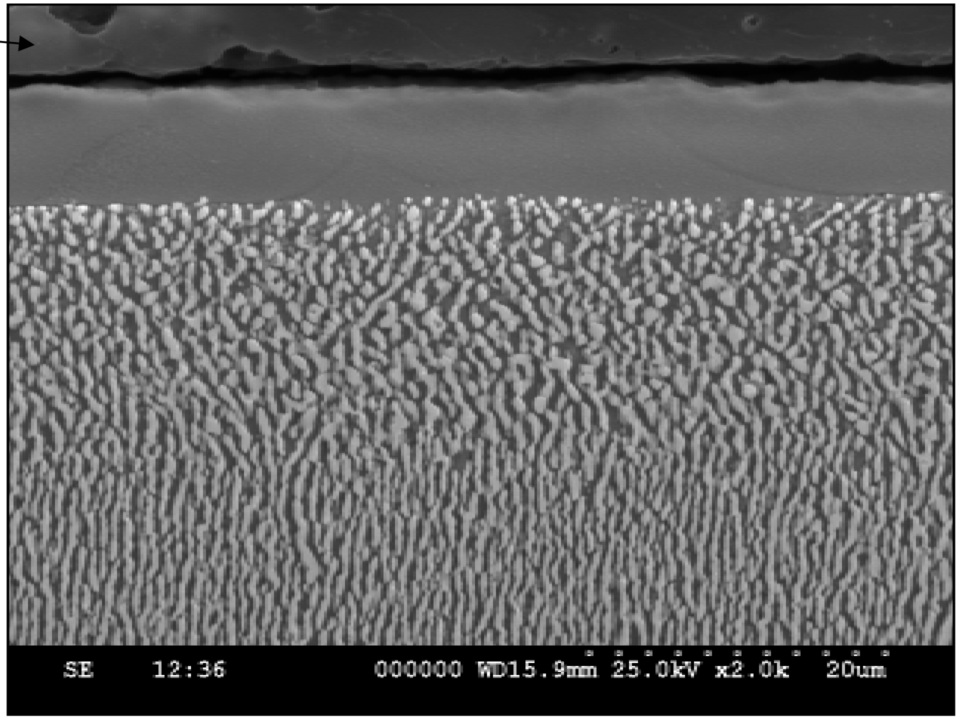
Chemical composition evolution

Microstructural evolution due to selective oxidation of the substrate (MC2)

S.Dryepondt PhD Thesis

Oxide scale

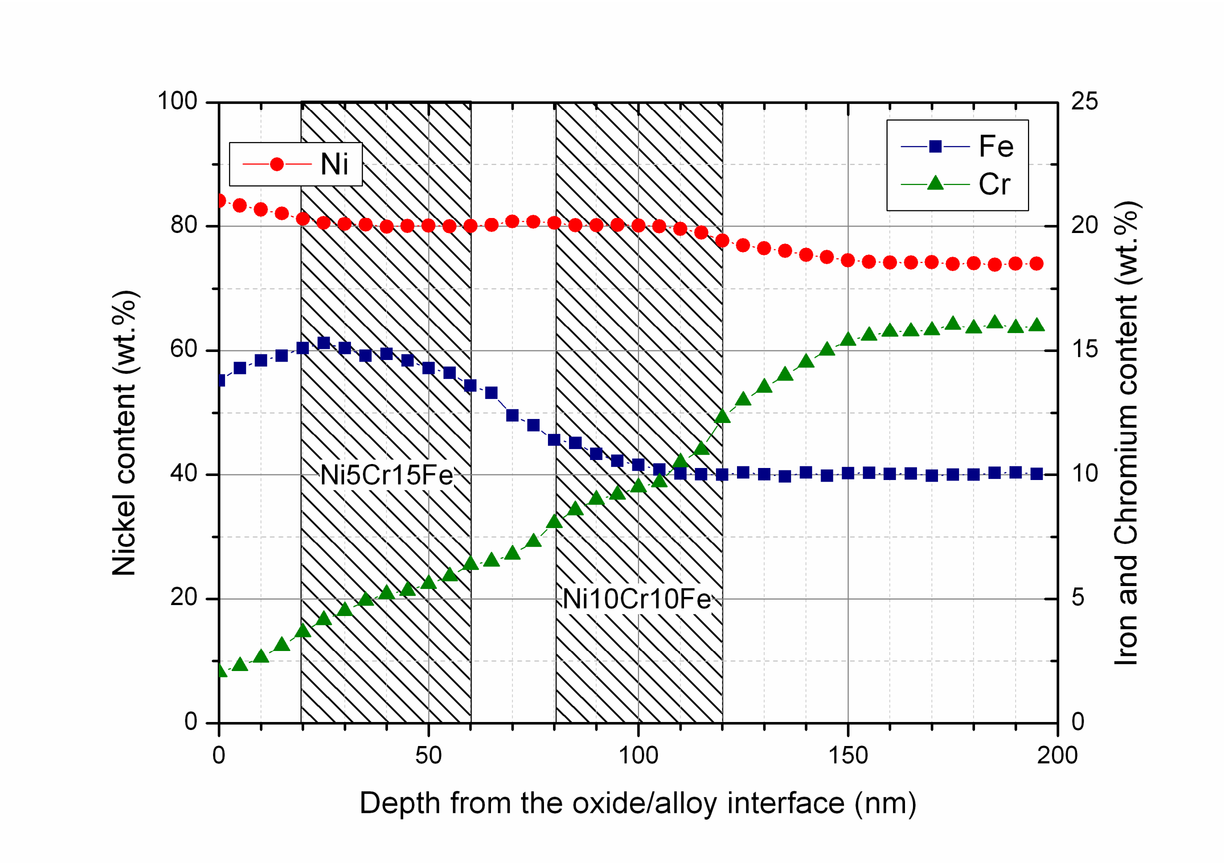
Affected zone



γ' are dissolved and coarsening is modified

Chemical composition evolution in alloy 600

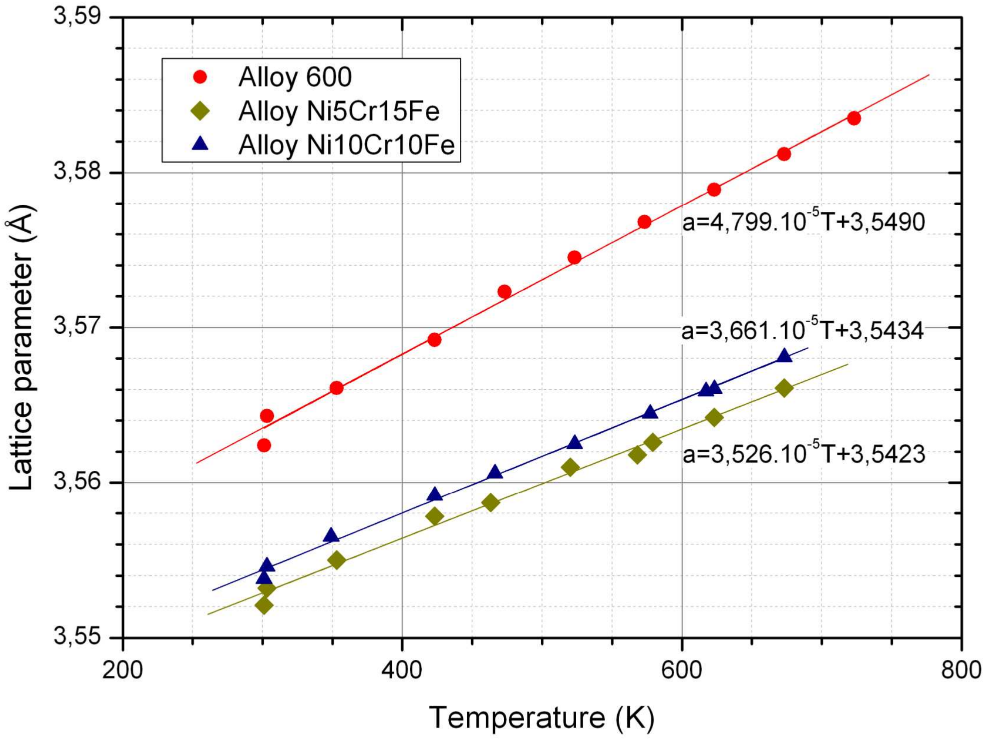
exposed to simulated PWR environment



Adapted from PhP Thesis J.Panter

Chemical composition evolution

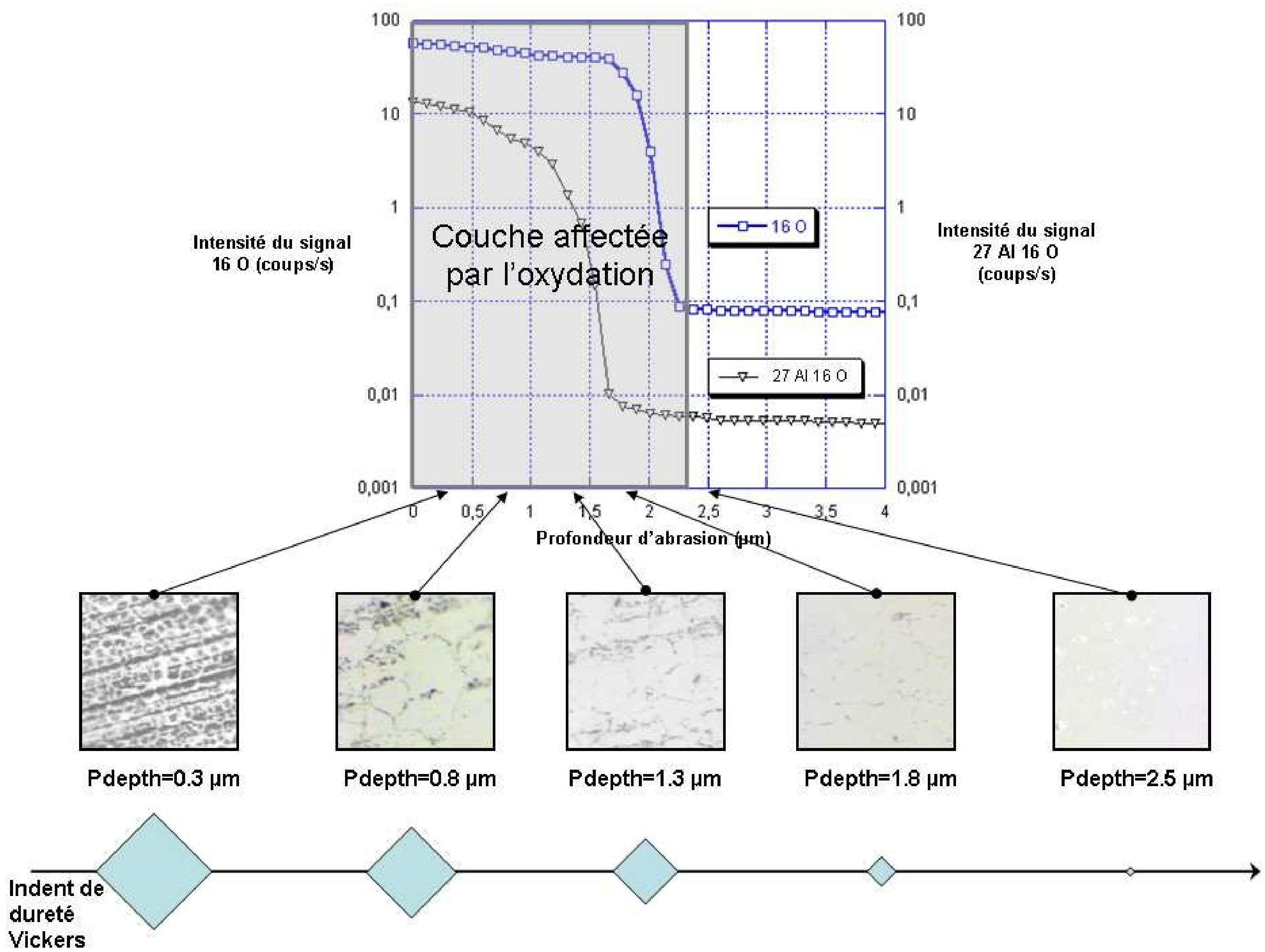
Consequences on the local physical properties



- Thermal expansion coefficient is modified
- Lattice parameter is reduced
- Internal stresses and thermal stresses are expected

Intergranular oxidation of a nickel based superalloy

SIMS profiles compared with optical microscopy images during incremental polishing

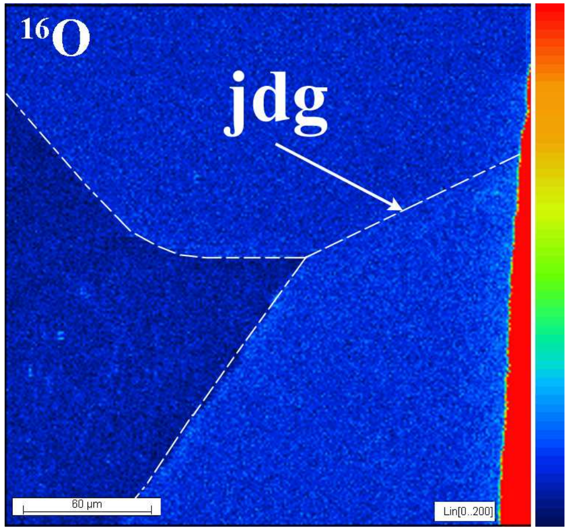


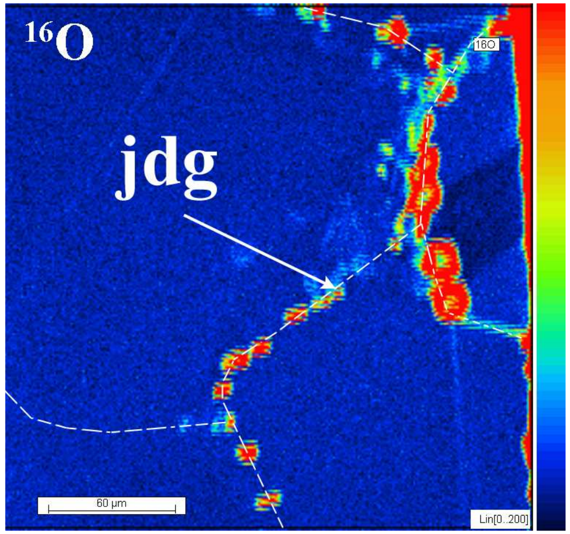
Intergranular oxidation

1000°C/48h (e=1mm)

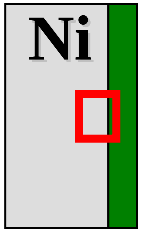
Oxidation 1 face

Oxidation 2 faces

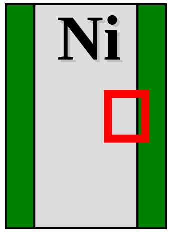


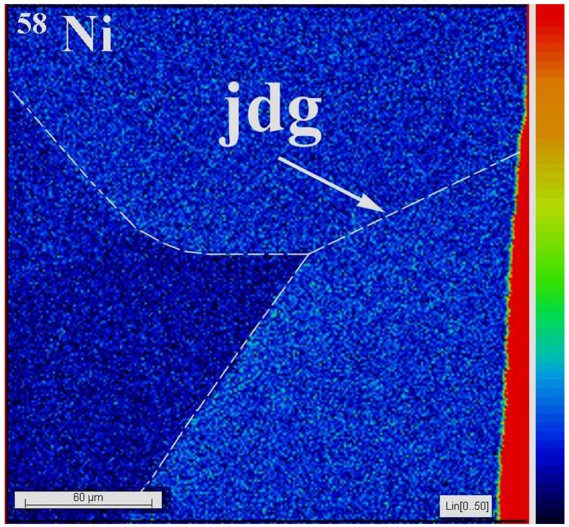


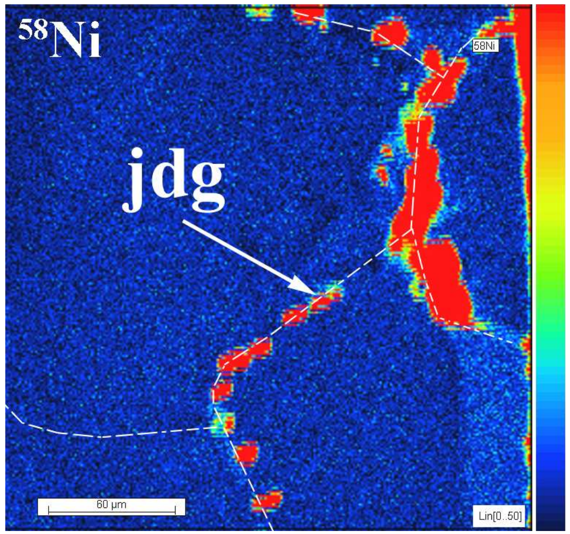
^{16}O



Interface Ni/NiO







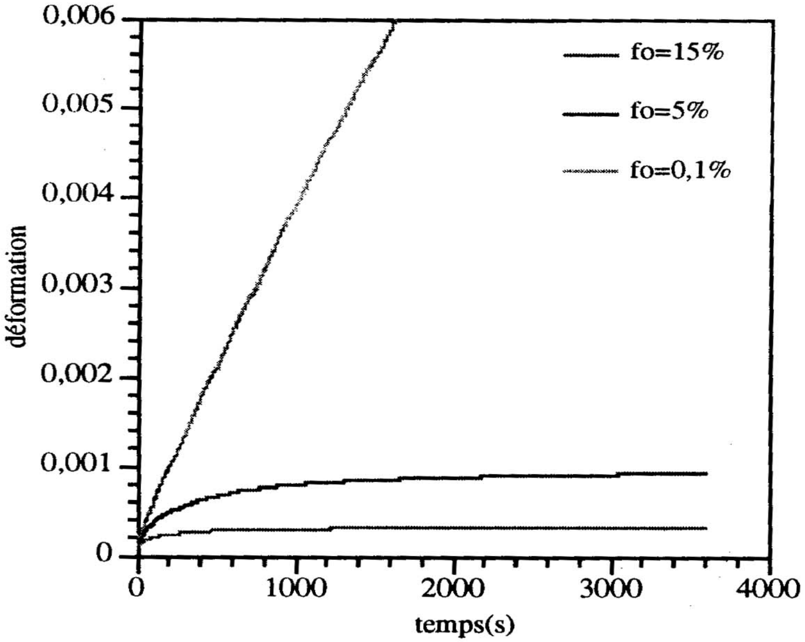
^{58}Ni

60 µm

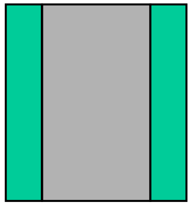
Effect of oxidation on the global mechanical behavior

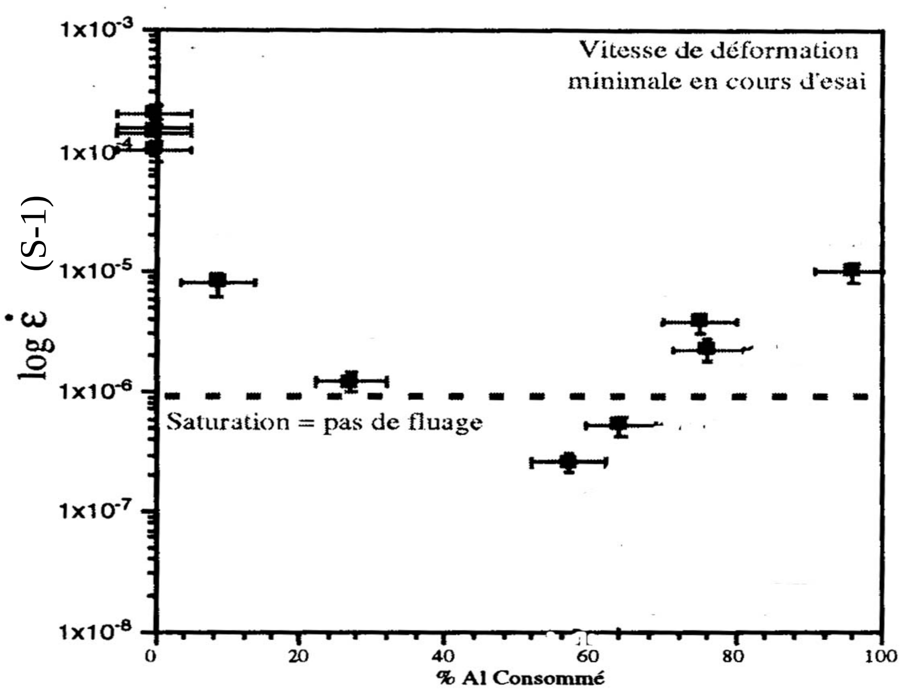
Composite effect

High temperature Creep (1000°C) of thin plate (40 µm) made of FeCrAl.



Effect of the oxide scale thickness



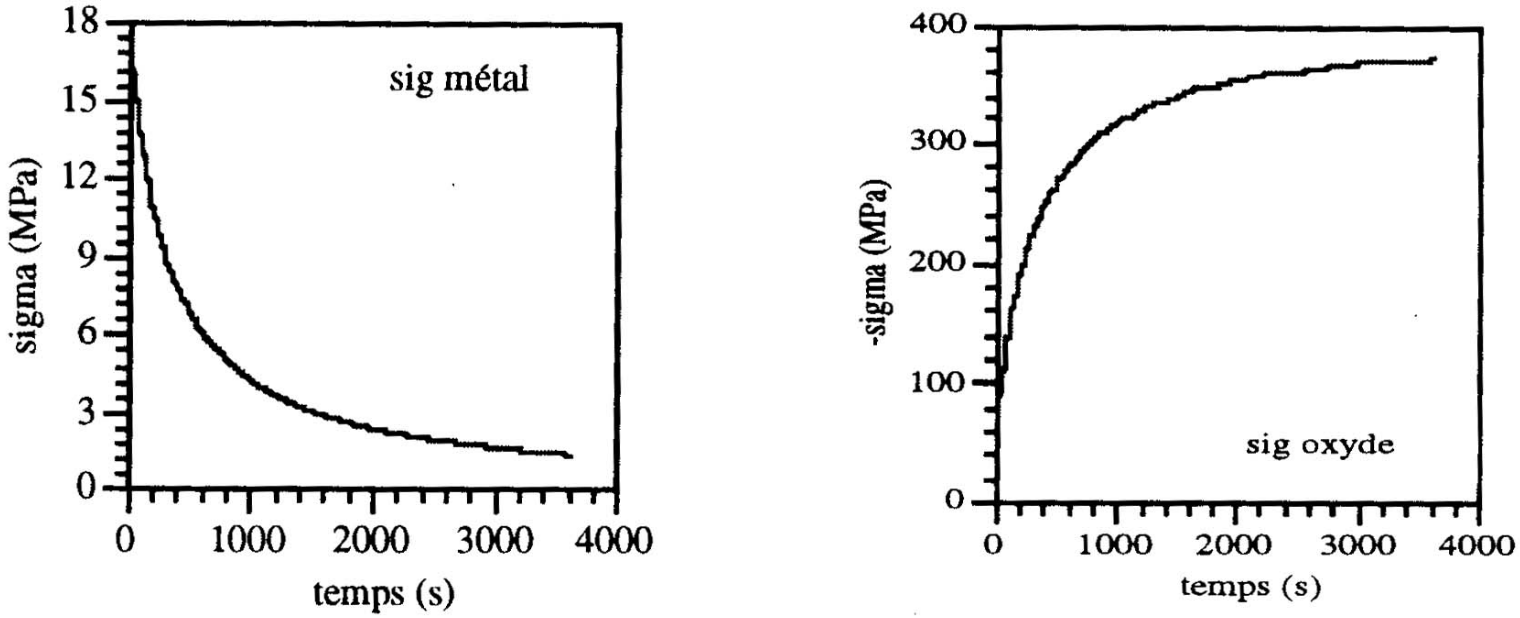


PhD Thesis A.Germidis ENSMP

$$\sigma_m = \left[(n-1)At + \left(\sigma_m^0\right)^{1-n}\right]^{1/1-n}$$

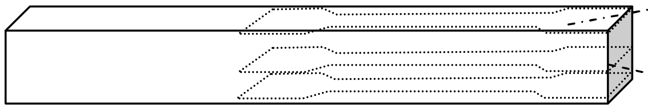
Where $A = f_0 K \overline{E}$ et $\overline{E} = \dfrac{E_m E_0}{f_m E_m + f_0 E_0}$

and σ_m^O initial stress in the metal at t=O

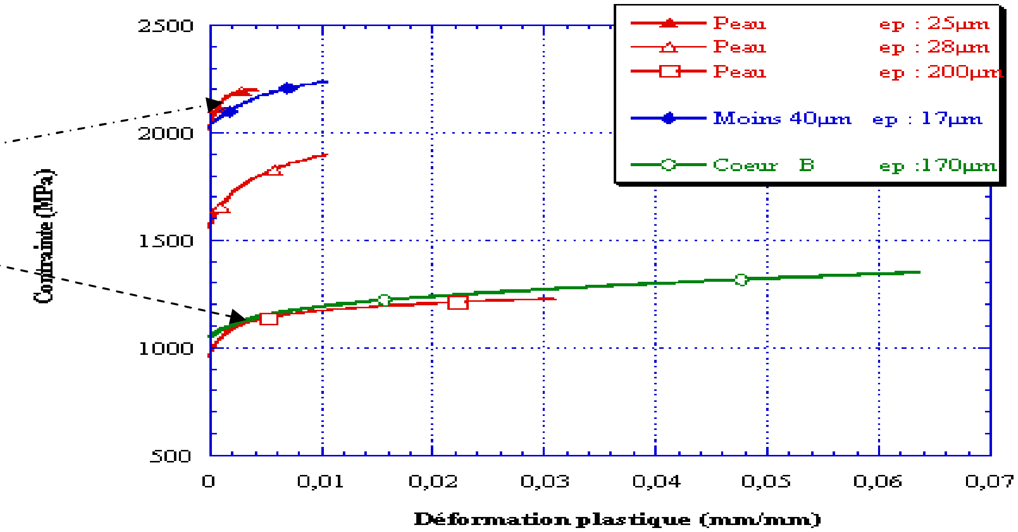


Stresses evolution in the two components of the system.

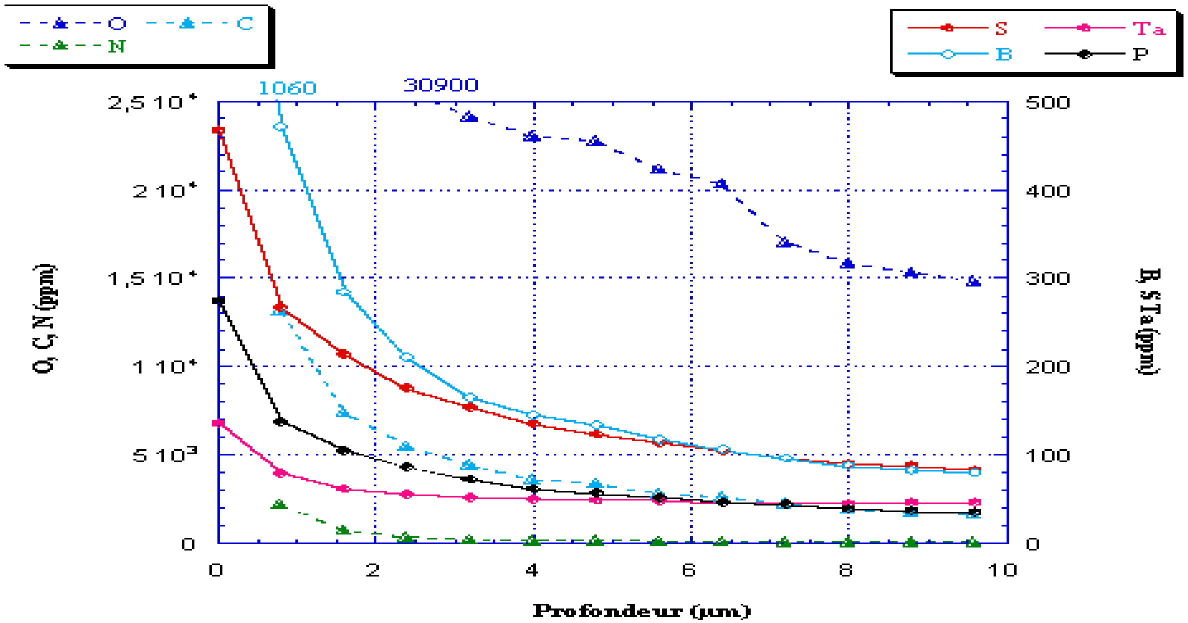
Effect of interstitial elements enrichment due to manufacturing conditions



Local mechanical properties
in the as-rolled product



Alloy 718



GDMS analysis of the chemical composition evolution
from the surface to the bulk.

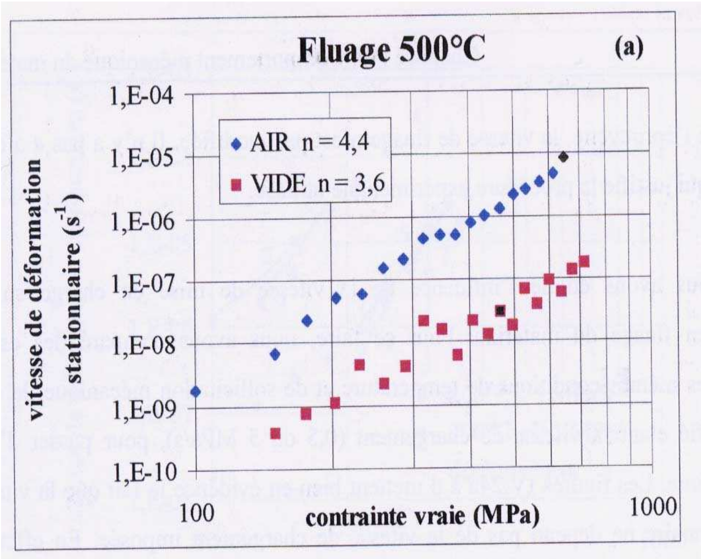
PhD Thesis O.Brucelle INPT

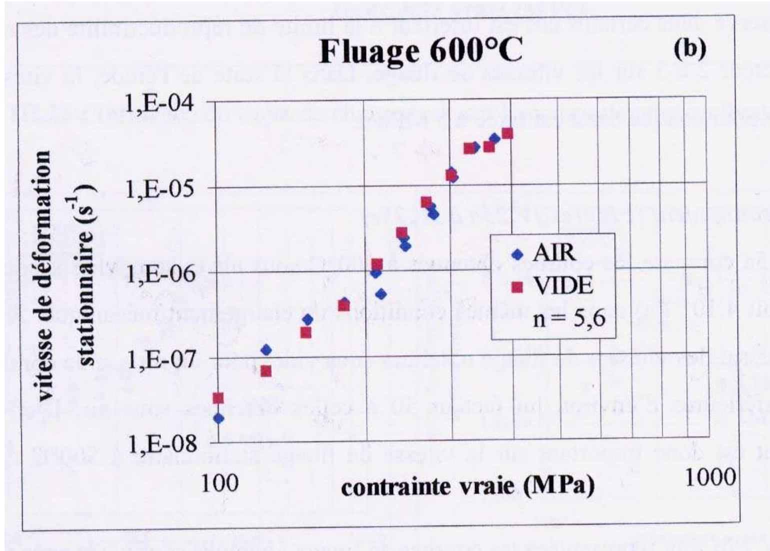
Dynamic coupling between oxidation and creep

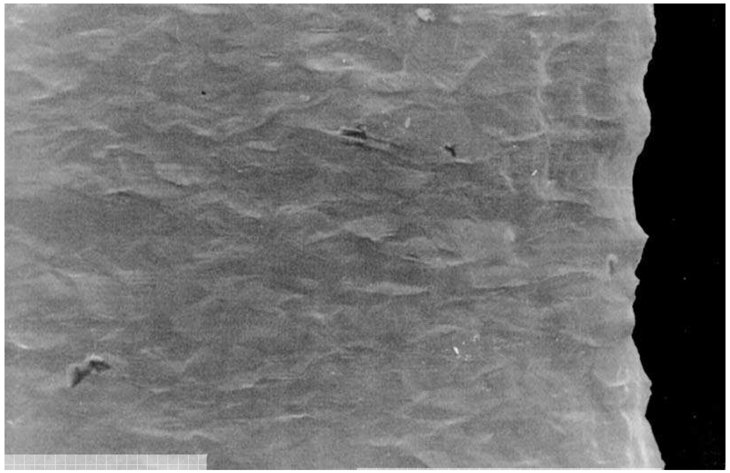
- NiCr thin plates
- MC2 thin walls
- Alloy 718 thin plates

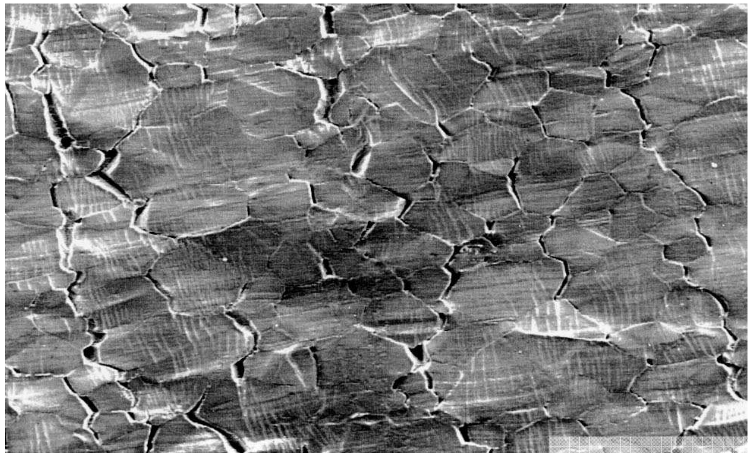
NiCr alloy (80/20)

PhD G.Calvarin 1998 ENSMP





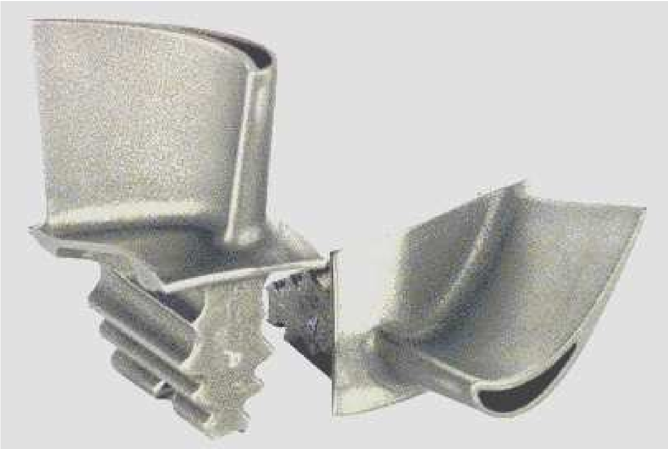




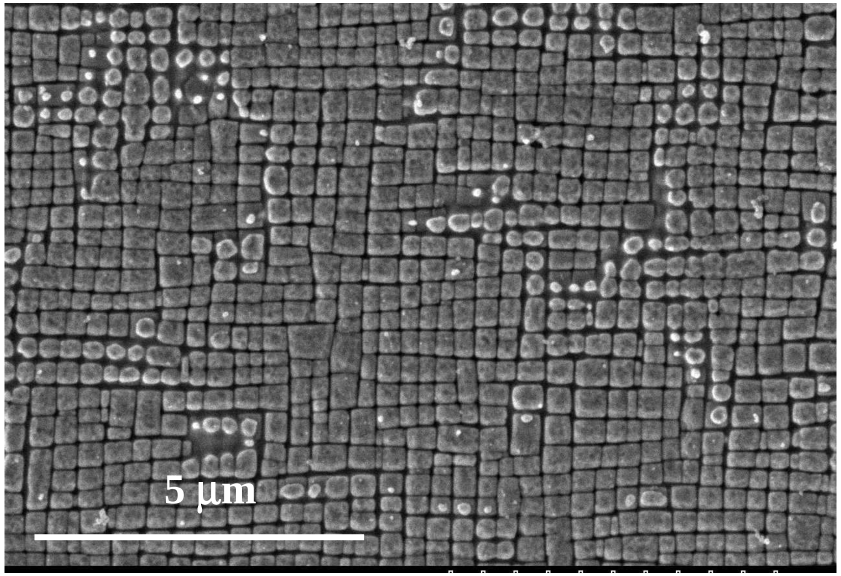
20µm

$t=t_{rupt}$ T<500°C

$t=t_{rupt}$



Thin wall problems

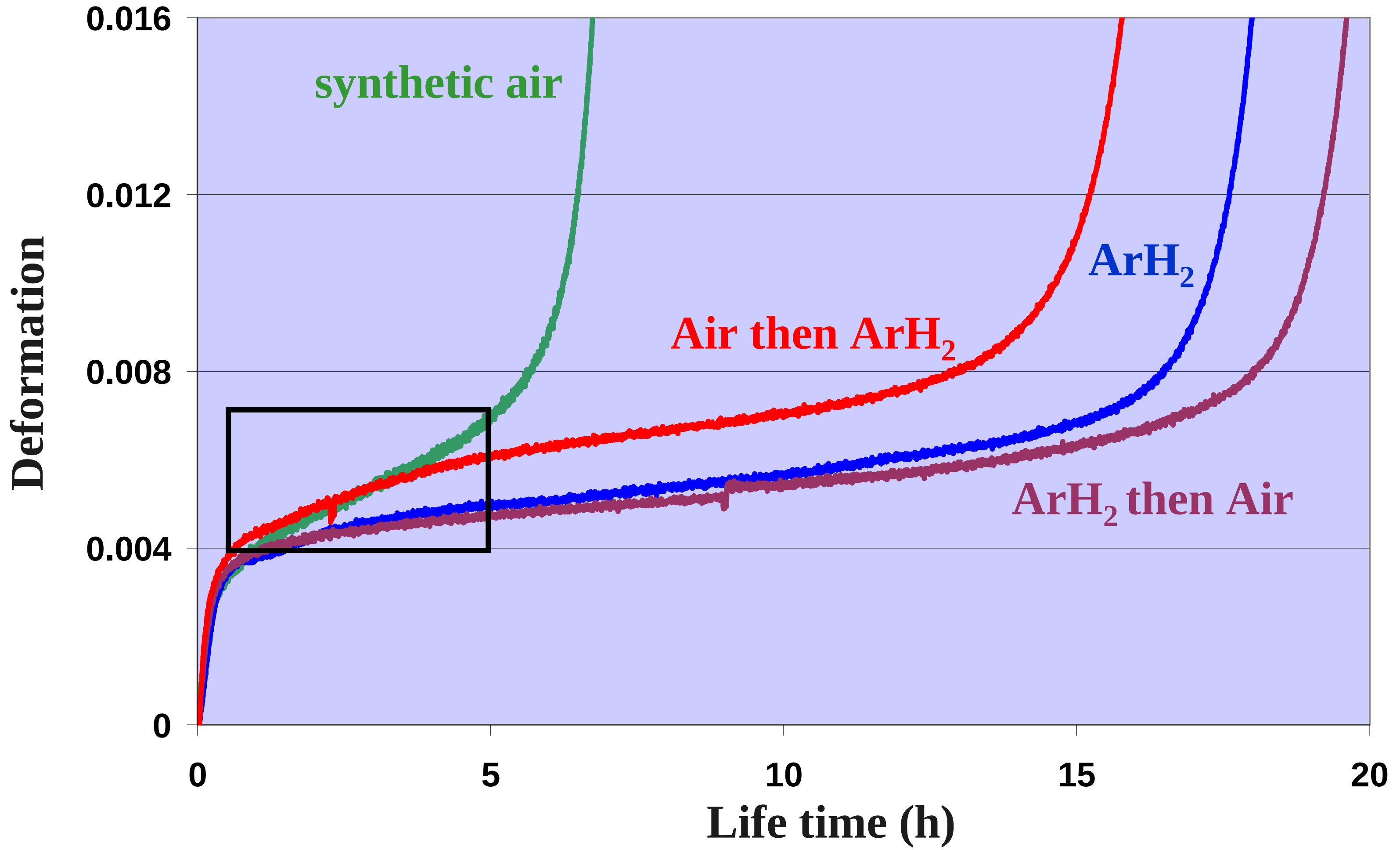


MC2 superalloy

Alliage	Ni	Cr	Co	Mo	W	Al	Ti	Ta	C
AM3	Base	8	5.5	2.25	5	6	2	3.5	
MC2	Base	8	5	2	8	5	1.5	6	<100 ppm

Creep resistant
up to 1100°C

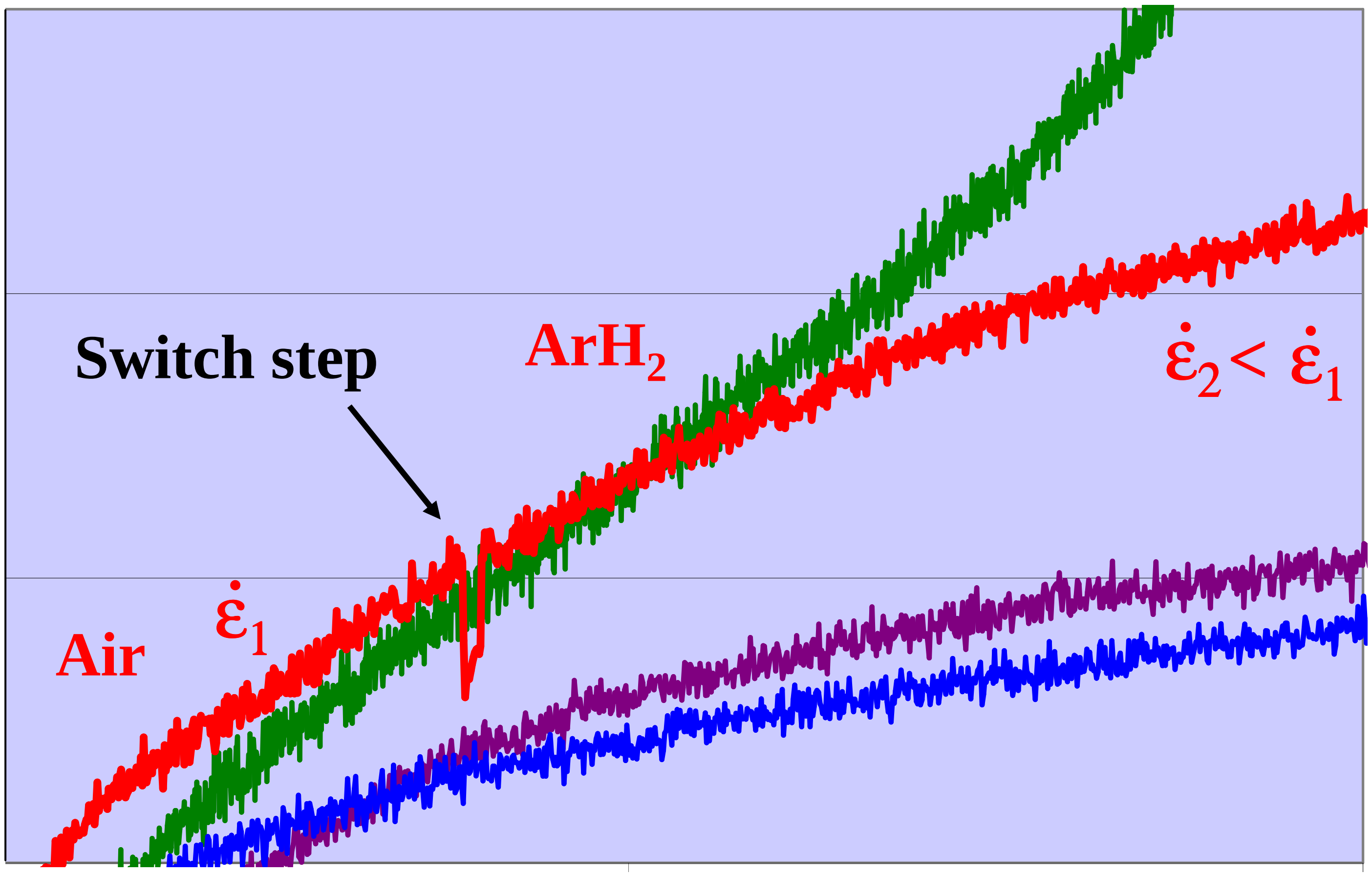
Effect of environment on creep behavior of MC2



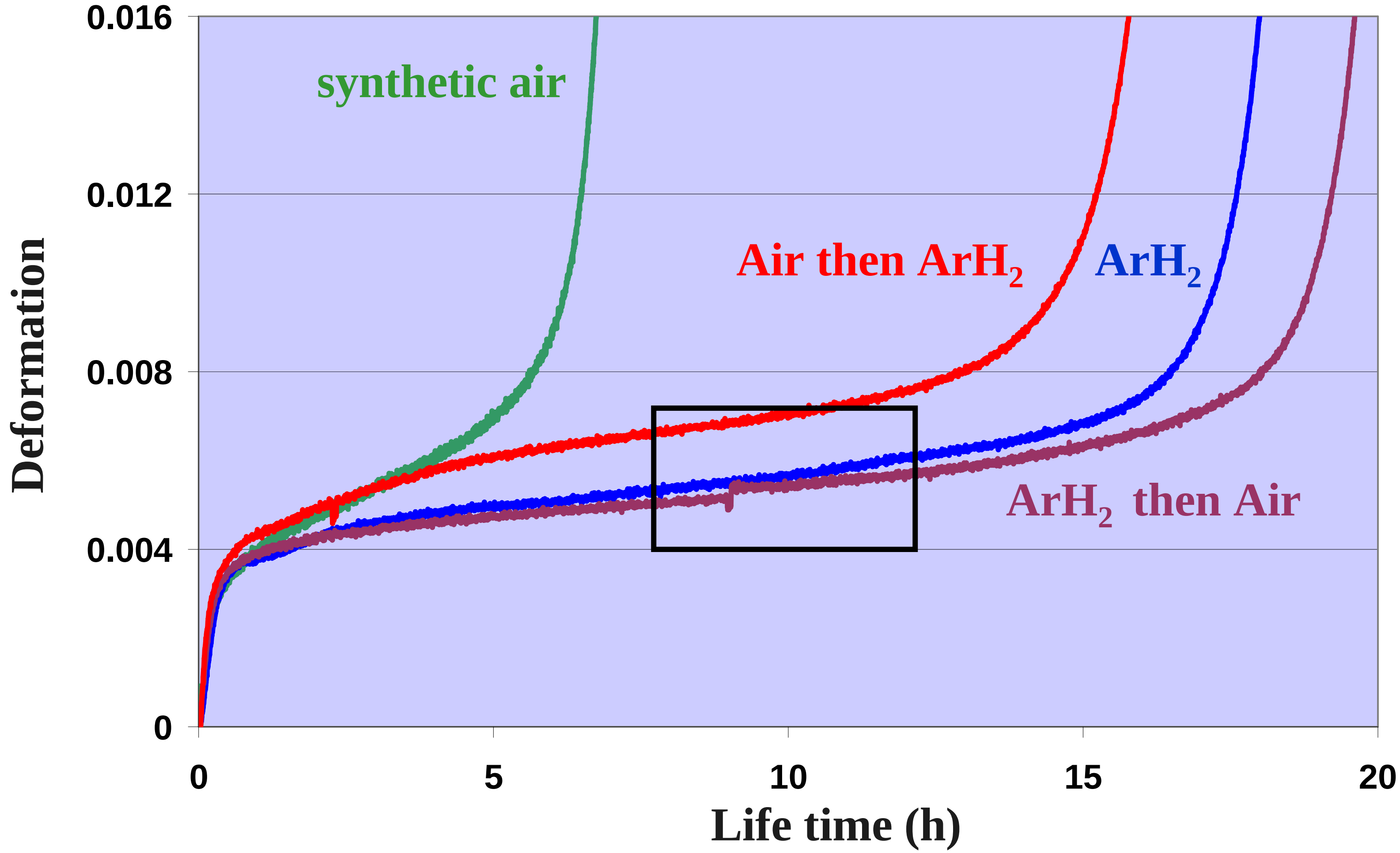
Creep of MC2 at 1150°C, 80 MPa on polished samples

S.Dryepondt PhD Thesis

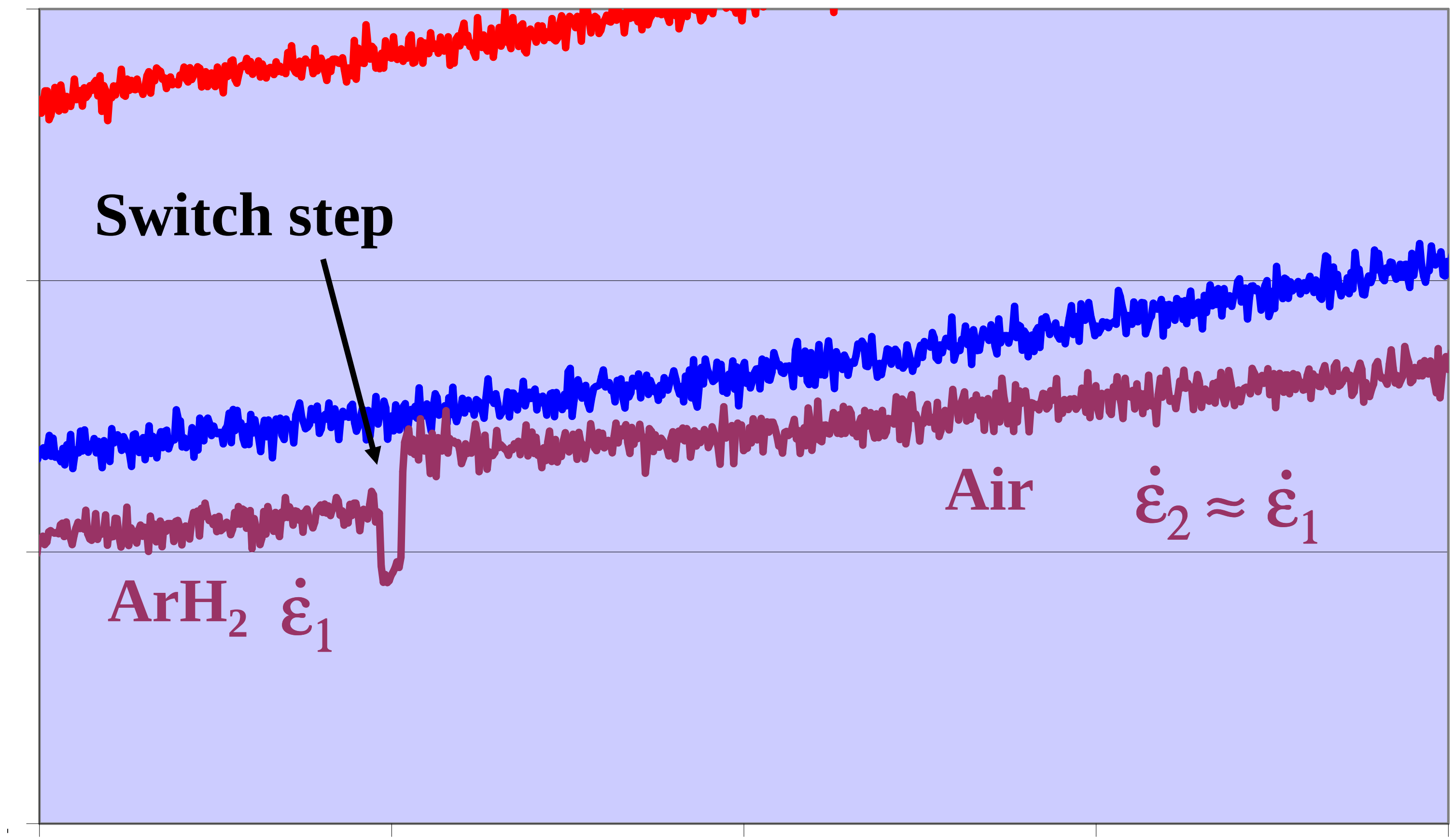
Effect of switching environment during the test



Creep strain rate variation after the switch step

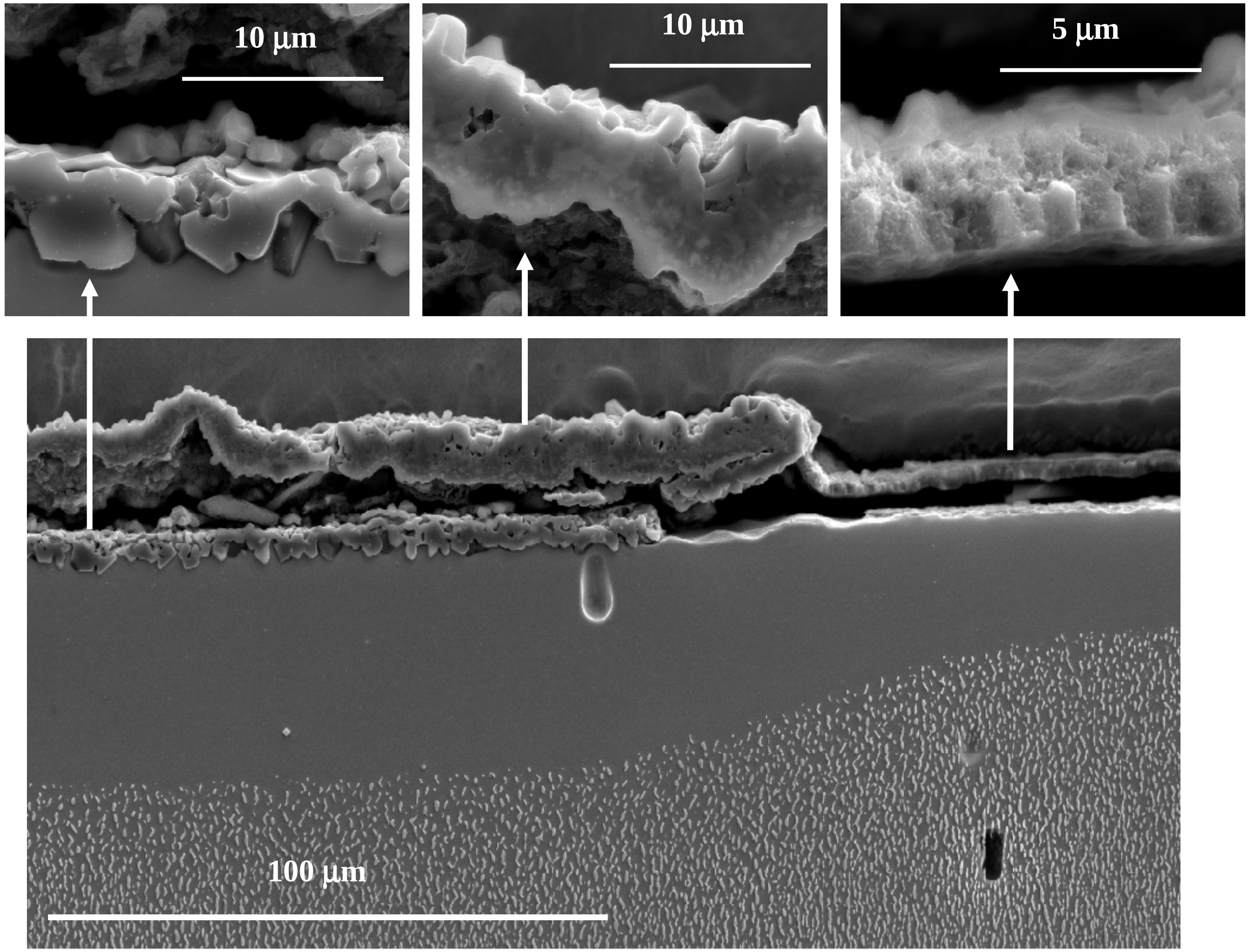


synthetic air
Air then ArH2
ArH2
ArH2 then Air
Deformation
0.016
0.012
0.008
0.004
0
0
5
10
15
20
Life time (h)



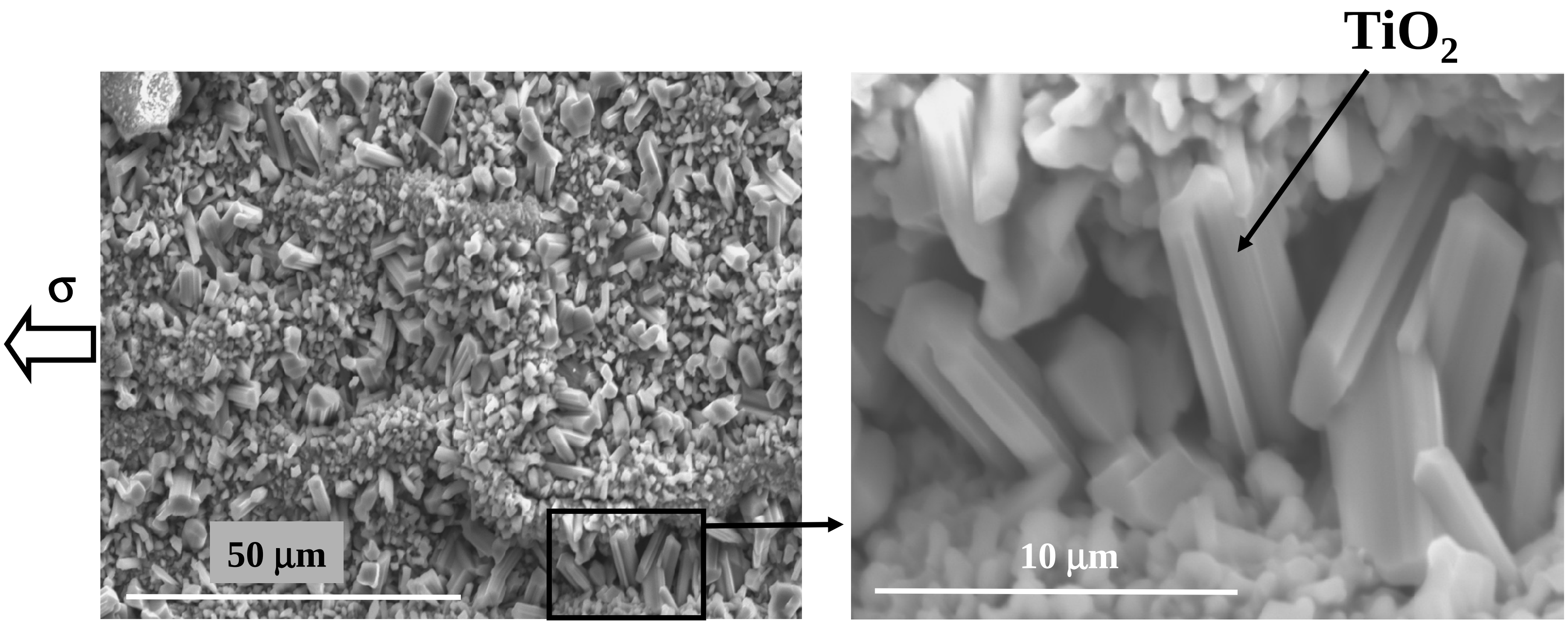
$\dot{\varepsilon}$ **remain unchanged after the switch step**

Creep under synthetic air conditions



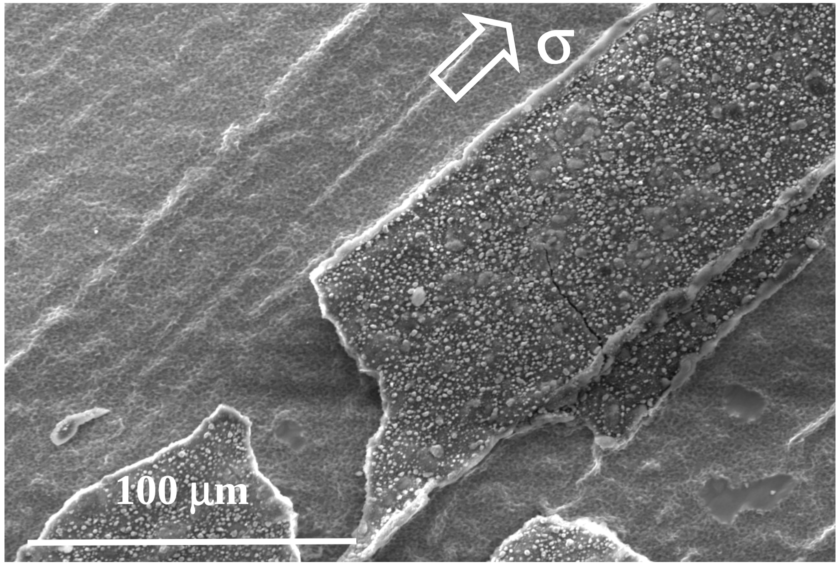
PFZ related to the local oxide scale thickness

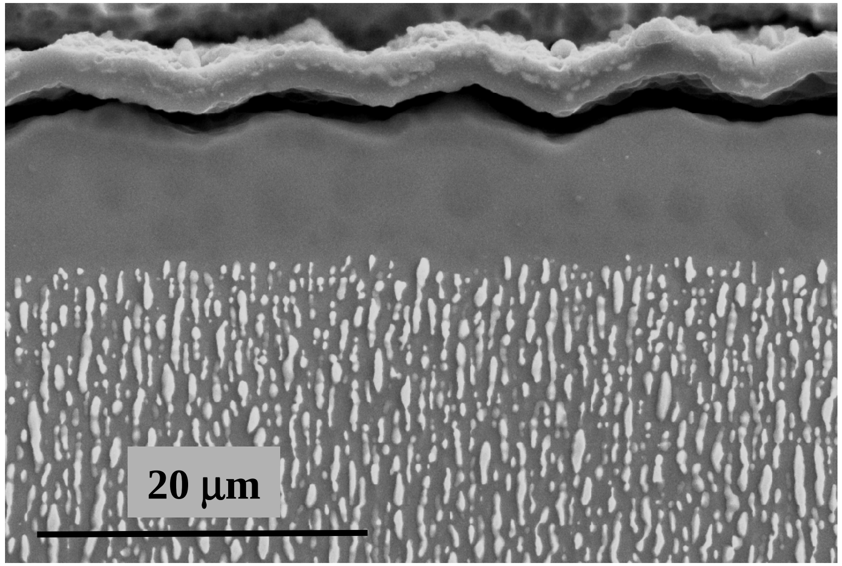
Creep under synthetic air



Alumina scale with other oxides

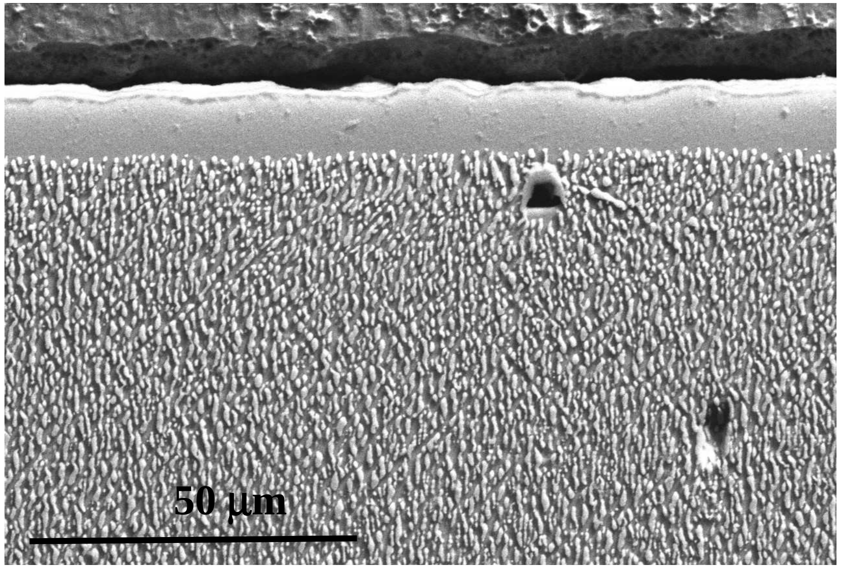
Creep under ArH_2

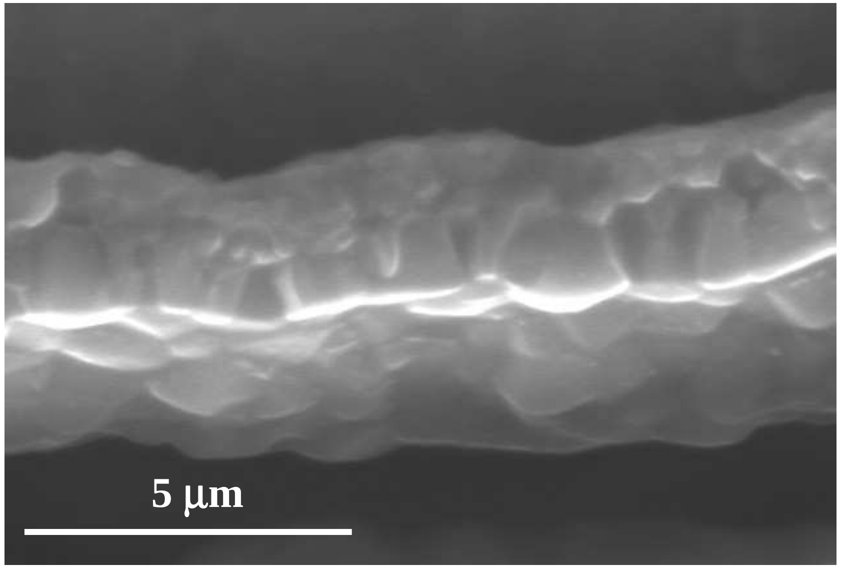




Alumina scale only

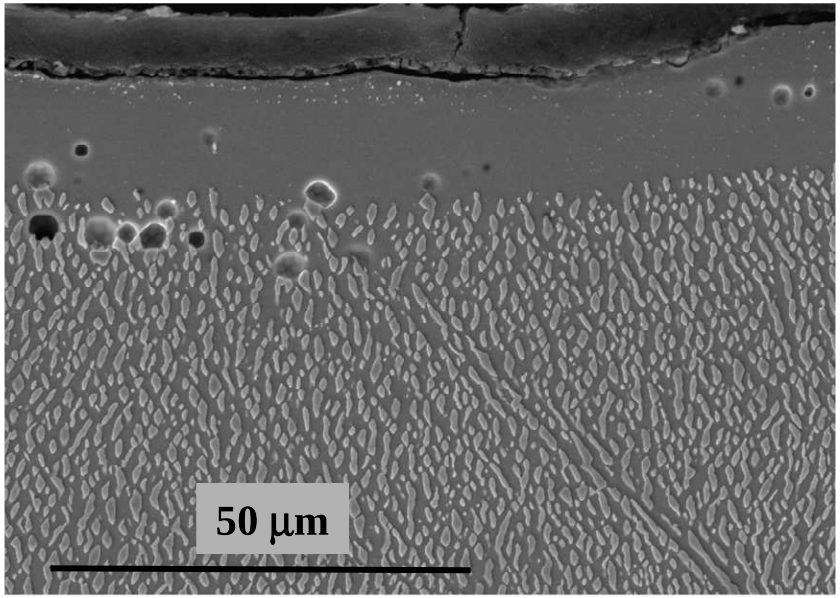
Creep under ArH_2 then under air testing conditions



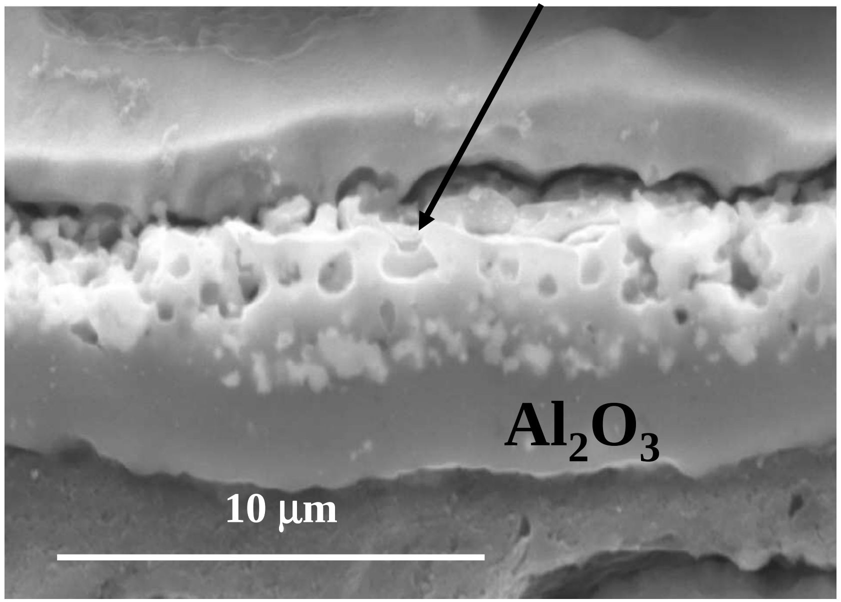


No difference with creep under ArH_2

Creep under air then under ArH_2 testing conditions



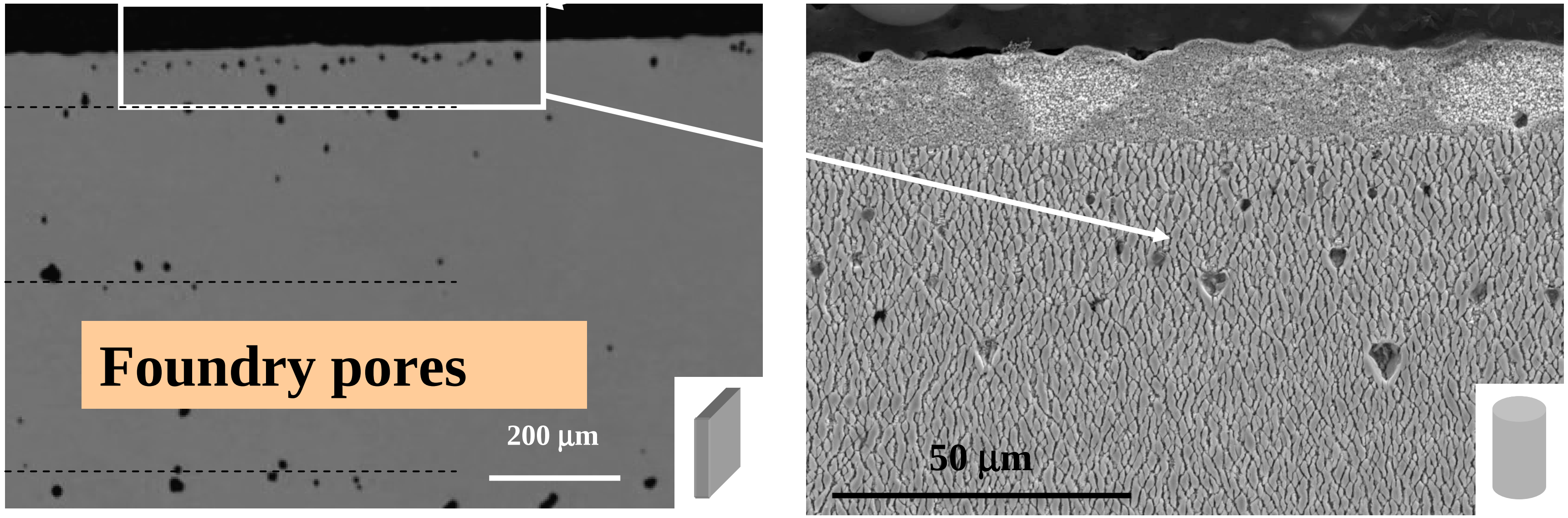
Ni, Ti, Cr oxides



Oxide microstructural evolution between the air and ArH_2 testing conditions.

Vacancies injection

Pores occurrence
[Hancok 76, Perusin 2004]



Effect of vacancies injection on diffusion processes and on dislocations climbing component [Gourgues 99]

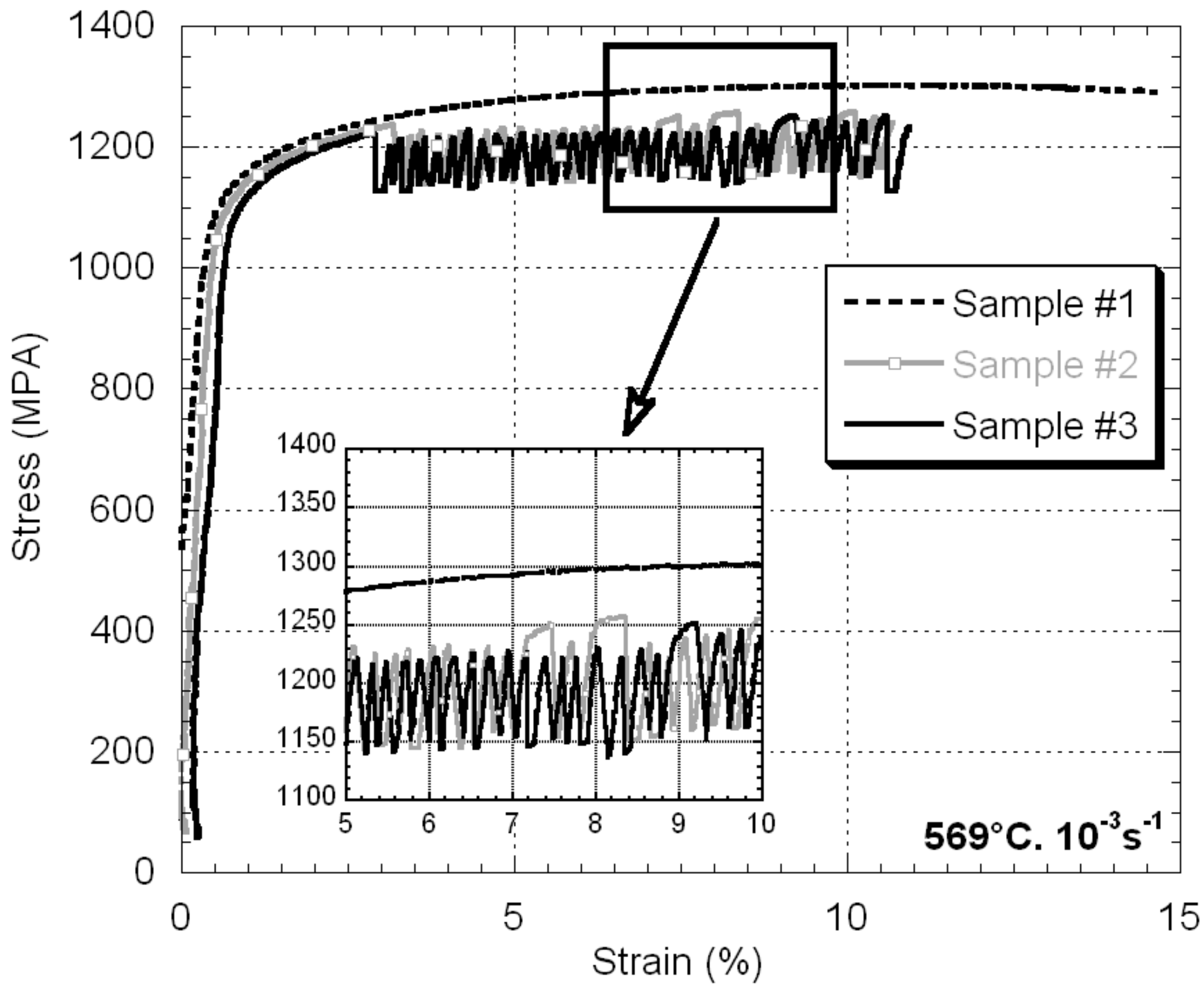
Coupling oxidation- deformation mode - fracture mode

Alloy 718

The effect of interstitial elements

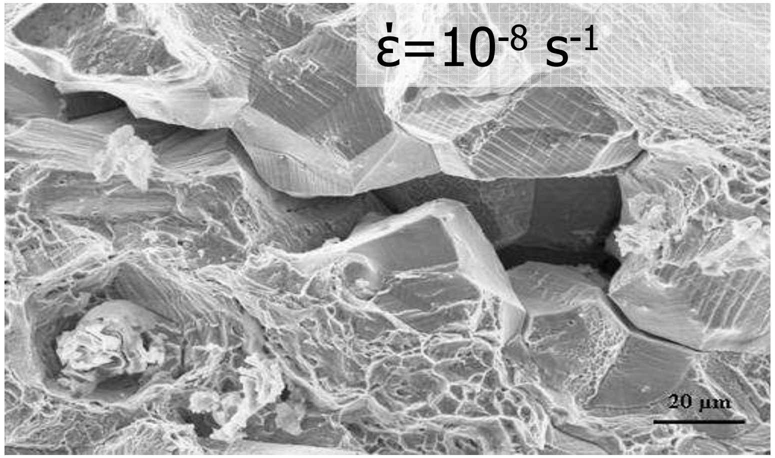
PhD Thesis: V.Garat, J.Deleume, B.Ter Ovanessian, B.Max, F.Galliano
INPToulouse

Dynamic Strain Aging and Portevin-Le Châtelier close to the triggering threshold



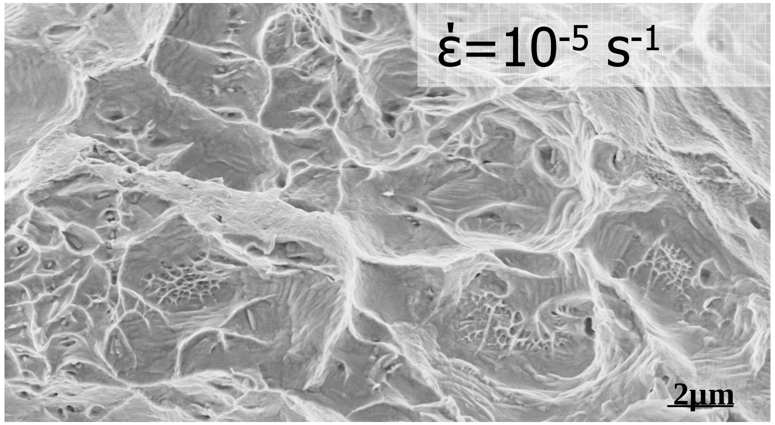
- Stress oscillations amplitude: 50 MPa.
- Under air testing conditions intergranular cracks initiate only in the DSA regime
- A critical deformation is needed to trigger PLC instabilities

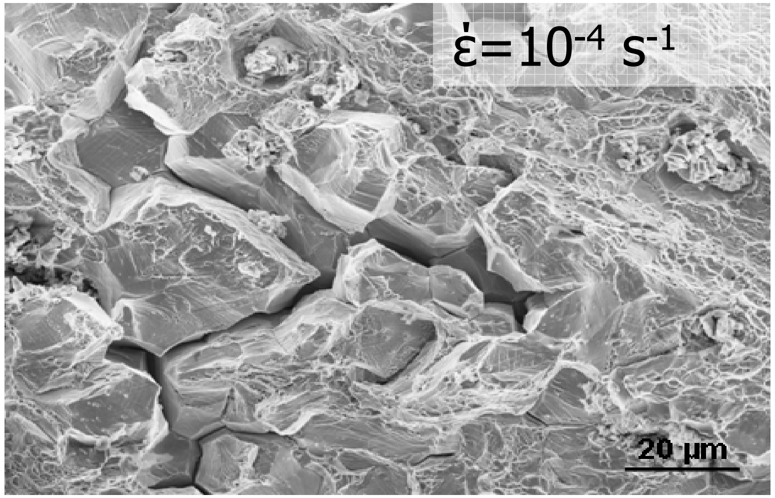
Similarities between fracture processes at different temperatures.



350°C

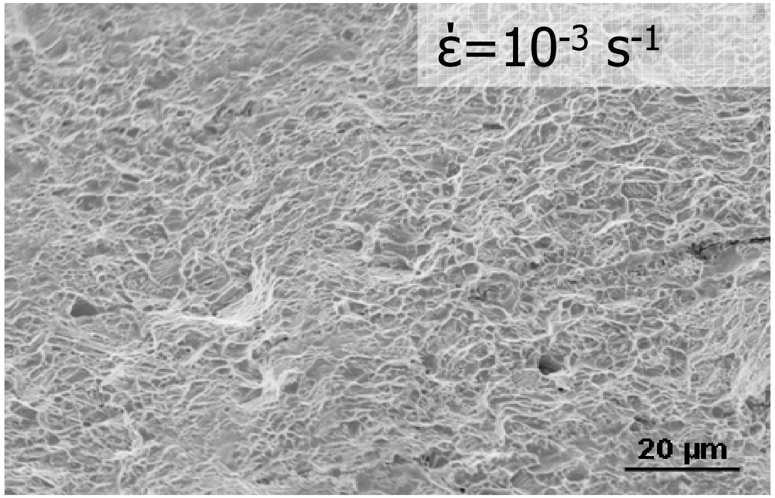
EAU





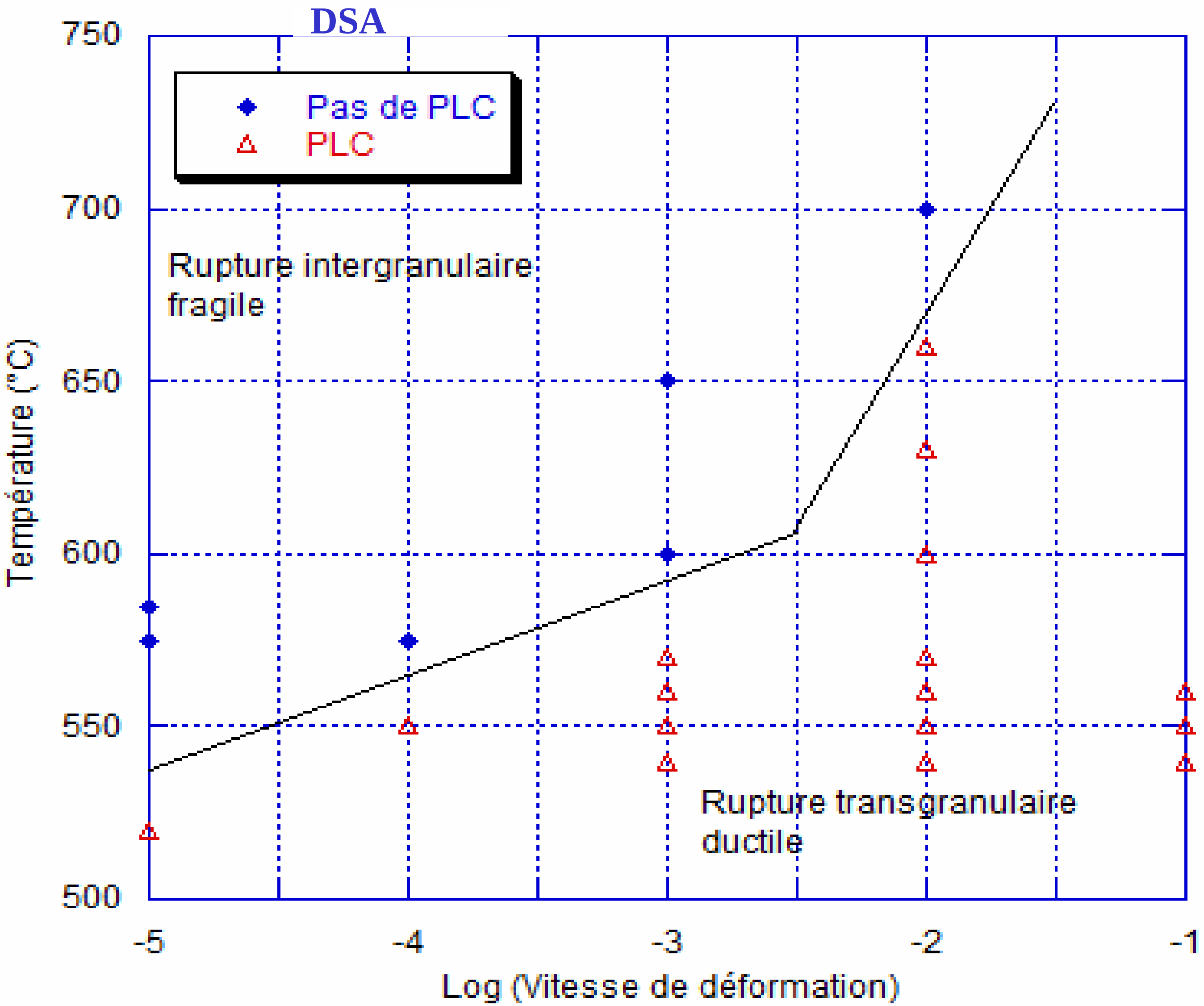
650°C

AIR

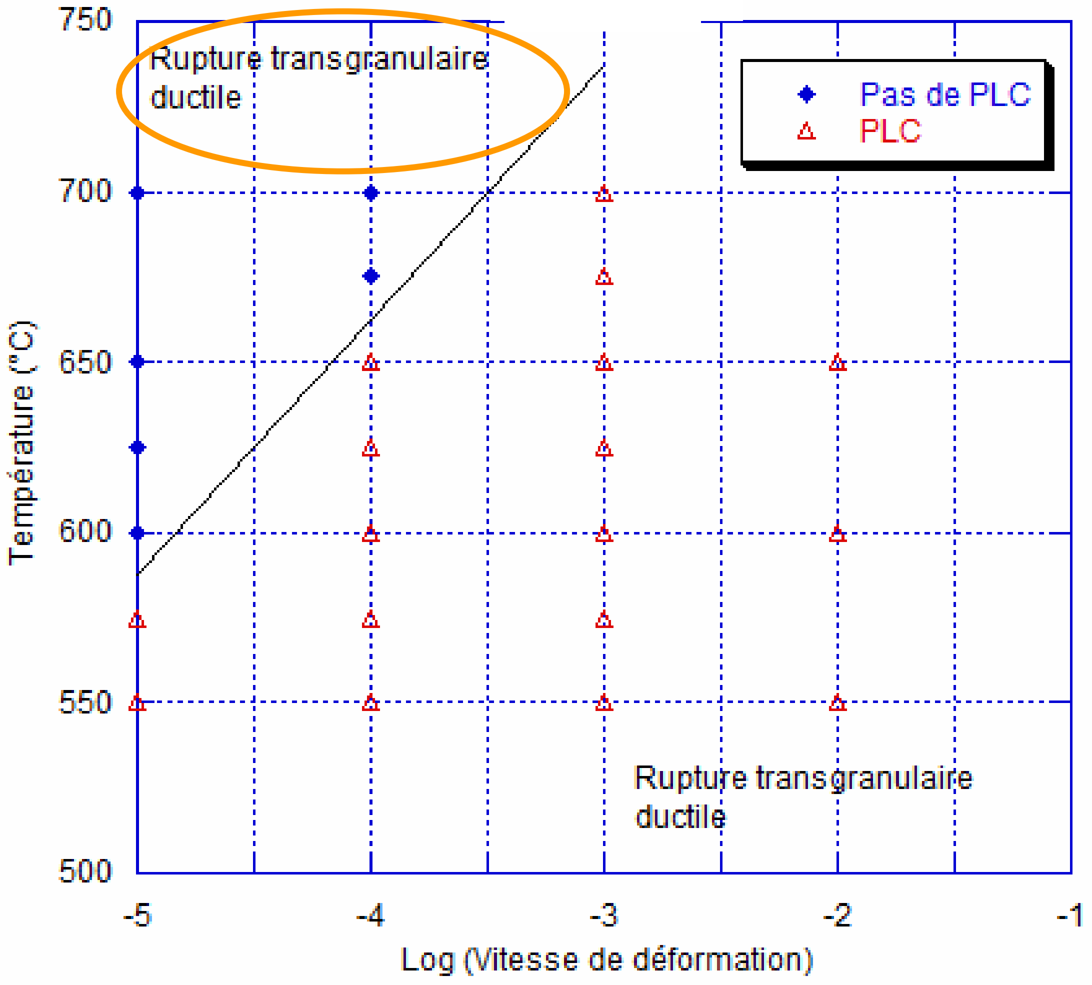


Tensile tests under air testing conditions:

Fracture mode changes from intergranular to transgranular at the border between DSA and PLC deformation modes.



Low carbon alloy



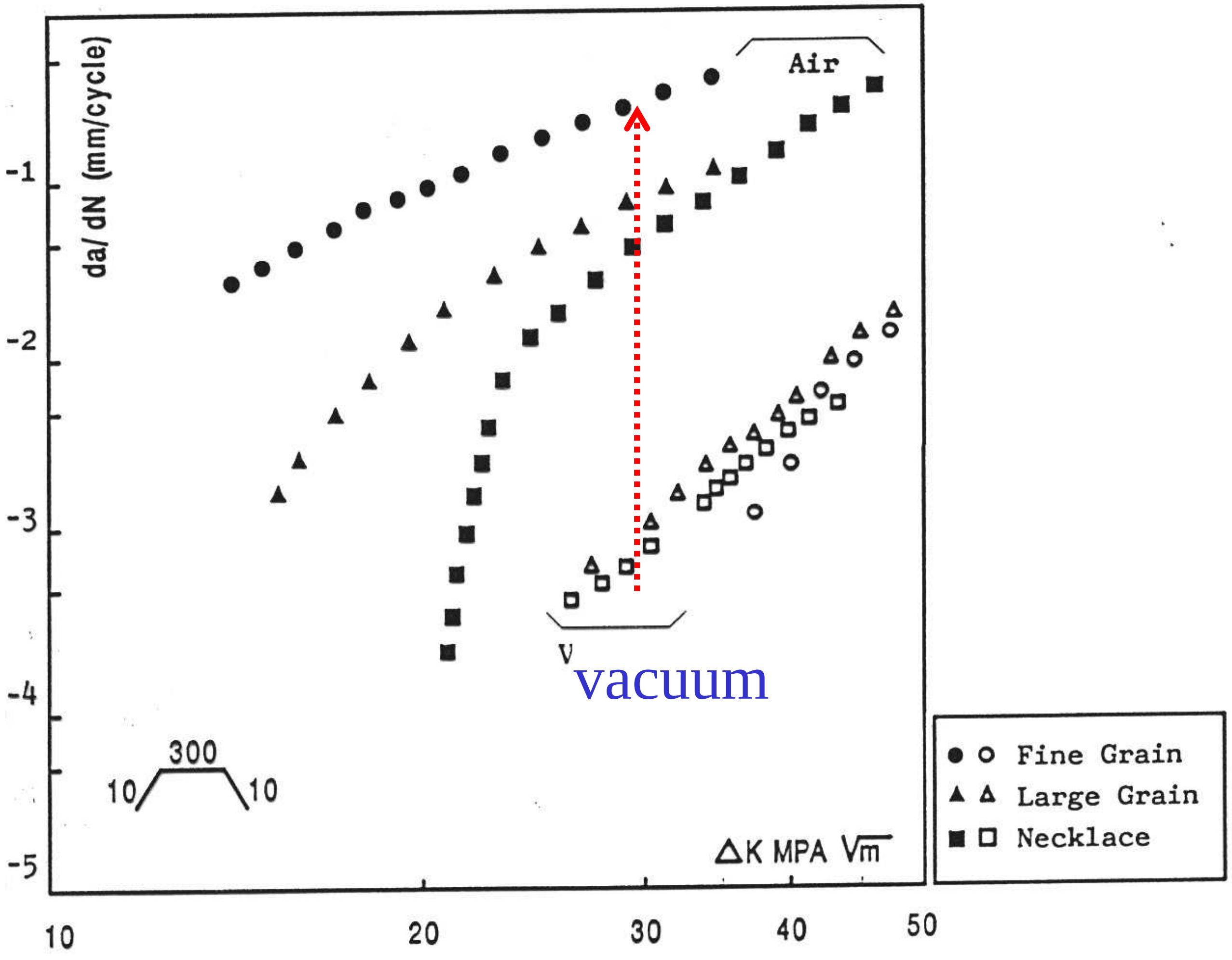
Effect of oxidation

on the local mechanical behavior

PhD Thesis ENSMP: R.Molins, A.F Gourgues, G.Hochstetter, J.C Chassaigne

PhD Thesis INPT: J.Deleume, B.Ter Ovanessian

Effect of the microstructure on the creep-fatigue crack growth rate of alloy 718 at 650°C

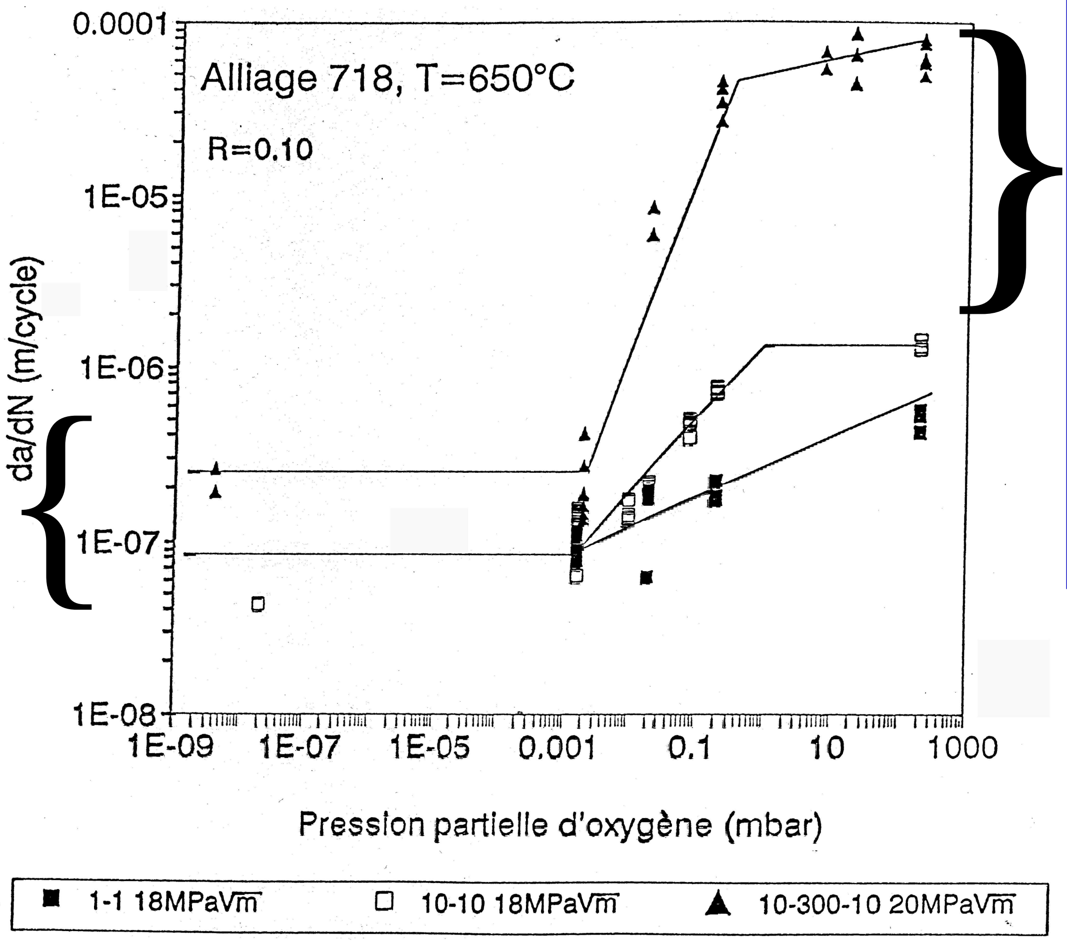


(Pineau-Pedron 1982)

Effect of the oxygen partial pressure on the crack growth rate under constant ΔK

•Trangranular fracture

•Selective oxidation of chromium



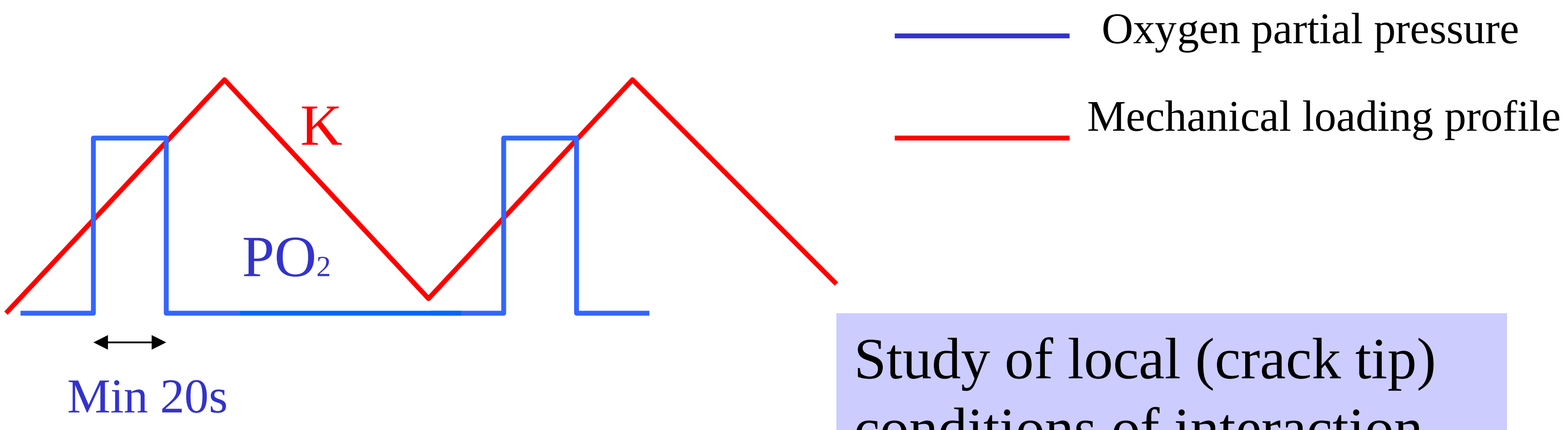
•Intergranular fracture

•The first oxide to grow is: (Ni,Fe)O

•Cationic growth process

•high oxide growth rate (transient regime)

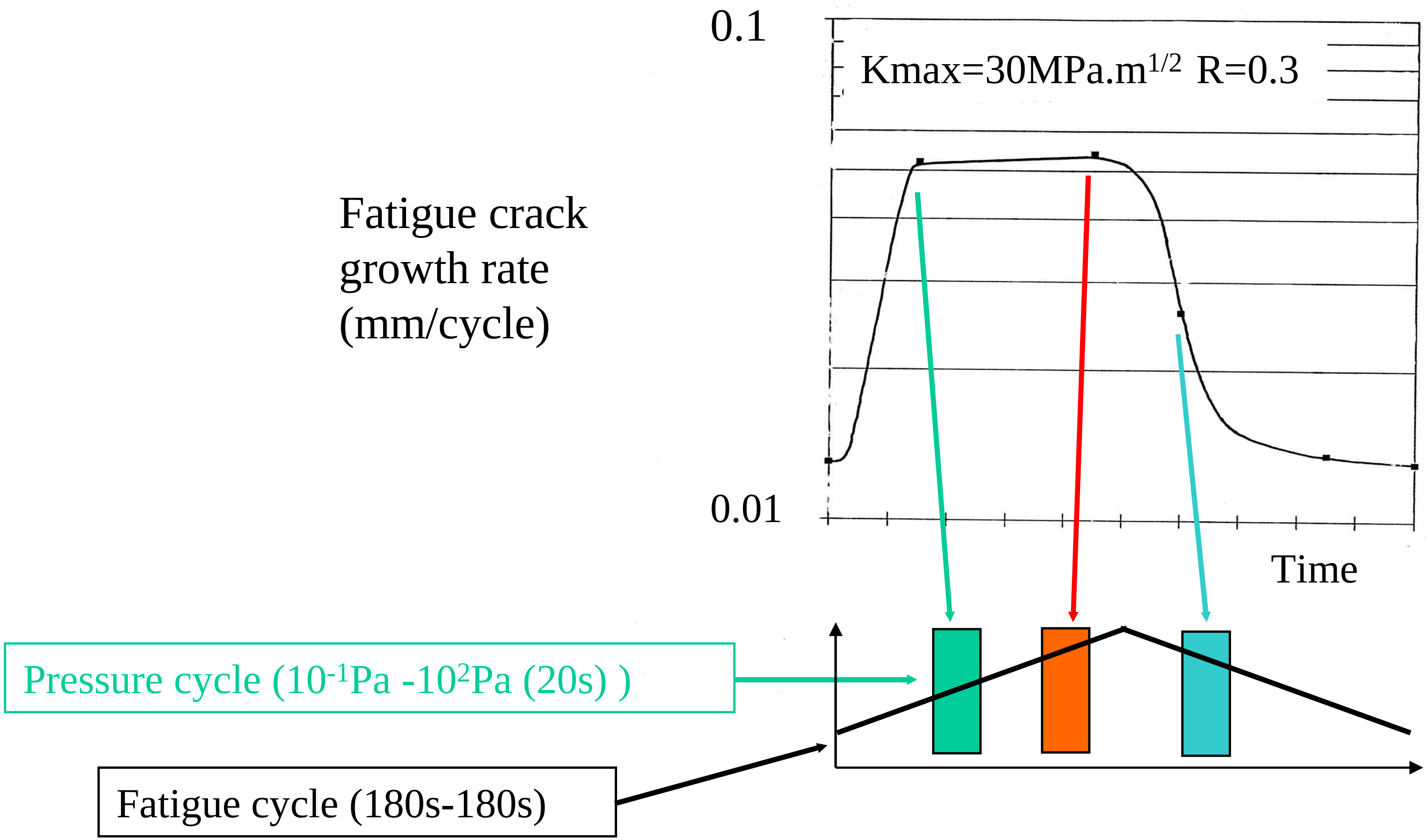
Types of experiments carried out on several nickel base superalloys



Study of local (crack tip) conditions of interaction between oxidation and deformation

Creep-fatigue

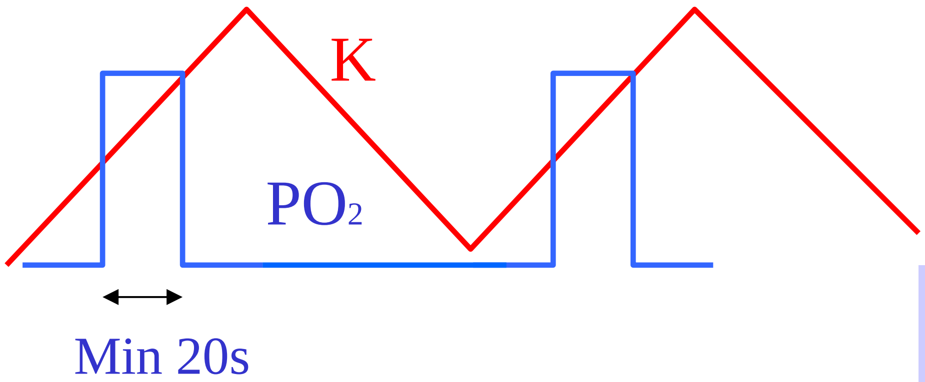
Effect of the relative position of an oxygen pressure cycle on the fatigue crack growth of alloy 718 at 650°C



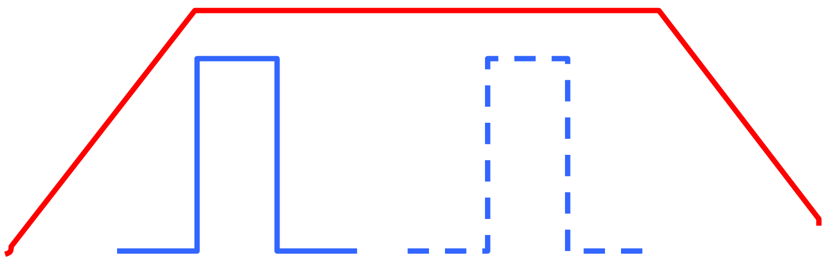
Types of experiments carried out on several nickel base superalloys

Oxygen partial pressure

Mechanical loading profile



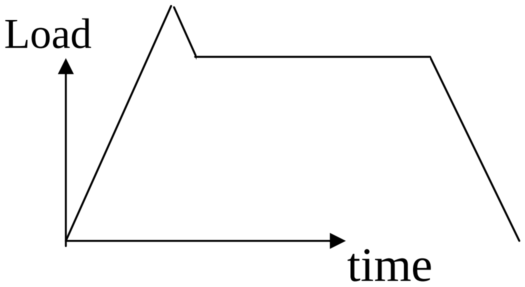
Study of local interactions between oxidation and deformation at a crack tip



Creep-fatigue

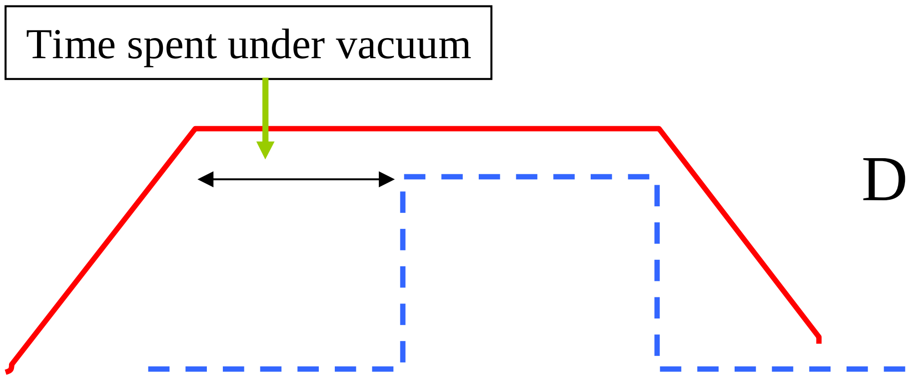
Is there a way to stop Oxidation Assisted Cracking?

For creep resistant alloys (Ni based superalloys)



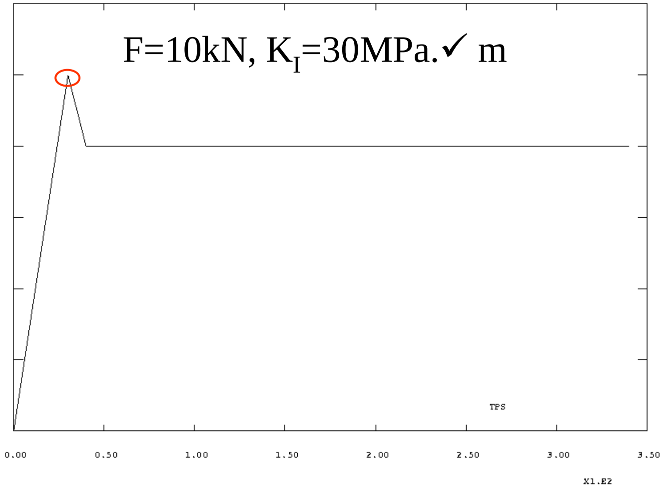
Unloading between 5 and 10%

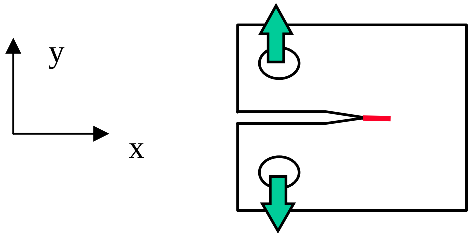
Compression stresses at the tip of the crack

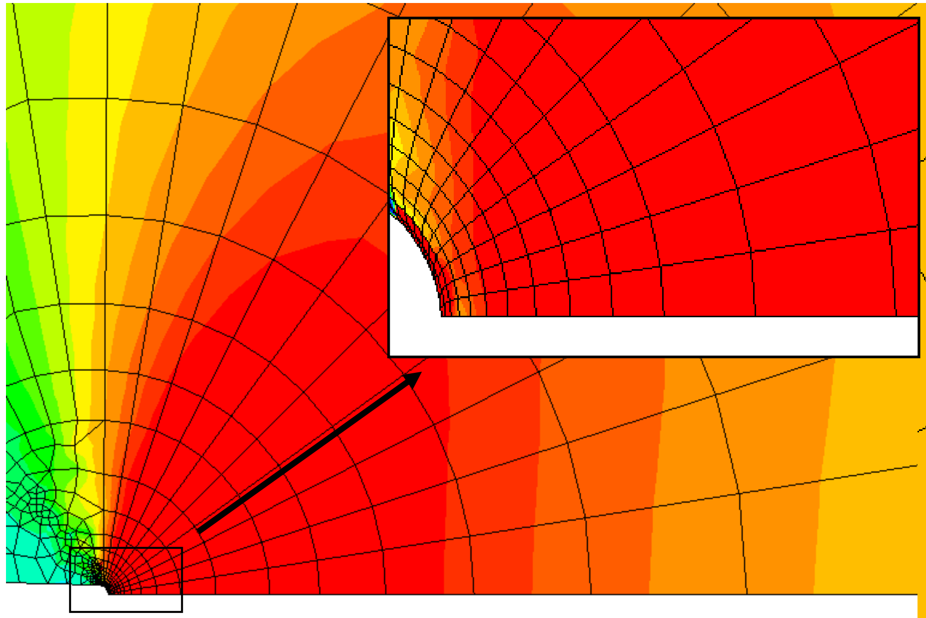


Delaying the oxidation effect

$\dot{\varepsilon}_{local}$ is reduced

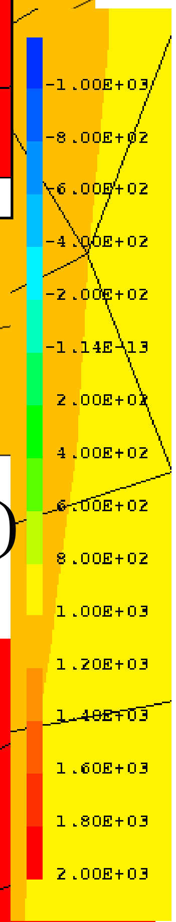


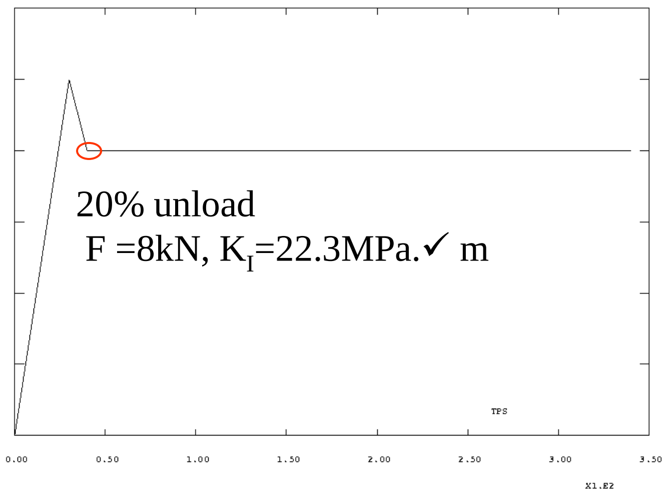


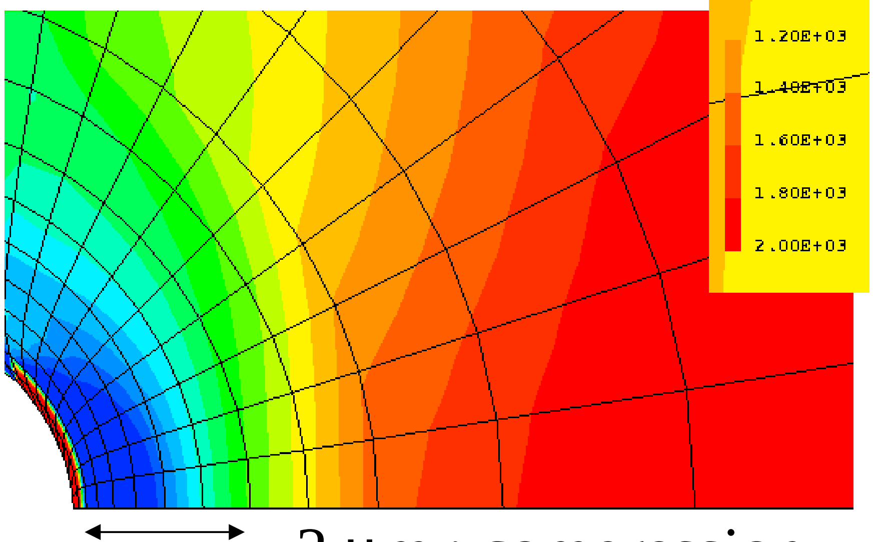


CT specimen (FEM 2D & plane strain)
Stress at the crack tip

σ_{yy}(MPa)



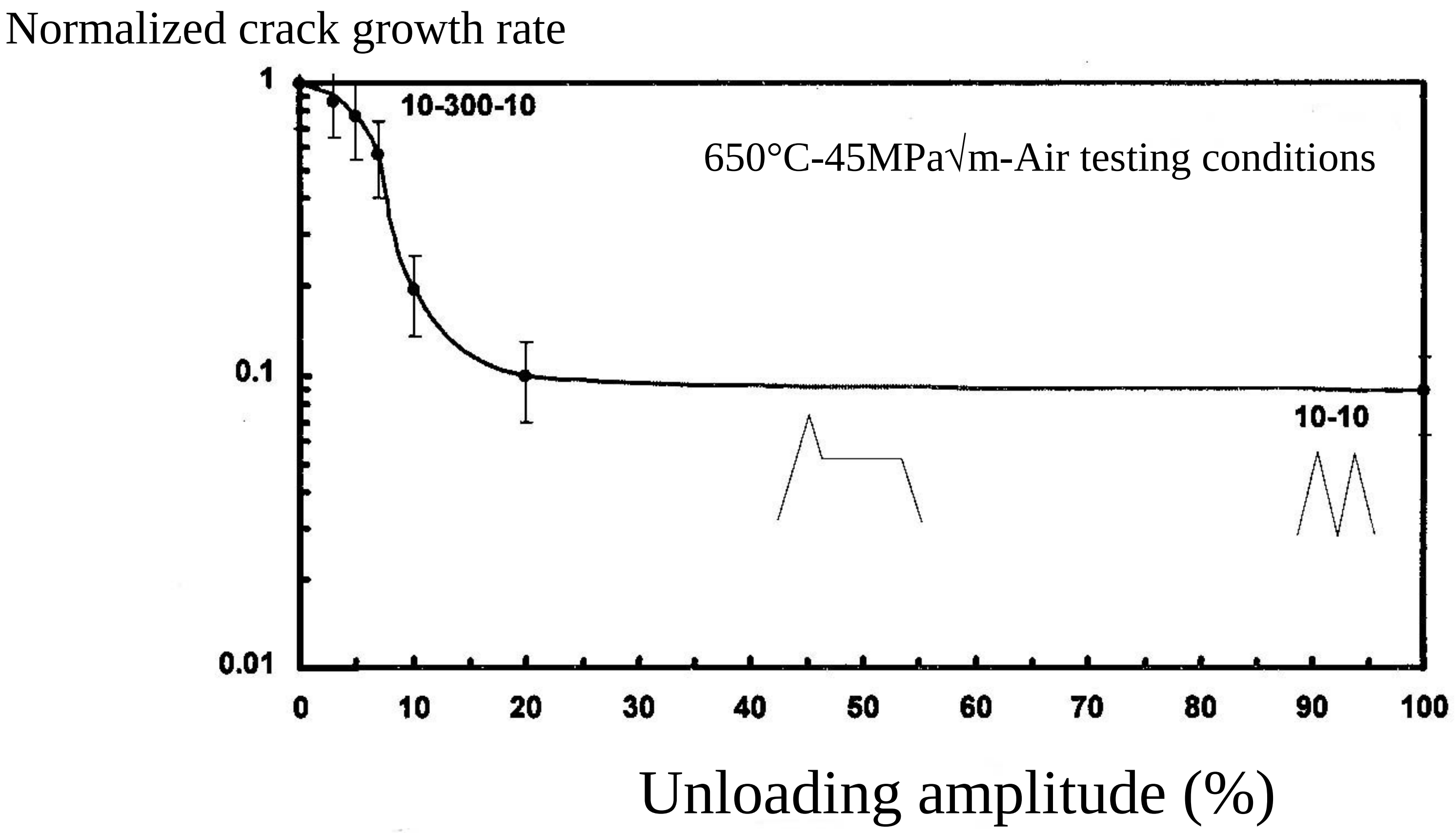




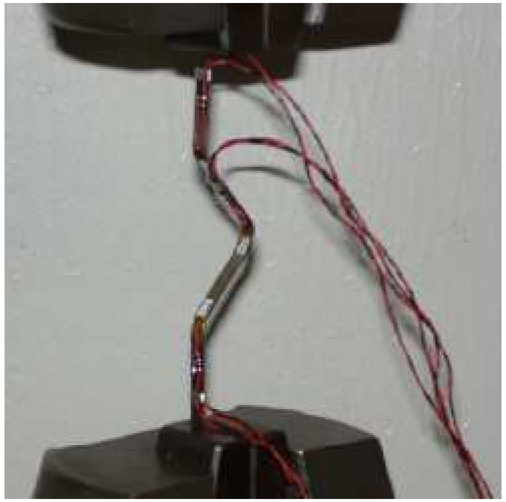
2 µm : compression

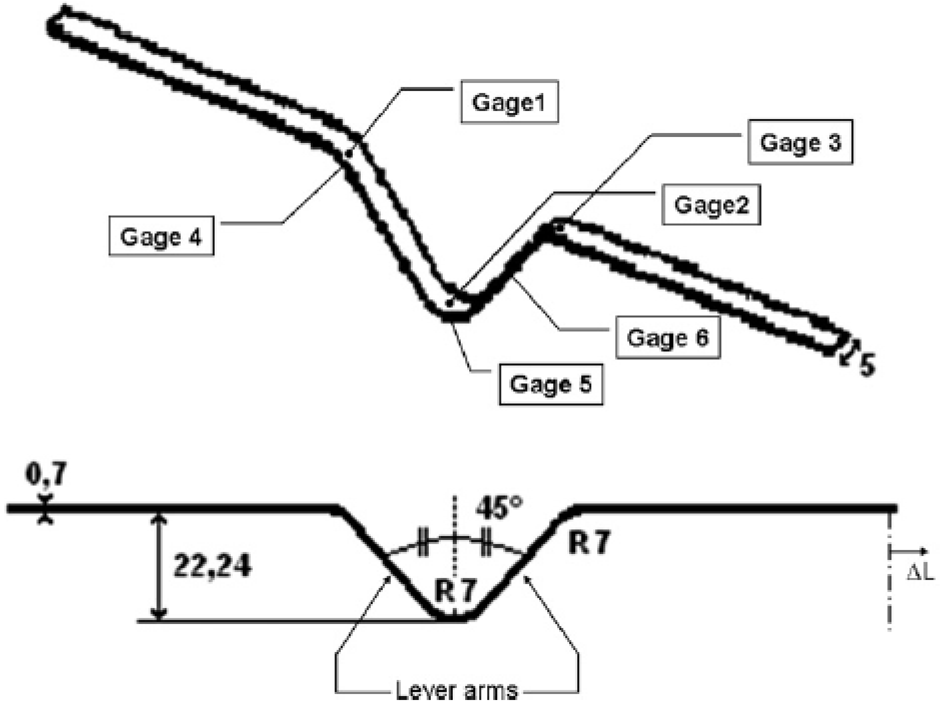
D.Poquillon   CIRIMAT

Effect of an unloading at the beginning of the hold time at Kmax on the creep-fatigue cracking resistance of N18

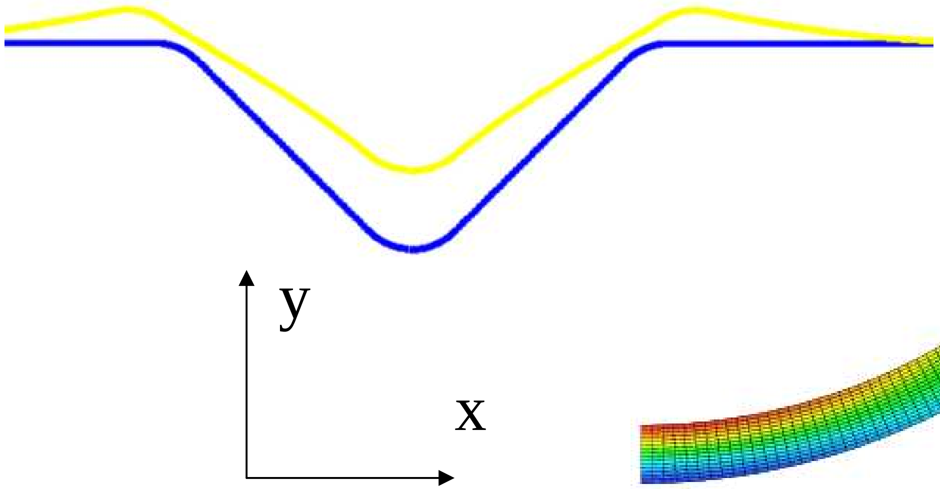


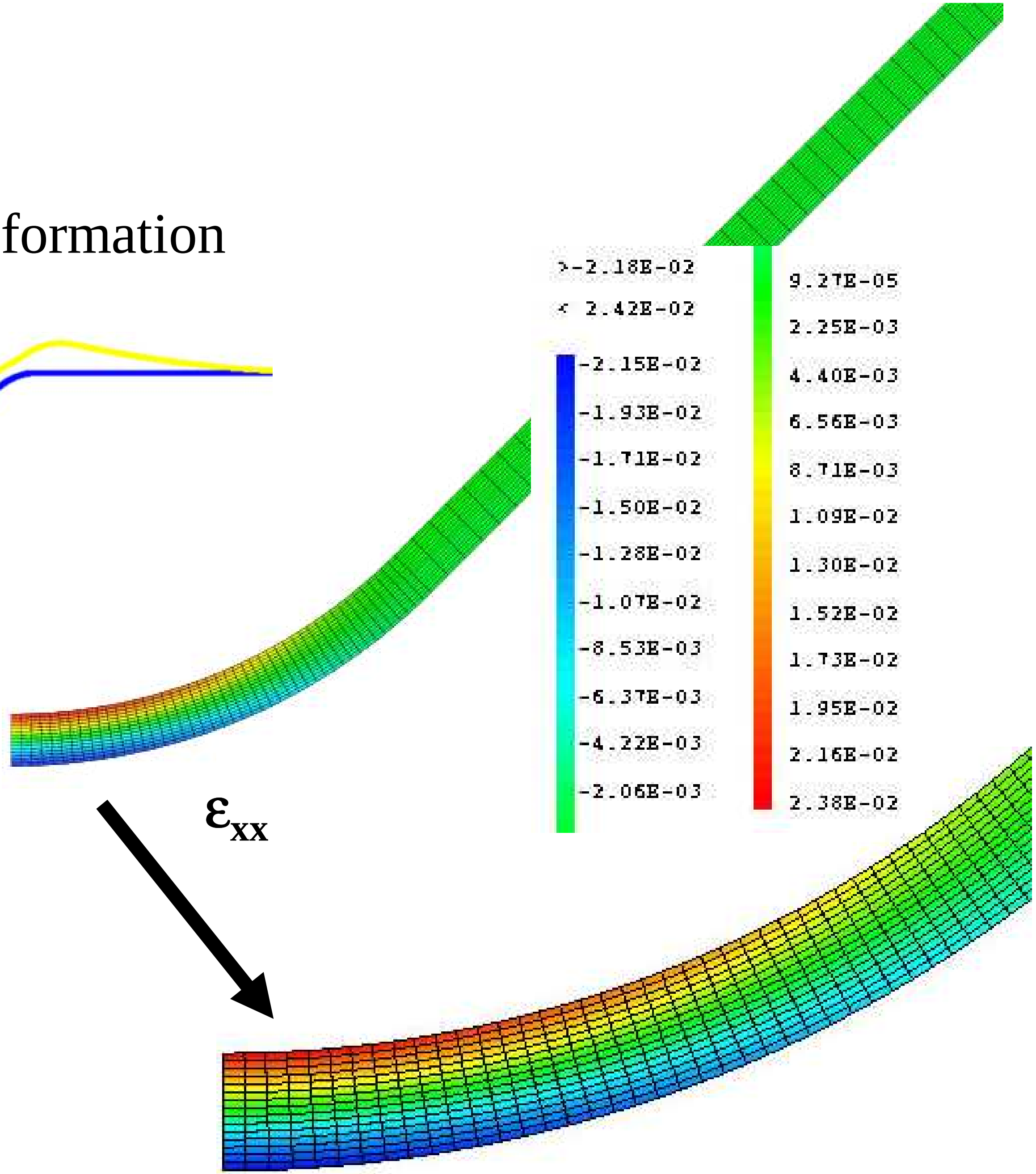
Development of a V shape specimen in order to explore the coupling between oxidation and mechanical behavior when deformation localisation occurs.





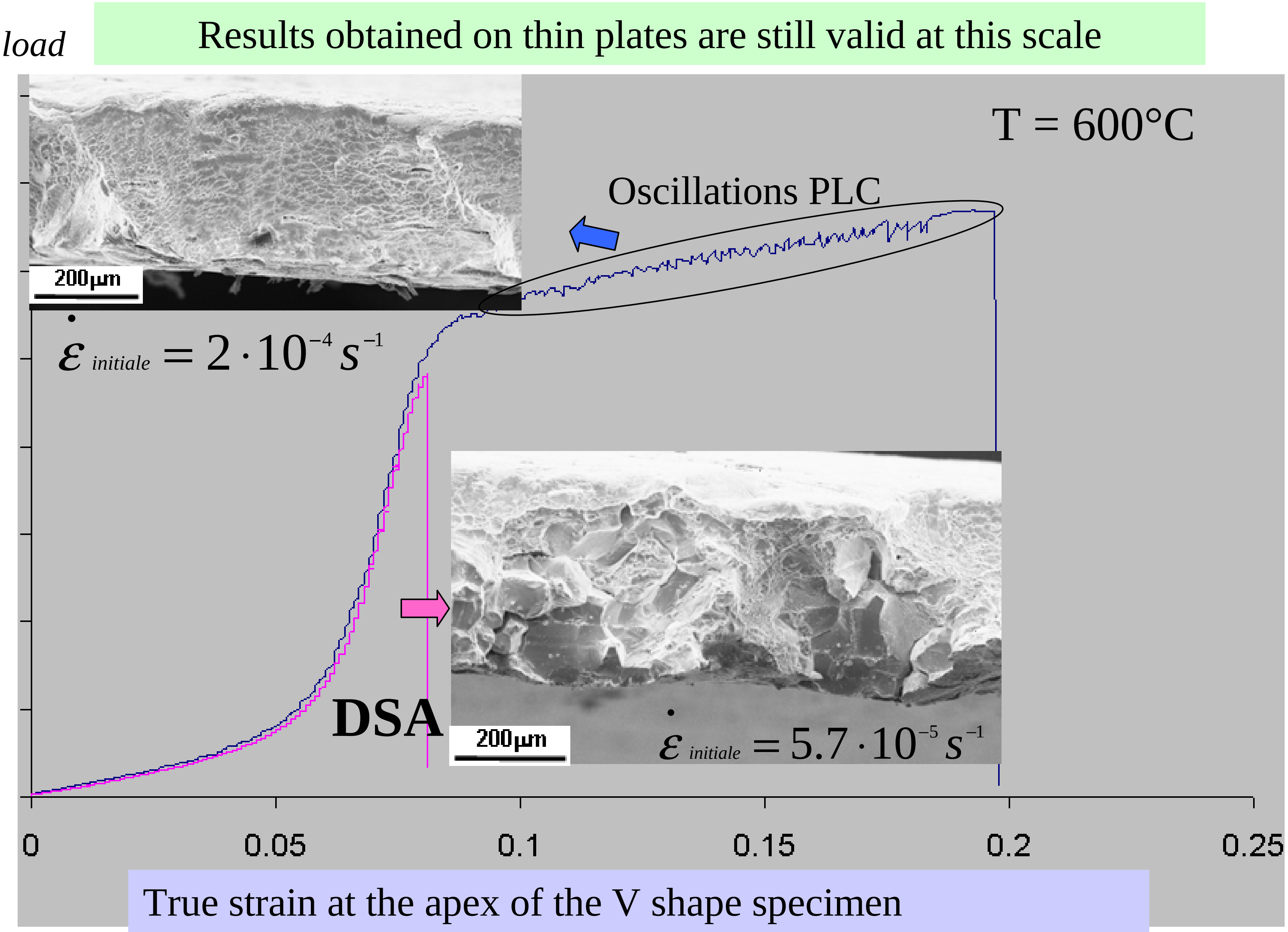
Localisation of the deformation





>2.5%

D.Poquillon Cirimat


load
Results obtained on thin plates are still valid at this scale
T = 600°C
Oscillations PLC
200µm
$\dot{\varepsilon}_{initiale} = 2 \cdot 10^{-4} s^{-1}$
DSA
200µm
$\dot{\varepsilon}_{initiale} = 5.7 \cdot 10^{-5} s^{-1}$
0
0.05
0.1
0.15
0.2
0.25
True strain at the apex of the V shape specimen

Perspectives and challenges

Design of new experiments in order to validate models (DFT, etc..)

Design of new materials or surface treatments to improve structural integrity

To Increase the data base in this scientific field

To federate other researchers in order to cover the different aspects and damaging processes of these interactions.