Mesures autour des couplages oxydation haute température-mécanique

Compiègne, 5-6 juin 2019

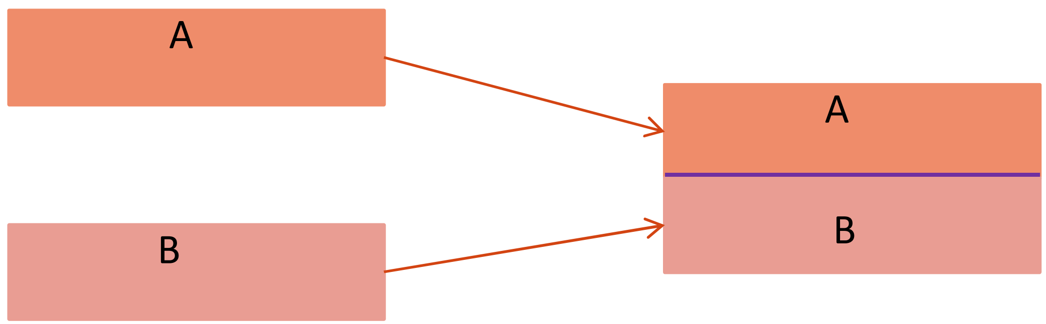
Relations entre microstructure induite par l'oxydation et adhérence : cas de l'acier Fe-17,5Cr-8,1Ni (304 L)

Valérie PARRY, Muriel BRACCINI, Céline PASCAL, Guillaume PARRY

Mesure d'adhérence ?

G_c toughness or fracture energy ($J.m^{-2}$)

$$G_c = W_{adhesion} + W_{dissipative}$$

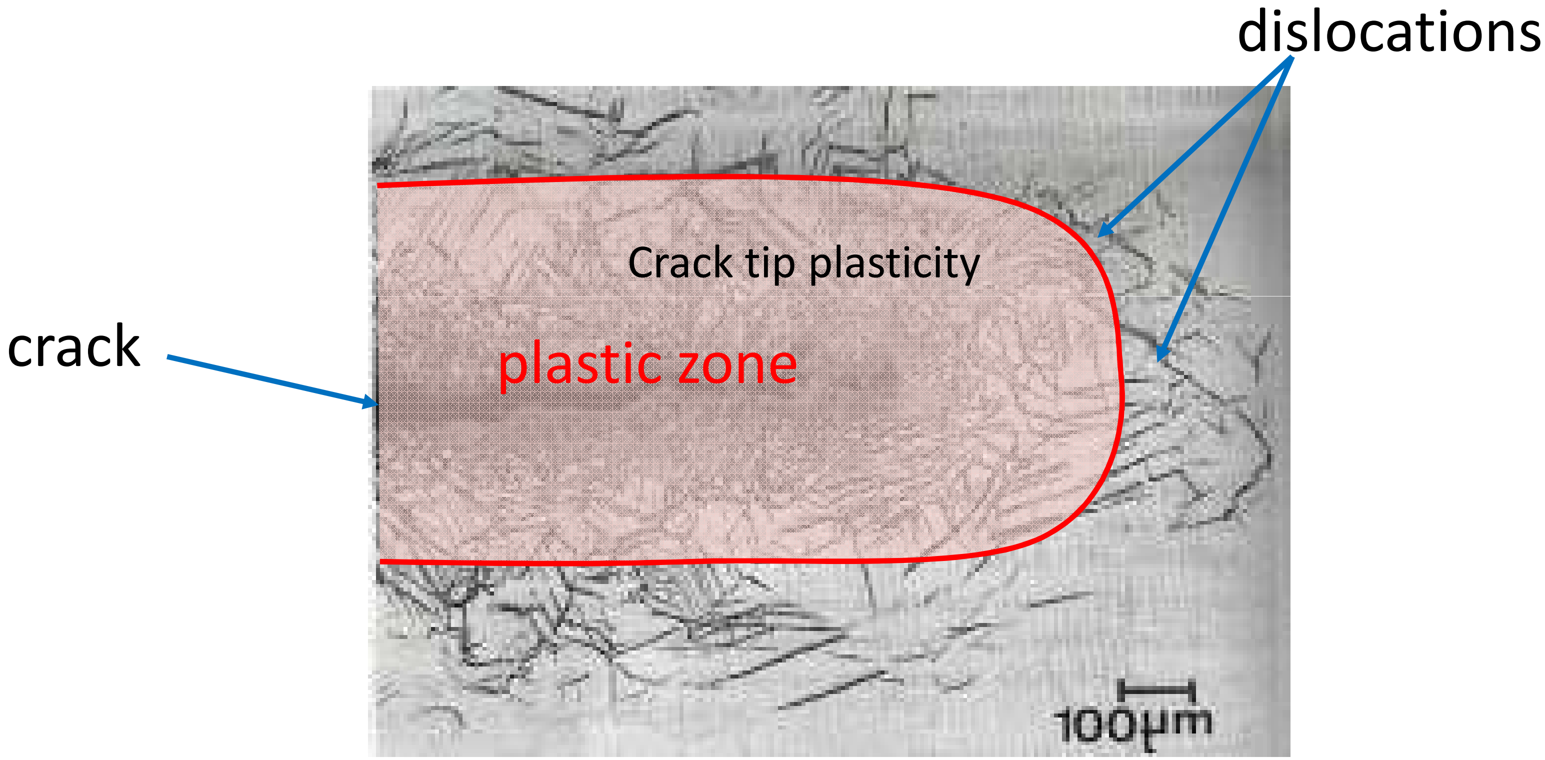


$$W_{adhesion} = \gamma_A + \gamma_B - \gamma_{AB} \cong 1\,\mathrm{J.m^{-2}}$$

Mesure d'adhérence ?

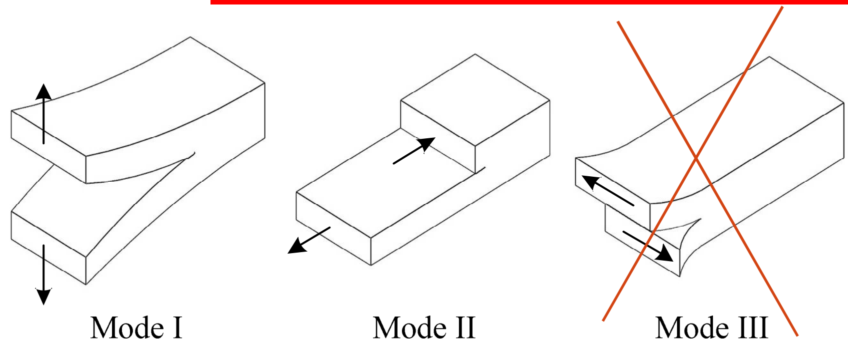
G_c toughness or fracture energy ($J.m^{-2}$)

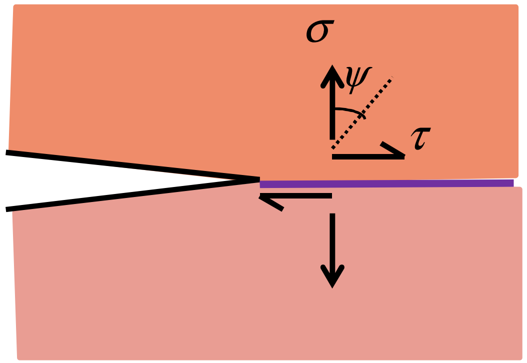
$$G_c = W_{adhesion} + W_{dissipative}$$

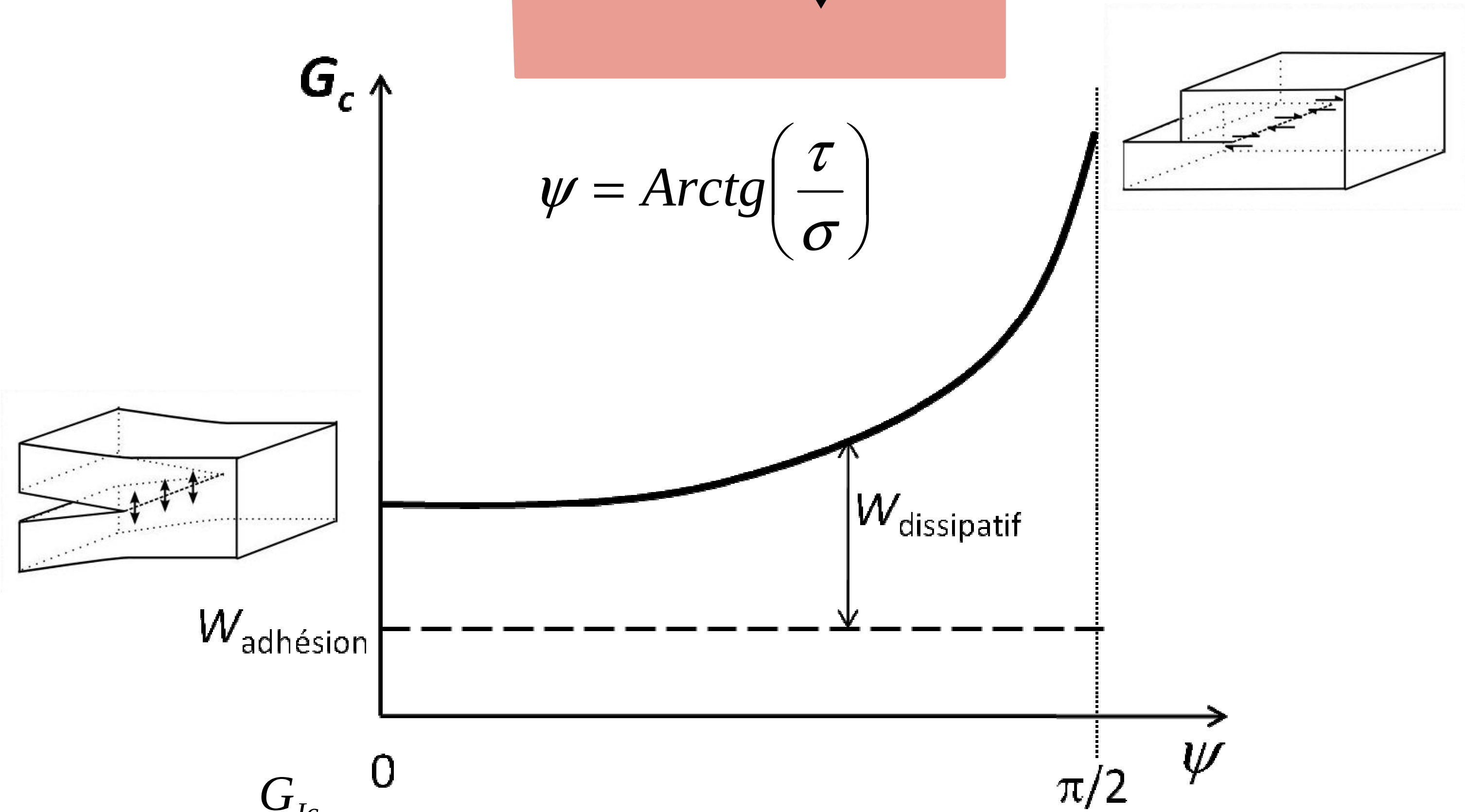


$$W_{adhesion} << W_{dissipative}$$

Propagation d’une fissure interfaciale





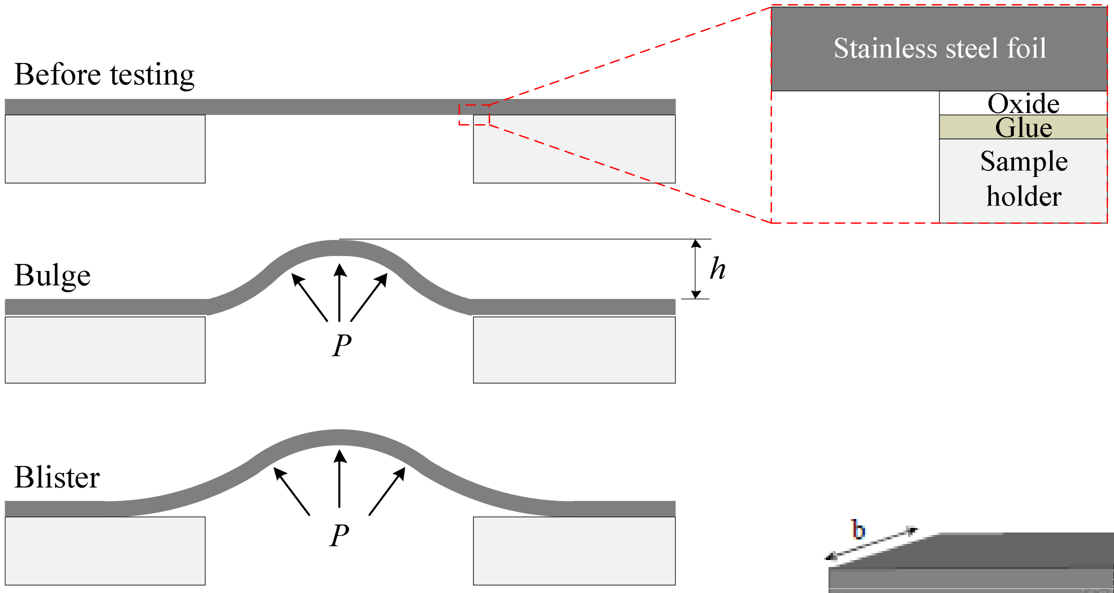


$$\psi = Arctg\left(\frac{\tau}{\sigma}\right)$$

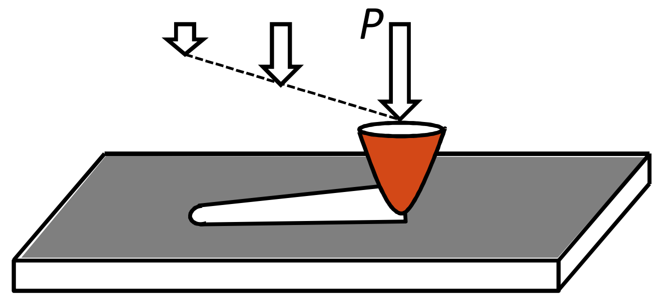
$$G_c = \frac{G_{Ic}}{\cos^2\psi + \lambda\sin^2\psi} = \frac{G_{Ic}}{1+(\lambda-1)\sin^2\psi} \qquad 0 \le \lambda \le 1$$

O'Dowd N.P., Stout M.G., Shih C.F., Phil. Mag. A66 (1992) 1037-1064

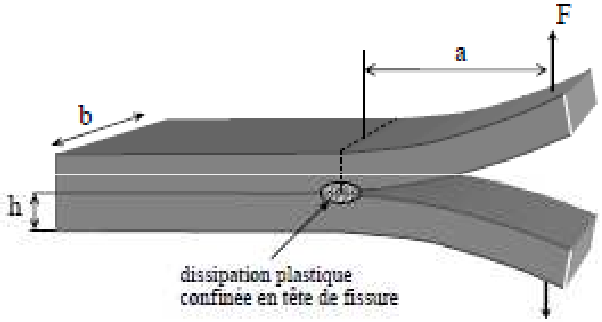
Adhesion measurement methods



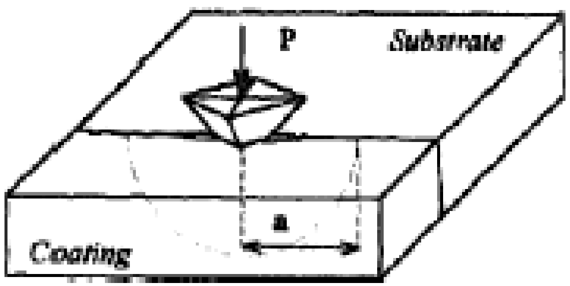
Inverted blister test



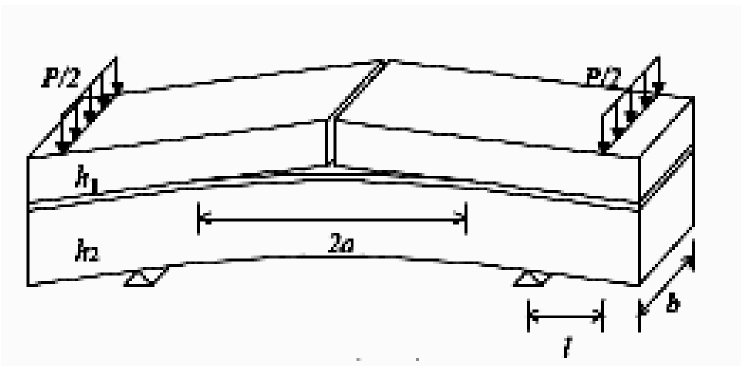
Scratch test



Wedging



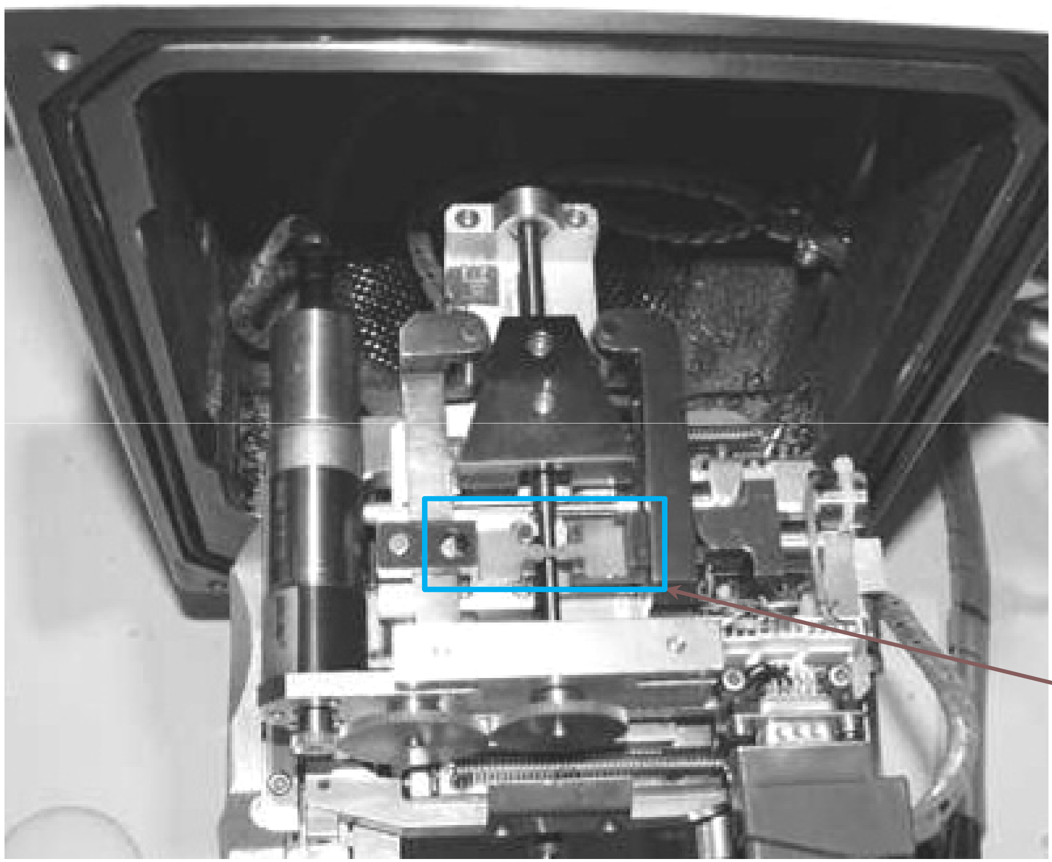
Interfacial indentation test



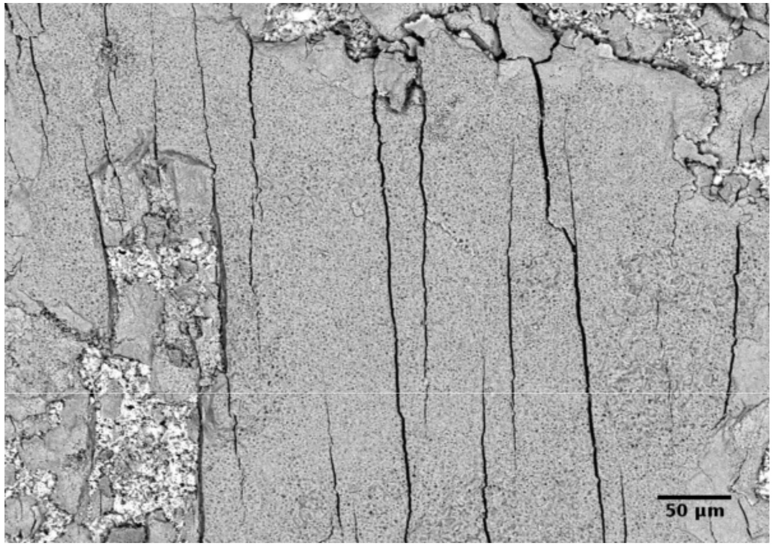
3- or 4-point bending

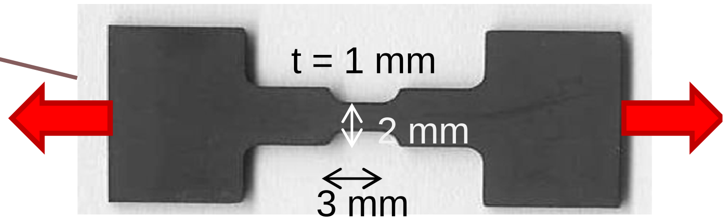
Adhesion measurement methods

- **SEM *in-situ* tensile testing**



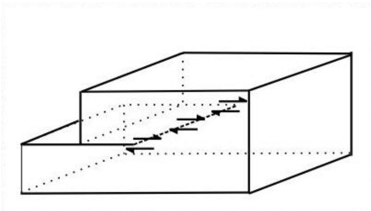
AISI 303 - 50 h – 1000 °C - air



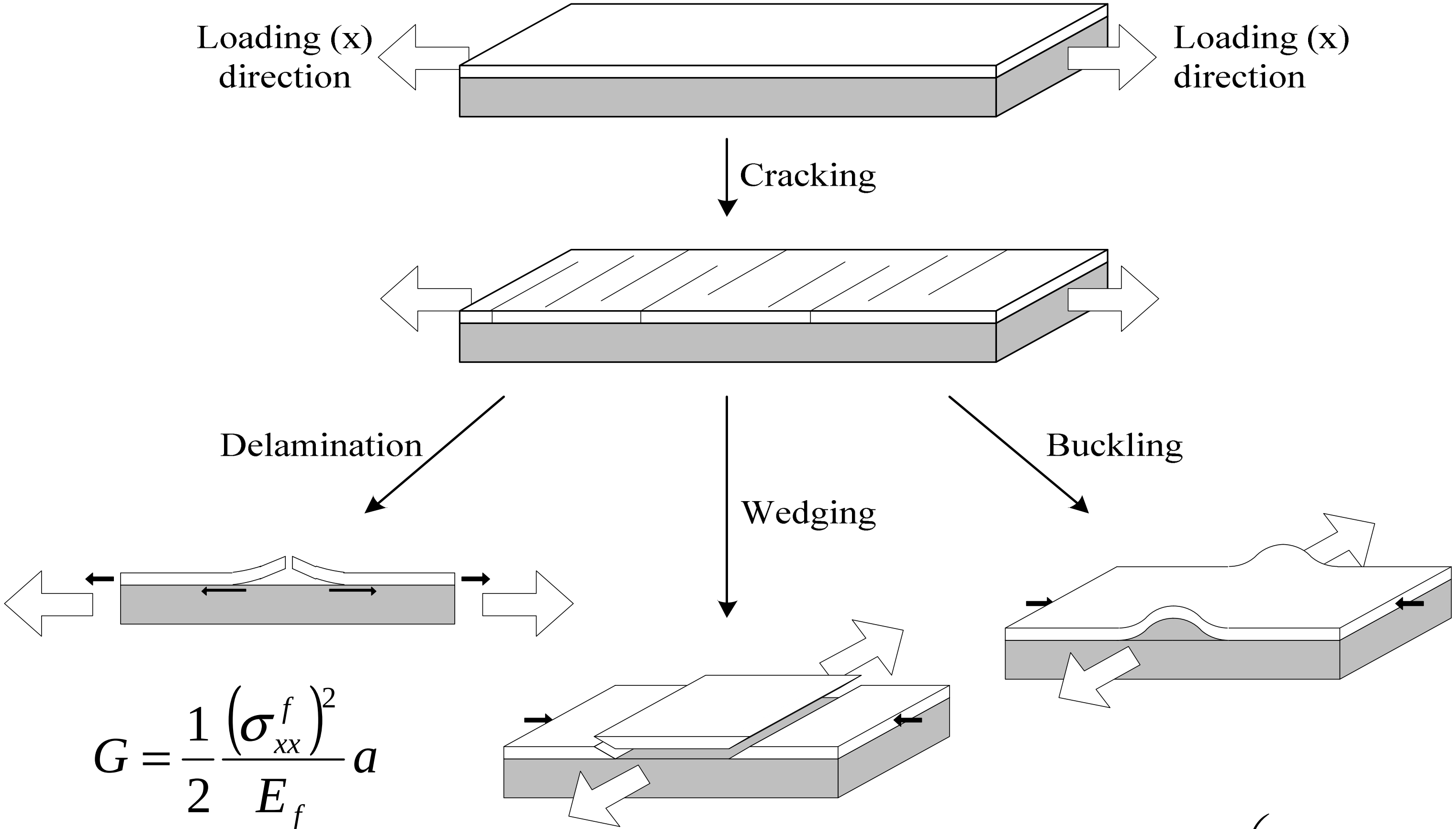


displacement rate = 50 µm.min^{-1}

Mode mixity $\Psi \Rightarrow \pi/2$



Tensile testing



$$G = \frac{1}{2}\frac{\left(\sigma_{xx}^{f}\right)^{2}}{E_f}a$$

$$G = \frac{1-\nu_f}{E_f}\left(\sigma_{yy}^{f}\right)^{2}h_f$$

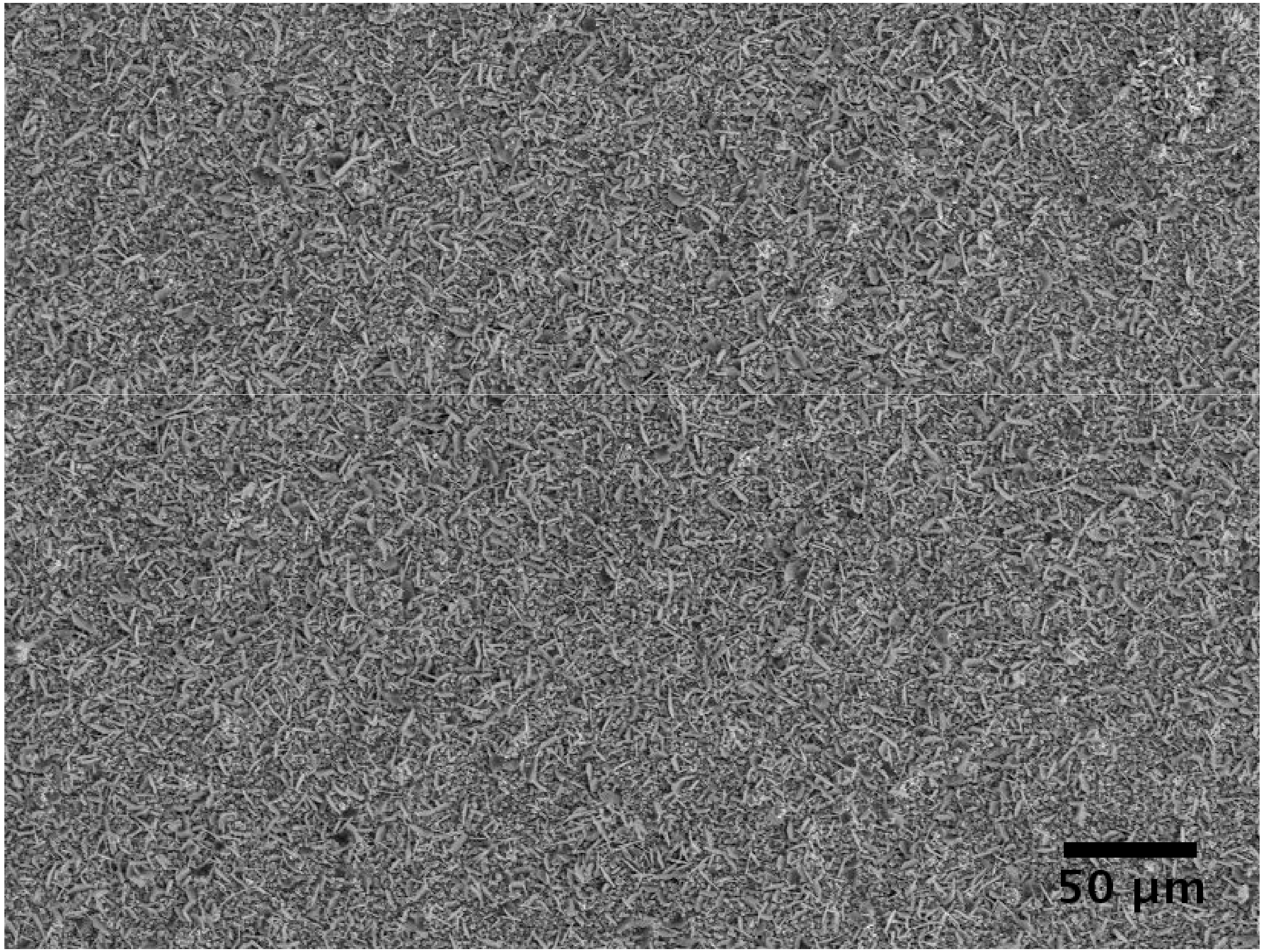
$$G = G_o\left(1-\frac{\sigma_{buckling}}{\sigma_{yy}^{f}}\right)\left(1+3\frac{\sigma_{buckling}}{\sigma_{yy}^{f}}\right)$$

$$\sigma_{buckling} = \frac{\pi^2}{12}E_f^{*}\left(\frac{h_f}{b}\right)^{2}$$

[Thouless M. D., Journal of Vacuum Science & Technology A9, 2510, 1991]
[Evans H.E., Int Mater Rev 40(1) 1-40, 1995]
[Hutchinson J.W., Suo Z., Adv. Apll. Mater. 29 63-191, 1992]

Understanding tensile testing pattern

AISI 304L previously oxidised at 900 °C for 50h air
SEM (SE mode) *in-situ* tensile testing experiment 0->63% strain

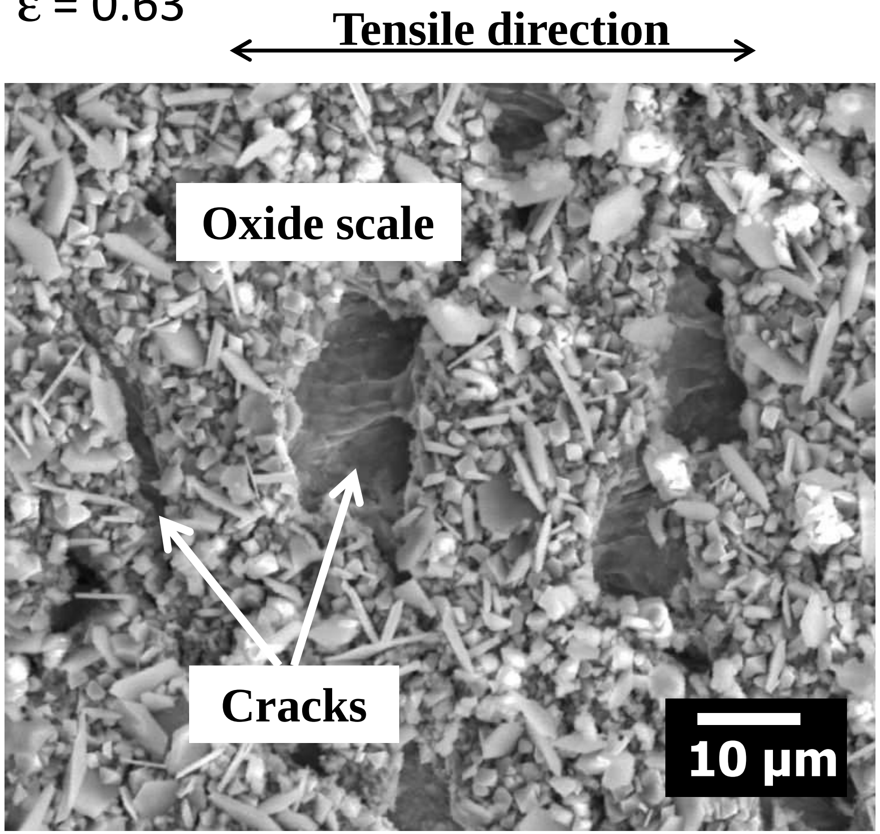


E. Fedorova et al. Corrosion Science. 103, 145 (2016)
V. Parry et al. Oxid Met 88, 29–40 (2017)
C. Pascal et al. Material Characterization 127, 161–170 (2017)

Understanding tensile testing pattern

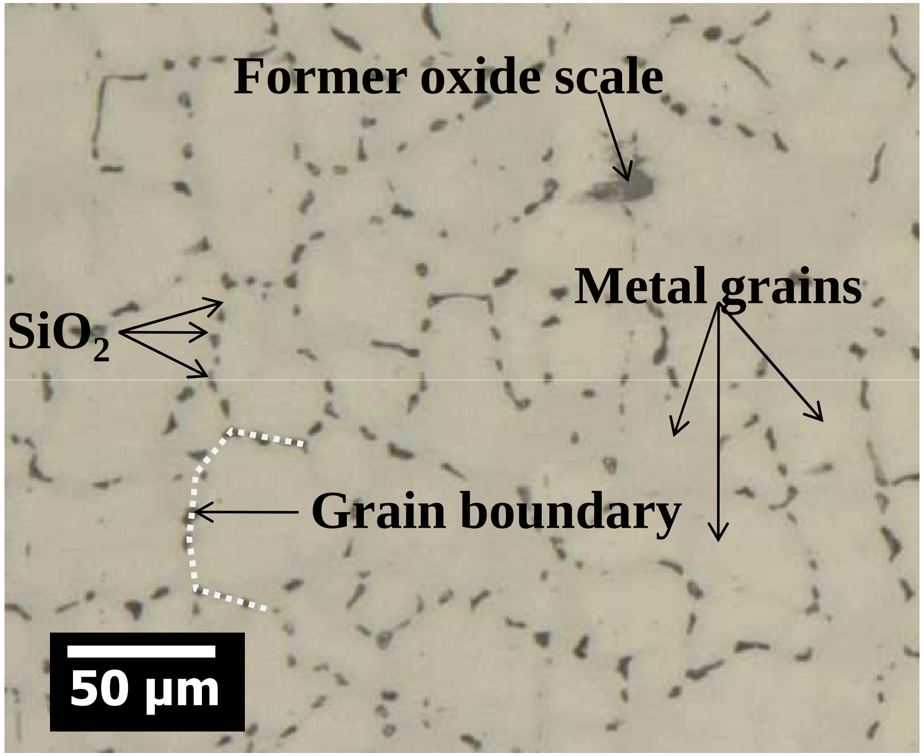
SEM Surface view (BES)

$\varepsilon = 0.63$



- Cracks opening in relation with the plastic deformation of the substrate
- Some cracks are not open

Optical microscope
(after oxide scale polishing)

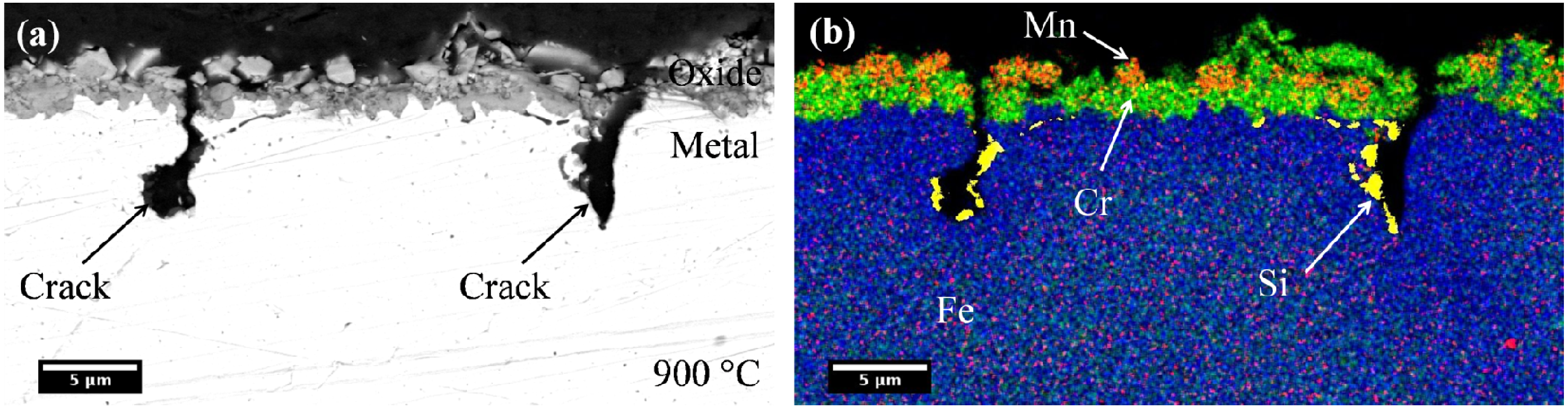


-strings of small SiO_2 precipitates decorate the substrate grains boundaries

Understanding tensile testing pattern

• Internal oxidation of Si plays a key role on the overall mechanical behavior during tensile testing.

•The cracks are linked to the silica embrittlement of the substrate grain boundaries



Can we predict this failure pattern ?

i.e. oxide scale spallation vs. inclusion debonding

=> Objectives : investigation of the relevant parameters controlling the silica/alloy interface debonding.

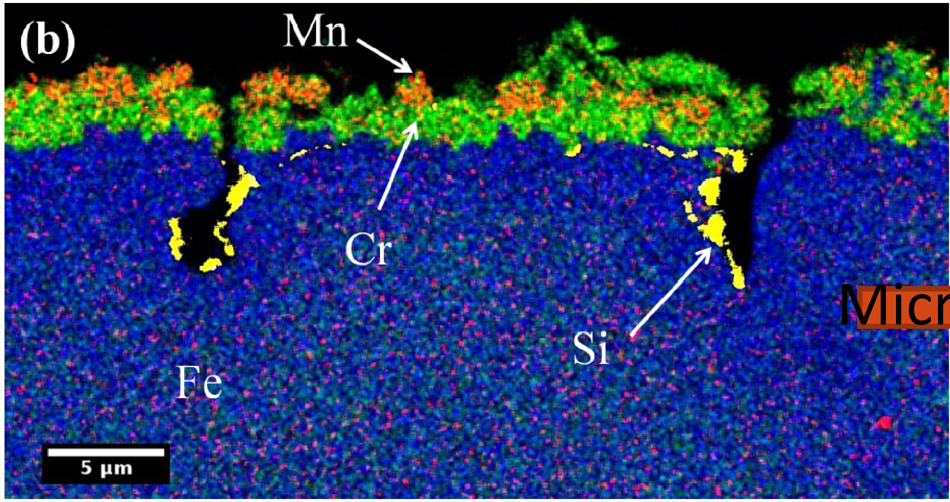
=> Tools : micromechanical modeling using cohesive zone models

E. Fedorova, M. Braccini, V. Parry, C. Pascal, M. Mantel, F. Roussel-Dherbey, D. Oquab, Y. Wouters, D. Monceau, *Corrosion Science*. **103**, 145 (2016)

V. Parry, C. Pascal, M. Braccini, El. Fedorova, M. Mantel, Y. Wouters, D. Oquab, D. Monceau, R. Estevez, G. Parry, *Oxidation of Metals.* **88**, 29 (2017)

Micromechanical modeling

Experiments

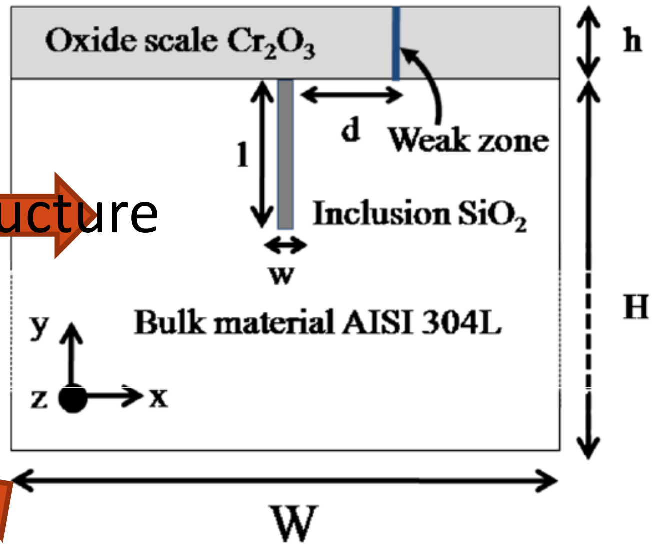


The cracks are linked to the silica embrittlement of the substrate grain boundaries

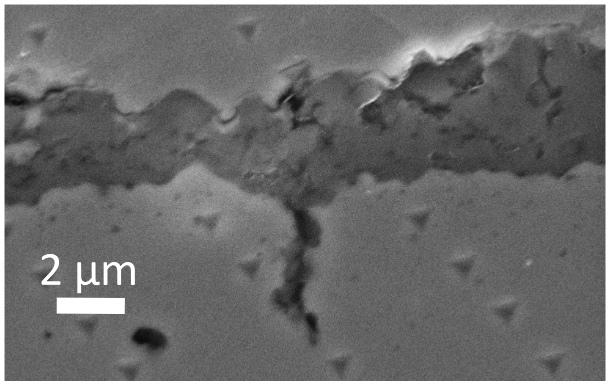
Model

Collab G. Parry

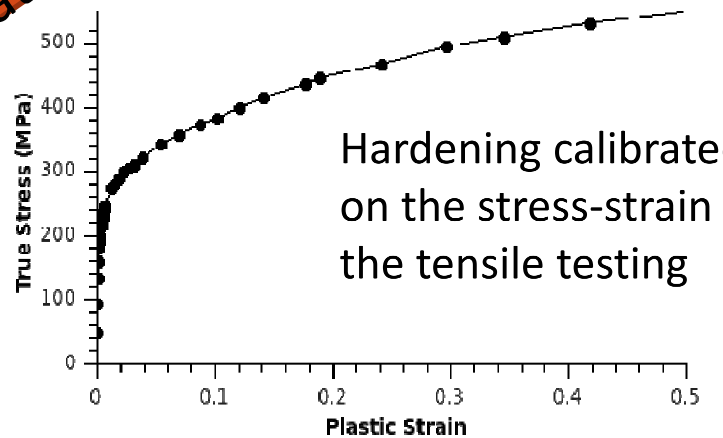
Microstructure



Materials



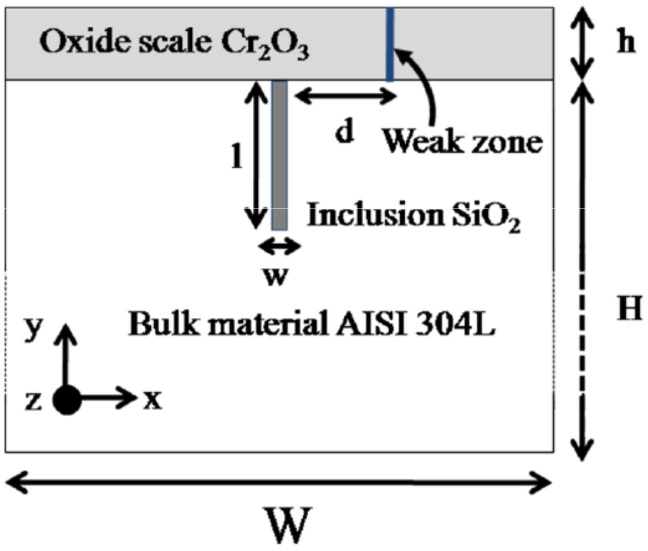
Young moduli measurements

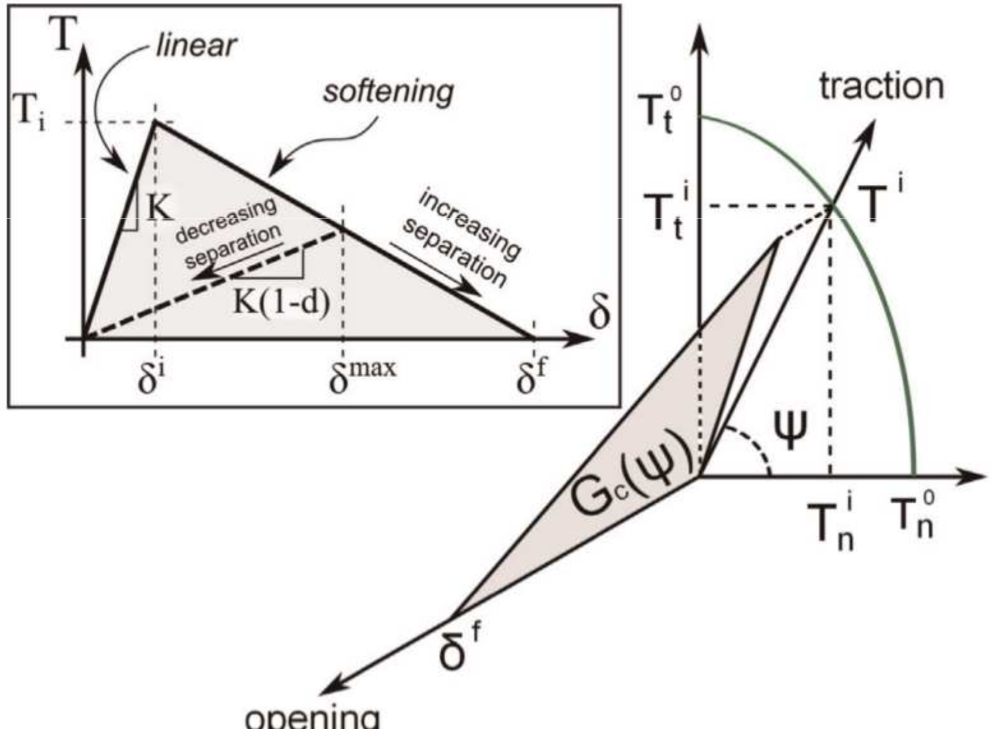


Hardening calibrated based on the stress-strain curve of the tensile testing

Cohesive zone model

- **Allow accounting for crack initiation and propagation**
- **Traction *versus* separation law describing interface fracture behavior**
- **Can include mixed mode dependence**





2 parameters: G_c and T_{max}

Peak maximum traction

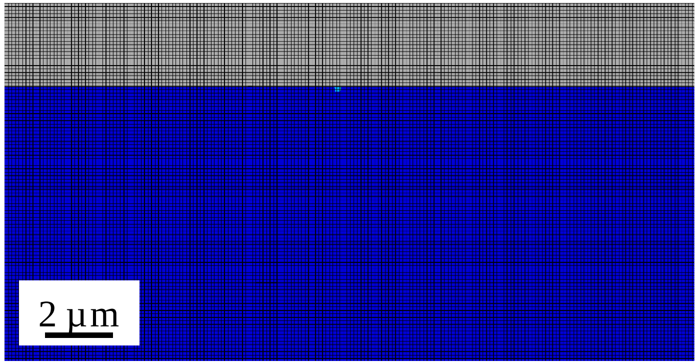
Faou et al. Journal of the Mechanics and Physics of Solids 75 (2015) 93–103

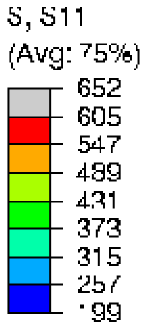
Results – conditions for interfaces decohesion

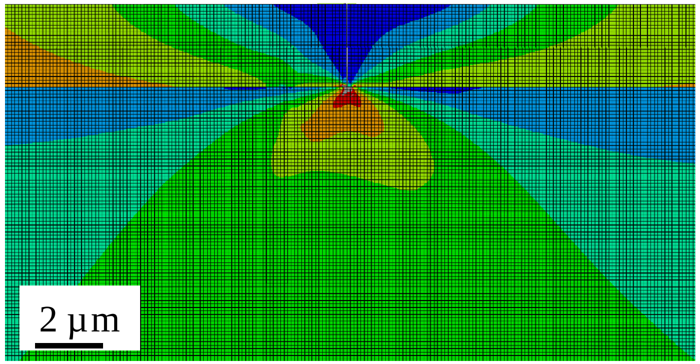
Right before oxide scale cracking

Right after oxide scale cracking

Normal stress S11 (MPa)







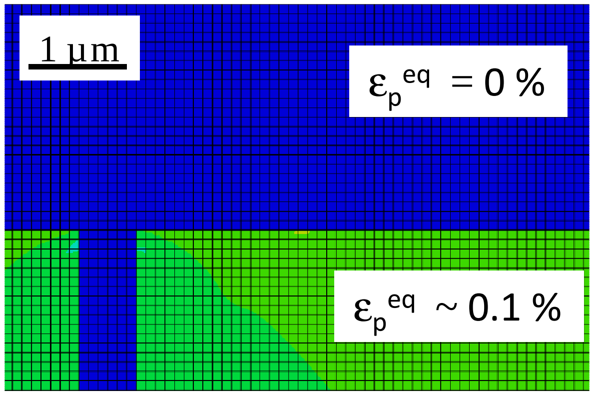
- High stress level in the oxide scale

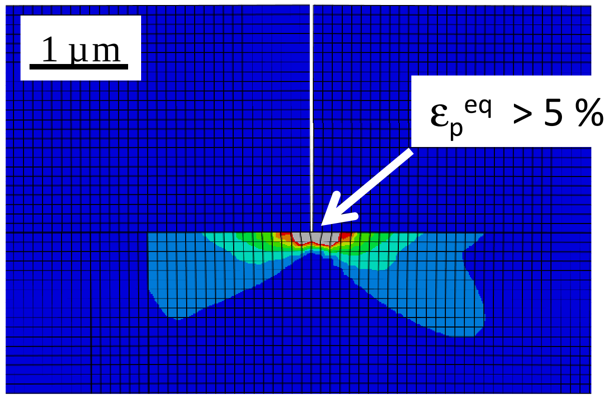
- Stress in the oxide scale is relaxed
- Stress concentration in the metal beneath the crack tip.

Applied strain ε = 0.26 %

Applied strain ε = 0.28 %

Equivalent plastic strain ε_p^{eq}





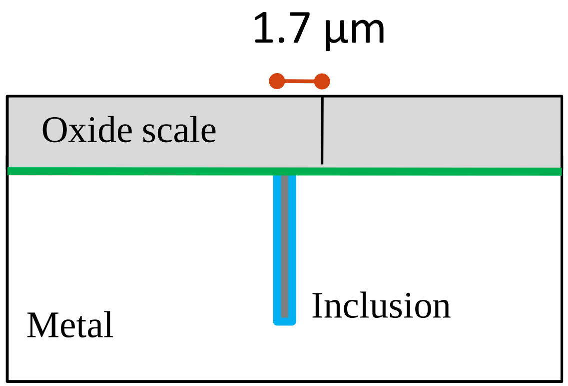
- The plastic strain in the metal propagates with increasing loading from the stress concentration zone at crack tip into the bulk and can reach the inclusion

Results – conditions for interfaces decohesion

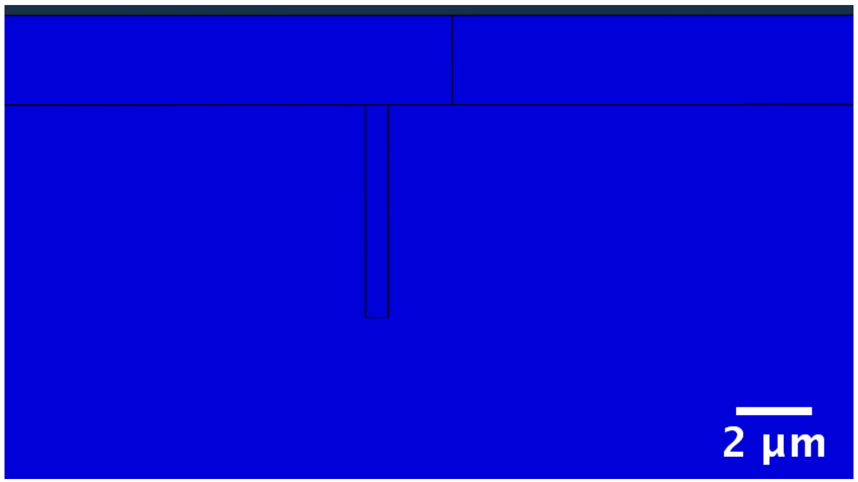
$$R = \frac{\sigma^{max}\ \text{metal inclusion interface}}{\sigma^{max}\ \text{metal oxide interface}}$$

With :

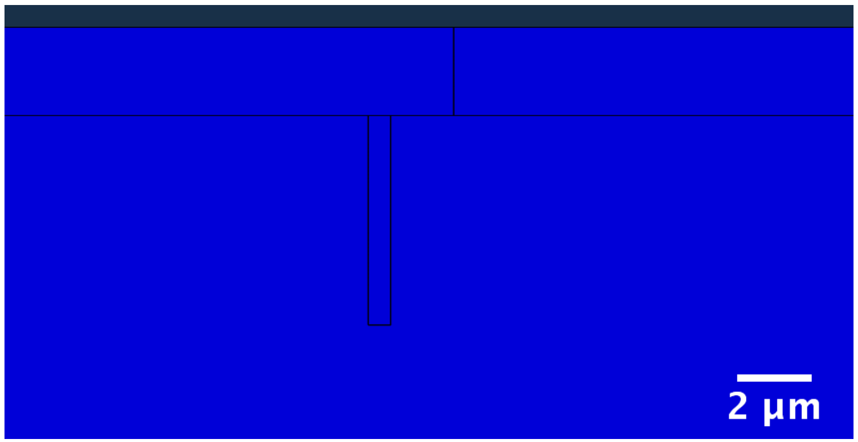
$\sigma_{n,MO}^{max}$	600 MPa	$\sigma_{n,MI}^{max}$	From 0.1 to 1.5 $\sigma_{n,MO}^{max}$
$\sigma_{t,MO}^{max}$	$5 \times \sigma_{n,MO}^{max}$	$\sigma_{t,MI}^{max}$	$5 \times \sigma_{n,MI}^{max}$
$G_{c,MO}$	3 $J.m^{-2}$	$G_{c,MI}$	From 0.5 to 1 $G_{c,MO}$



R < 1 Inclusion debonding



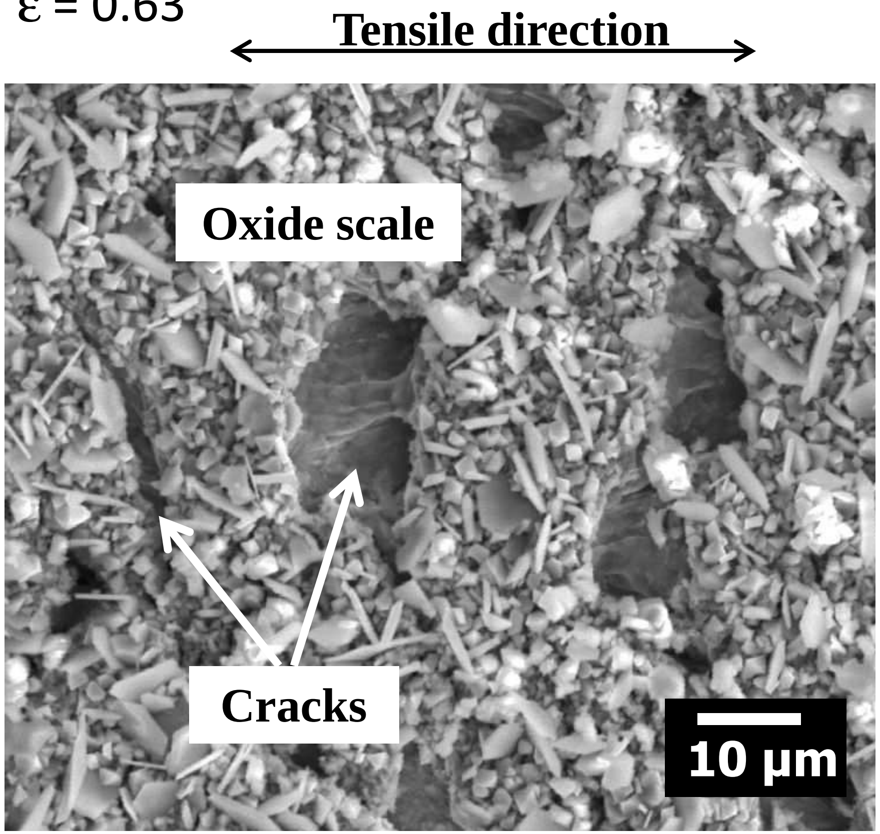
R > 1 Oxide scale debonding



What about the other cracks ?...

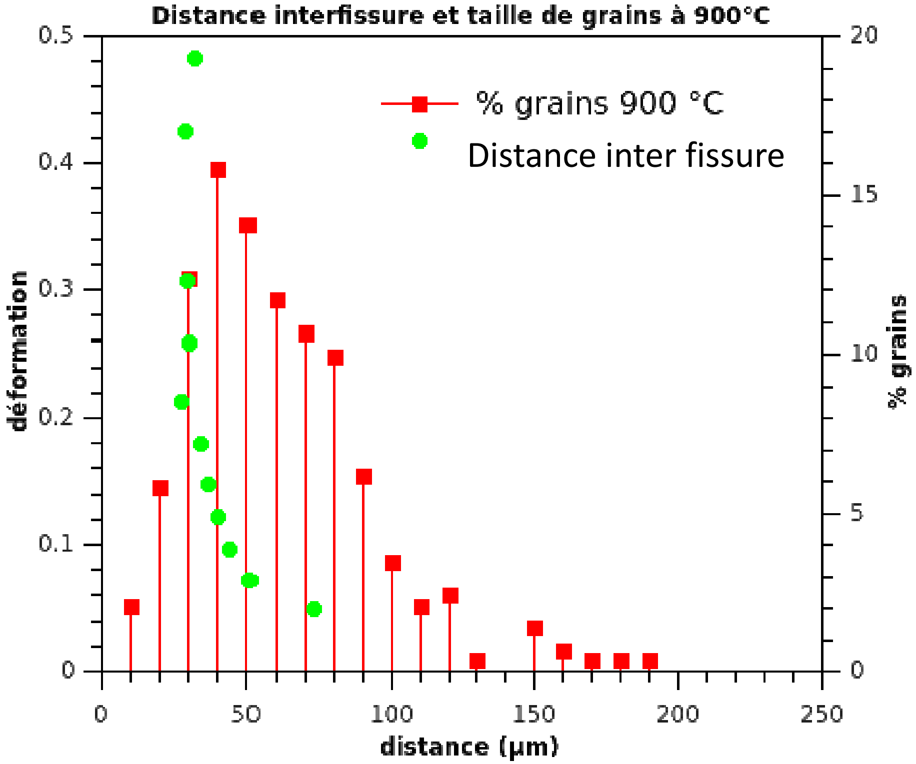
SEM Surface view (BES)

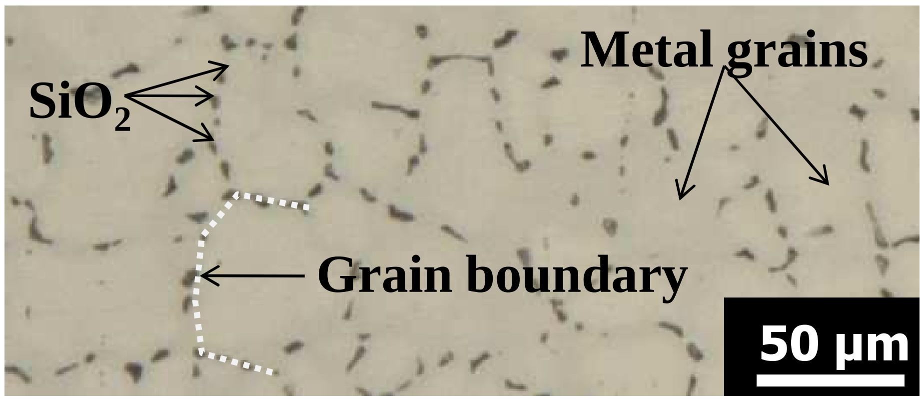
$\varepsilon = 0.63$



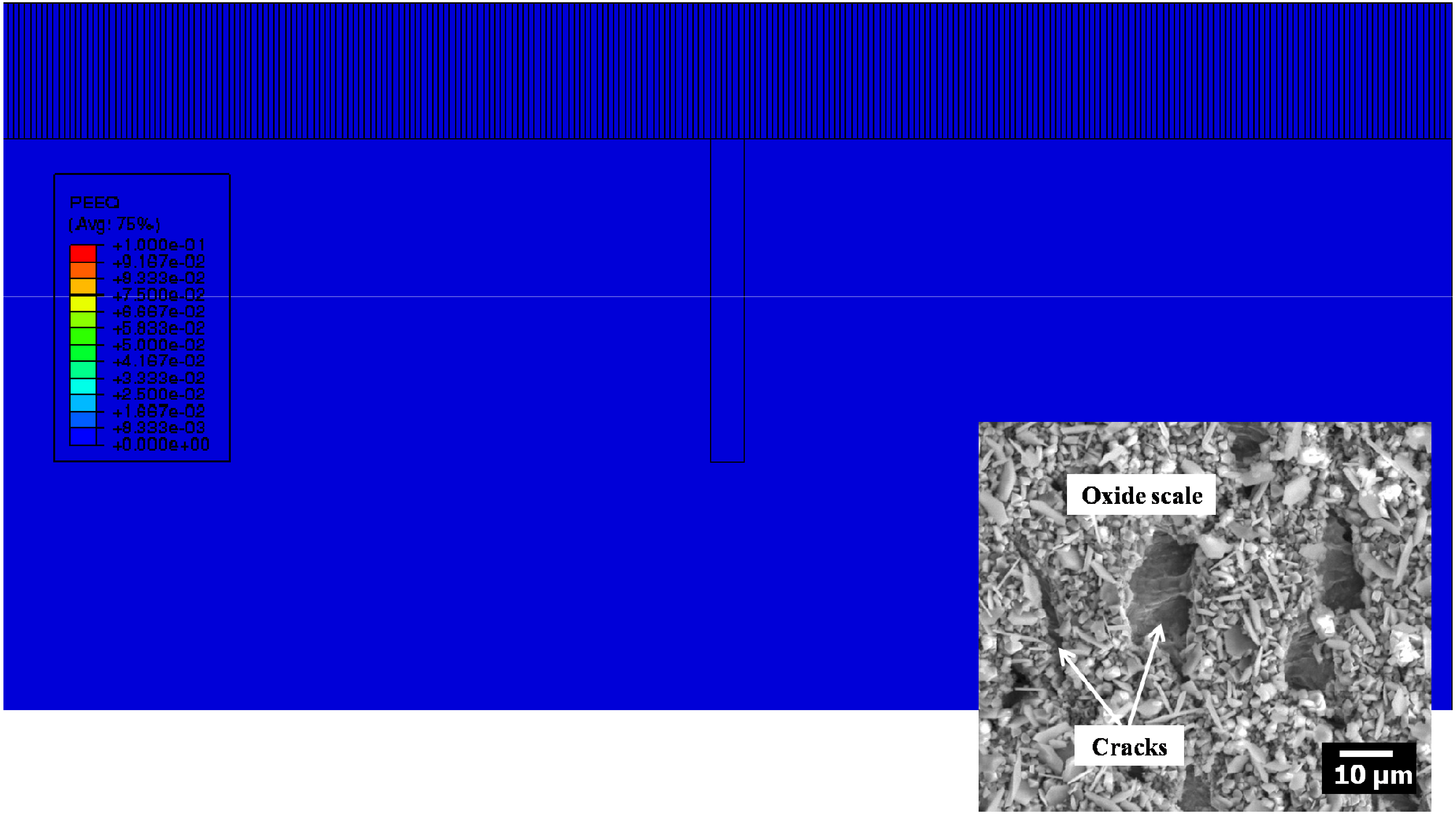
- Cracks opening in relation with the plastic deformation of the substrate
- Cracks not open

d_{sat} = 29 µm < grain size



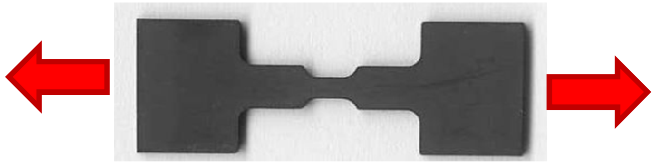


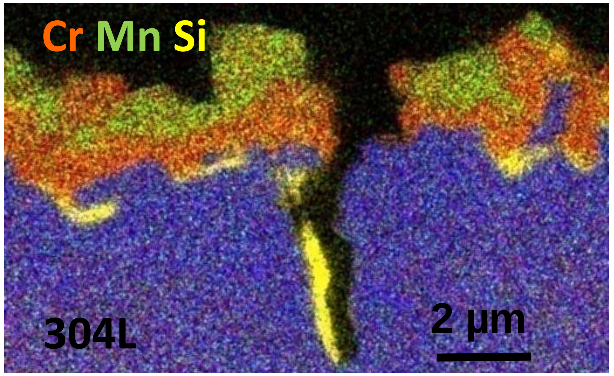
CZM in the oxide with a random distribution of maximum traction values

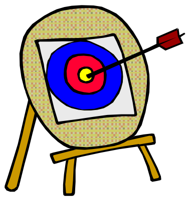


Bilan

Mesure d'adhérence de la couche d'oxyde par essai de traction *in-situ* sous MEB







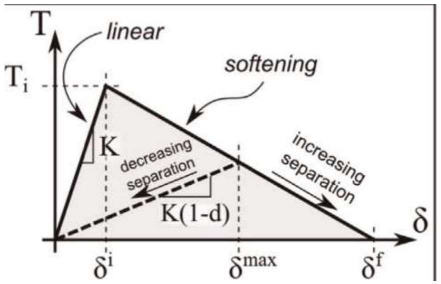
La formation de silice le long des joints de grains modifie le comportement mécanique de la zone affectée par l'oxydation

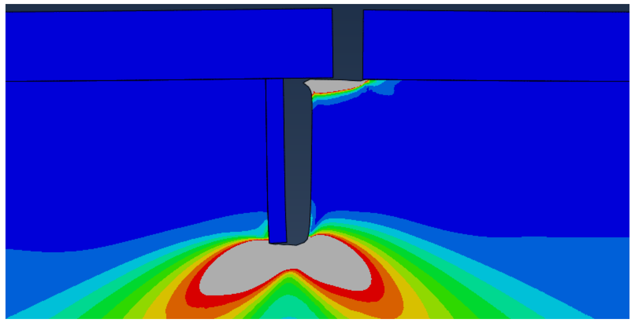
Lien entre mesure globale (adhérence) et effet local (microstructure) ??

Quid si sollicitation mécanique différente (e.g. blister test) => Effet d'ancrage ?

Compétition entre décohésion métal/oxyde et métal/silice

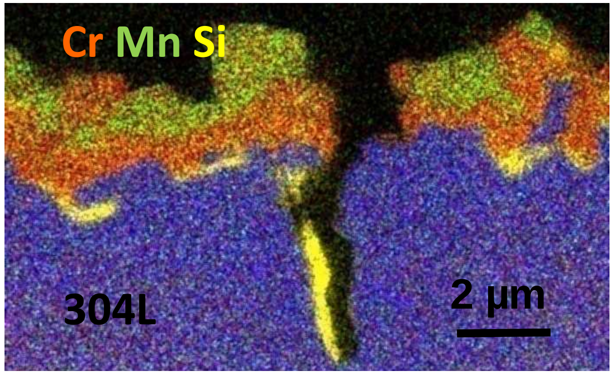
=> modèle micromécanique utilisant des CZM pour décrire le pattern de fissuration.

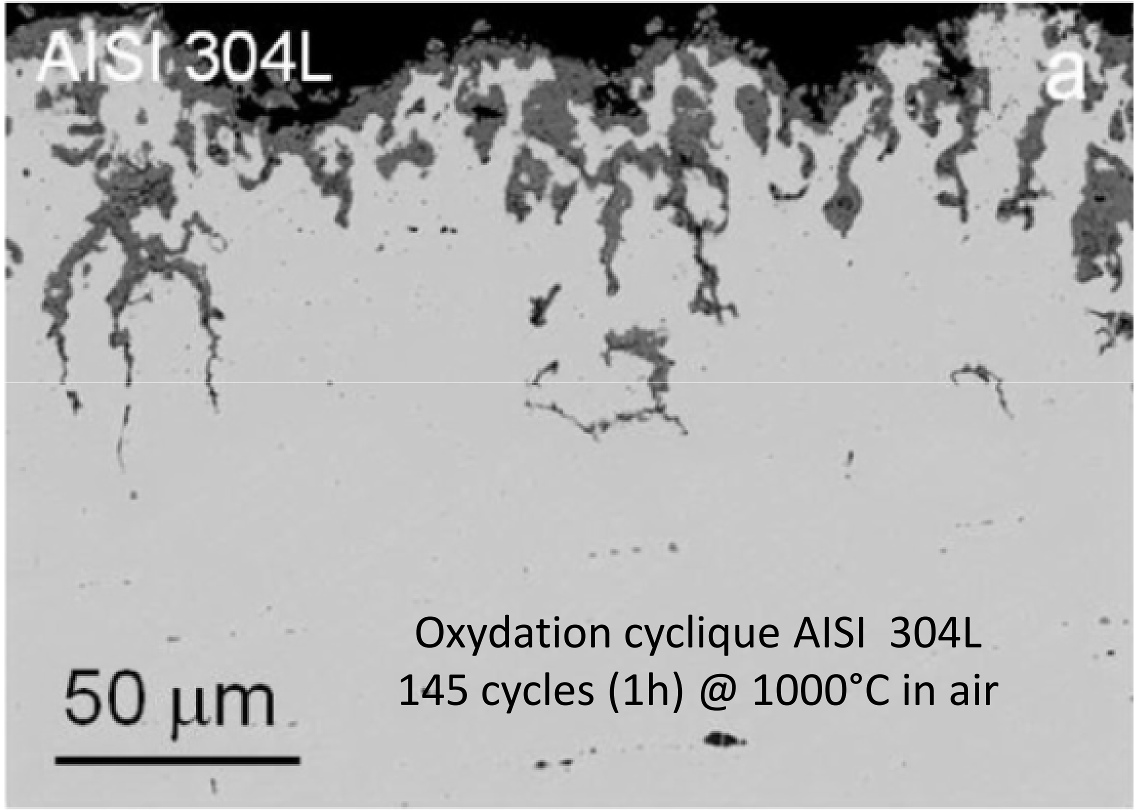




Essai de traction *in-situ* sous MEB

Oxydation isotherme AISI 304L @1000°C





E. Fedorova et al. Corrosion Science. 103, 145 (2016)

Améliorer les modèles et simulations

en intégrant la géométrie (ondulation)

Quantitatif

Effet thermique