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Nuclear corrosion: achievements et challenges

D. Feron, F. Legendre

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MAIN ACTIVITIES OF THE “CEA CORROSION UNIT”

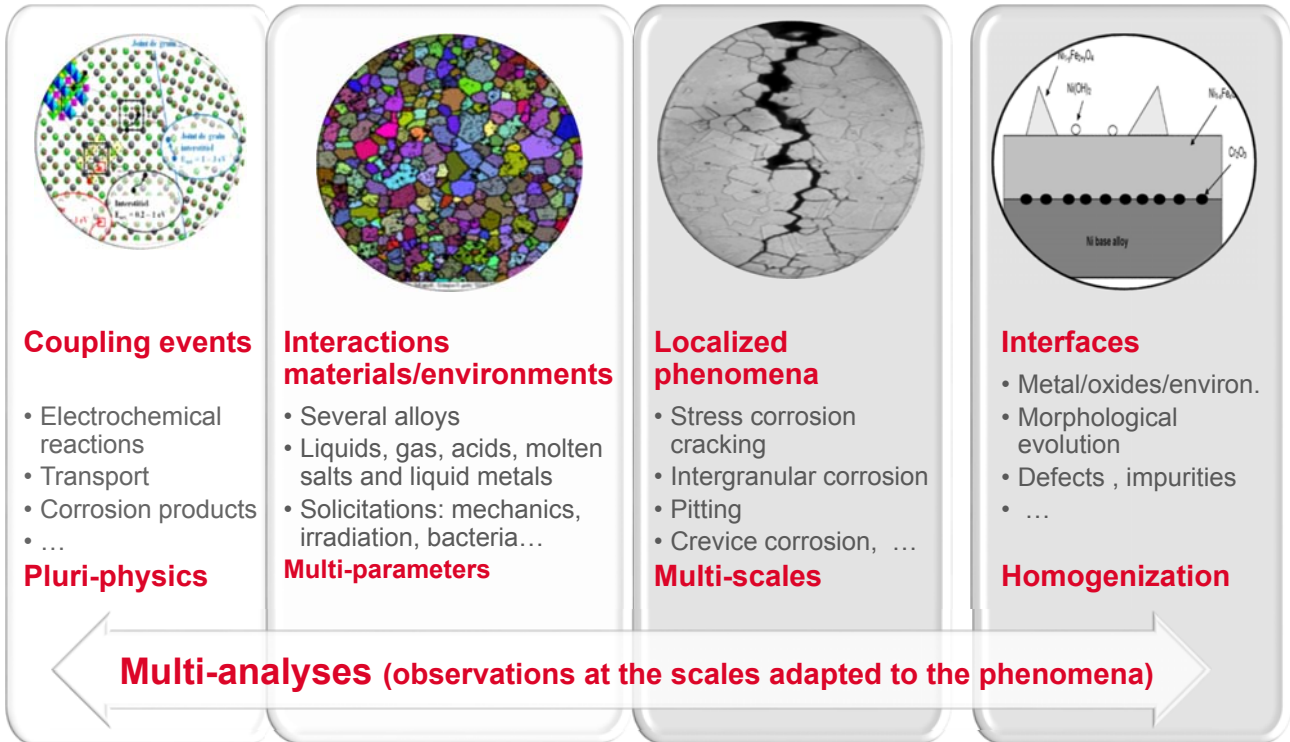
NUCLEAR CORROSION: ACHIEVEMENTS & CHALLENGES

| **Damien Féron & Fabrice Legendre**
Service de la corrosion et du comportement des matériaux dans leur environnement
Département de Physico-Chimie, Direction de l'Energie Nucléaire
Commissariat à l'énergie atomique et aux énergies alternatives
French atomic energy and alternatives energies Commission
Saclay, France

Department of Quantum Science and Energy Engineering
Tohoku University, Sendai, Japan
February 17, 2016

- ❑ Introduction: CEA Corrosion Unit
- ❑ Gen 2&3 (Pressurized Water Reactor – PWR)
- ❑ Gen 4 (Liquid metals, gas & supercritical water cooled reactors)
- ❑ Nuclear waste storage
- ❑ Decommissioning & conclusive remarks

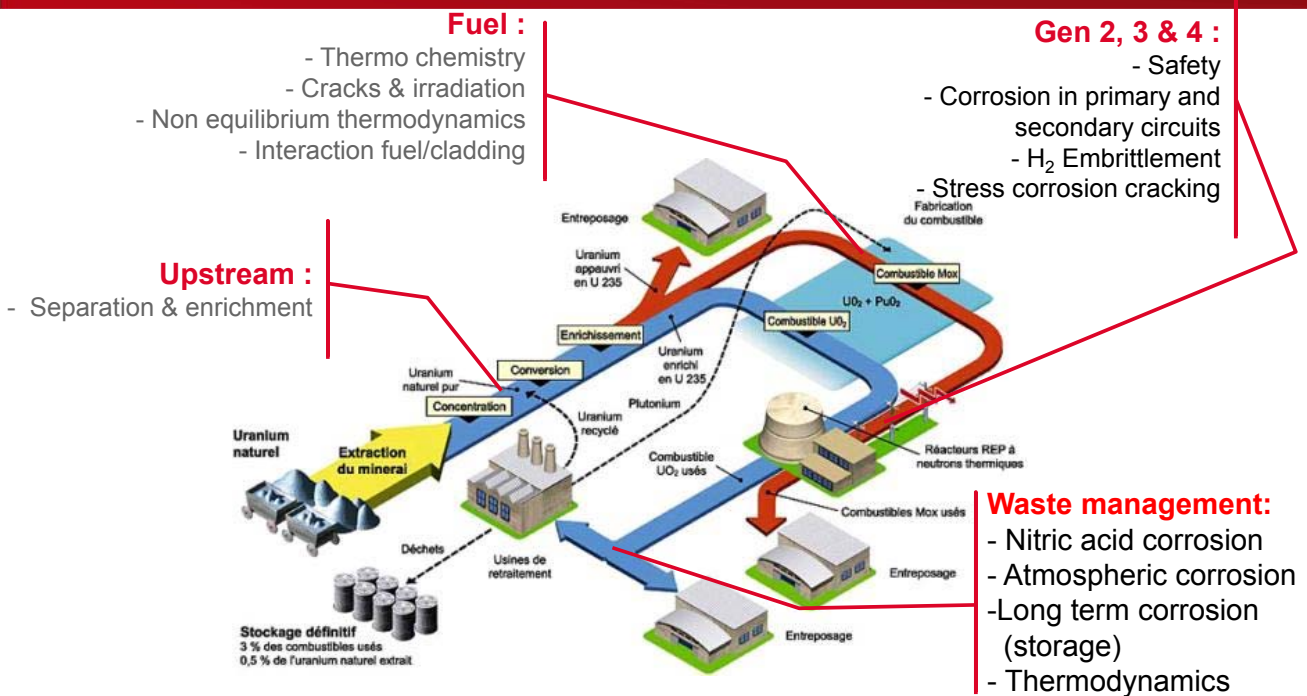
⇒ Complexity of corrosion alteration phenomena



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CORROSION ACTIVITIES: NUCLEAR FUEL CYCLE



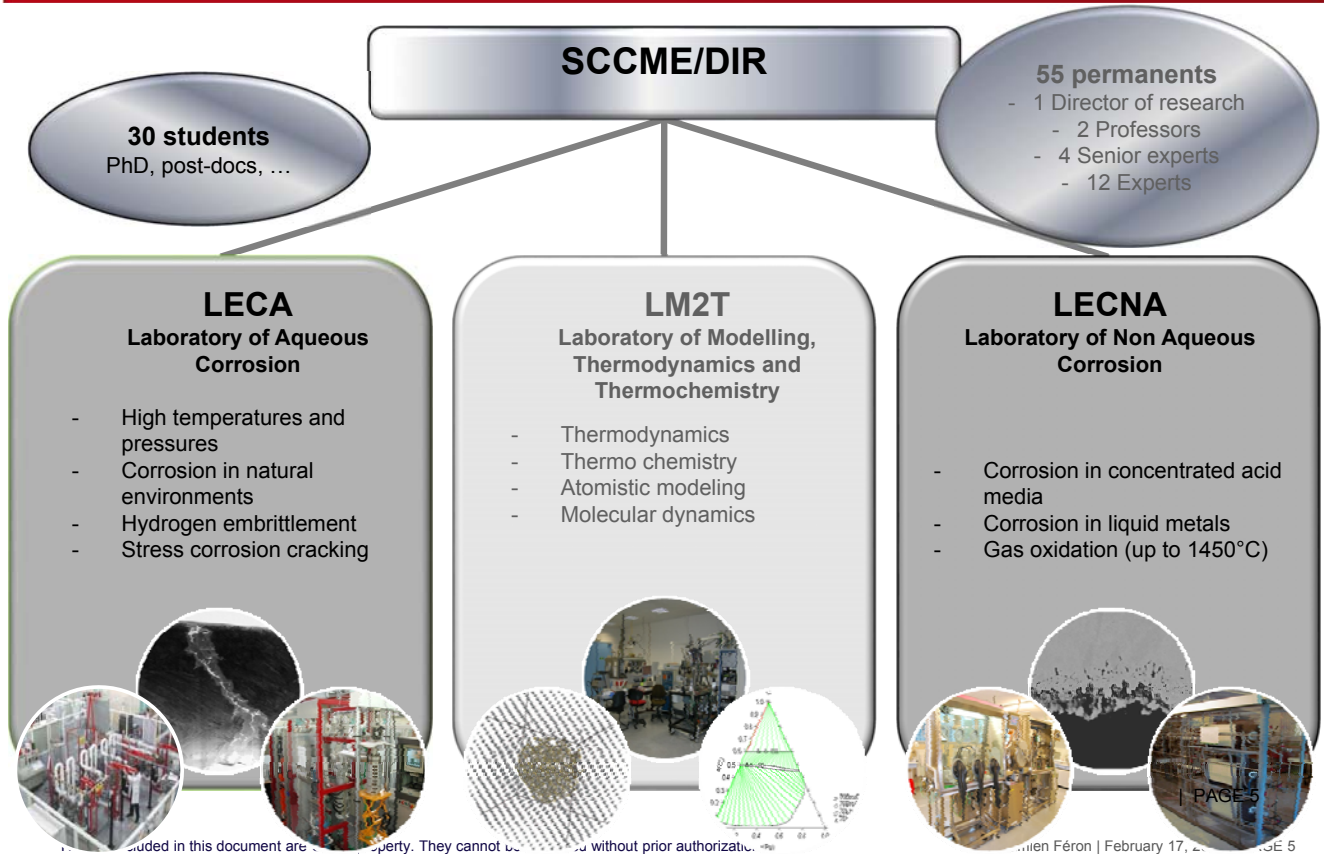
Main Industrial Partners:

- EdF, ANDRA, AREVA, IRSN, CNE, ONDRAF, ENGIE (GdF-Suez),

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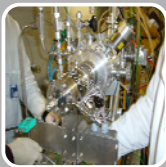
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« **CEA corrosion unit** »



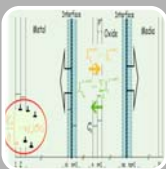
METHODOLOGY

Approach and methodology



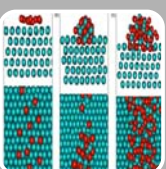
Data acquisition & surface analysis

- Specific facilities (autoclaves, loops...)
- **Specific methods (isotopic tracers,...)**
- Surface analyses
- Semi-empirical modeling



Mesoscopic modeling

- Uniform corrosion, Intergranular corrosion...
- Estimation of the Kinetics of Oxidation
- Diffusion Poisson Coupled Model



Simulations

- Thermodynamics (Calphad, PhreeCea,...)
- Cellular automata
- Atomistic & molecular dynamics

Illustrations



Facilities at the "CEA corrosion department"



View of the corrosion hall at CEA-Saclay (B. 458)

Artist view of the corrosion equipment (Juliette Plisson)



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Some specific experimental facilities

Sodium



CORRONa 1&2
(300-650°C)

Stress Corrosion Cracking

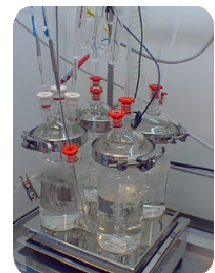


Autoclaves & loops with tensile machines



VENUS

HNO₃



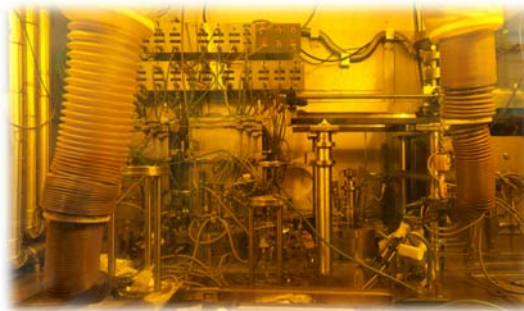
Electrochemical cells

Oxidation & H₂



HAWAI

Irradiated materials (stainless steel & zircaloy)



Facility with 3 autoclaves and one tensile machine in hot cell (HP & HT)

Irradiation & electrochemistry

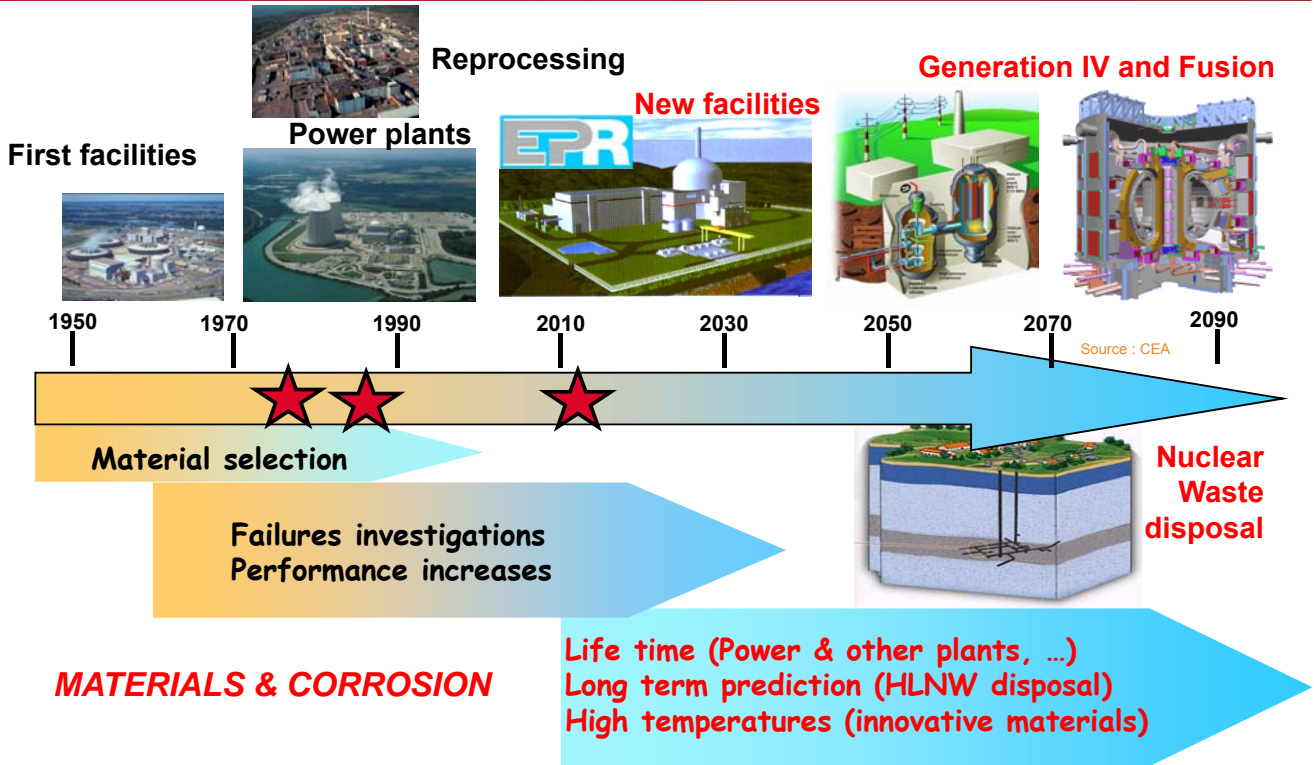


HP & HT cell (e & α irradiations)

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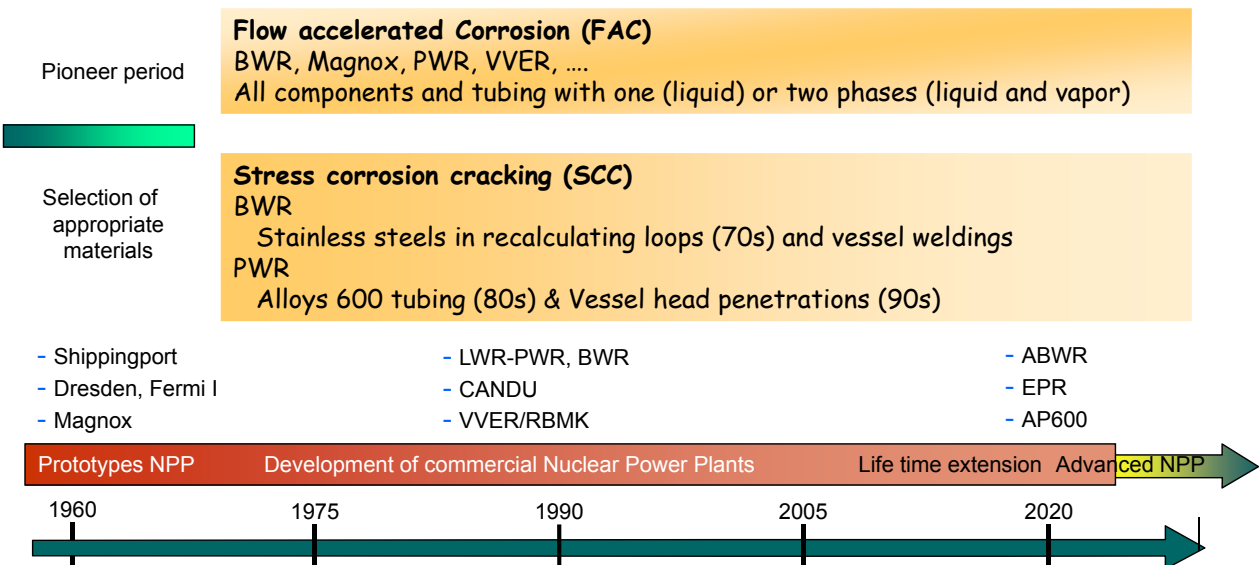
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NUCLEAR ENERGY & CORROSION: from yesterday to tomorrow



Main Corrosion Challenges

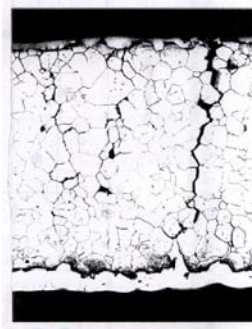
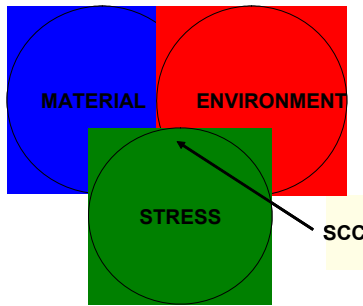
“solved” in Nuclear Power Plants but still occurring



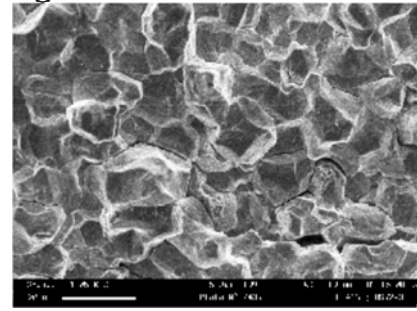
Gen 2&3 illustrations

- Flow accelerated corrosion
- Stress corrosion cracking
- Zirconium oxidation

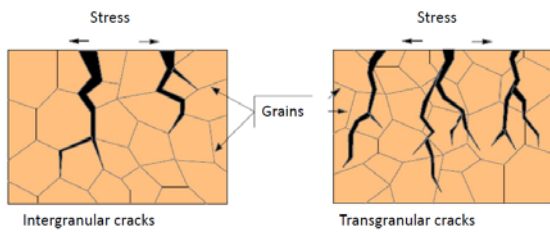
STRESS CORROSION CRACKING



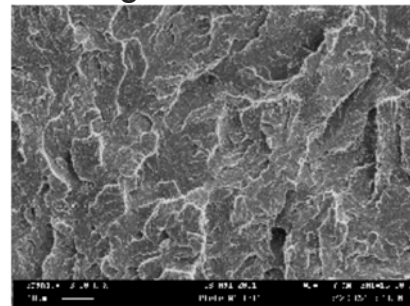
Intergranular SCC



Alloy 600, SG tube, primary water

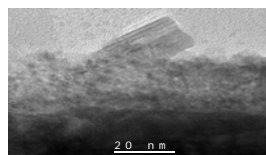


Transgranular SCC

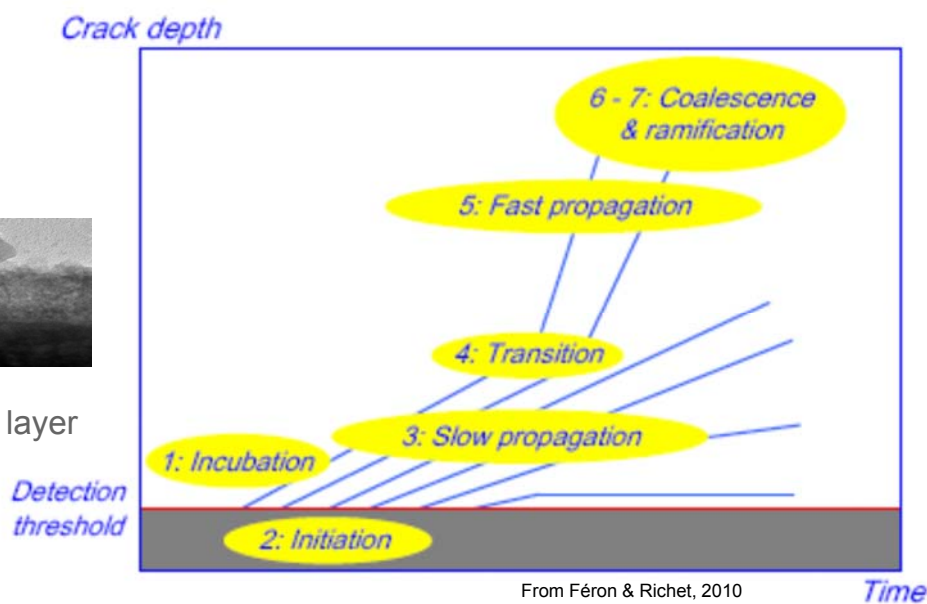


17-4 PH Stainless steel, 300°C

STRESS CORROSION CRACKING



1: Passive layer



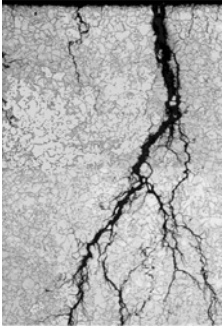
From Féron & Richet, 2010

2: Breakdown of the passive layer

3-4: Inside and outside cracks (anodic and cathodic reactions)

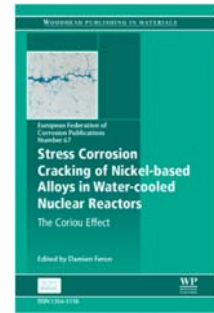
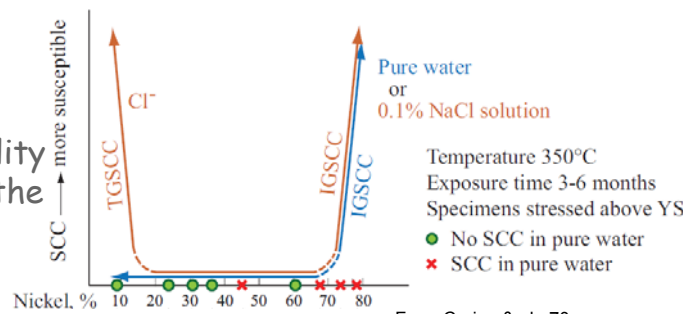
STRESS CORROSION CRACKING OF NI ALLOYS

Historical background SCC of Alloy 600 or the "Coriou effect"



- ✓ 1959: *Coriou et al.* published that Alloy 600 (nickel based alloy (75% Ni & 15% Cr) is sensitive to SCC in pure water at 350°C.
- ✓ 1959 - 1980s: Confirmation of the phenomena in pure water and in PWR primary water, named also "PWSCC" for Pure or Primary Water Stress Corrosion Cracking".
- ✓ 1980s - 1990s: generic phenomena occurring on Alloy 600 components and particularly on SG tubes.
- ✓ 1991 (in France) - 2002 (US, then Japan ...): cracking of vessel head penetrations (Alloy 600 tubes, Weld made of Alloy 182 or 82).

SCC susceptibility as function of the nickel content



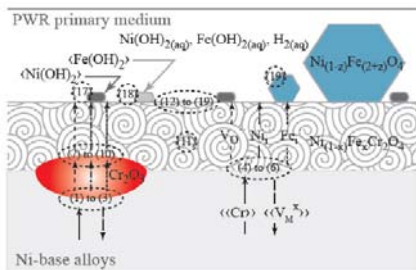
From Coriou & al., 70s

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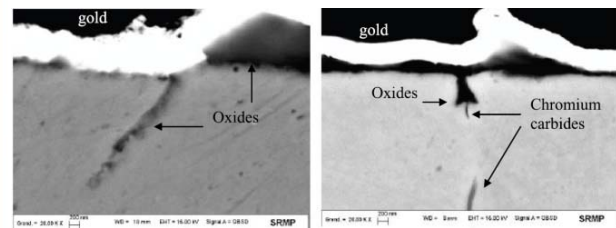
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STRESS CORROSION CRACKING OF NI ALLOYS

Incubation



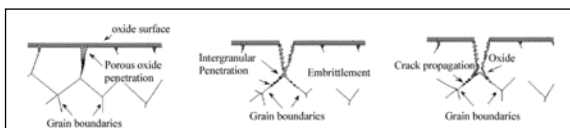
Initiation



On model alloy with intergranular carbides, intergranular oxidation much shorter

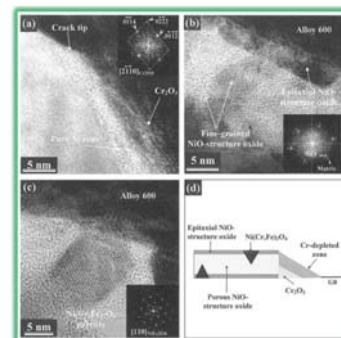
P. Laghoutaris & al., JNM 393 (2009) 254-266
M. Sennour & al., JNM, 402 (2010) 147-156
C. Guerre & al., Environmental Degradation, TMS, 2011, 1477-1488
M. Dumerval & al., Corrosion Science 85 (2014) 251-257,
L. Marchetti & Al., Corrosion Science, 102 (2016) 24-35

Internal oxidation model



Crack propagation: oxidation & hydrogen

Propagation

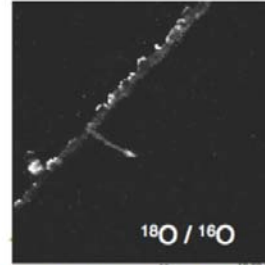
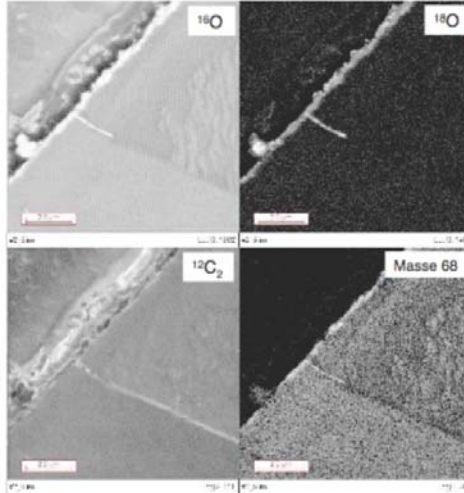


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Use of tracers (^{18}O) to investigate the growth of the oxide of the passive film and in the crack

Evidence and role of oxide at the crack tip (1)



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

\Rightarrow Oxygen and hydrogen can diffuse in the crack and in an IG attack

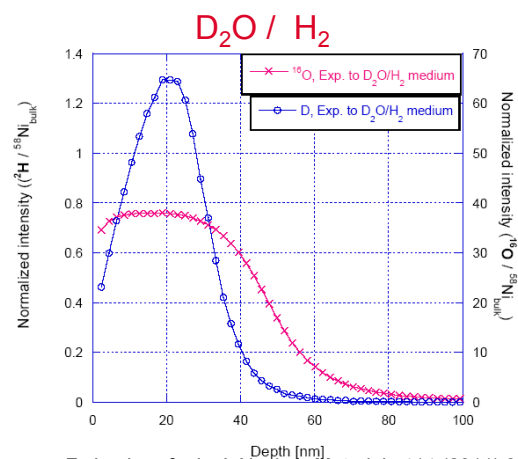
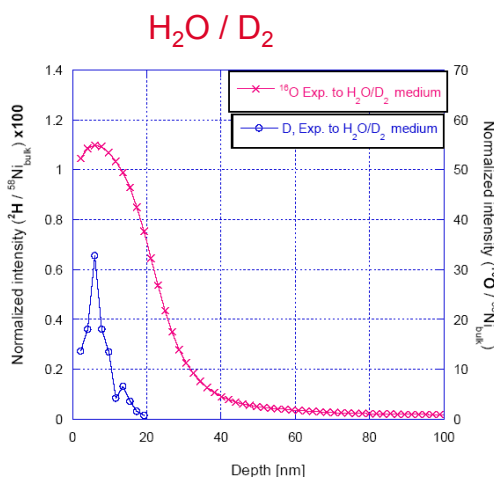
P. Laghoutaris & Al., 2009-2016

Pre-exposure in primary water conditions followed by an exposure period in H_2O_{18} under same conditions

Hydrogen origin

Two series of experiments were done, in which the specimens are exposed to:

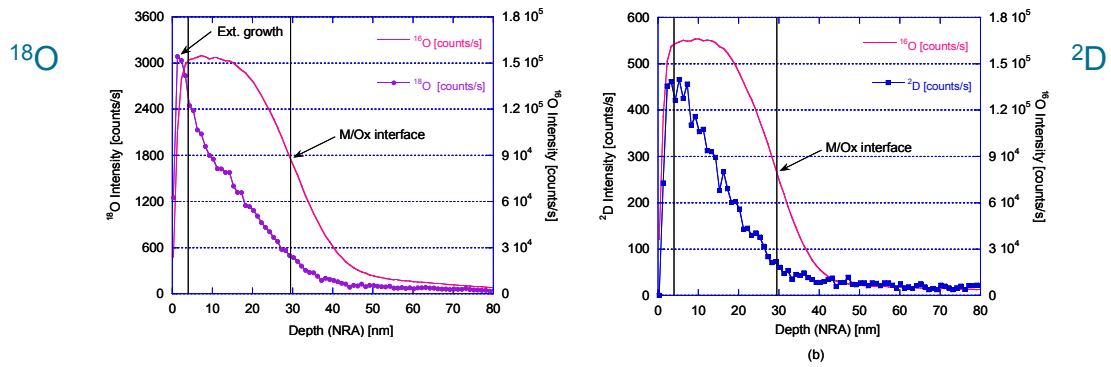
- a classical primary medium (with H_2O), pressurized by deuterium gas (D_2).
- a PWR primary medium in which the water is replaced by deuterated water D_2O , pressurized by classical hydrogen gas (H_2).



F. Jambon & al., J. Nuclear Materials 414 (2011) 386–392

Hydrogen absorption is associated with the cathodic reaction
($\text{H}_2\text{O} + \text{e}^- \rightarrow \text{H}^* + \text{OH}^-$)

STRESS CORROSION CRACKING OF NI ALLOYS



SIMS profiles of the isotopic tracers through the oxide scale as a function of the recalculated depth (the oxide scale is represented by the ^{16}O): (a) ^{18}O tracer (subplot: total oxygen signal's second derivative used to identify the domains); (b) ^2H tracer.

respectively $D_{sc}=(4.6\pm 0.9) \times 10^{-17} \text{ cm}^2/\text{s}$ for ^{18}O , and $D_{sc}=(5.2\pm 1.2) \times 10^{-17} \text{ cm}^2/\text{s}$ for ^2H

⇒ **diffusion of OH^- in the oxide layer**

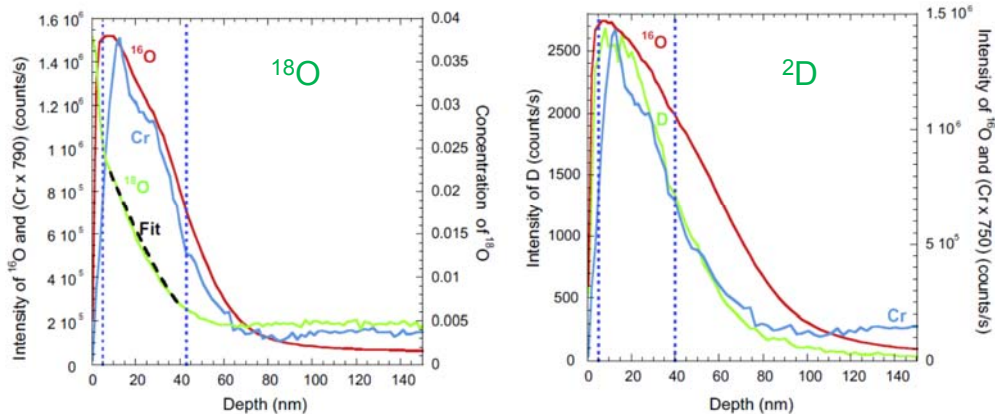
F. Jambon & al., Solid State Ionics, 231 (2013) 69-73

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STRESS CORROSION CRACKING OF STAINLESS STEELS

SIMS profiles of the isotopic tracers through the oxide scale as a function of the recalculated depth: 2-stages (600h+16h) corrosion experiments, 325°C, with ^{18}O tracer and ^2H tracer during the second stage.



Oxygen and hydrogen transport is not coupled

Short-circuit diffusion coefficient of ^{18}O : $D_{sc}=(9\pm 1) \times 10^{-17} \text{ cm}^2/\text{s}$

M. Dumerval & al., Corrosion science 85 (2014) 251-257

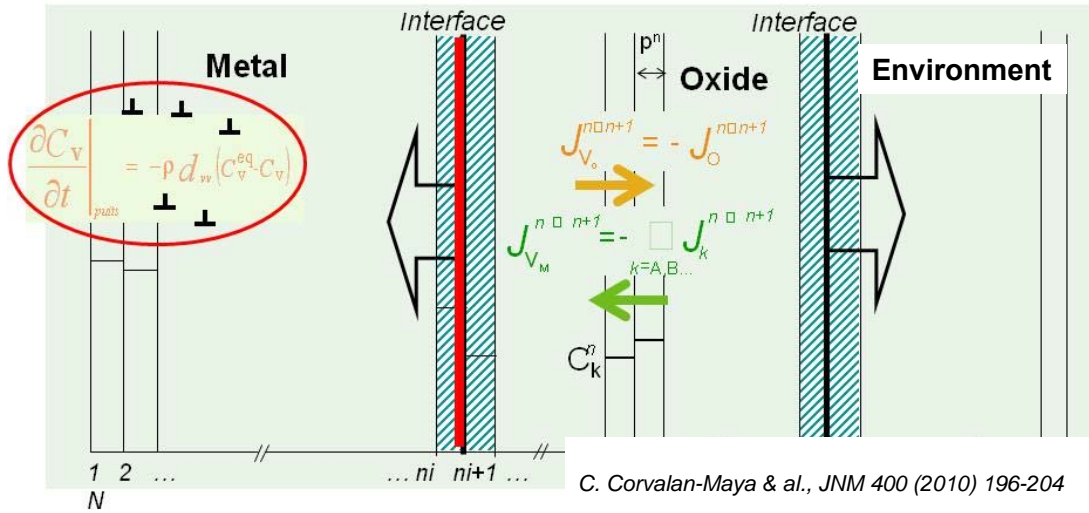
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FUEL CLADDING EVOLUTION DURING LOCA

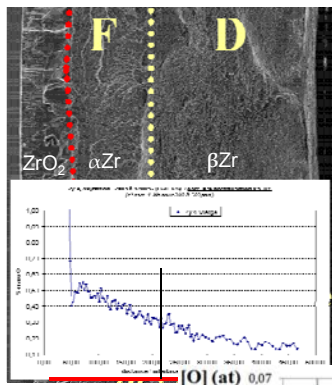
Zircaloy oxidation at high temperature

EKINOX (Estimation of the Kinetics of Oxidation)
Calculations to anticipate the evolution in the substrate during alloy oxidation



FUEL CLADDING EVOLUTION DURING LOCA

Numerical tool: EKINOX-Zr



Steam oxidation,
1200°C, 187s, Zy4

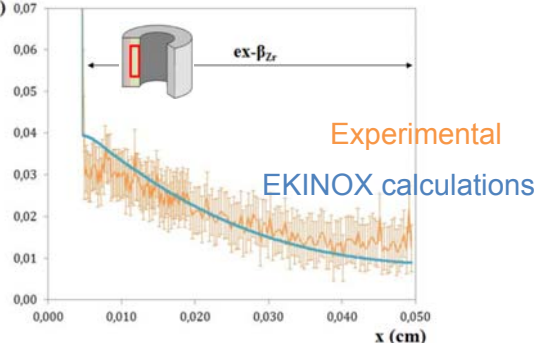
Steam oxidation,
1200°C, Zy-4

Critical duration leading to overall ductile to brittle

Analytical solution	EKINOX Zircobase	Experimental
41 min.	17.8 min.	17 min.

C. Corvalan-Maya & al.,
JNM 400 (2010) 196-204

B. Mazeres & al.,
Ox. Metals (2013)

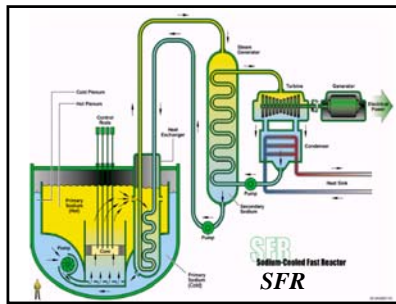


- ❑ Introduction: CEA Corrosion Unit
- ❑ Gen 2&3 (Pressurized Water Reactor – PWR)
- ❑ Gen 4
- ❑ Nuclear waste storage
- ❑ Decommissioning
- ❑ Conclusion: modelling

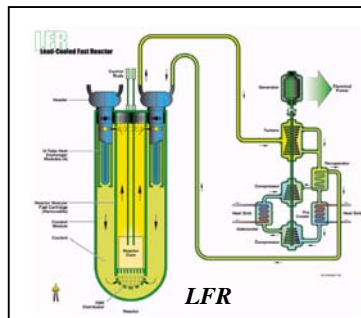
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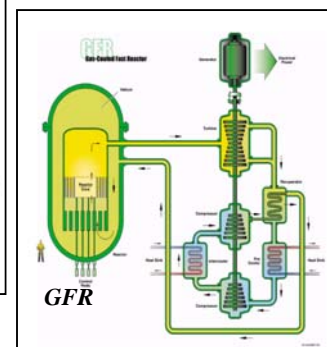
GENERATION IV CONCEPTS



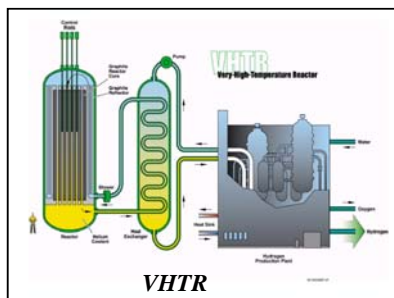
Sodium Fast reactor



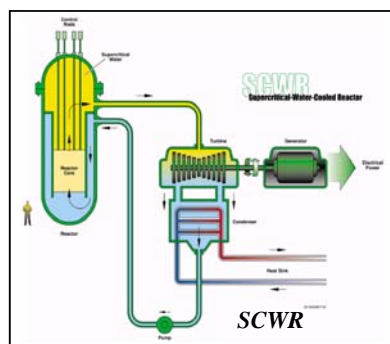
Lead Fast Reactor



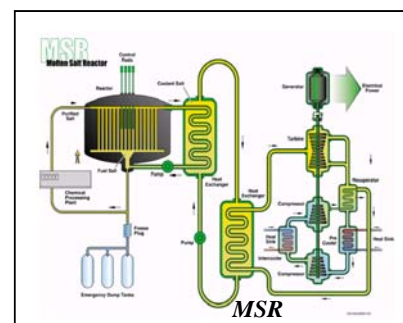
Gas Fast Reactor



Very High Temperature Reactor



SuperCritical Water Reactor



Molten Salt Reactor

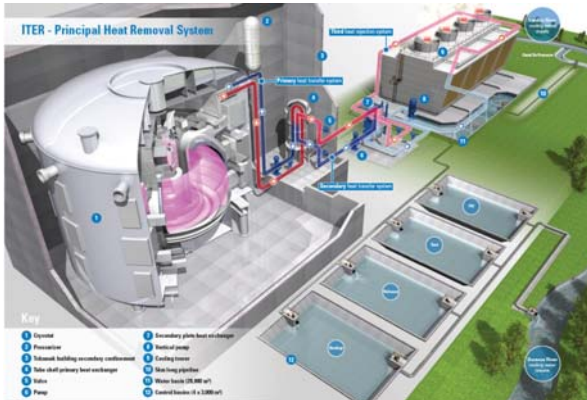
Source : AIEA

Characteristics: high and very high temperatures / Corrosive environments

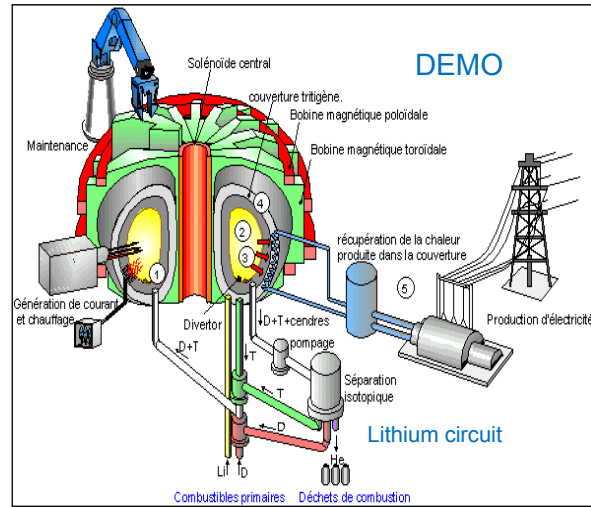
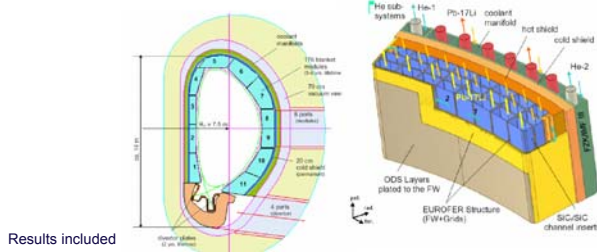
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Sodium & lead Fast reactor (SFR and LFR) & fusion (ITER)



Pb-17Li/Helium Dual Coolant Blanket



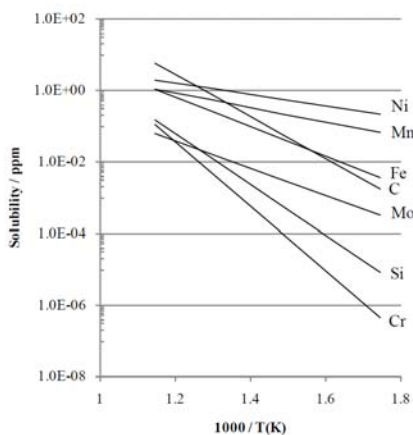
Liquid metals or gas (helium)
High dose rates
High temperatures

uthorization.

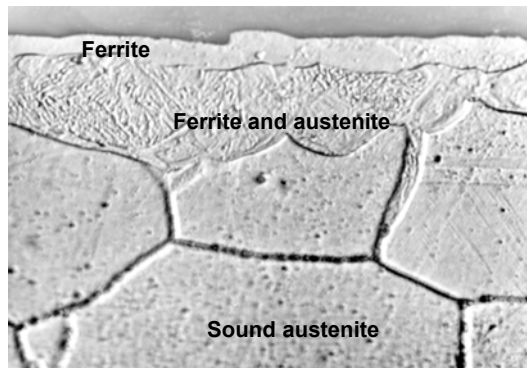
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LIQUID METAL CORROSION

Corrosion in reducing liquid metals: dissolution (Li, Pb-Li, Na & Pb with low oxygen content)

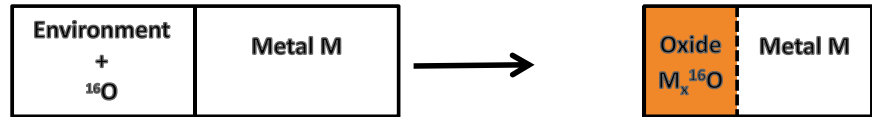
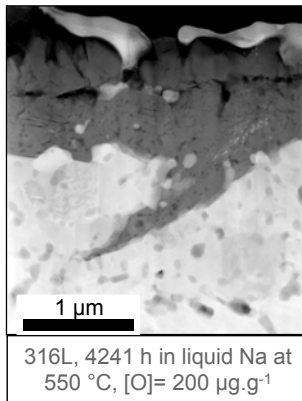


Solubility of alloy elements in liquid sodium

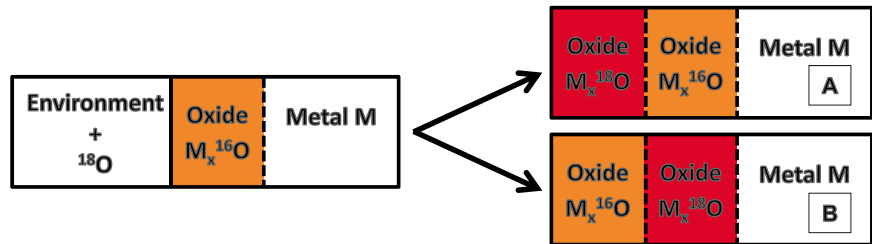


Austenitic stainless steel in liquid sodium with low level of oxygen -preferential dissolution of nickel-

Corrosion in “oxidising” liquid metals: oxidation & dissolution
(Na, with high oxygen content)



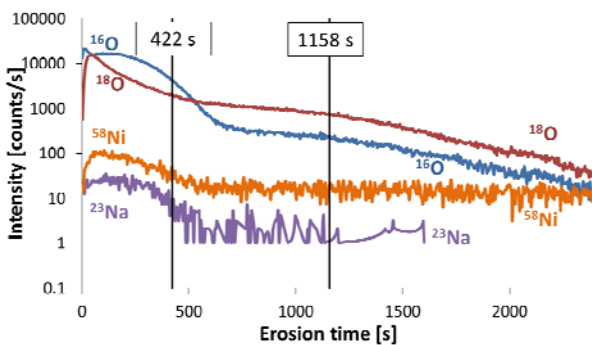
1st stage of isotopic tracer corrosion test



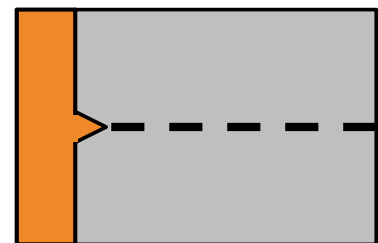
2nd stage of isotopic tracer corrosion test

“Two stages” Experiments with oxygen 18 to know where the oxide growth occurs

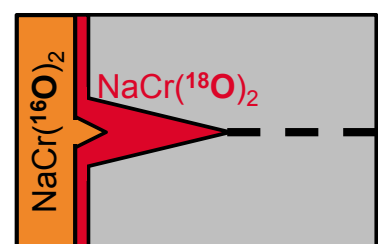
Corrosion in “oxidising” liquid metals: oxidation & dissolution
(Na with high oxygen content)



^{18}O profile on grain boundary
550 °C
[O] = 200 $\mu\text{g.g}^{-1}$
(^{16}O : 500 h,
 ^{18}O : 960 h)

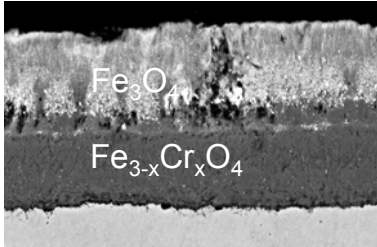


Initial stage (^{16}O , 500h)



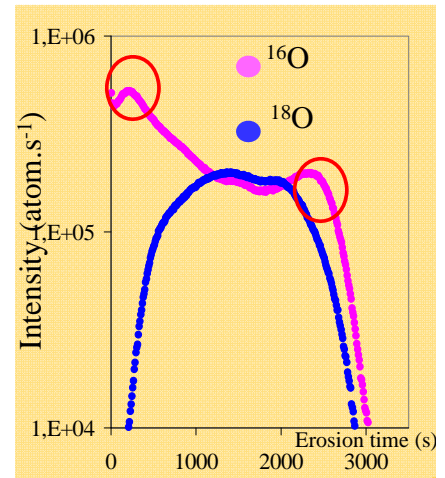
Final stage (^{18}O , 960h)

Corrosion in "oxidising" liquid metals: oxidation & dissolution (Pb-Bi with high oxygen content)



Ferritic steel (9% Cr) in Pb-Bi at 500°C with oxygen : formation of two oxide layers (protective)

Use of tracers to determine the growth mechanism (¹⁶O and ¹⁸O)
First with ¹⁶O and ¹⁸O dissolved in liquid Pb-Bi, followed by an exposure only with ¹⁶O.



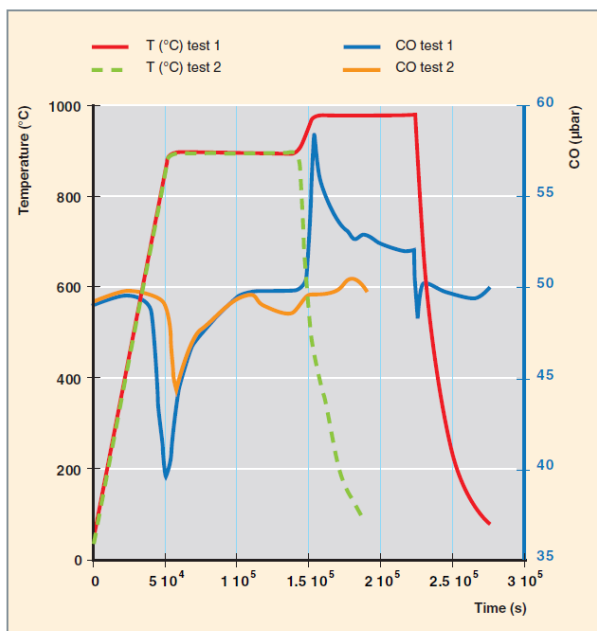
From L. Martinelli & al.



SIMS analysis : peaks of ¹⁶O at the external interface and at the metal interface

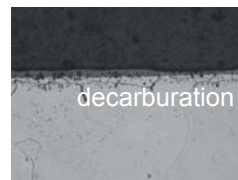
- >> Growth of magnetite layer at the interface with Pb-Bi
- >> Growth of the spinel layer at the metal/spinel interface

High temperature corrosion in helium systems (major role of the impurities / example with CO)



Evolution of CO concentration with time and temperature during the exposure of a nickel base alloy in an helium flux (other impurities constant)

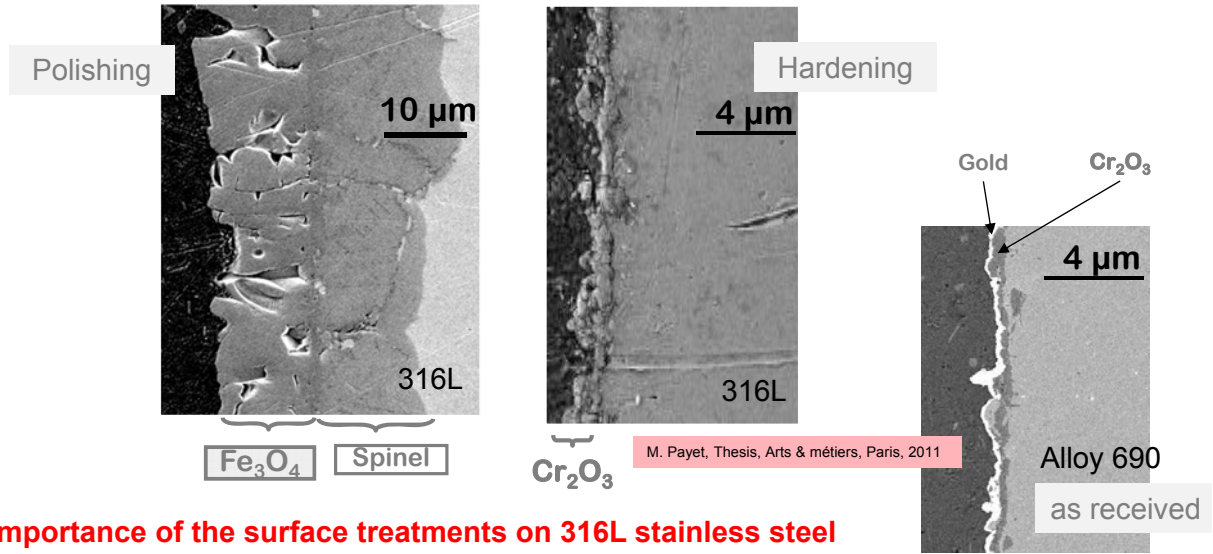
- Carburation between 650 and 900°C
- Decarburation at 980°C



From C. Cabet & al.

PASSIVE ALLOYS IN SUPERCRITICAL WATER

316L stainless steel exposed 335h at 600°C, 25 MPa in ultra pure water with H₂



Importance of the surface treatments on 316L stainless steel

- High corrosion rates with duplex oxide layer on polished surfaces
- Formation of a protective chromium oxide layer on hardened or cold rolled surfaces linked to the preferential diffusion of chromium via internal defects due to the hardening.

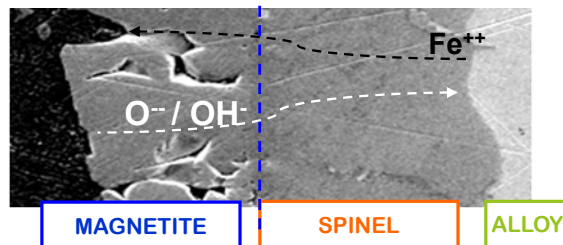
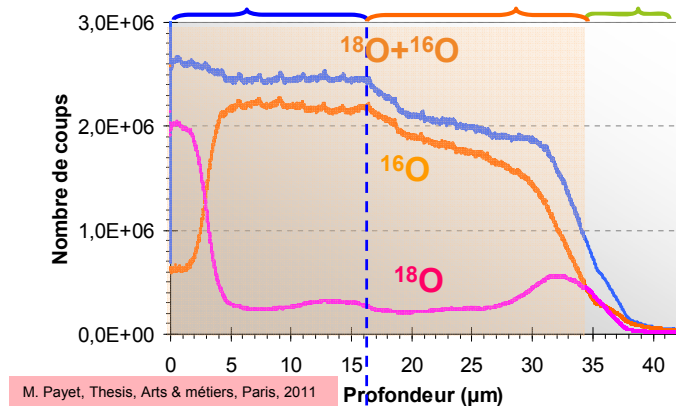
Good behavior of 690 nickel base alloy (higher chromium content)

Alloy 690, 840h

SUPERCRITICAL WATER

Mechanism of the formation of the double layer observed in SCW on stainless steels: use of tracers, ¹⁸O

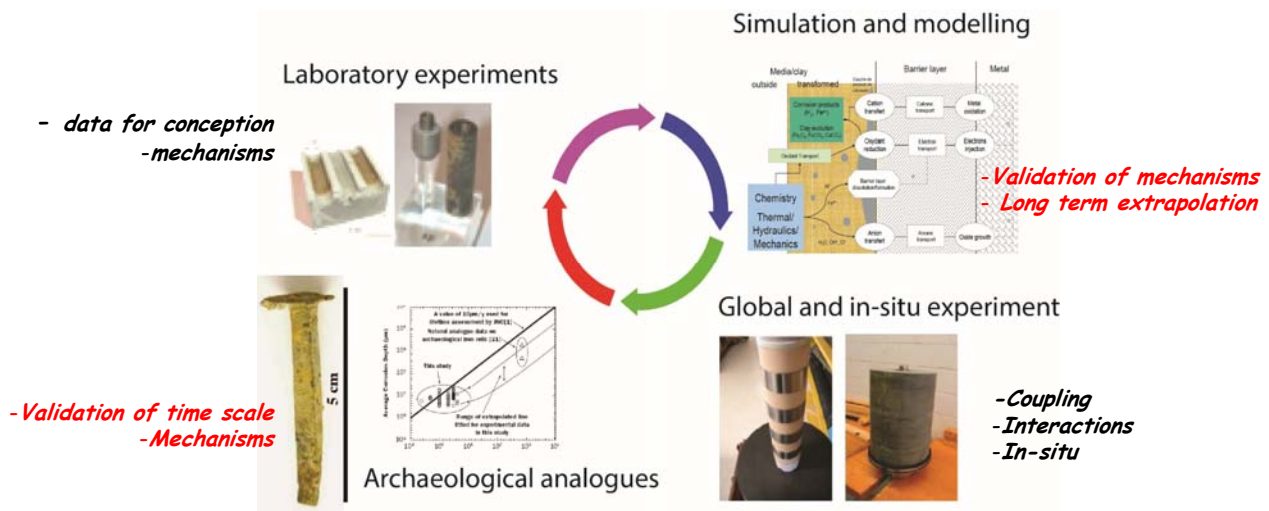
- First oxidation
760h, 600°C, 25 MPa,
H₂¹⁶O
- Second oxidation
305h, H₂¹⁸O, idem
- SIMS analyses to locate ¹⁸O which is found at two locations
 - Magnetite/SCW
 - Spinel/alloy
- Growth of the oxide layers at the two interfaces
 - Magnetite/SCW
 - Spinel/alloy
 - Modelling



- ❑ Introduction: CEA Corrosion Unit
- ❑ Gen 2&3 (Pressurized Water Reactor – PWR)
- ❑ Gen 4
- ❑ Nuclear waste storage
- ❑ Decommissioning
- ❑ Conclusion: modelling

LONG TERM CORROSION

How to predict corrosion over millennia ?



- Communication
- Technology
- Science

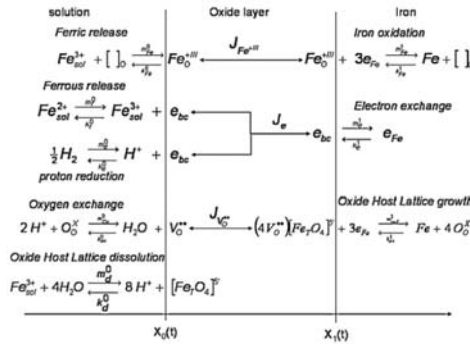
D. Féron & al., *Corrosion* (2009) 65-3, 213-223
P. Dillmann & al., *CEST* (2014) 49-6, 567-576

LONG TERM CORROSION - MODELING

Corrosion modeling of an iron based alloy in passive conditions



Arcorr experiments



In situ experiments (Mont Terri and Bure underground laboratories)

Fig. 3. Flow chart for electrons, octahedral cations and oxygen vacancies. Kroger-Vink notation is used. []_o corresponds to a free octahedral site.

C. Bataillon & al. , *Elect. Acta* 55 (2010) 4451-4467

- Corrosion modeling based on Fick and Poisson equations with moving interfaces (Diffusion Poisson Coupling Model - DPCM)
- Electrochemical experiments for data acquisition and verification
- Coupling with geochemical models
- Clay and concrete

Results included in this document are CEA's property. They cannot be disclosed without prior authorization.

LONG TERM CORROSION - MODELING

Corrosion modeling of an iron based alloy in passive conditions Example of obtained results

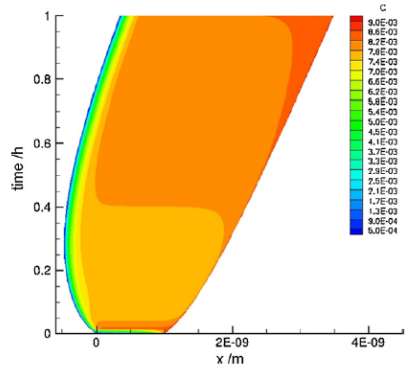
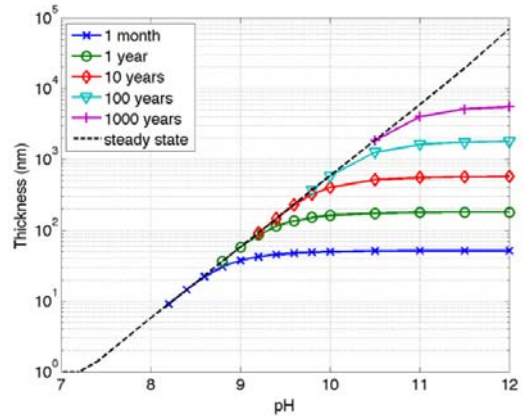


Fig. 36. Evolution of the location of the outer and inner interfaces with time for $V=0.05$ V/SHE. The concentration of oxygen vacancies is indicated by the color on the right band. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

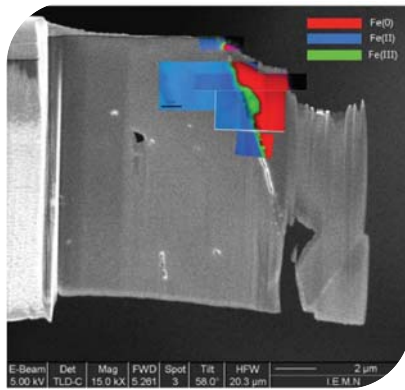


C. Bataillon & al. , *Elect. Acta* 55 (2010) 4451-4467

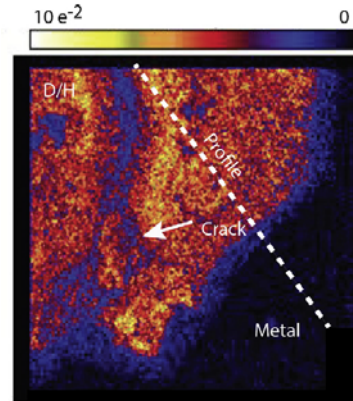
C. Bataillon & al. , *J. Comput. Physics* 231 (2012) 6213-6231

This model is under implantation in the nuclear waste simulations codes

USE OF ARCHEOLOGICAL ARTEFACTS



D/H ratio map obtained at the metal/corrosion product interface

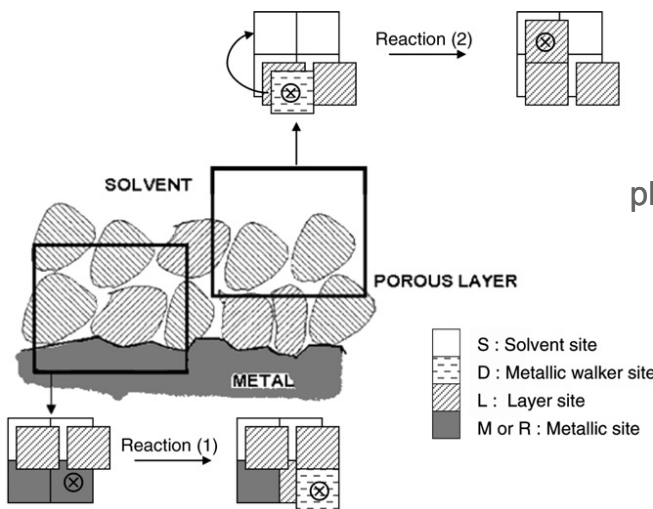


A. Michelin & al., JAAS (2012)
 Michelin et al., J. Analytical Atomic Spectrometry, 2013
 Leon et al., Corrosion Science (2014)

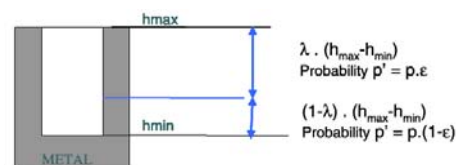
- Corrosion products on a 450 year-old archaeological iron nail in anoxic environment were investigated at the nanometer level using STXM
- **Interfacial layer of 100nm to few µm at the interface metal/oxide interface (magnetite + maghemite, low porosity)**
- Support the hypothesis of a nanolayer controlling the corrosion process (DPCM, Point Defect Model and associated models)

STOCHASTIC MODELING & CORROSION

Use of cellular automata to model morphological evolutions during corrosion



Aqueous corrosion is a stochastic phenomena regarding the occurrence of anodic and cathodic reactions

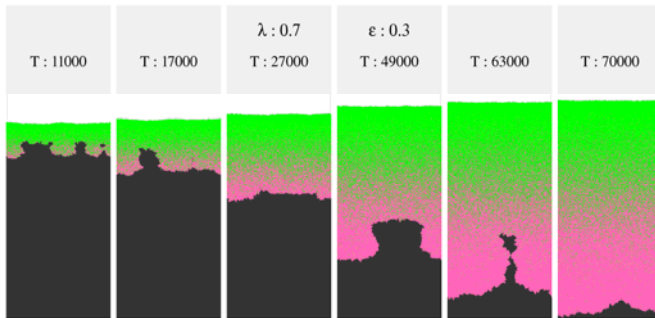


Reaction 1: formation of the oxide layer
 Réaction 2: dissolution of the oxide

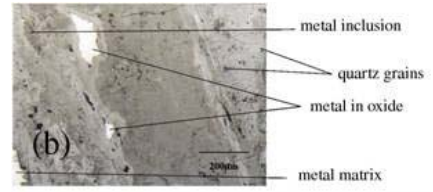
Probability of reaction 1 is function of the corrosion depth

D. di Caprio & al.,

Use of cellular automata to model corrosion phenomena



Archeological analogue with the presence of metallic inclusions inside the oxide layer



Continuous evolution of the interface morphology from uniform corrosion to more localized corrosion and creation of metal inclusions as observed on archeological analogues $\lambda = 0.70$ and $\varepsilon = 0.30$

D. di Caprio & al., Corrosion Science 53 (2011) 418–425

CONTENT

- Introduction: CEA Corrosion Unit
- Gen 2&3 (Pressurized Water Reactor – PWR)
- Gen 4
- Nuclear waste storage
- Decommissioning
- Conclusion: modelling

NEW CHALLENGE: DECOMMISSIONING

Generic activities

- Atmospheric corrosion (from mechanisms to corrosion code)
- Chloride pitting on passive alloys

Specific programs

- Graphite
- Corrosion in supercritical fluids (SCWO and SCCO₂)
- Mg alloys corrosion in concrete or geo-polymer matrix

Expertise & consulting

- Transport
- Interim storage



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9 EDF reactors are under decommissioning

- 1 réacteur à eau pressurisée (REP)
Chooz A (300MW) : 1967-1991
- 1 réacteur à eau lourde (REL)
Brennilis (70 MW) : 1967-1988 (EDF/CEA)
- 6 réacteurs de la filière Uranium naturel - graphite-gaz (UNGG)
Chinon A1 (70MW) : 1963-1973
Chinon A2 (200MW) : 1965-1985
Chinon A3 (480MW) : 1966-1990
Saint-Laurent A1 (480MW) : 1969-1990
Saint-Laurent A2 (515MW) : 1971-1992
Bugey 1 (540MW) : 1972-1994
- 1 réacteur à neutrons rapides (RNL)
Creys-Malville (1240MW) : 1986-1997

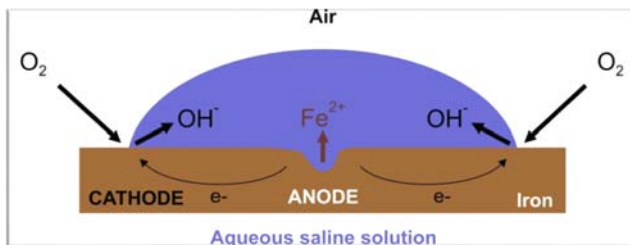
Specificity: 6 UNGG reactors (graphite moderator, carbon dioxide, natural uranium)

IFREMER - Plateforme OceanLab/CEA

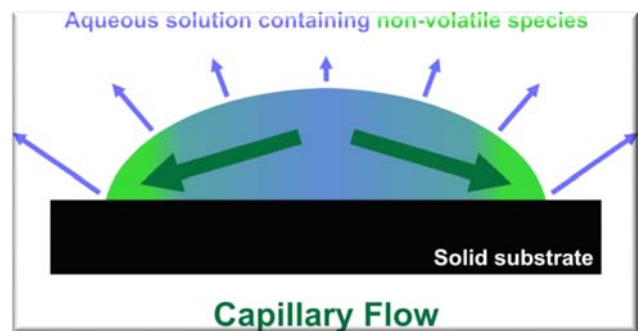
28/11/12



ATMOSPHERIC CORROSION / BASICS



The Evans droplet model (static conditions)



Capillary flow towards the three-phase contact line

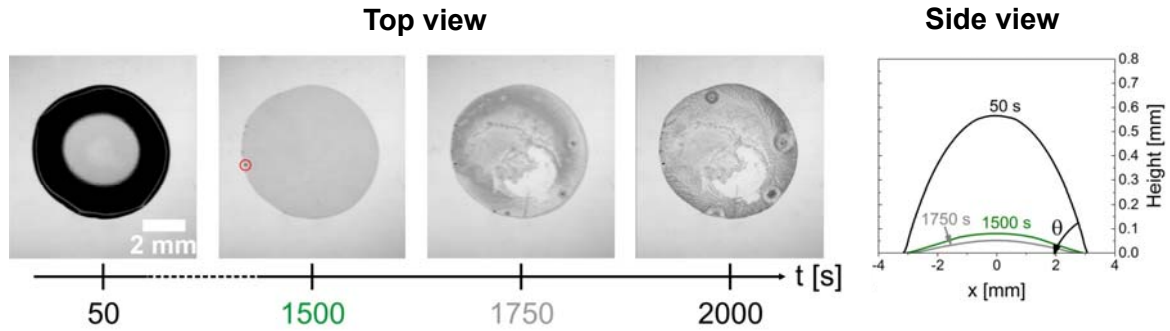
« Coffee-ring » effect



R. D. Deegan. *Nature*, **1997**, 389, 827-829
H. Hu. *J. Phys. Chem.*, **2002**, 106, 1334-1344

Corrosion under Evaporating Salty Sessile Droplets

Relative Humidity: $RH = 40\%$ ($T = 23^\circ\text{C}$)
 Gaseous phase: Air
 Initial volume: $V_0 = 10 \mu\text{l}$
 Salt initial concentration: 10^{-3} M NaCl
 Substrate: Iron

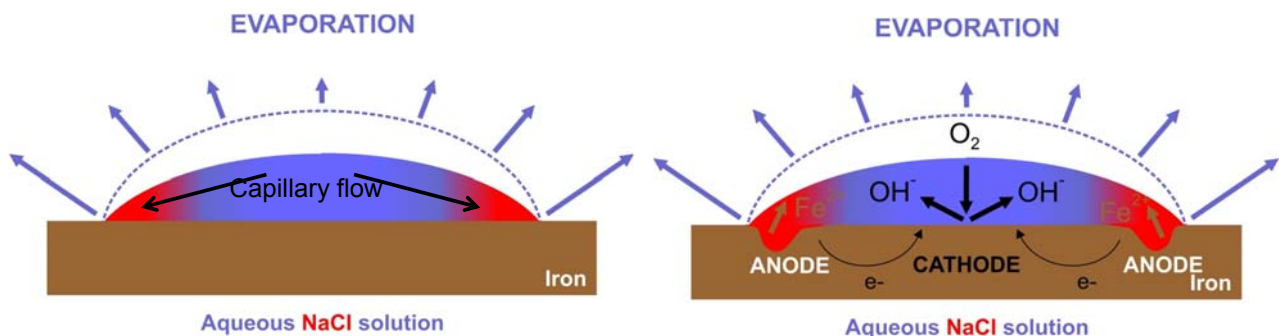


Pitting corrosion starting near the contact line

V. Soulié, thèse Université de Montpellier, 2014

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- Evaporation of pinned salty sessile droplets causes peripheral salt enrichment
- Local chloride enrichment decreases oxygen solubility and promotes the initiation of corrosion

Inversion of the classical Evans droplet scenario

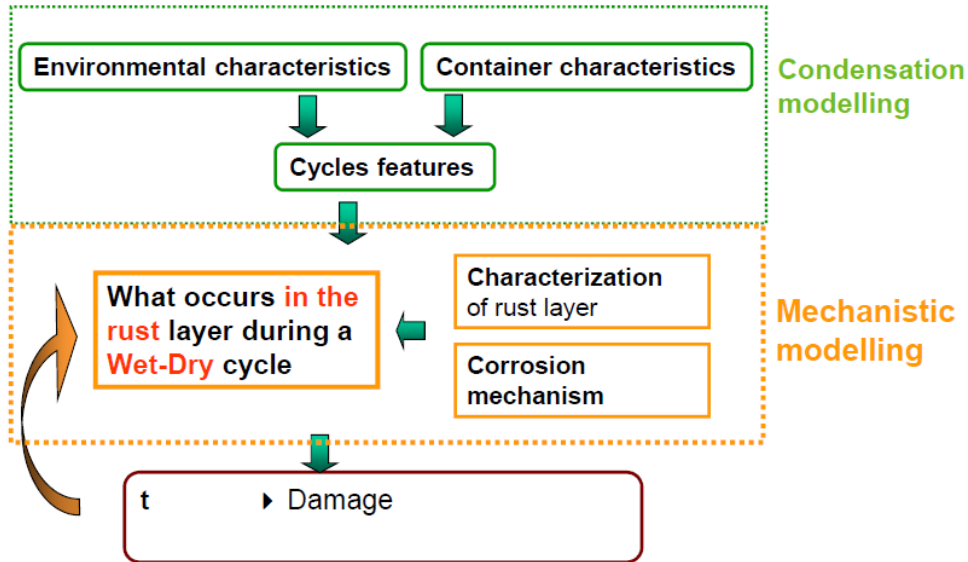
V. Soulié, thèse Université de Montpellier, 2014

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❑ « Calculations » based on ISO standards (9223 & 9224)

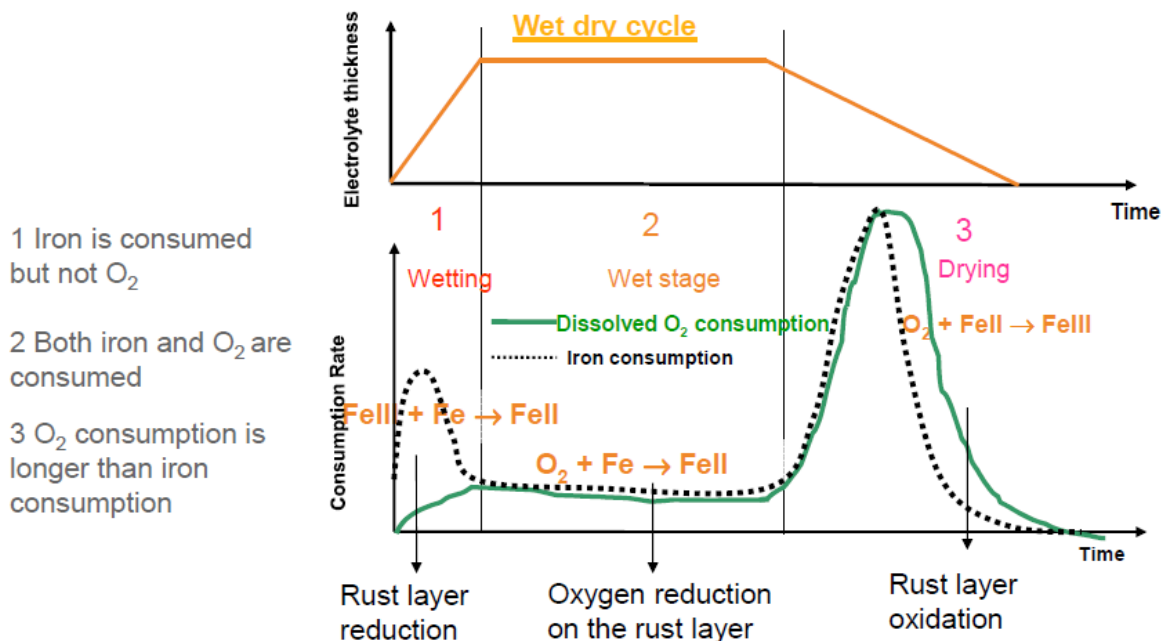
❑ Mechanistic modelling



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Corrosion mechanism during a wet dry cycle (Based on Stratman works)

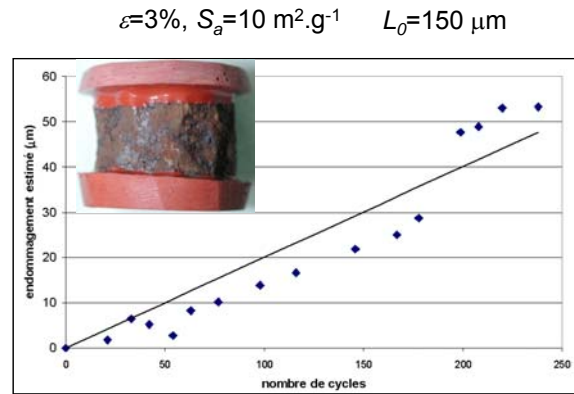
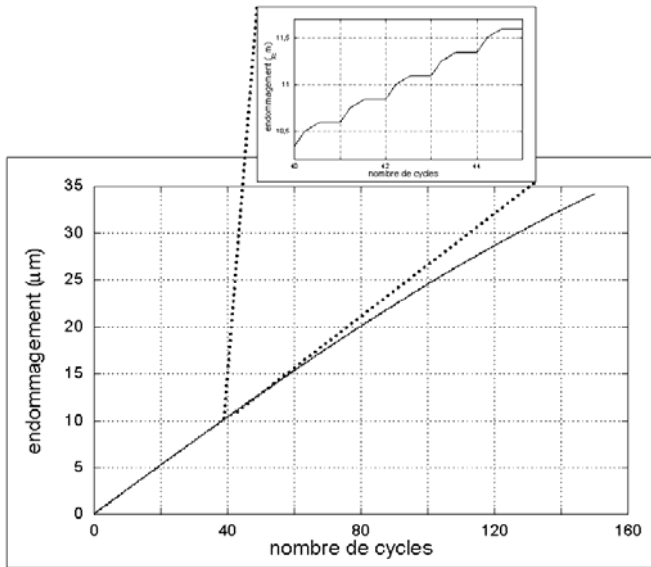


E. BURGER, & AL. CORROSION SCIENCE, V. 53(6), 2011, 2122-2130

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Verification of the atmospheric mechanistic modelling



calculation of n wet dry cycles

E. BURGER & AL. CORROSION SCIENCE, V. 53(6), 2011, 2122-2130

Results included in this document are CEA's property. They cannot be disclosed without prior authorization.

Experimental data:
Ageing in climatic chamber

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**ATALANTE / DELOS
DESTRUCTION OF CONTAMINATED ORGANIC LIQUID WASTES**

- Treatment of spent fuel contaminated organic liquids
- C.H.O.N., **TBP/alcane**, amines, aromatics...

Waste (+ H₂O)

H₂O + O₂/N₂

Effluents Outlet

Stirrer

Confinement Tube

Magnetic HP drive

corrosion
4,2 µm/h
HTO of dodecane / TBP
5%

Initial thickness
1mm

Joussot-Dubien C., Turc H.A., Didier, G., Brevet FR 2814967, 2000
S. Sararde, 11th International Symposium on Supercritical Fluids, October 11-15, 2015, Seoul, Korea

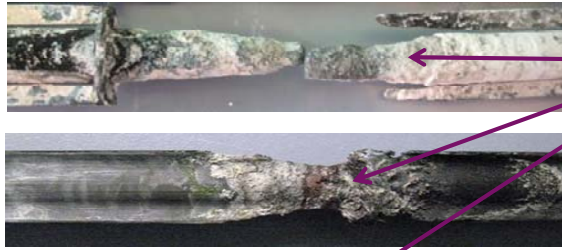
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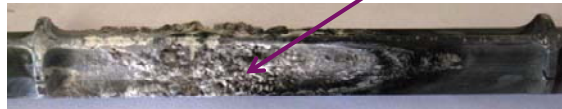
SUPERCRITICAL WATER OXIDATION

Combustion of dodecane/tributylphosphate/dichloromethane
Material : Ti Grade 2

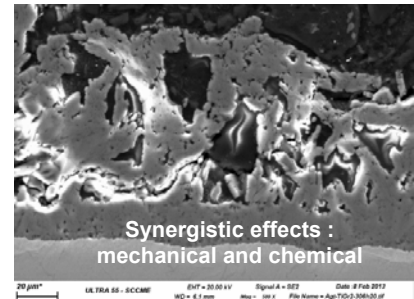
Welding design



Massive design



TiP₂O₇, Ti(OH)PO₄



Final Scale up

DAMAGE MANAGEMENT & FINAL PROCESS

100 h



300 h



1000 h

Corrosion < 5 μm/h

Corrosion < 5 μm/h
Mechanical Strength

Industrial Conditions
6 months Batch
1 hour maintenance

Joussot-Dubien C., Turc H.A., Didier, G., Brevet FR 2814967, 2000

S. Sararde, 11th International Symposium on Supercritical Fluids, October 11-15, 2015, Seoul, Korea

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CONCLUSIVE COMMENTS

CORROSION CHALLENGES

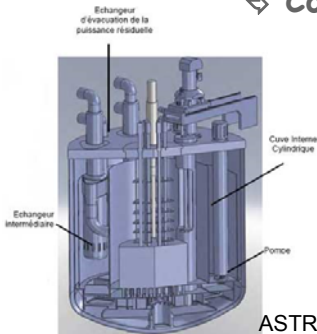
- ↪ Extension of life time of NPPs
life time extension (FAC & SCC)
- ↪ Nuclear waste disposals
Long term prediction (methodology)
- ↪ Generation 4 & fusion
high temperatures & aggressive media
- ↪ Decommission



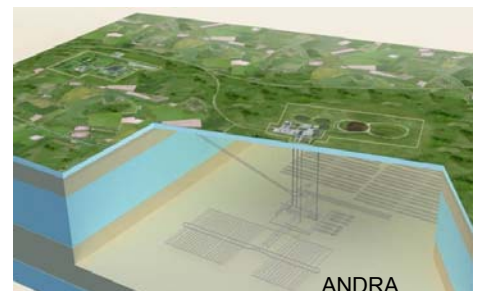
Source : EDF

↪ Common approaches & processes

- experimental (use of tracers)
- modelling



ASTRID, CEA



ANDRA

Major challenges linked with corrosion behaviour



Institut national des sciences
& techniques nucléaires

Materials Simulation & Engineering

Winter School

“ Modelling of Corrosion ”

INSTN CEA Saclay, December 10-14, 2012

Spring School

“ Modelling of corrosion for Nuclear Reactors ”

INSTN CEA Saclay, March 31-April 4, 2014

Fall School

“ Modelling of Corrosion ”

INSTN CEA Saclay, October 26-30, 2015

**“School for Advanced
Thermodynamic Assessments”**

Summer school

June 23-27, 2014

Centre Port-Royal, France

July 6 —10, 2015

Centre Port-Royal, France

HTCorrosion
Toulouse 2013
Spring School

HTCorrosion
Biarritz 2014
2nd Spring School

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Thank you for your attention



FROM RESEARCH TO INDUSTRY



French Alternative Energies and Atomic Energy Commission

Alternative Energies and Atomic Energy Commission
Centre de Saclay | 91191 Gif-sur-Yvette Cedex
T. +33 (0)1 64 50 10 00 | F. +33 (0)1 64 50 11 86

Direction
Département
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