Oxidation of a cold-worked 316L stainless steel in pressuriezd water reactor primary water environment

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OXIDATION OF A COLD-WORKED 316L STAINLESS STEEL IN PRESSURIZED WATER REACTOR PRIMARY WATER ENVIRONMENT

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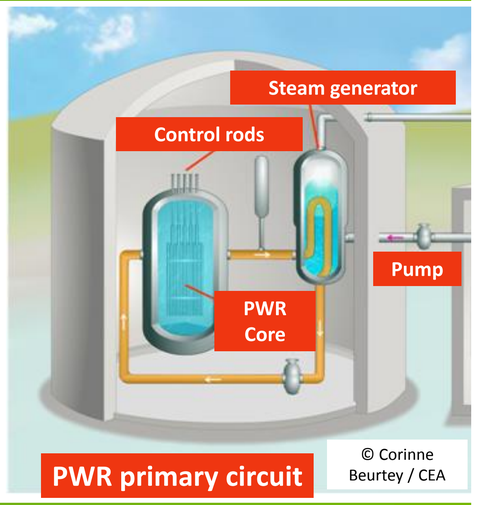
Introduction

- Pressurized Water Reactors (PWR) represent 76.3 % of the electrical power produced in 2015 in France [1]
- **Operational feedback → intergranular Stress Corrosion Cracking (SCC)** affecting cold-worked stainless steel components in PWR primary water [2]
- PWR primary water : pure hydrogenated and deaerated water, with Li and B additions, 150 bars and 290-340 °C
- **Oxygen transients** (oxygen injections in PWR primary water [3, 4]) **= Possible detrimental factor**
- Dissolved oxygen in PWR primary water has an impact on the :
  → Location of initiation sites (trans- or intergranular sites) [5]
  → Oxide layer properties (inner layer morphology and composition) [6]
- However, **the oxygen transient effect on SCC susceptibility has not been extensively investigated to date**

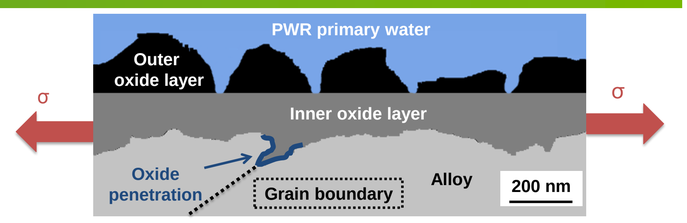


Schematic representation of the primary circuit of a PWR

Objectives

General aim: Determine the effect of dissolved oxygen on SCC susceptibility of a cold-worked stainless steel in PWR primary water

- Hypothesis: Link between oxidation and SCC susceptibility
- In particular, localized oxide penetrations are suspected to play a crucial role in crack initiation



Oxidation of a stainless steel specimen exposed to PWR primary water

First step: effect of dissolved oxygen in PWR primary water
→ On oxidation kinetics and surface oxide layers nature
→ On localized oxide penetration kinetics, morphology and nature

} Pre-strained / non cold-worked 316L stainless steel specimens

Material and Experimental Conditions

Composition (weight %)	C	S	P	Si	Mn	Ni	Cr	Mo	Cu	N
316L – this study	0,016	0,0009	0,026	0,62	1,86	10	16,54	2,03	-	0,022

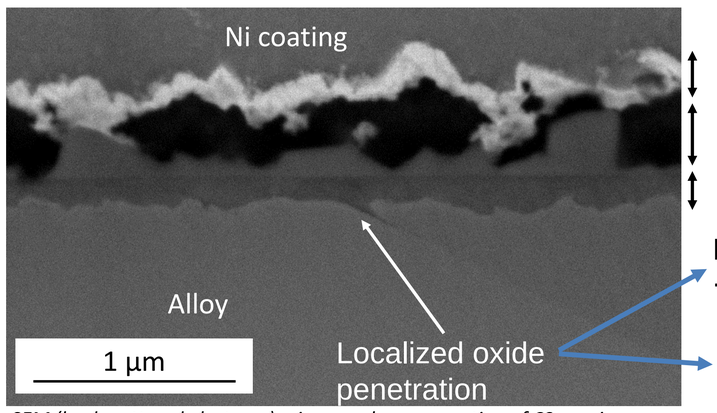
Annealing treatment (1050°C, Ar overpressure, 1h) applied to our specimens
→ Isotropic austenite grains (50 ± 10 µm) and residual ferrite (1-5 % in volume)
→ Non-sensitized material

Nominal PWR primary water (pure water, 150 bars, 340°C, pH = 7.0 to 7.2)					
Element	B	Li	H	O	Chlorides, sulfates, fluorides
Concentration	1000 ppm	2 ppm	25-35 mL/kg (TPN)	< 0,01 ppm	< 0,05 ppm

Oxidation in nominal PWR primary water →
- C2: Oxidation time = 500 h, Non cold-worked
- C1: Oxidation time = 1000 h, Non cold-worked
- EP1: Oxidation time = 500 h, Pre-strained (11%)

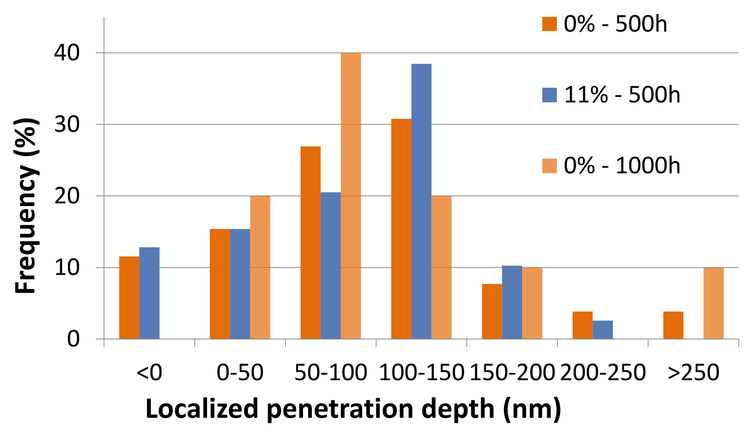
Results

Oxidation kinetics

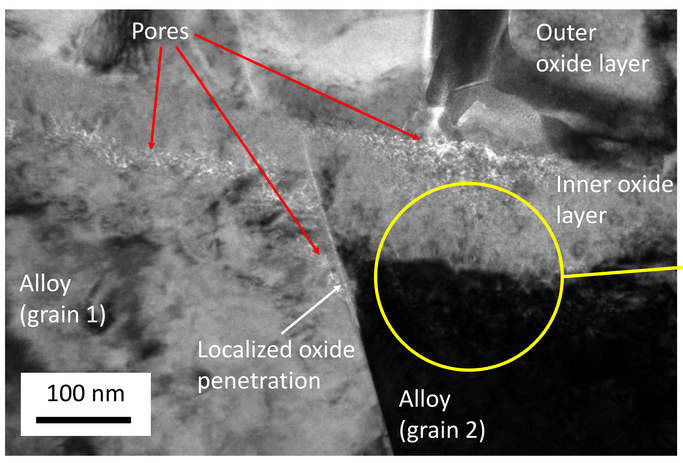


SEM (backscattered electrons) micrograph, cross-section of C2 specimen

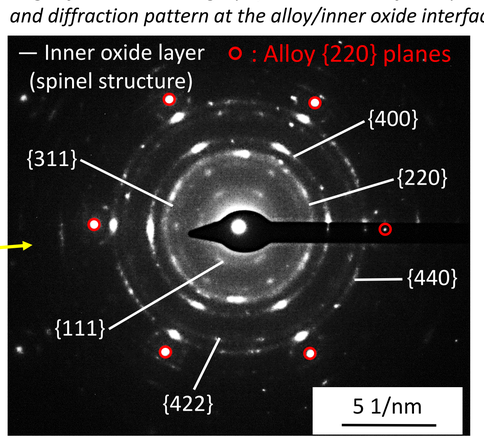
Specimen	Mean oxide thickness (nm)	Mean localized penetration depth (nm)
500 h	140 ± 41	92 ± 78
11% - 500 h	89 ± 43	87 ± 64
1000 h	146 ± 42	105 ± 74

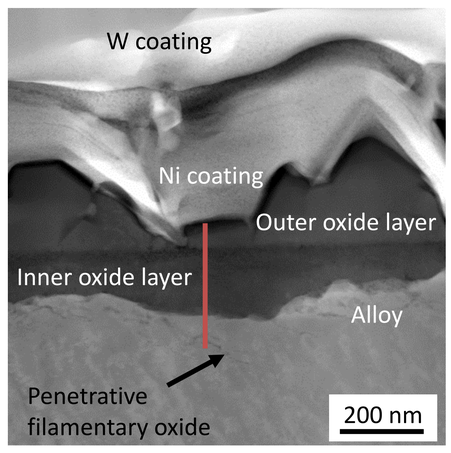


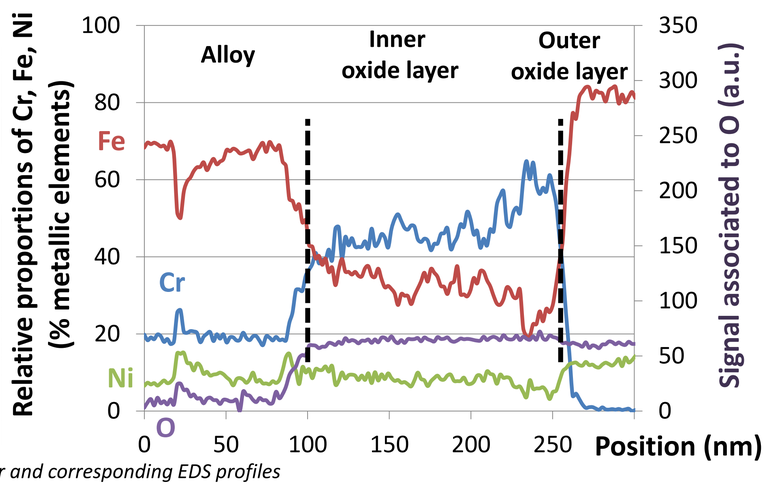
TEM characterization of a non cold-worked specimen oxidized 500h at 340° C



Bright-field TEM micrograph, cross-section of the specimen and diffraction pattern at the alloy/inner oxide interface







STEM-HAADF micrograph of the surface oxide layer and corresponding EDS profiles

Conclusions

SEM observations after oxidation tests in nominal PWR primary water →
- Duplex surface oxide layer
- Localized oxide penetrations at grain boundaries or at slip bands
- Cold-working → lower mean oxide thickness
- Increasing oxidation times → increase of the deepest localized penetration density

TEM characterization of a non cold-worked specimen oxidized 500h at 340°C →
- Inner oxide layer: randomly-oriented nano-grains of spinel structure, composition close to $(Fe,Ni)(Fe,Cr)_2O_4$
- Outer oxide layer: Fe-rich, possibly magnetite Fe_3O_4 (to be confirmed)

Next steps →
- Tests in nominal PWR primary water: oxidation kinetics and impact of cold-working on oxidation
- Adjustment of the experimental methodology for tests in aerated PWR primary water
- Tests in aerated PWR primary water → effect of dissolved O_2

[1] : Commissariat à l'Energie Atomique, Mémento sur l'énergie 2016, 2016
[2] : ILEVBARE, G., et al, Fontevraud 7, Avignon, 2010
[3] : COMBRADE, P., et al, Dossier Technique de L'ingénieur, bn3756, 2014
[4] : BETOVA, I., et al, rapport VTT-R-00699-12, 2012
[5] : HUIN, N., et al, EnvDeg 2015
[6] : TERACHI, T., et al, 61st NACExpo, Paper 06608, 2006