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Corrosion Behavior of a AISI-316 Stainless Steel Grade Type in Nitrogen Under Pressure (180 bar) at 515 °C

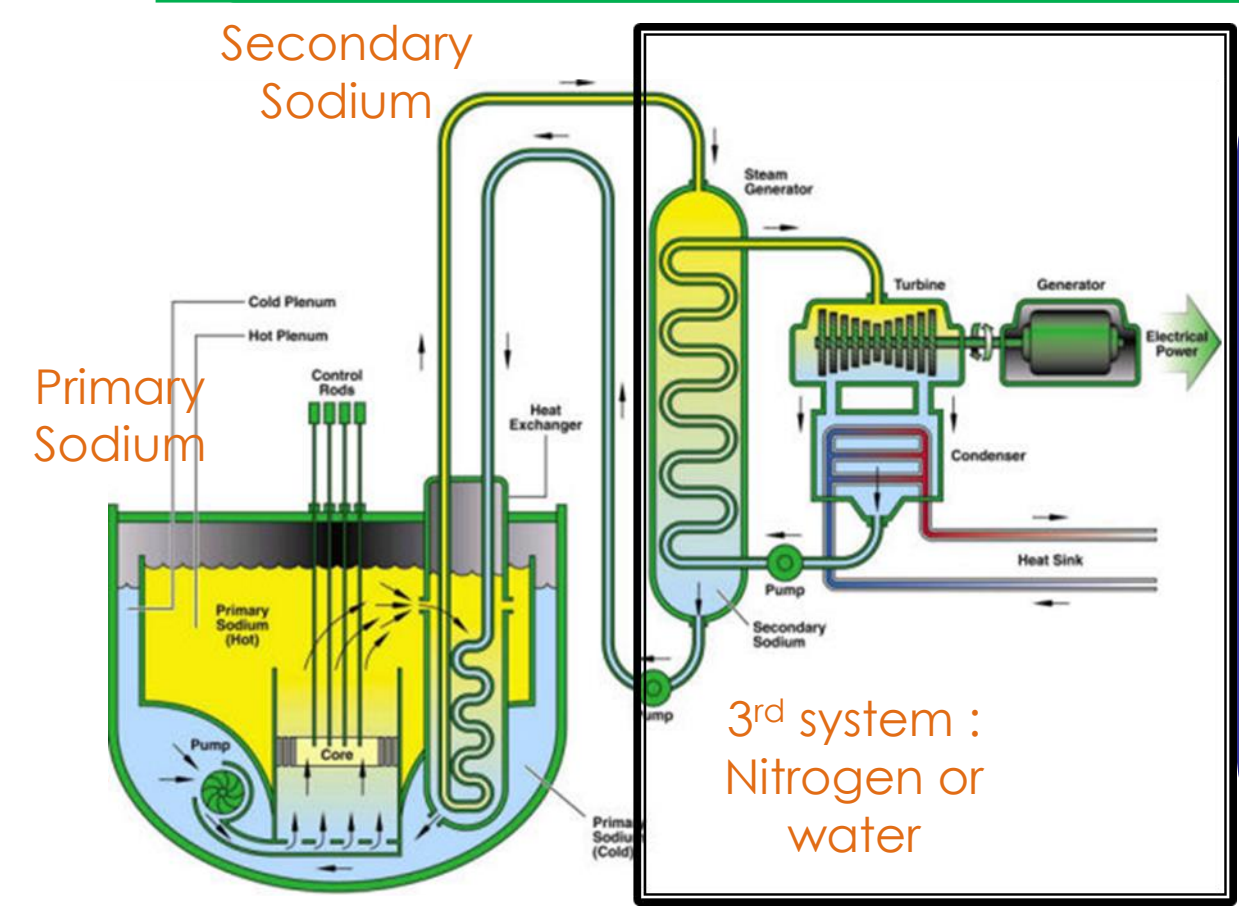
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CONTEXT: Generation IV for nuclear power plants: Energy Conversion System (ECS) for ASTRID project

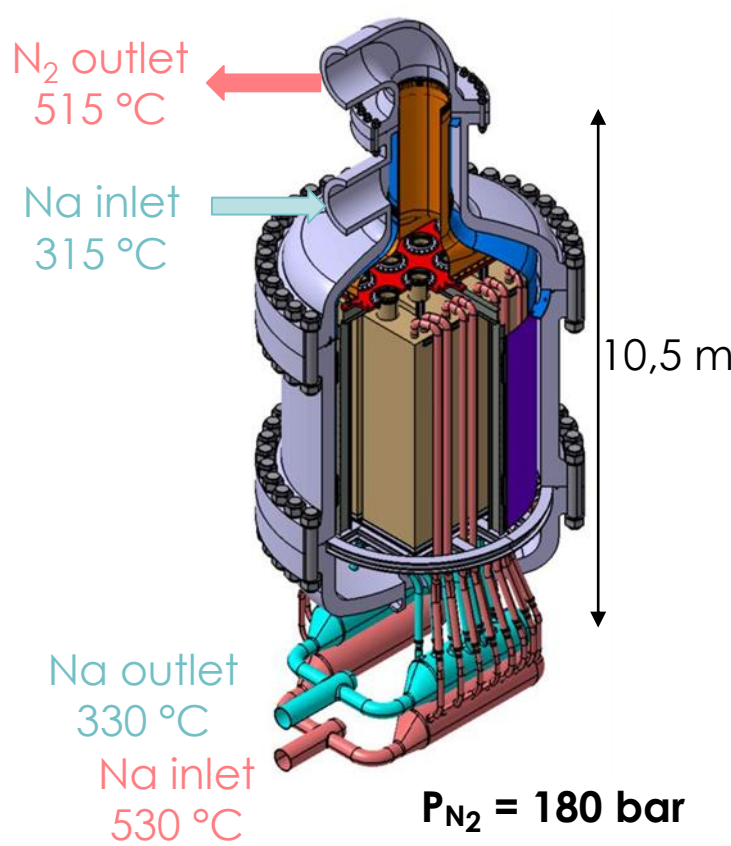


ASTRID : prototype advanced fast neutron reactor

- Sodium-cooled Fast reactor
- Conceived of feedback of earlier installations (Phenix and Superphenix)
- With technological innovations **for enhanced safety**

Energy Conversion System with Gas is preferred to ECS with Water

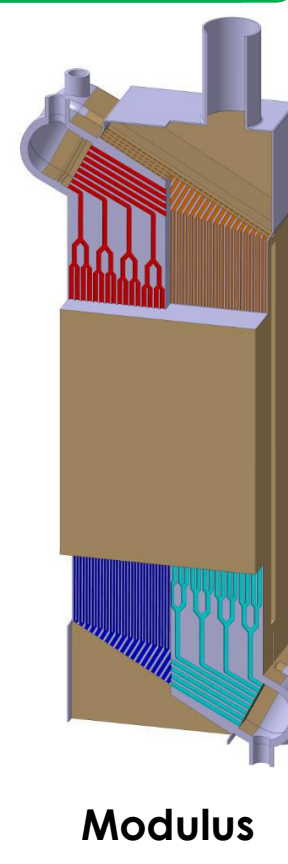
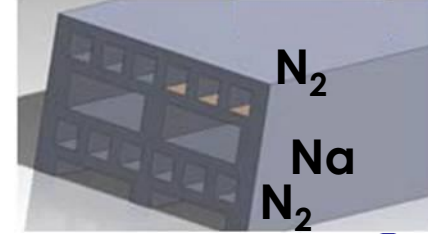
→ Sodium-Water Reaction eliminated



Sodium-Gas Energy System

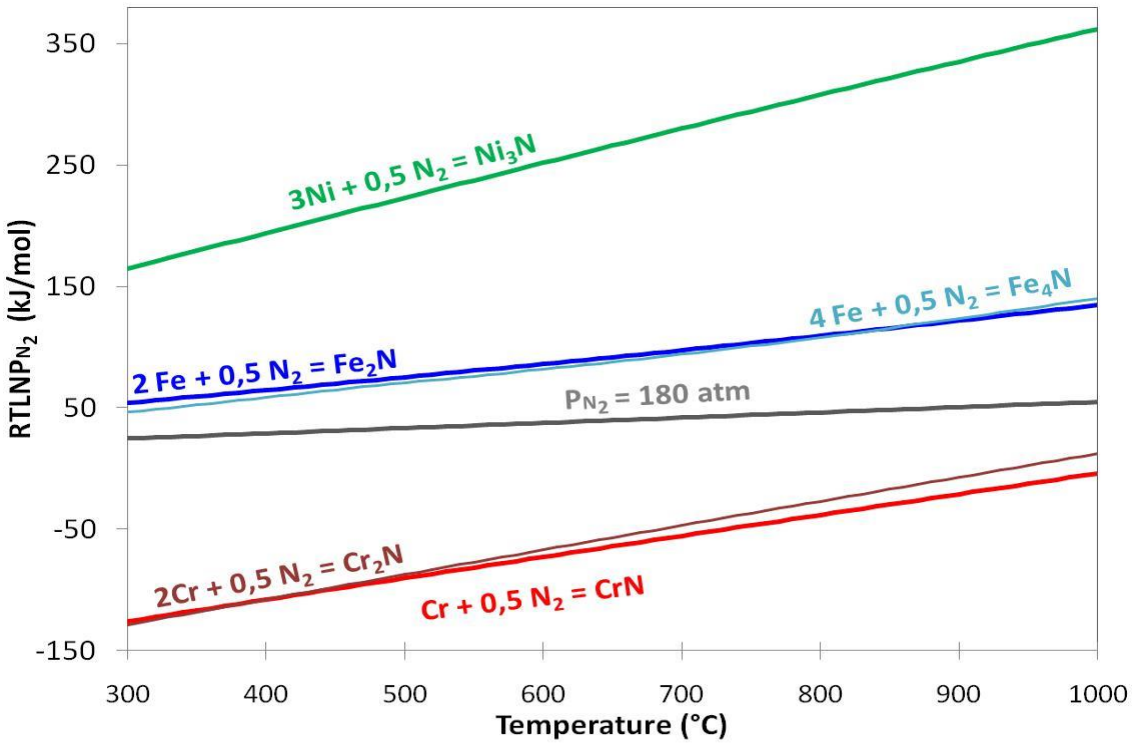
- 8 modulus in under pressure vessel with hot gas collector pipe
- Based on the principle of plate heat exchanger: Na and N₂ flow in counter-current
- Specific design with 2 channel networks : heat transferred through 1 mm thick wall

Does nitridation occur and what would the involved depth ?



OBJECTIVES

Performance of the ECS has to be guaranteed for **30 years** → Compatibility of the 316L with N₂ environment at 515°C and 180 bar has to be checked.



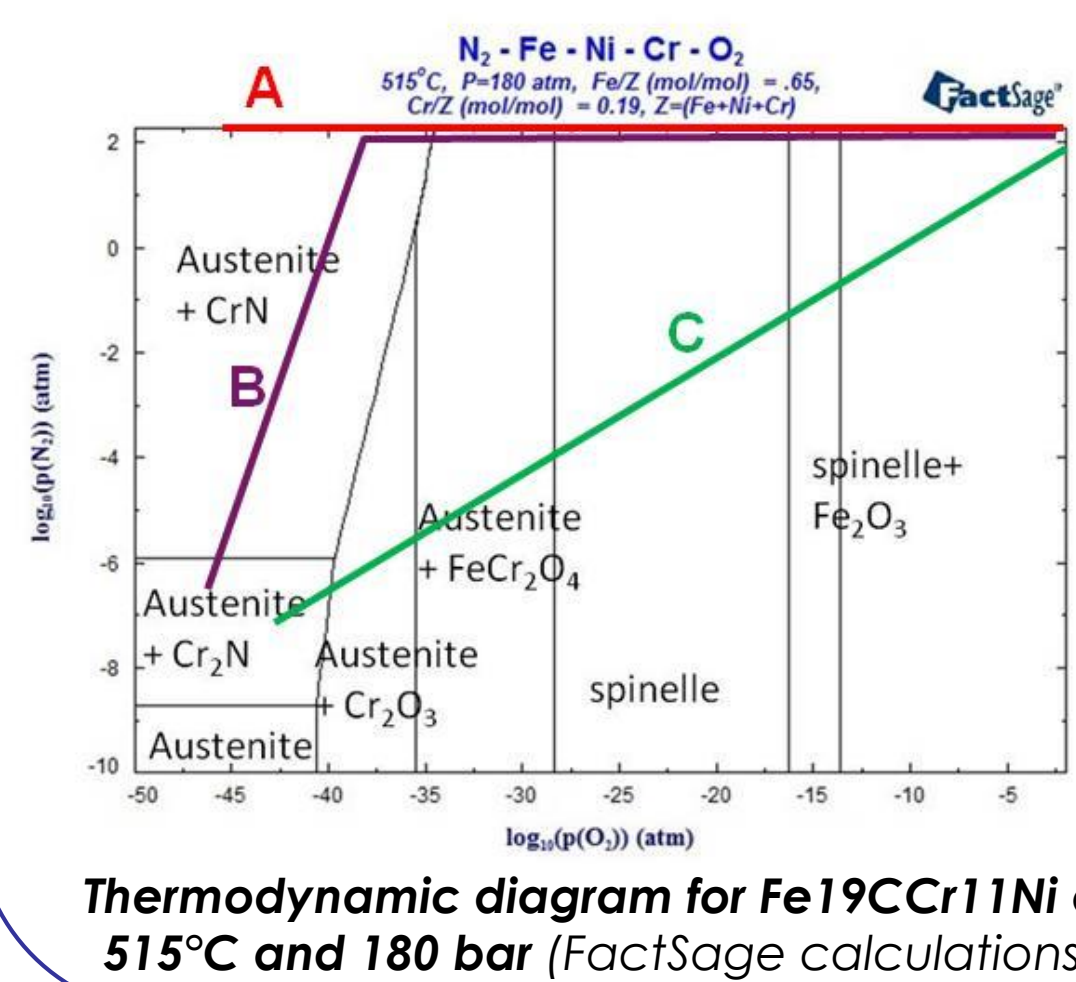
Main nitrides Ellingham diagram
Thermodynamic data from HSC database.

- Few relevant data within the ECS conditions
- Thermodynamic forecasting : CrN and Cr₂N
- Embrittlement by increasing hardness
- Nitrides growth controlled by diffusion
- But large scattering of diffusion coefficients in literature

→ Nitridation thickness (after 30 years) : up to 4 to 10 mm!

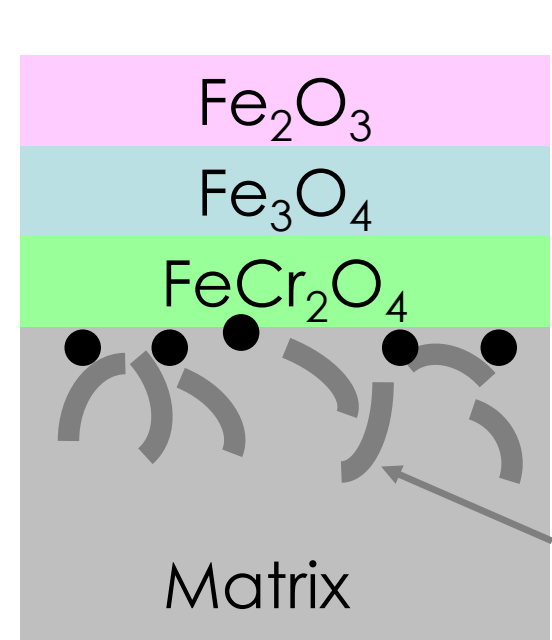
Need of refining data

Oxidation susceptibility: competition between oxidation and nitridation

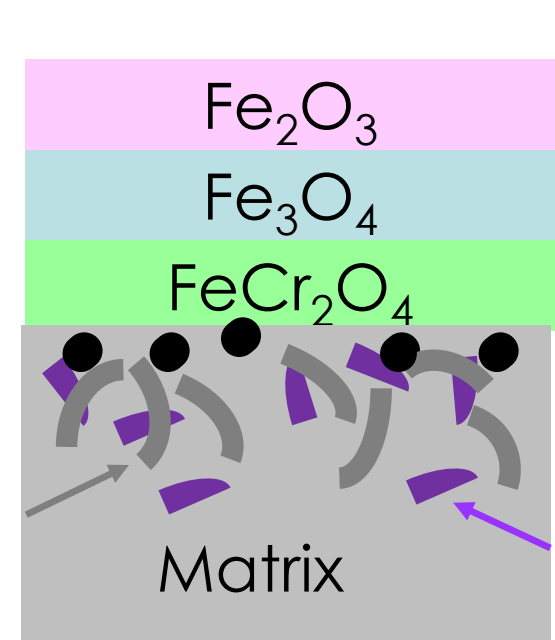


3 scenarios suggested by thermodynamic

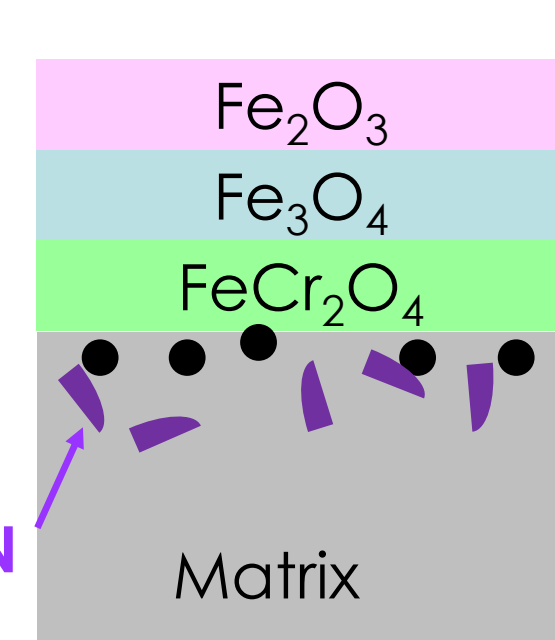
Path A



Path B



Path C



• Cr₂O₃ internal oxidation

Literature do not rule on the absence of damaging on 316L in ECS conditions.
Experimental program set up to acquire long term corrosion kinetics (up to 5000 hours)

EXPERIMENTAL

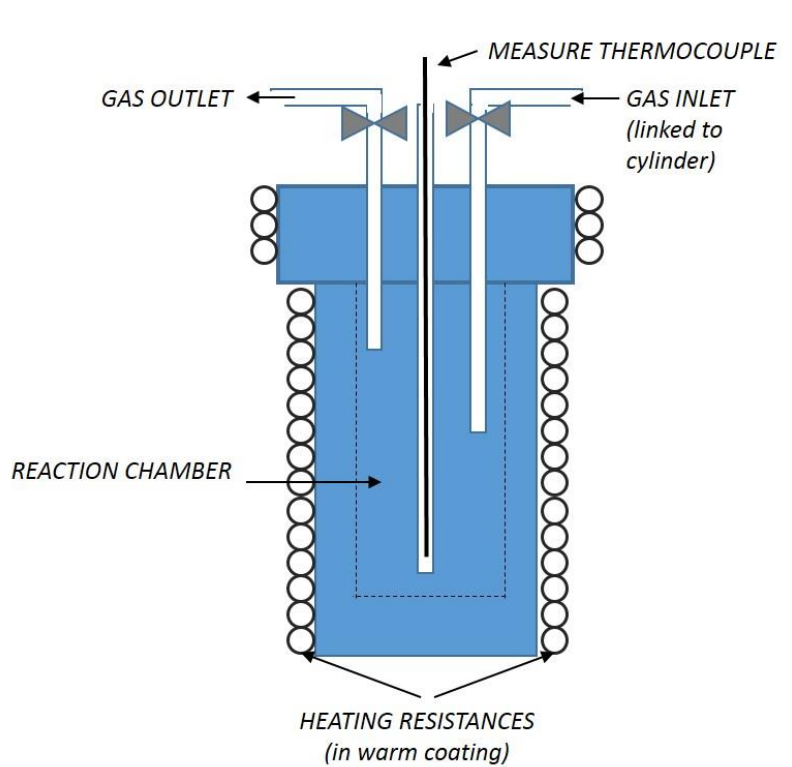
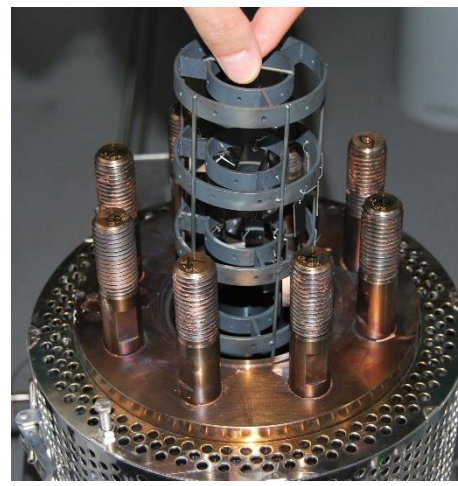
MATERIAL

EN1.4404 : X2Cr-Ni-Mo-17-12-2

C	Si	Mn	P	S	Cr	Mo	Ni	N	Co	Fe
0,022	0,335	1,286	0,034	0,002	16,543	2,044	10,093	0,048	0,132	bal

Composition (weight %) of studied material

Surface sample polished (SiC grade 1200)

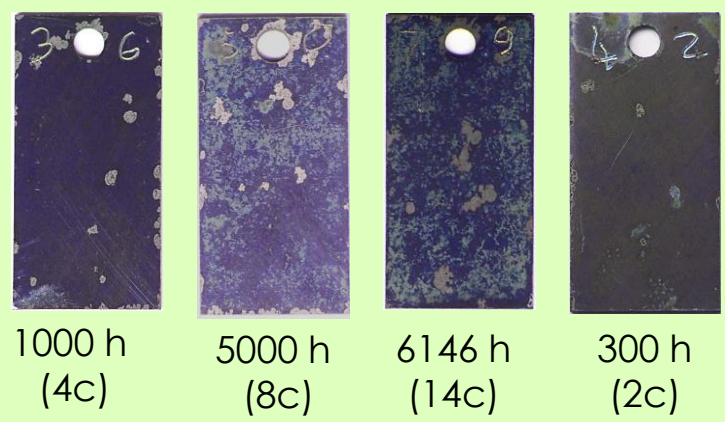


- ❑ Cylindrical pressure sealed vessel with up to 40 samples hung up to holder;
- ❑ Filled with 67 bar industrial N₂ (99,995 %) at RT
- ❑ Heating to 515°C
- ❑ Final Pressure = 180 bar
- ❑ Sequences of variable durations

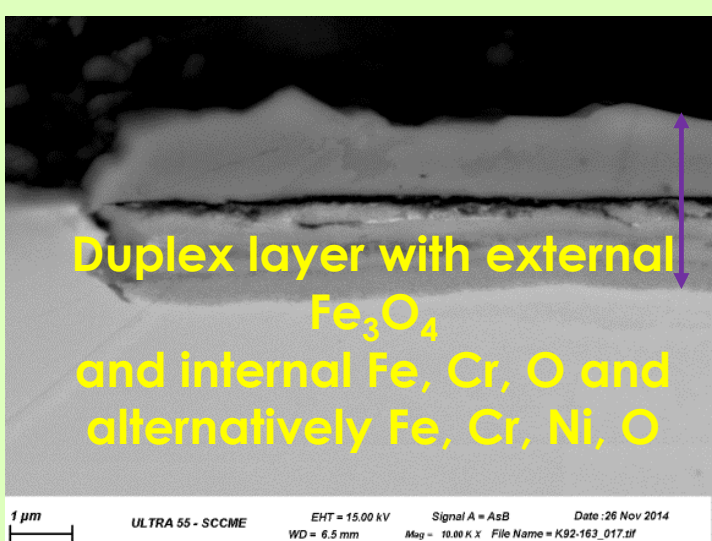
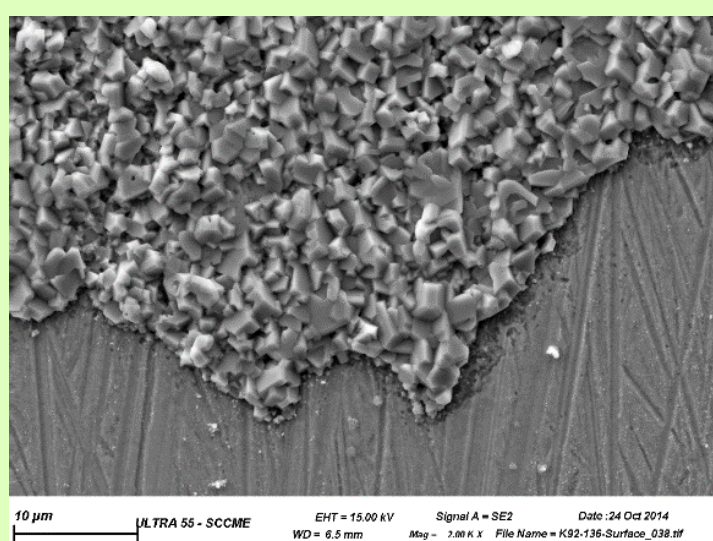
- ❑ Cumulative time up to 6000 hours
- ❑ Samples withdrawing after heating stop and gas purge;
- ❑ New samples introduced at each cycle as other samples withdrawn;
- ❑ Corrosion kinetics evaluated by weighting before and after test.

RESULTS

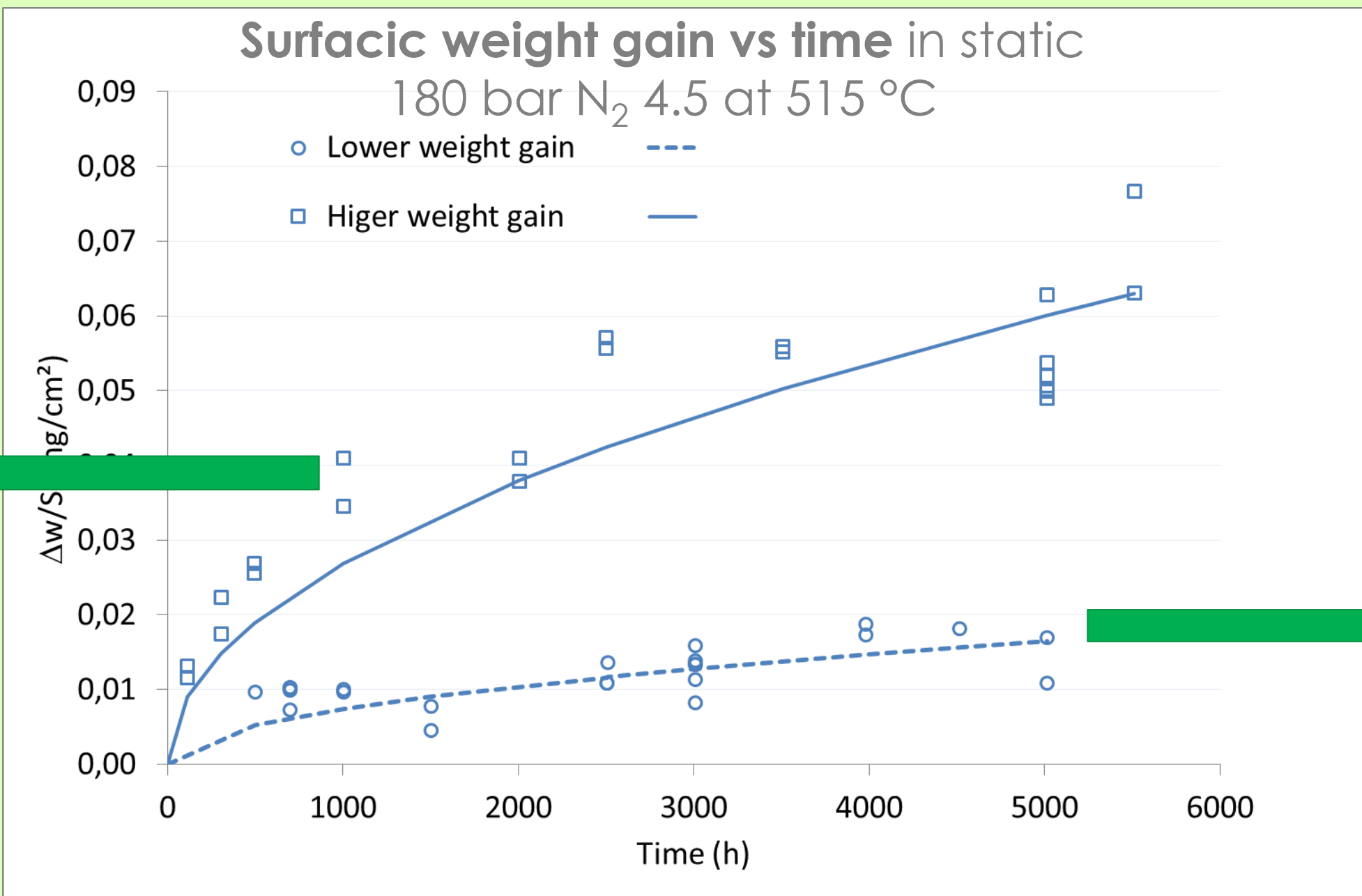
High weight gain



Appearance of samples after testing

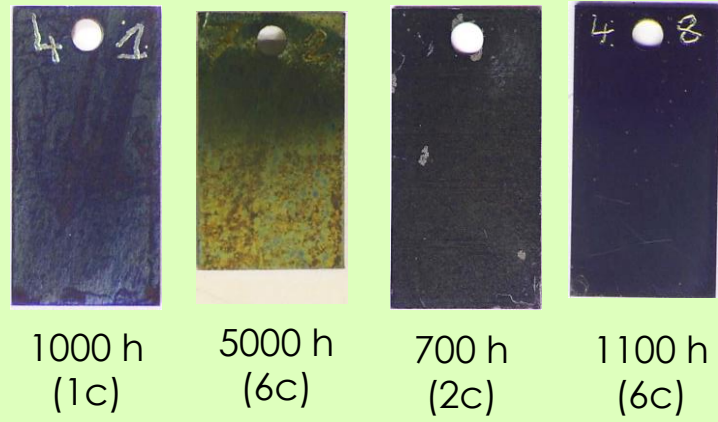


Heterogeneous spotted surface

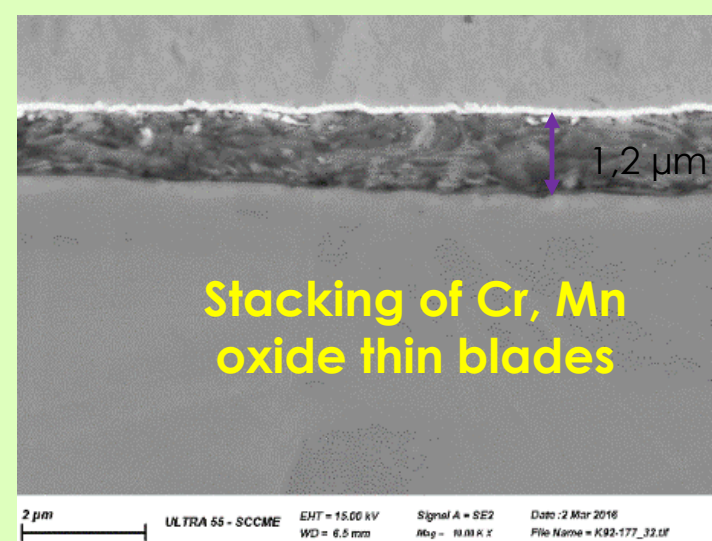
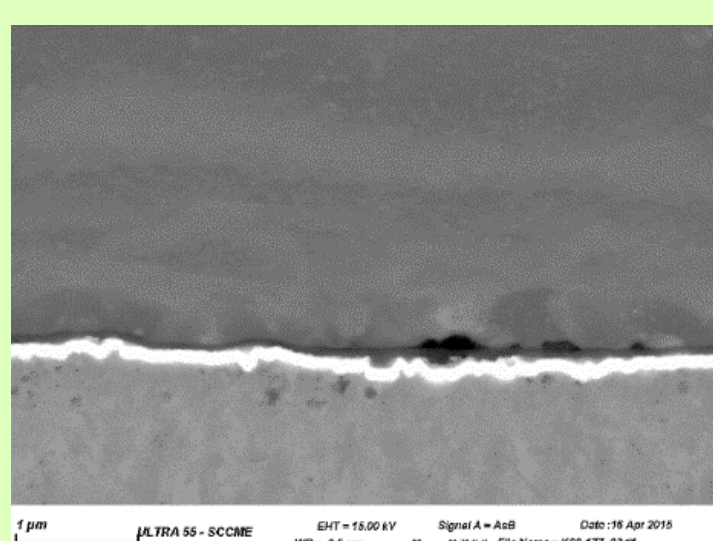


- ❑ Kinetics considered as parabolic $\frac{\Delta m}{S} = \sqrt{k_p t}$.
- ❑ 2 trends : "low weight gain" and "high weight gain"

Low weight gain



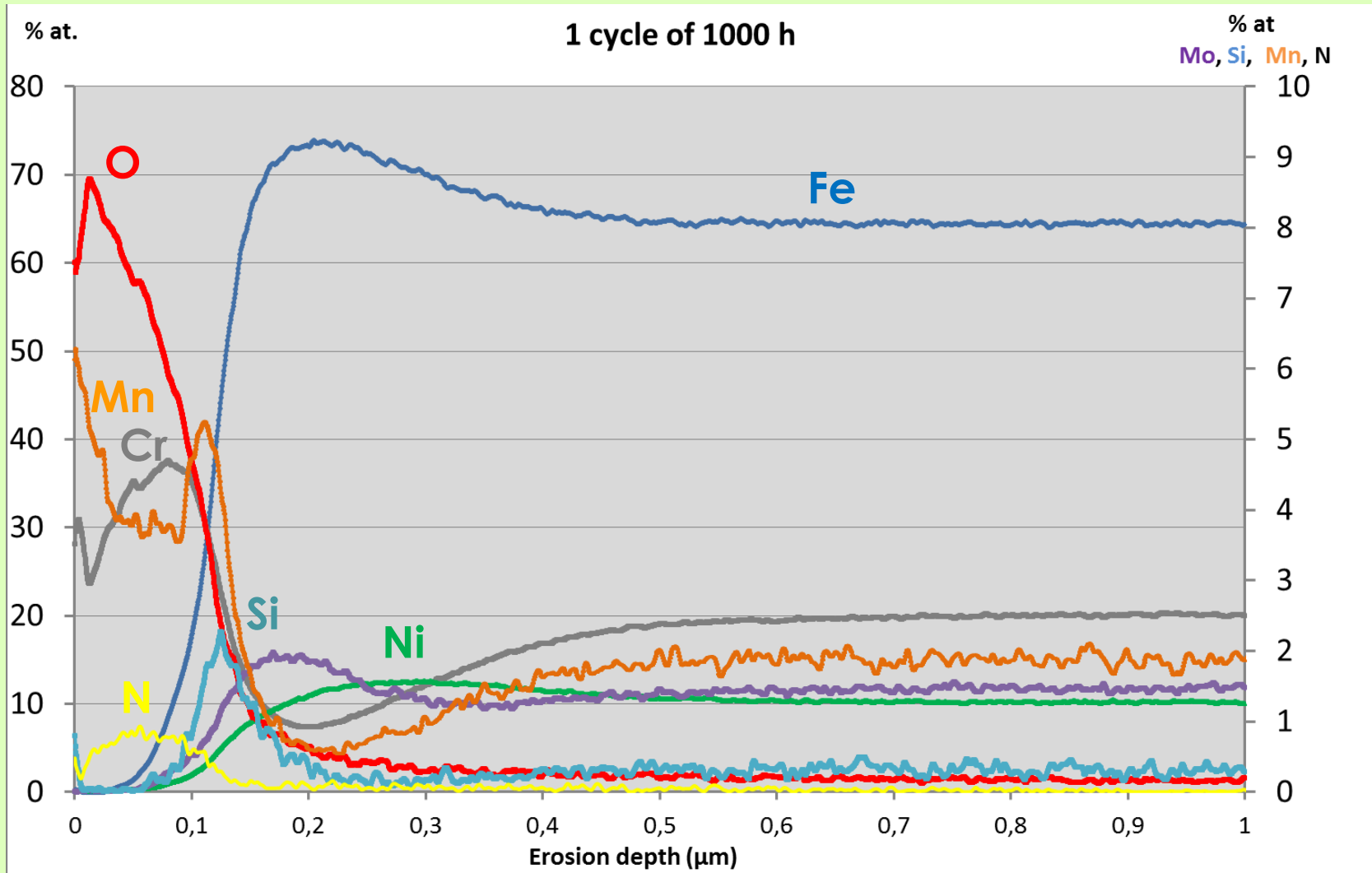
Appearance of samples after testing



Corrosion products: thin film (~100 nm) of Cr_{2-x}Mn_xO₃ and scattered duplex oxide

Corrosion products: thin and uniform film (~100 nm) of Cr_{2-x}Mn_xO₃ and thick non-compact oxide (undefined structure)

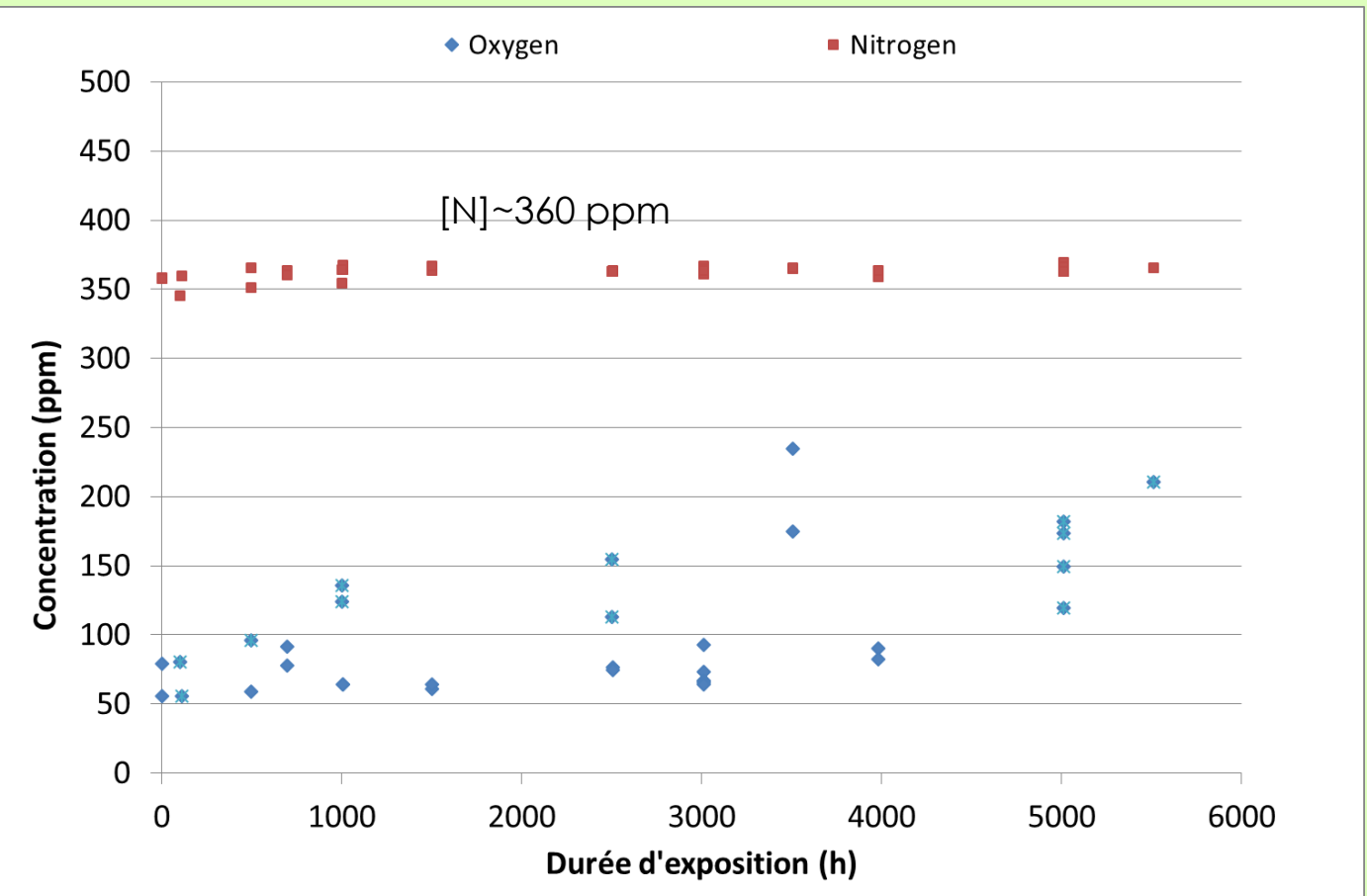
GD-OES



- ❑ Cr and Mn thin oxide (~100 nm) with N inside (few at %), **no N in the bulk material**

Inert gas fusion (bulk material)

- ❑ No growth of N global content with time;
- ❑ Well-marked increase of O global content with same trend than kinetics



Evolution of O and N content for all exposure conditions
Analysis by inert gas fusion technique

Low incidence XRD: phases identification

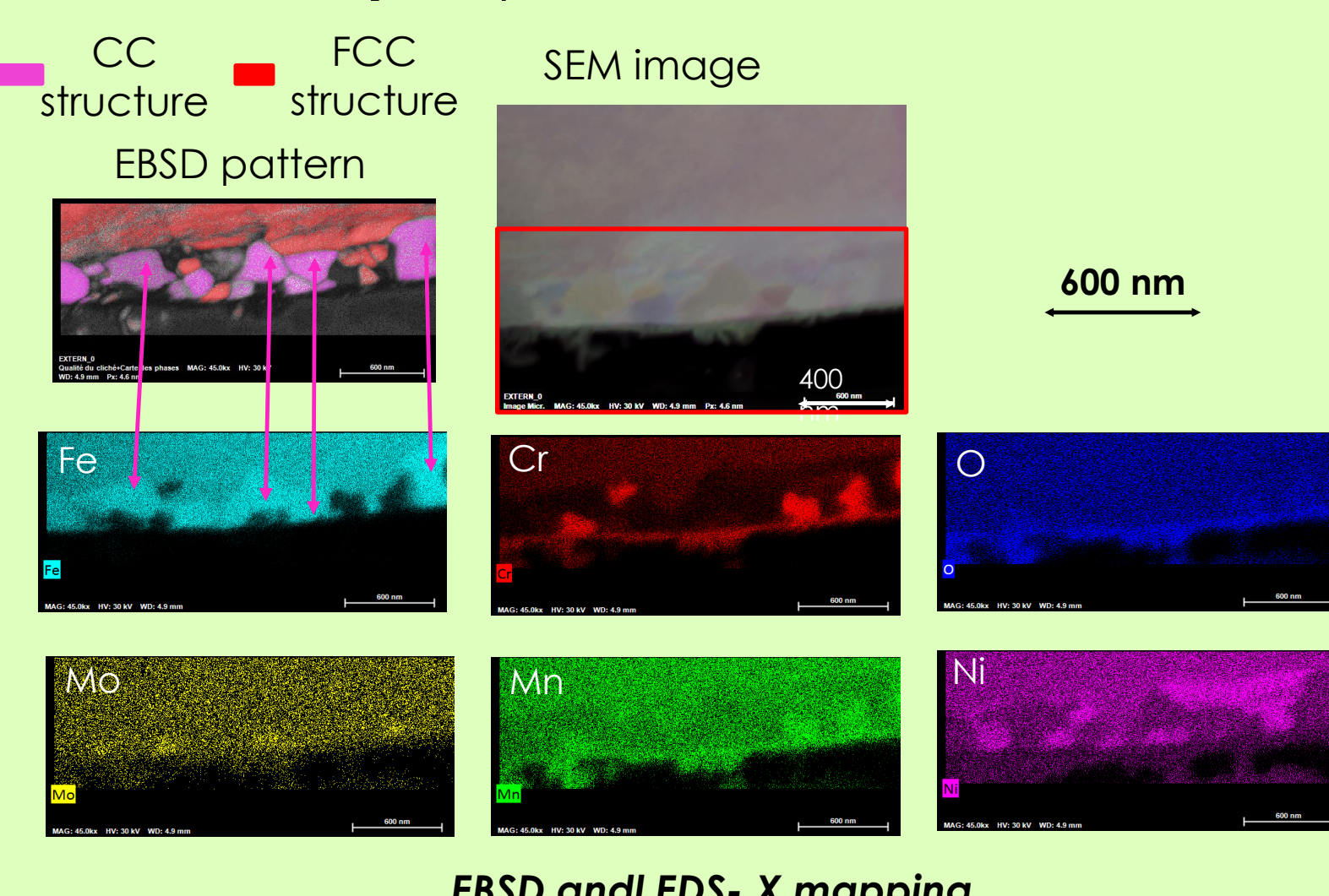


γ : austenitic matrix
α : centered cubic phase
Cor: corindon(Cr, Mn)₂O₃
Sp: spinel (CrMn)₃O₄ or Fe₃O₄

- ❑ Spinel (A₂BO₄) and corindon (M₂O₃) depth ~100 nm and increasing of cc phase in underlying material.

No nitrides

EBSD analysis: phases location



- ❑ Corroborates XRD : cc phase in underlying material and cc phase is Fe rich. **N is not detected** (< detection limit).

CONCLUSION

Nitridation risk in ECS conditions (180 bar N₂, 515 °C) can be **eliminated**.
Corrosion phenomena is due to **oxidation** caused by O₂ impurities contained in N₂.
2 kinetics corresponding to 2 morphologies : high weight gains sign the growth of scattered duplex oxides. Chemical cycling has an effect on oxide morphology.
History of samples with low and high weight gains differ : "high weight gain" underwent succession of short cycles (100 h) while "low" underwent longer cycles (mini 500 h).
In static conditions gas content is gradually depleted and conditions are **non-stationary**.

PERSPECTIVES

What are the conditions that facilitate duplex oxide formation and when does it form?
How do they evolve with O₂ depletion?

Weight gain is due to oxygen supply