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

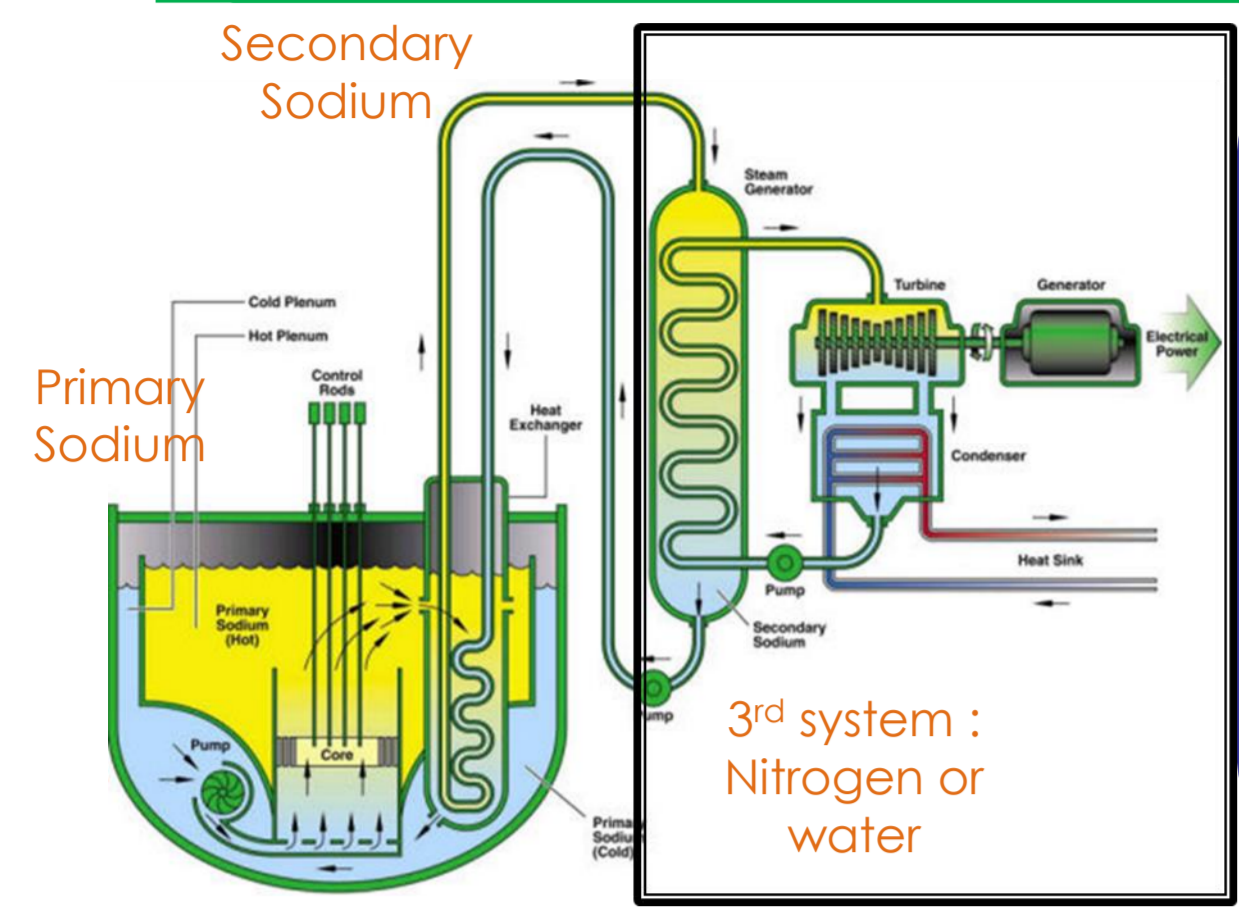
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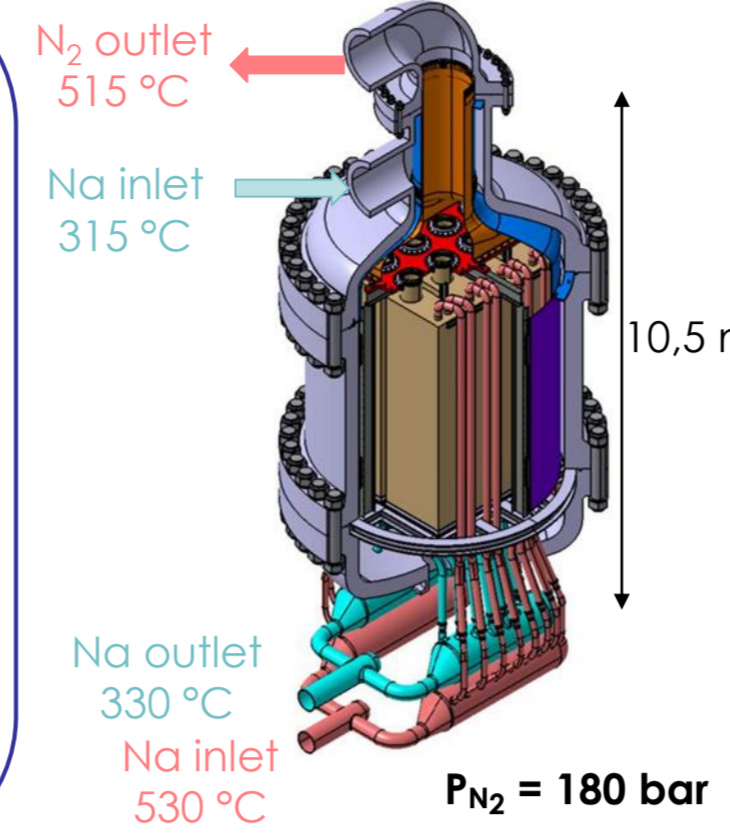
## CONTEXT: Generation IV for nuclear power plants: Energy Conversion System (ECS) for ASTRID project



**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**

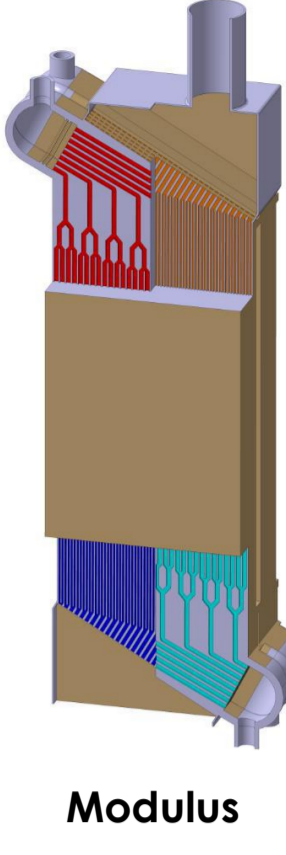
**Energy Conversion System with Gas is preferred to ECS with Water**  
**→ Sodium-Water Reaction eliminated**



**Sodium-Gas Energy System**

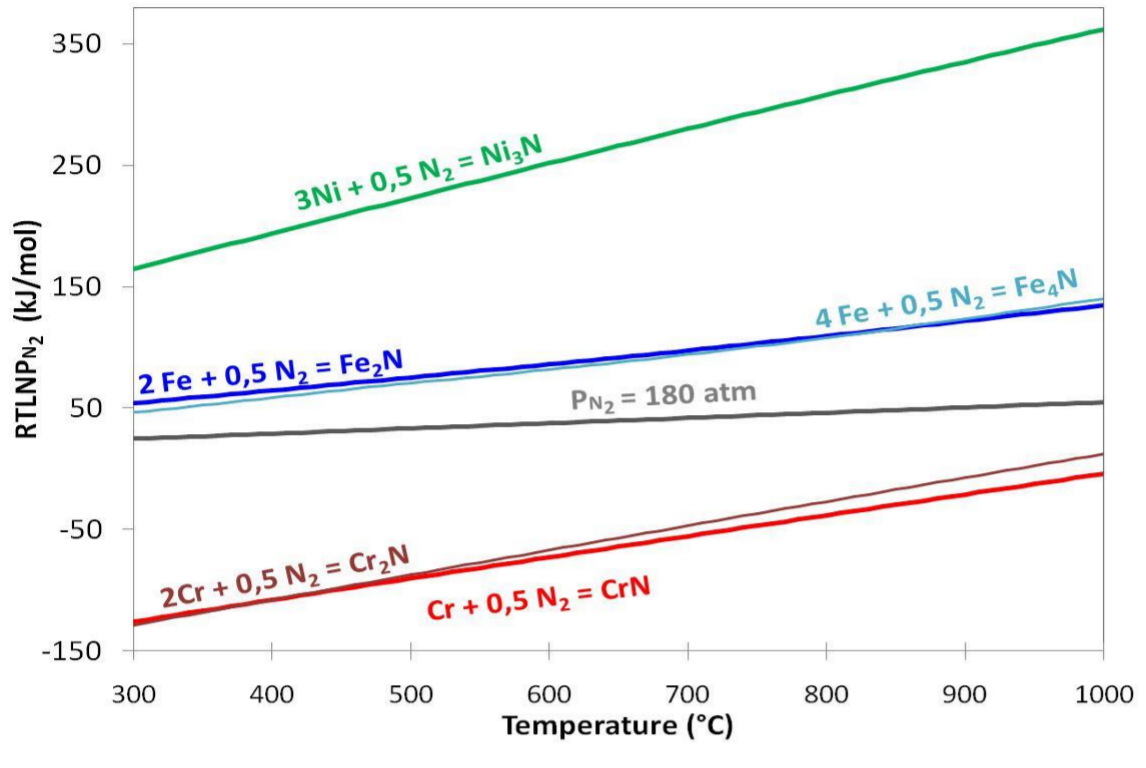
- 8 modulus in under pressure vessel with hot gas collector pipe
- Based on the principle of plate heat exchanger: Na and N<sub>2</sub> flow in counter-current
- Specific design with 2 channel networks : heat transferred through 1 mm thick wall

**Does nitridation occur and what would the involved depth ?**



## OBJECTIVES

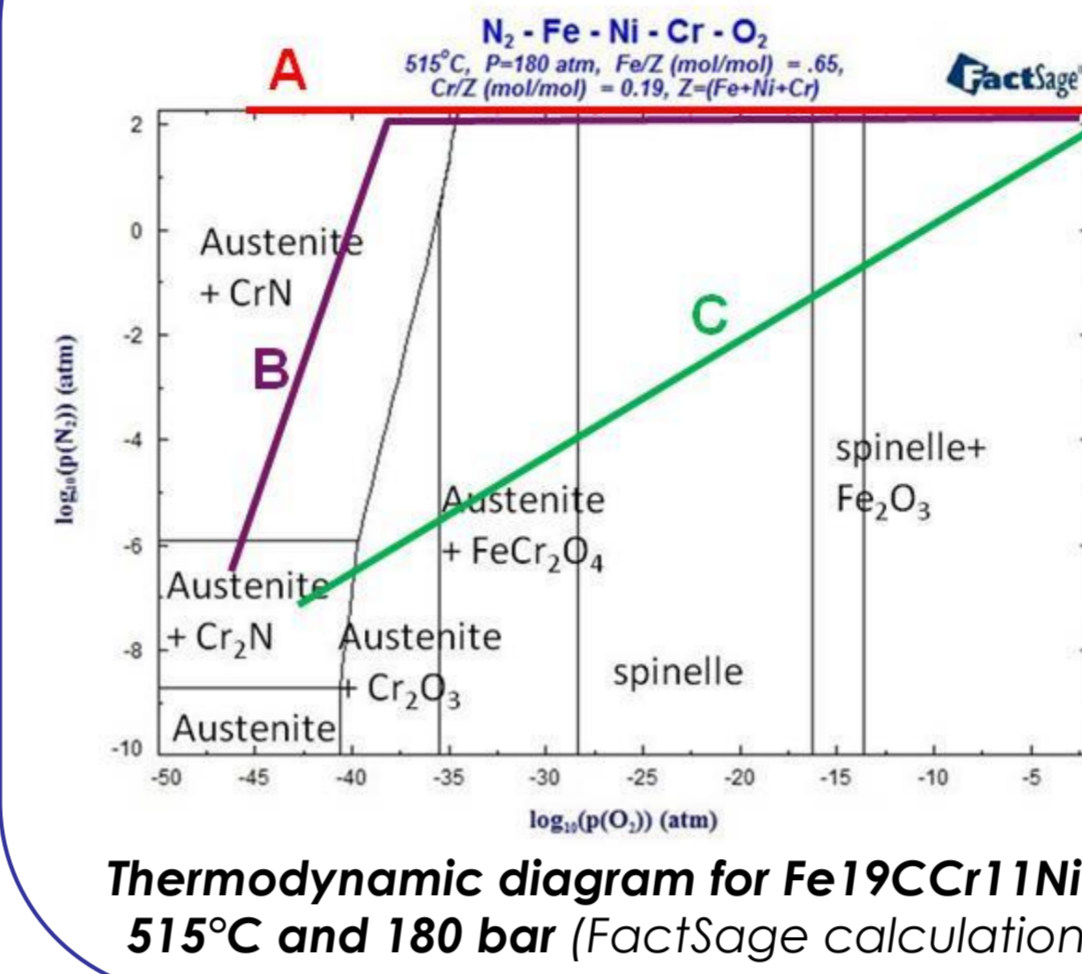
Performance of the ECS has to be guaranteed for **30 years** → Compatibility of the 316L with N<sub>2</sub> environment at 515°C and 180 bar has to be checked.



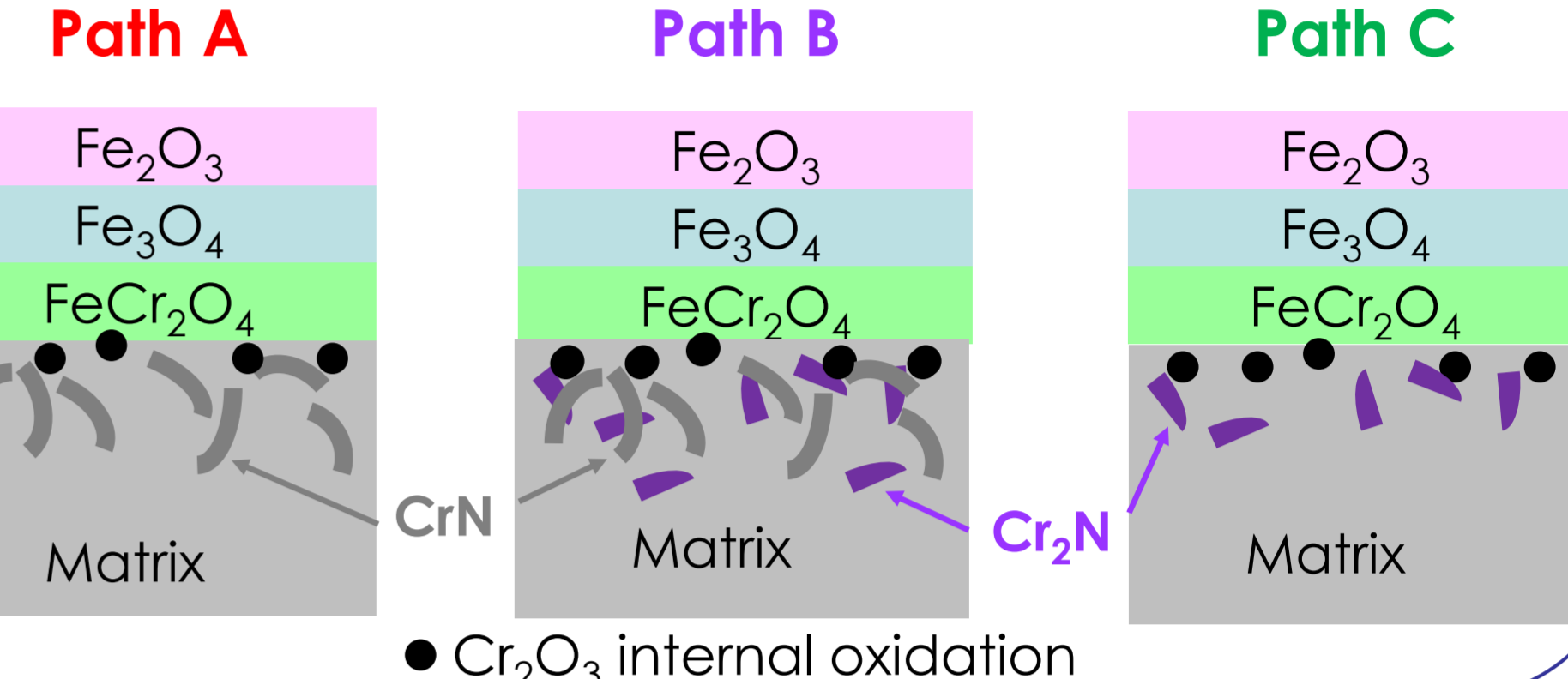
- Few relevant data within the ECS conditions
  - Thermodynamic forecasting : CrN and Cr<sub>2</sub>N
  - Embrittlement by increasing hardness
  - Nitrides growth controlled by diffusion
  - But large scattering of diffusion coefficients in literature
- Nitridation thickness (after 30 years) : up to 4 to 10 mm!

**Need of refining data**

**Oxidation susceptibility:** competition between oxidation and nitridation

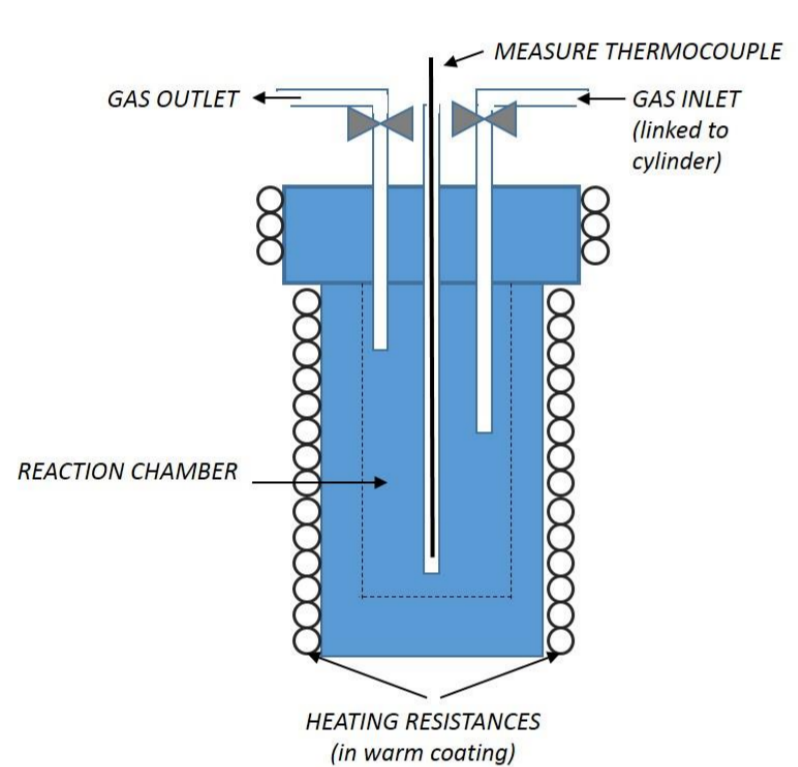


**3 scenarios suggested by thermodynamic**



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



- Cylindrical pressure sealed vessel with up to 40 samples hung up to holder;
- Filled with 67 bar industrial N<sub>2</sub> (99,995 %) at RT
- Heating to 515°C
- Final Pressure = 180 bar
- Sequences of variable durations

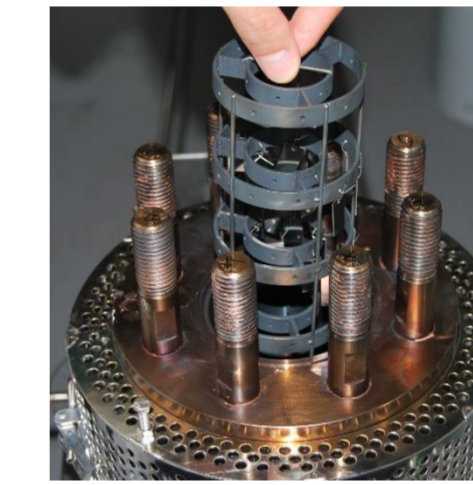
### MATERIAL

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

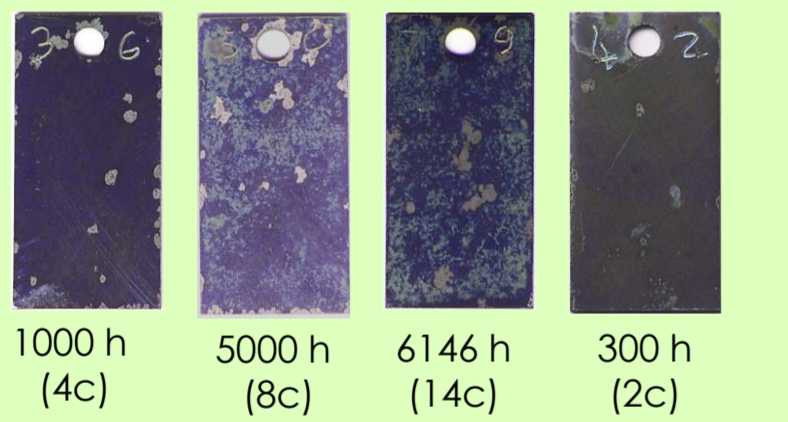
Surface sample polished (SiC grade 1200)



- 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 weighing before and after test.

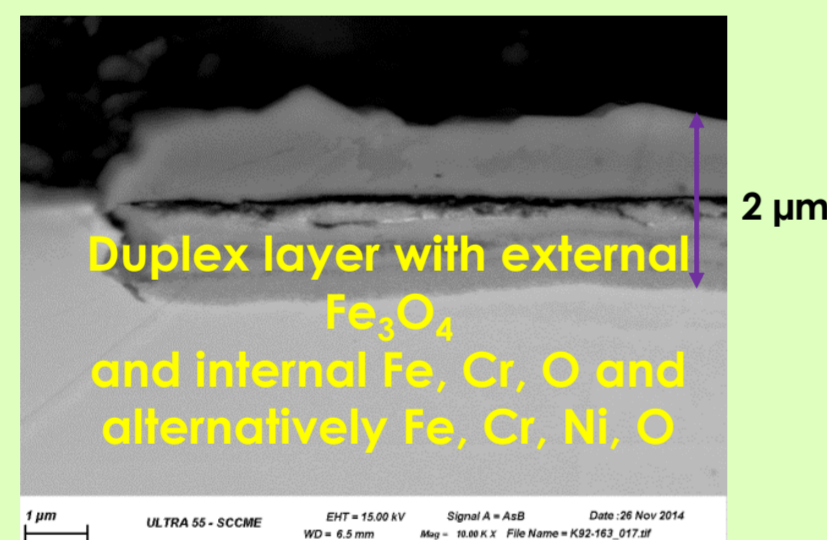
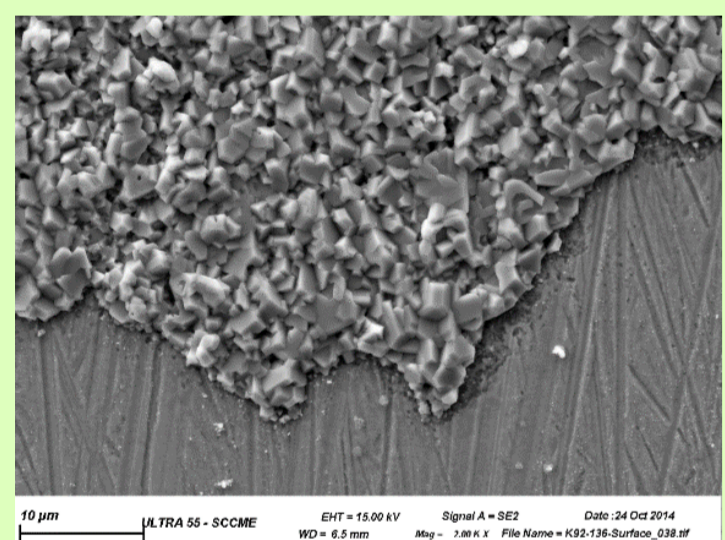
## RESULTS

### High weight gain

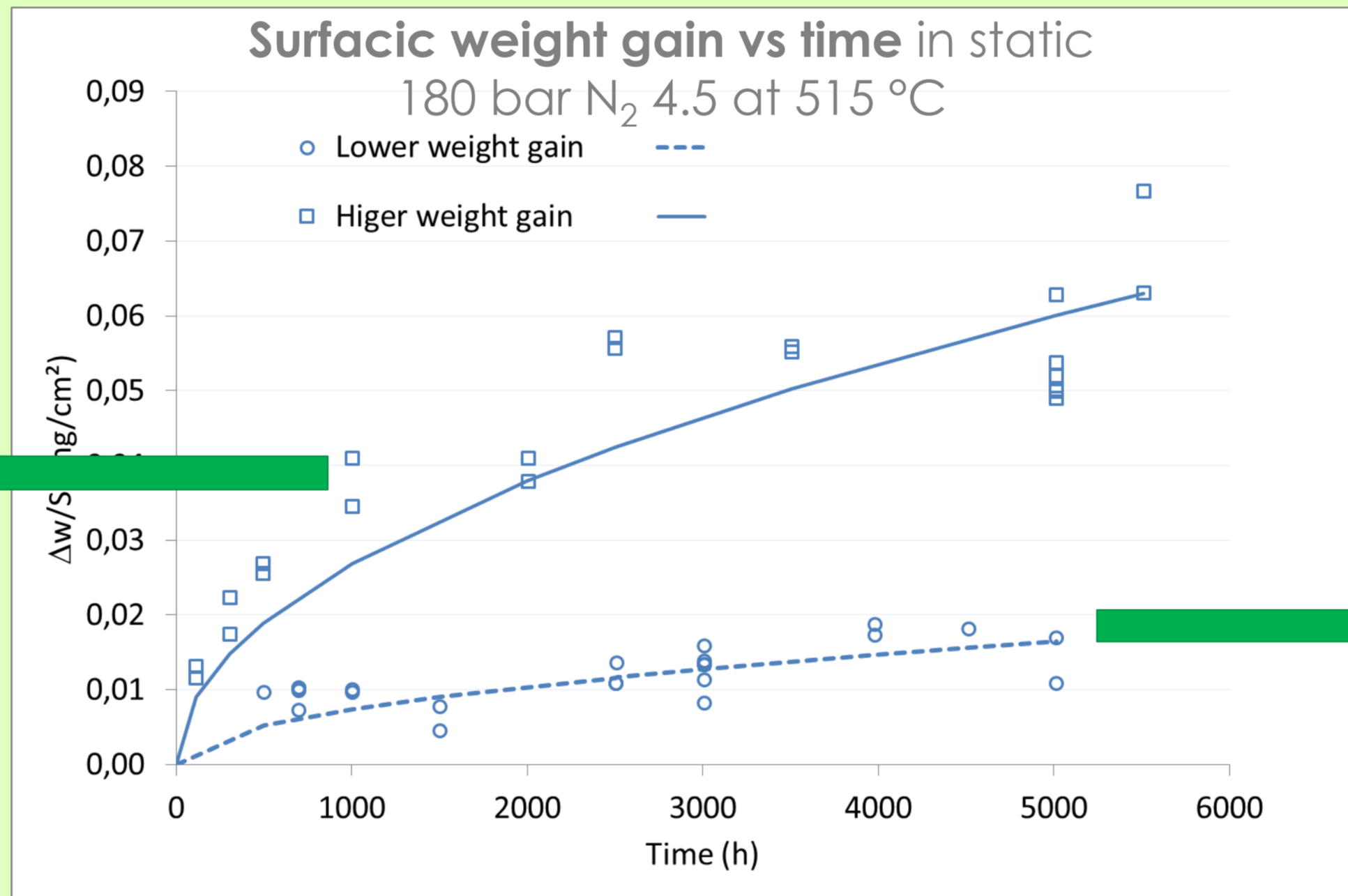


Heterogeneous spotted surface

Appearance of samples after testing

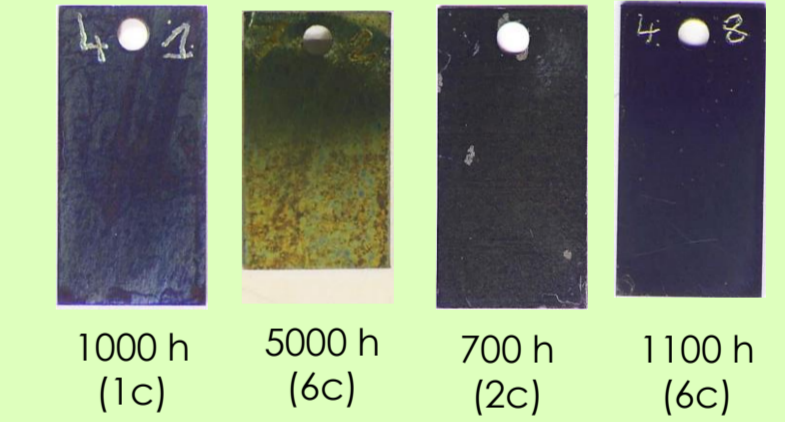


Corrosion products: thin film (~100 nm) of Cr<sub>2-x</sub>Mn<sub>x</sub>O<sub>3</sub> and scattered duplex oxide



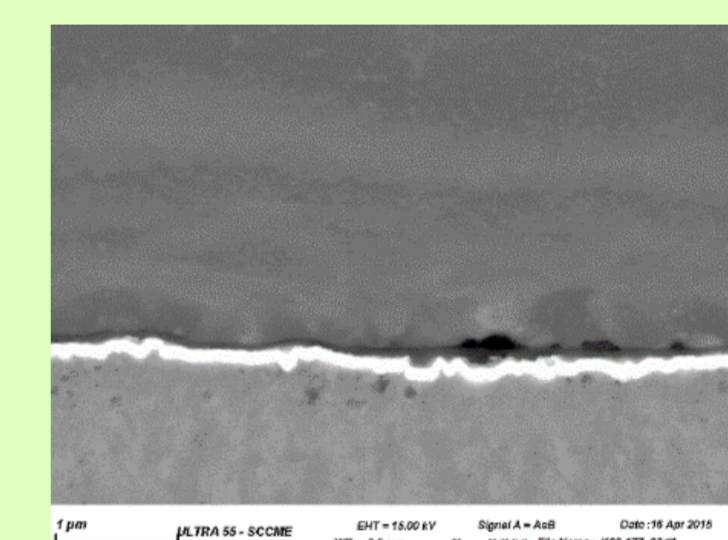
- Kinetics considered as parabolic  $\frac{\Delta m}{S} = \sqrt{k_p t}$
- 2 trends : "low weight gain" and "high weight gain"

### Low weight gain



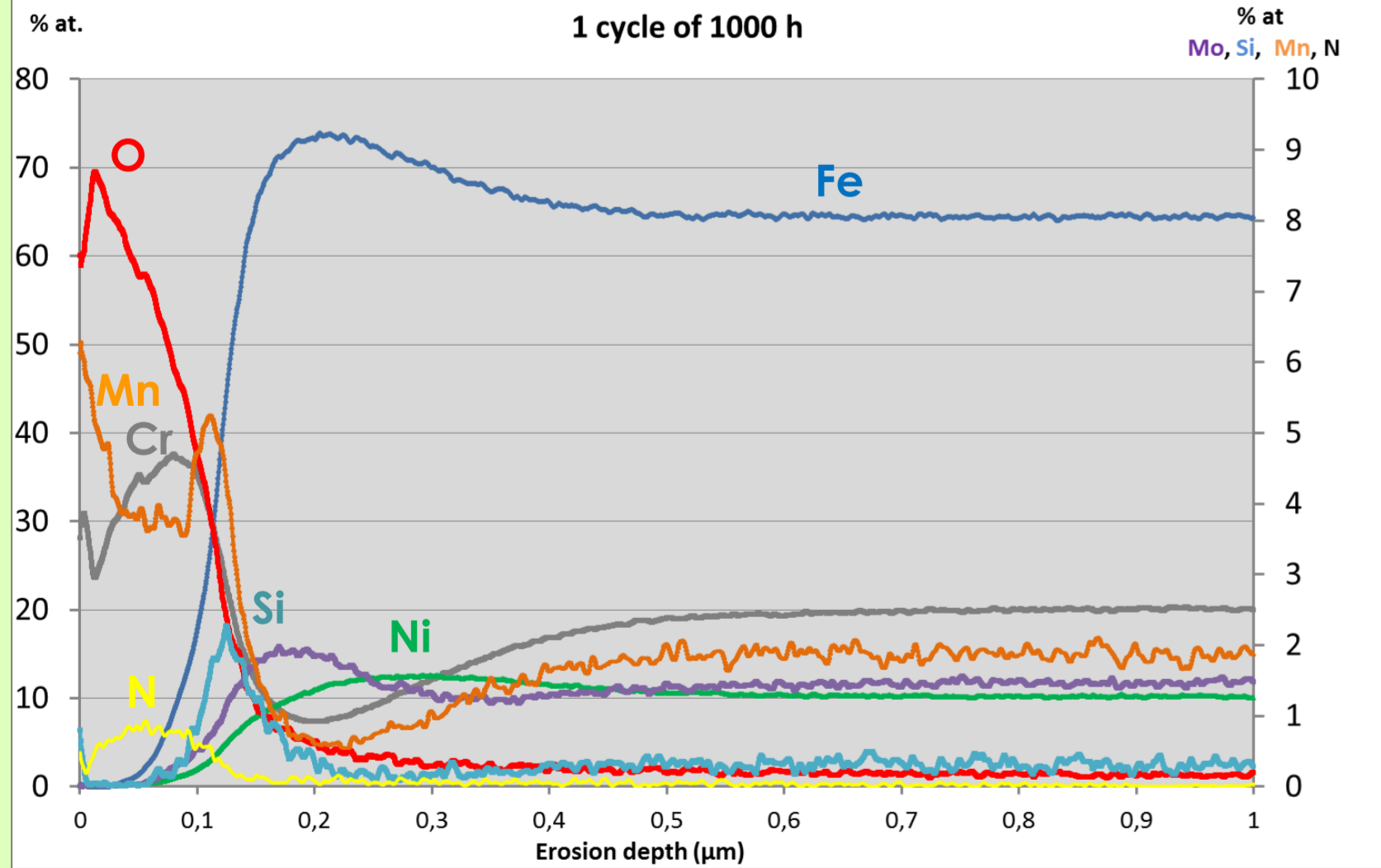
Even surfaces

Appearance of samples after testing



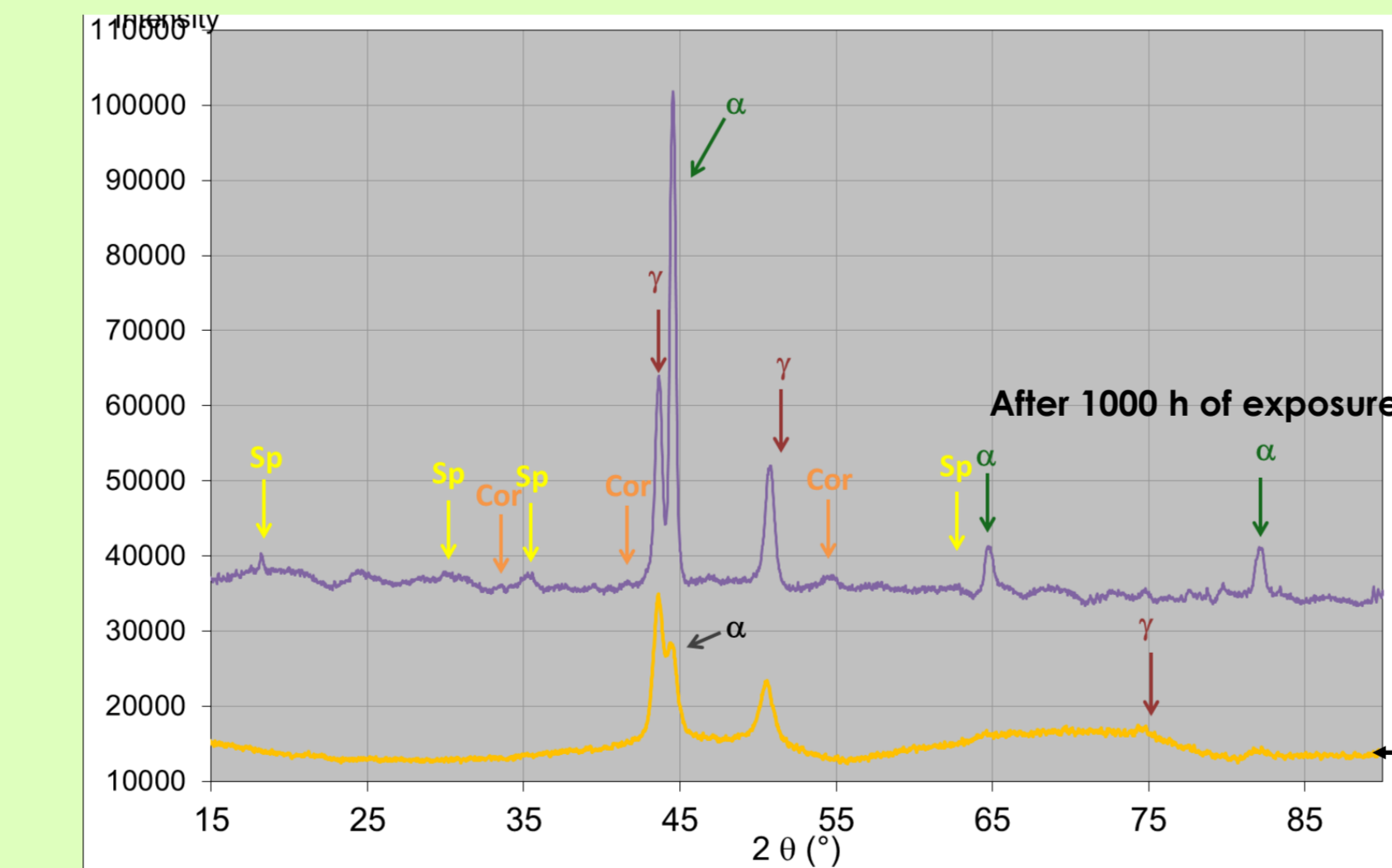
Corrosion products: thin and uniform film (~100 nm) of Cr<sub>2-x</sub>Mn<sub>x</sub>O<sub>3</sub> and thick non-compact oxide (undefined structure)

### GD-OES



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

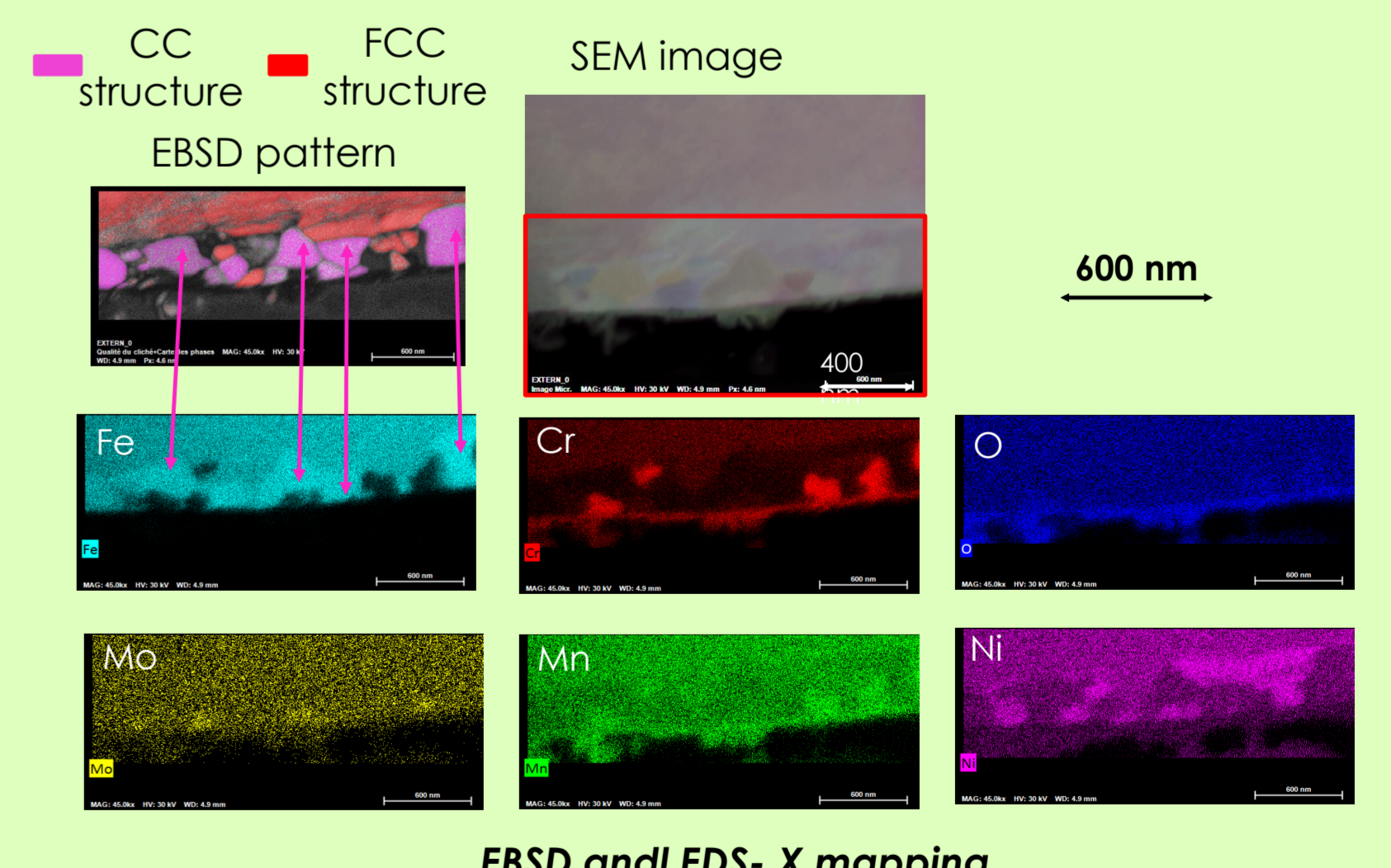
### Low incidence XRD: phases identification



- Spinel (A<sub>2</sub>BO<sub>4</sub>) and corindon (M<sub>2</sub>O<sub>3</sub>) depth ~100 nm and increasing of cc phase in underlying material.
- No nitrides**

- γ : austenitic matrix
- α : centered cubic phase
- Cor: corindon (Cr, Mn)<sub>2</sub>O<sub>3</sub>
- Sp: spinel (CrMn)<sub>3</sub>O<sub>4</sub> or Fe<sub>3</sub>O<sub>4</sub>

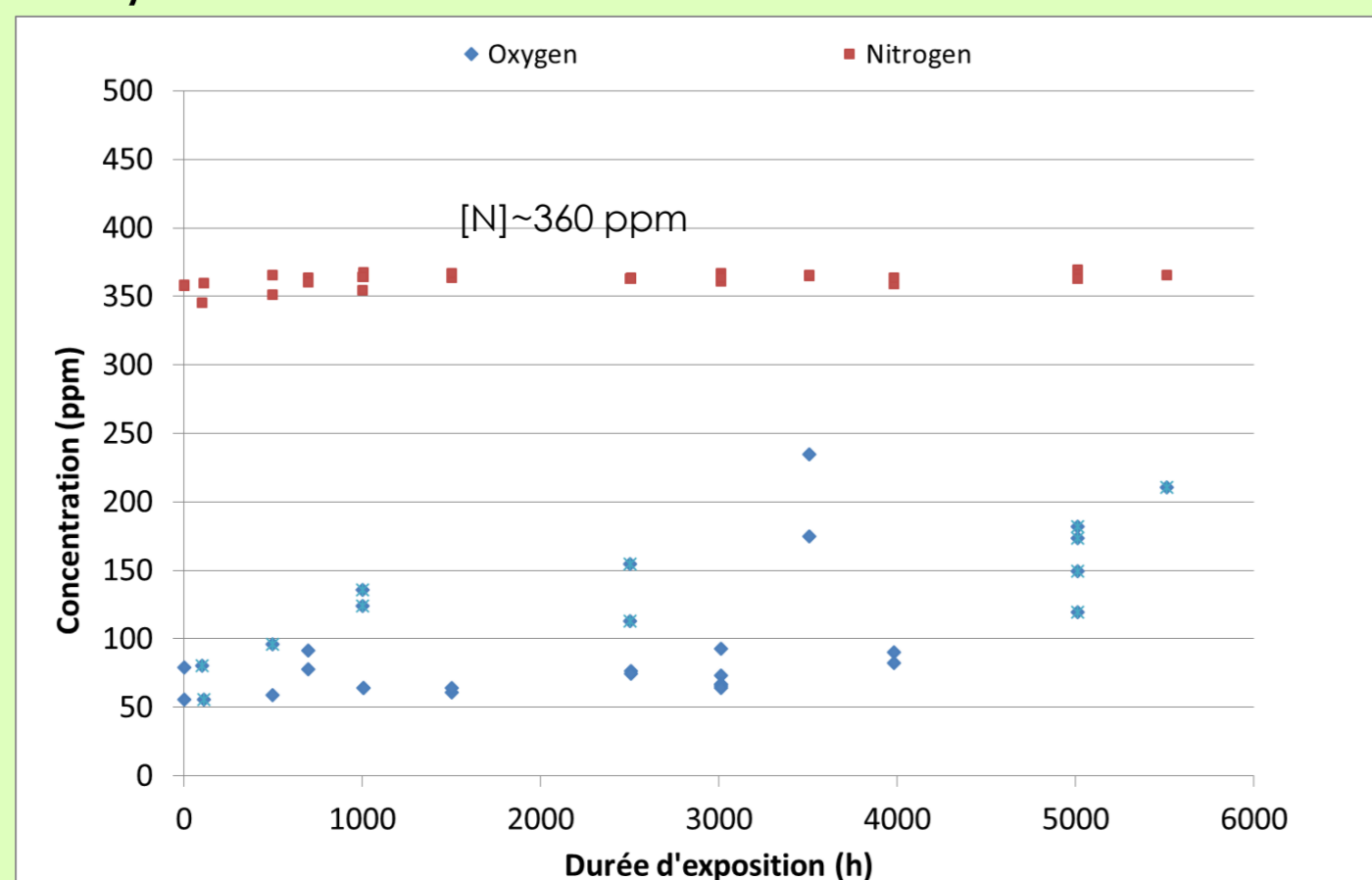
### EBSD analysis: phases location



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

### 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



**Weight gain is due to oxygen supply**

## CONCLUSION

**Nitridation risk in ECS conditions (180 bar N<sub>2</sub>, 515 °C) can be eliminated.**  
**Corrosion** phenomena is due to **oxidation** caused by O<sub>2</sub> impurities contained in N<sub>2</sub>.  
 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 O<sub>2</sub> depletion?