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(EDF-AREVA-CEA-CNRS)

EVALUATION OF ALUMINIDE COATINGS AS HARDFACING MATERIAL CANDIDATE IN SODIUM FAST REACTOR

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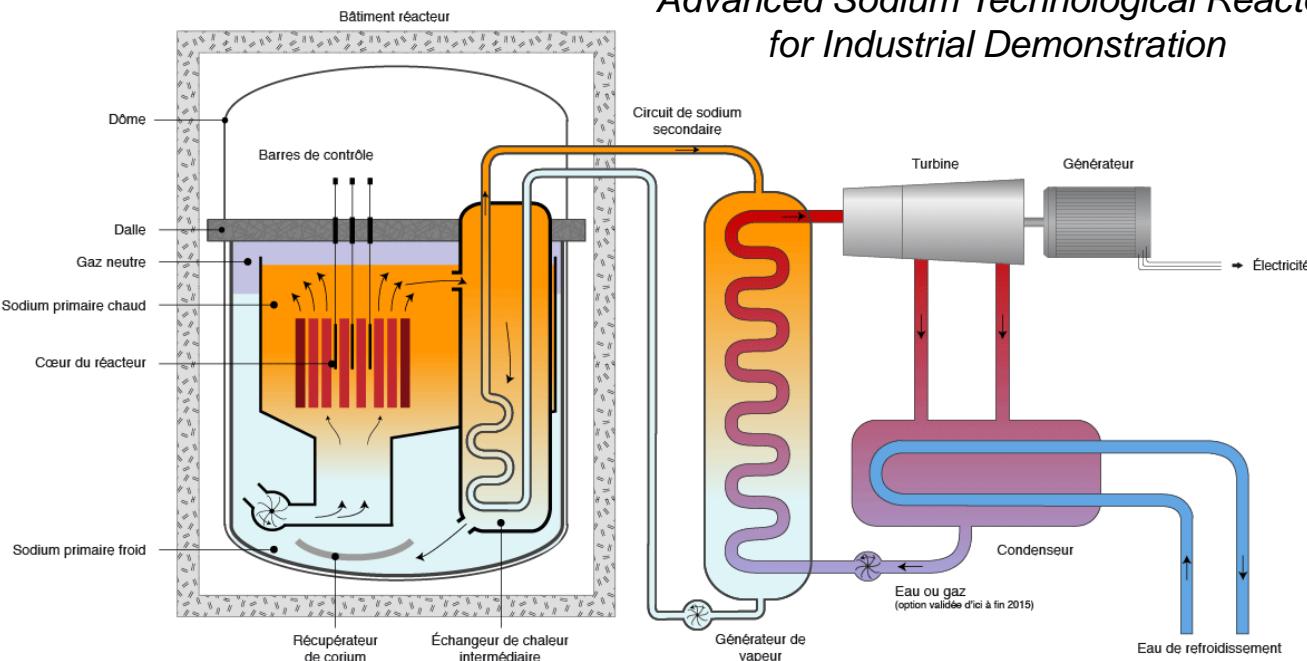
2- Jean Lamour Institute, University of Lorraine, Nancy, France

3- LASIE - University of La Rochelle, France

4- UMET - University of Lille, France

CONTEXT : NEW HARDFACING ALLOYS FOR TRIBOLOGICAL APPLICATIONS OF SFR

Sodium-cooled Fast Reactor ASTRID (600 MWe)



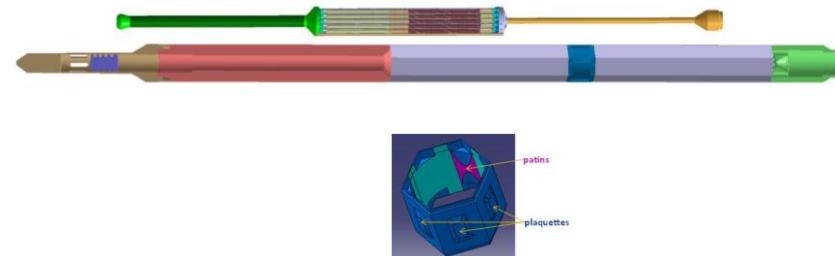
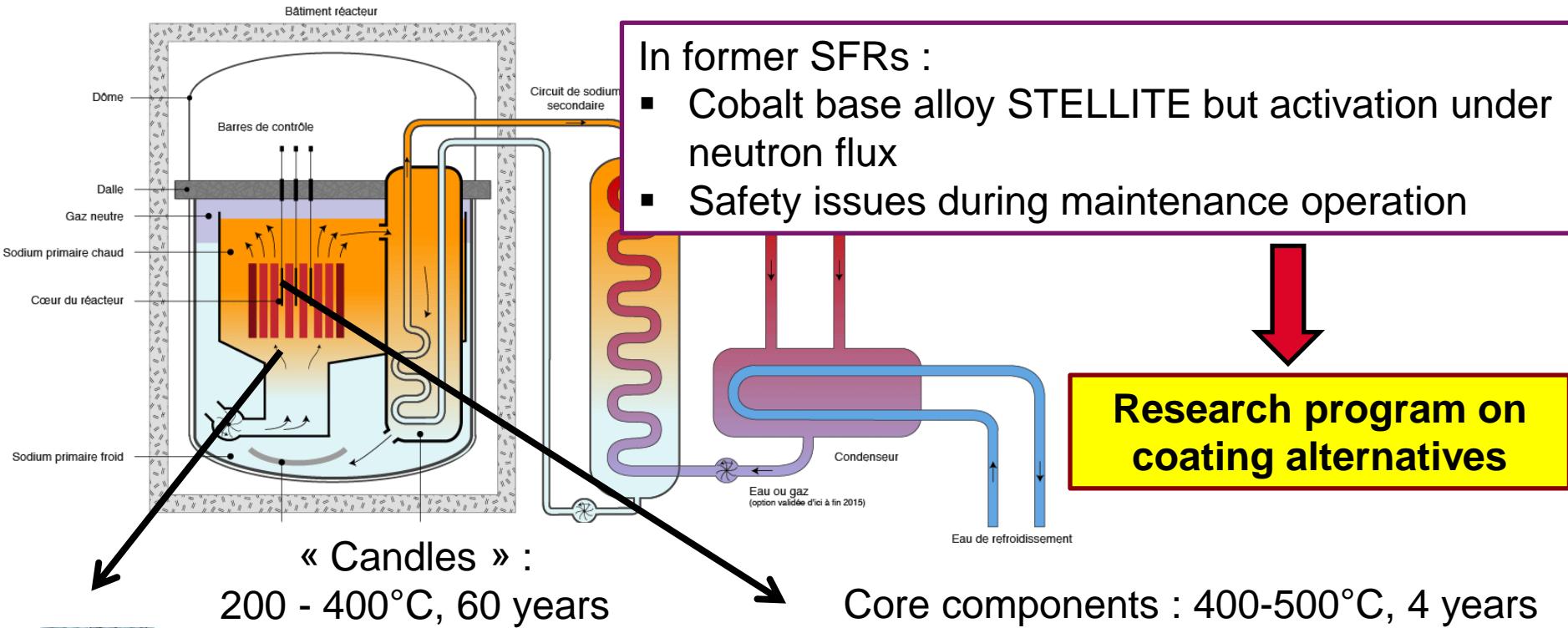
- Main goals of ASTRID:
- Multi-recycling of plutonium (uranium ressource preservation)
 - Minor actinide transmutation (nuclear waste reduction)
 - Enhanced safety (as for Gen III reactors)

Many components need to move during :

- nominal operation
- or during maintenance operation

CONTEXT : NEW HARDFACING ALLOYS FOR TRIBOLOGICAL APPLICATIONS OF SFR

Sodium-cooled Fast Reactor ASTRID (600 MWe)



EXPECTED PROPERTIES OF TRIBOLOGICAL MATERIAL

- Good friction coefficient
- Good wear resistance
- Good corrosion resistance in Na
- Good behaviour under irradiation
- Good mechanical behaviour : resistance to thermal and mechanical shocks
- Good adhesion

Fabrication process = robust and vendor = existing or expected in near future

CEA-AREVA-EDF program :

- Thick coatings (2 mm) : Nickel base alloys deposited by PTA or laser
- Thin coatings (<100 µm) : Aluminides**



- Good compatibility in Na from results obtained during the « golden age » of SFRs in the 70's and 80's.
- Good tribological properties : good friction coefficient associated to the formation of stable surface NaAlO_2 or $\text{Na}_2\text{Al}_2\text{O}_4$ in Na

JOINT WORK IN COLLABORATION BETWEEN 4 LABS



Aluminides coatings synthesis by *pack-cementation* (IJL) and *slurry* (University of La Rochelle)

- Na compatibility
- Tribological properties in Na
- Behaviour under irradiation (experiments conducted in Russian BOR60 reactor)

Mechanical testing on coated sample :
« Small Punch Test » in Na

JOINT WORK IN COLLABORATION BETWEEN 4 LABS



1

Aluminides coatings synthesis by *pack-cementation* (IJL) and *slurry* (University of La Rochelle)

2

- Na compatibility
- Tribological properties in Na
- Behaviour under irradiation (experiments conducted in Russian BOR60 reactor)

3



Mechanical testing on coated sample :
« Small Punch Test » in Na

1) ALUMINIDE COATINGS SYNTHESIS

STEEL GRADES AND COATING

%weight	Fe	Cr	Ni	C	Mo	N	Aluminide coating process
316L(N)	Bal	17	13.2	0.04	2.3	0.06	Pack cementation
T91	Bal	9	0.2	0.1	1	/	Pack cementation + slurry

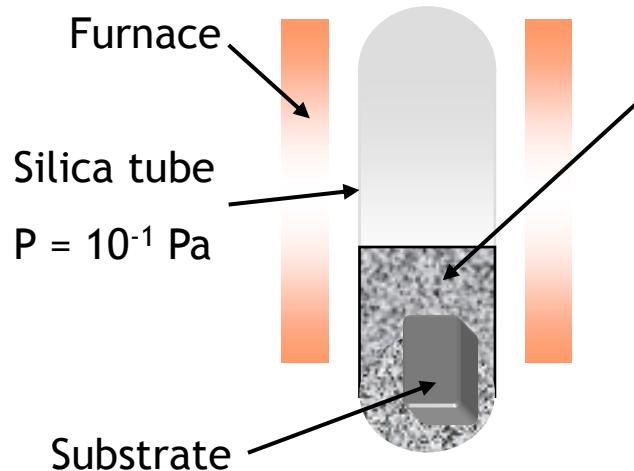
Both coating processes were realized without or with pre Ni plating treatment



Fe rich aluminides

Ni rich aluminides

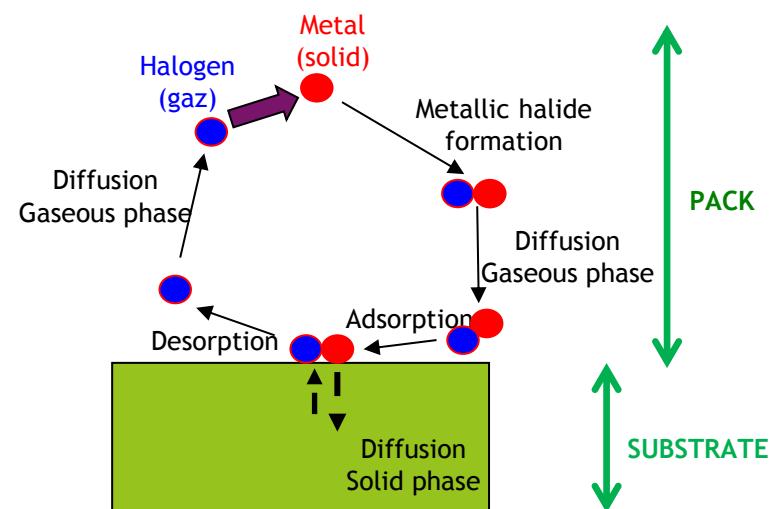
PACK-CEMENTATION – LOW AL ACTIVITY (PC)



“pack” composition :

- halide activator - CrCl_3 - 1 wt%
- inert powder (Al_2O_3) - 59 wt%
- Master alloy (Ni_2Al_3) - 40 wt%

980°C – 4h in air
(muffle furnace)

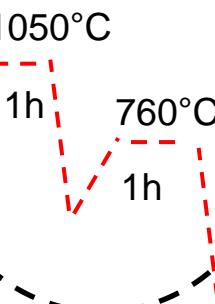
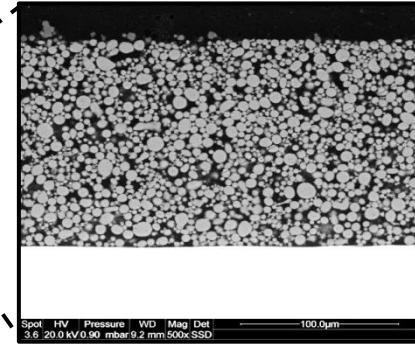


When pre nickel plated : 18 µm then vacuum annealed at 400°C before PC

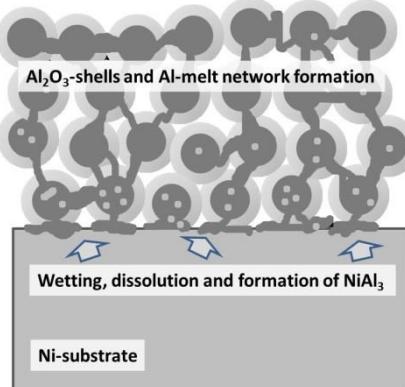
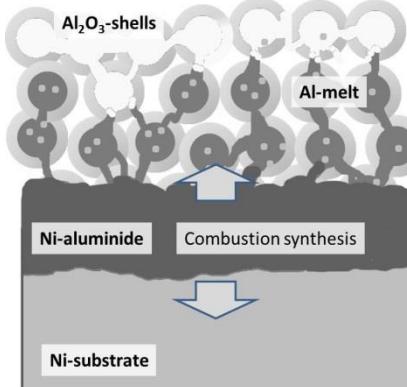
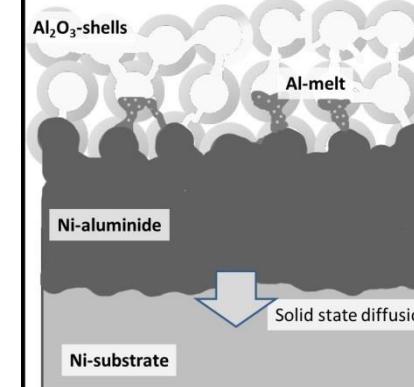
SLURRY – HIGH AL ACTIVITY (SL)



Thermal treatment



Final annealing step to recover
the microstructure and the
mechanical properties of T91
substrate

Wetting**Combustion synthesis****interdiffusion**

SLURRY – HIGH AL ACTIVITY (SL)

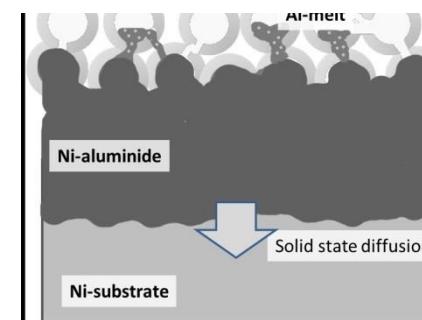
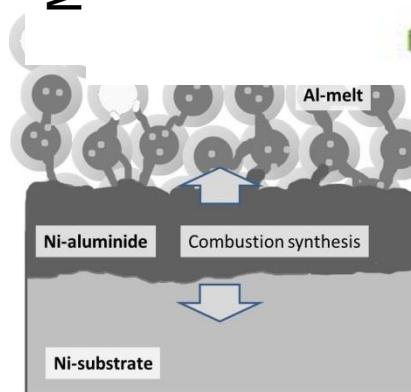
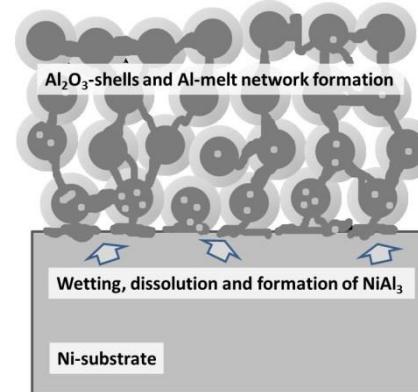
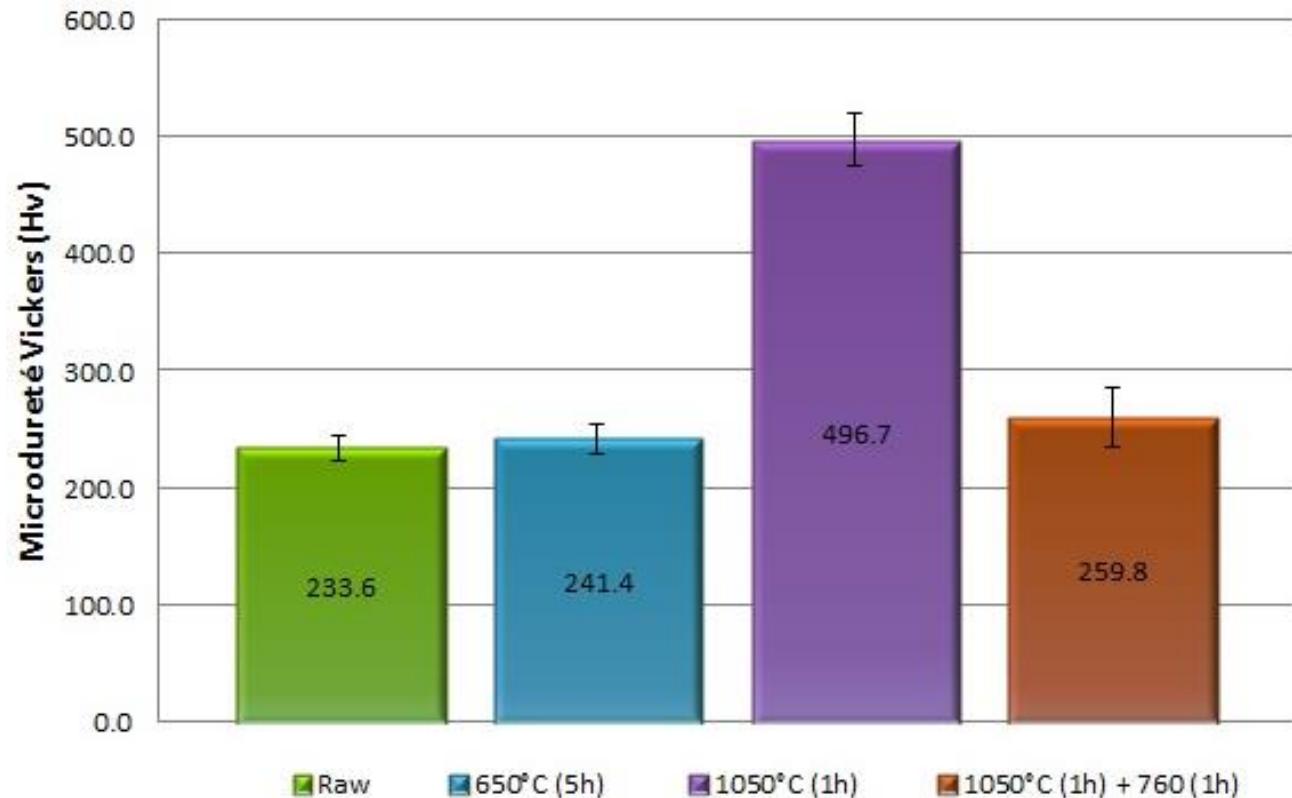


Thermal treatr

400°C
3h

Wetting

> C



Ni & Fe substrates

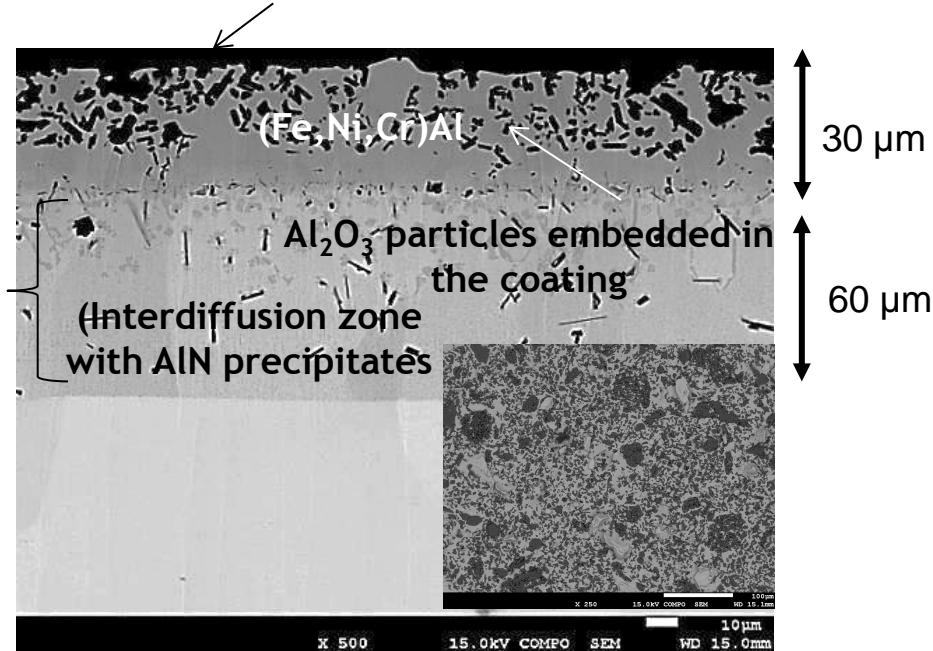


Last step : grit-blasting of the top coat to remove the residue (bisque)

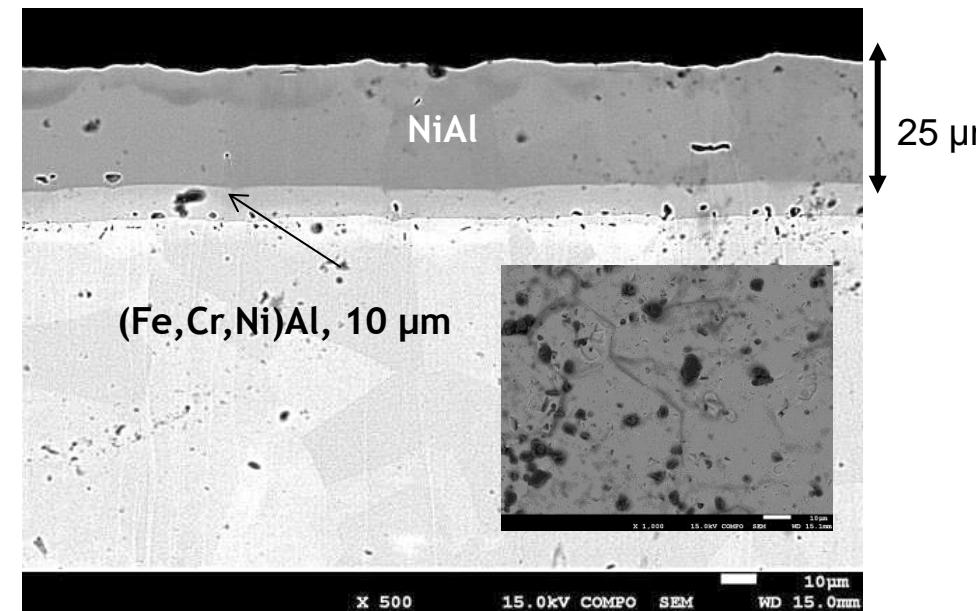
COATING OBTAINED BY PC ON 316L(N)

Without pre Ni plating

Al_2O_3 and Ni_2Al_3 particles stuck on the surface



With pre Ni plating



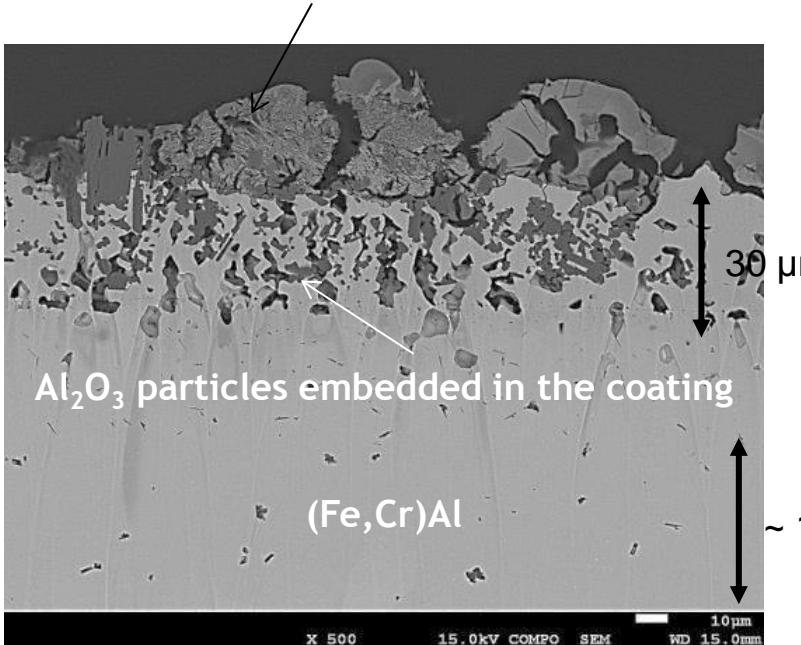
- Composite coating : B2-(Fe, Ni, Cr) Al with « hard » embedded Al_2O_3 particles
- Outwards aluminide growth

- NiAl
- Less particles stuck on the surface
- Lower outwards aluminide growth rate
- Thin interdiffusion zone

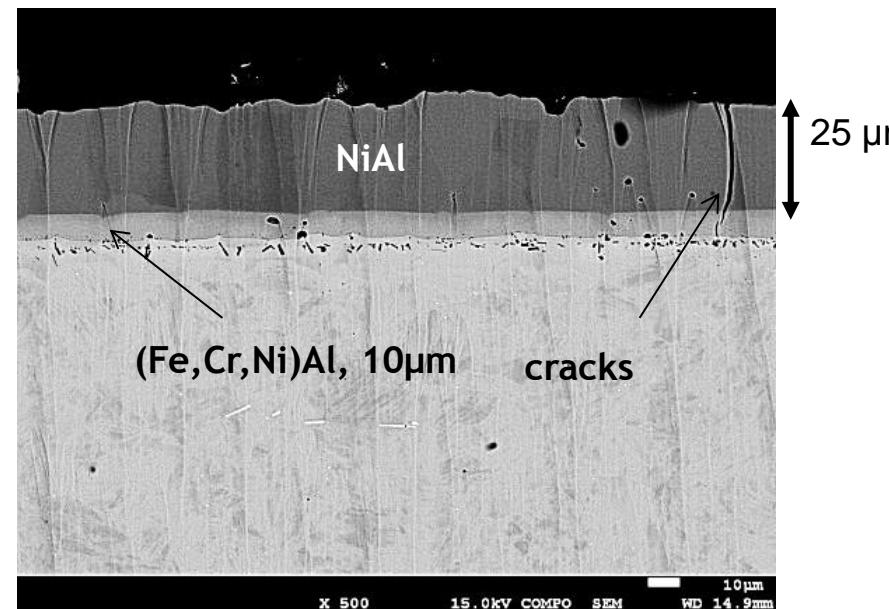
COATING OBTAINED BY PC ON T91

Without pre Ni plating

Al_2O_3 and Ni_3Al particles stuck on the surface



With pre Ni plating

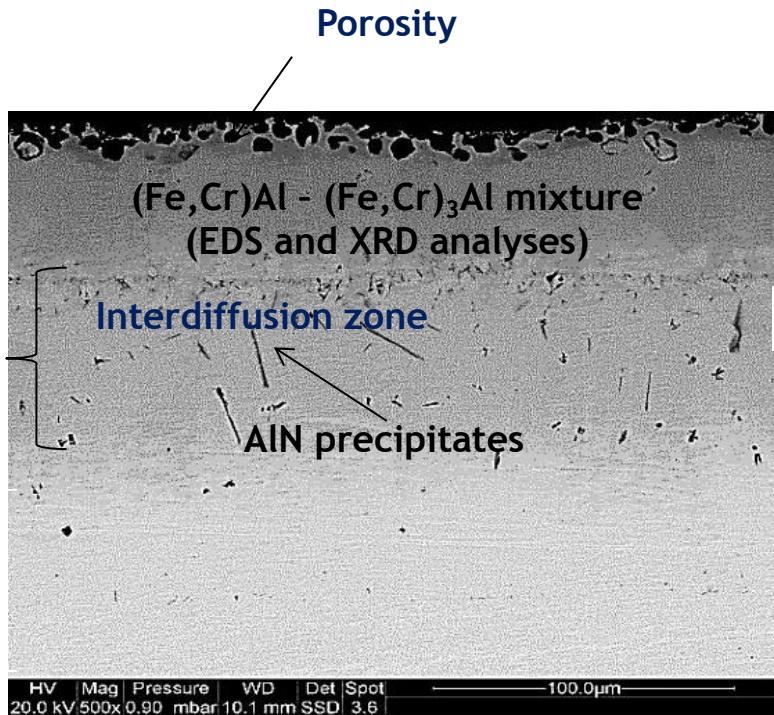


- **Composite coating : B2-(Fe, Cr) Al with « hard » embedded Al₂O₃ particles**
 - Outwards aluminide growth
 - Very large interdiffusion zone

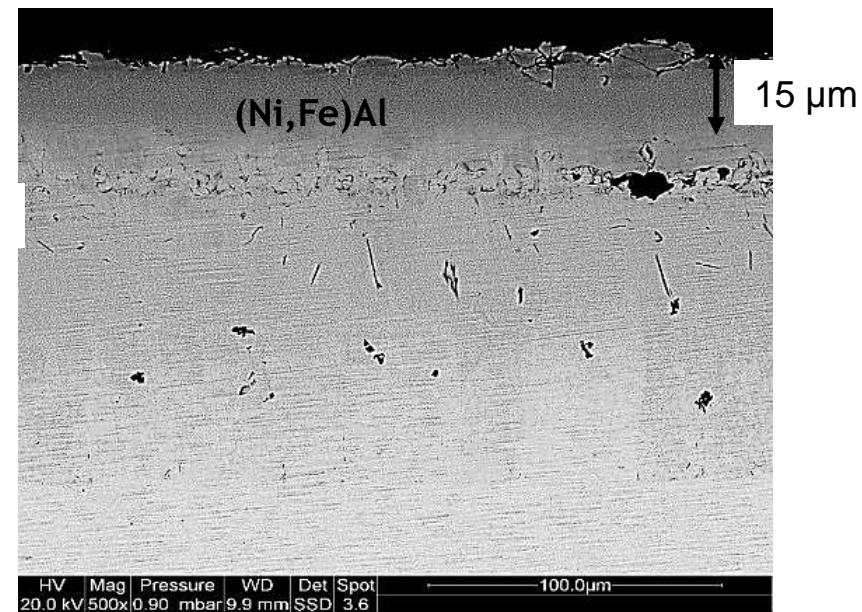
- NiAl
- Less particle stuck on the surface
- Thin interdiffusion zone
- **Cracks = due to higher thermal expansion difference between T91 and NiAl ?**

COATING OBTAINED BY SL ON T91

Without pre Ni plating



With pre Ni plating



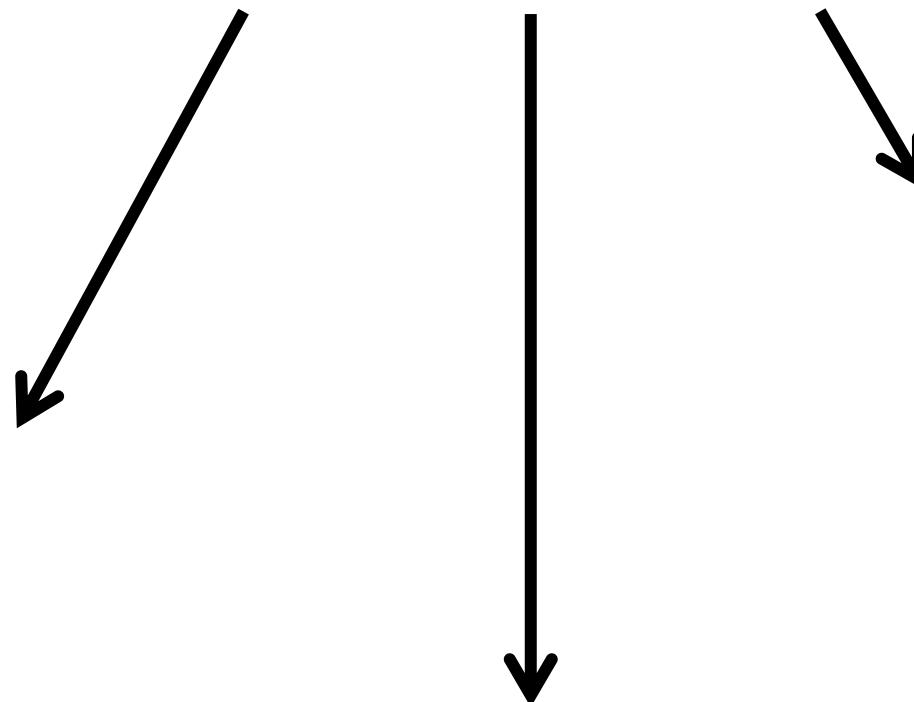
- B2-(Fe, Cr) Al with porosity at the surface
- Inwards aluminide growth
- Surface porosity

- NiAl
- Same thickness (preferential inwards Al diffusion)

2) COMPATIBILITY OF THE COATINGS IN NA

MAIN CORROSION PROCESSES IN HT NA

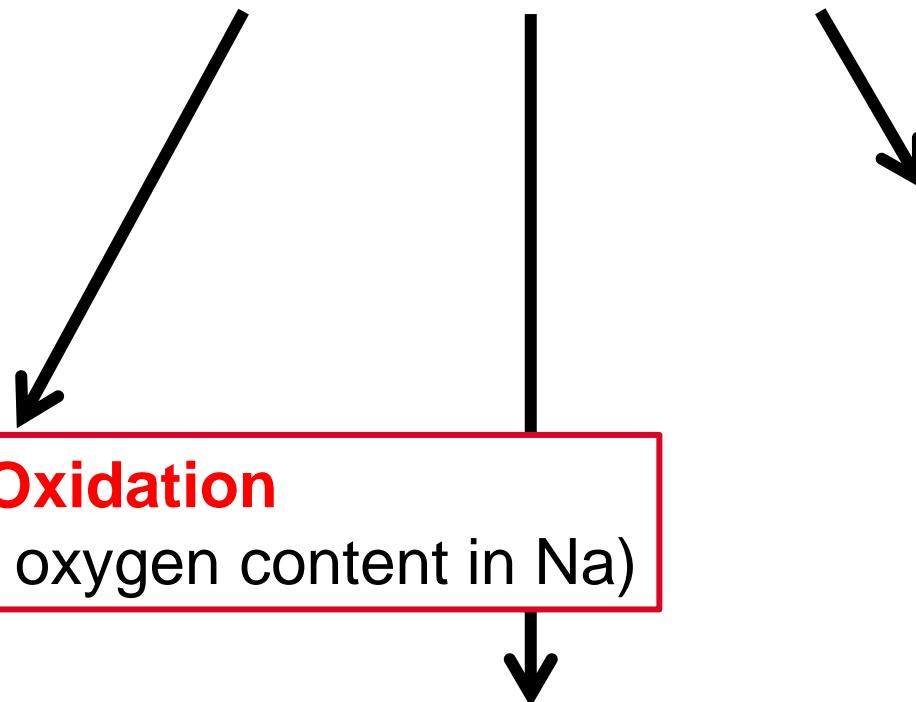
3 main corrosion processes



Dissolution
(governed by solubility)

MAIN CORROSION PROCESSES IN HT NA

3 main corrosion processes



Dissolution
(governed by solubility)

MAIN CORROSION PROCESSES IN HT NA

3 main corrosion processes

De- or carburisation
(governed by carbon content in Na)

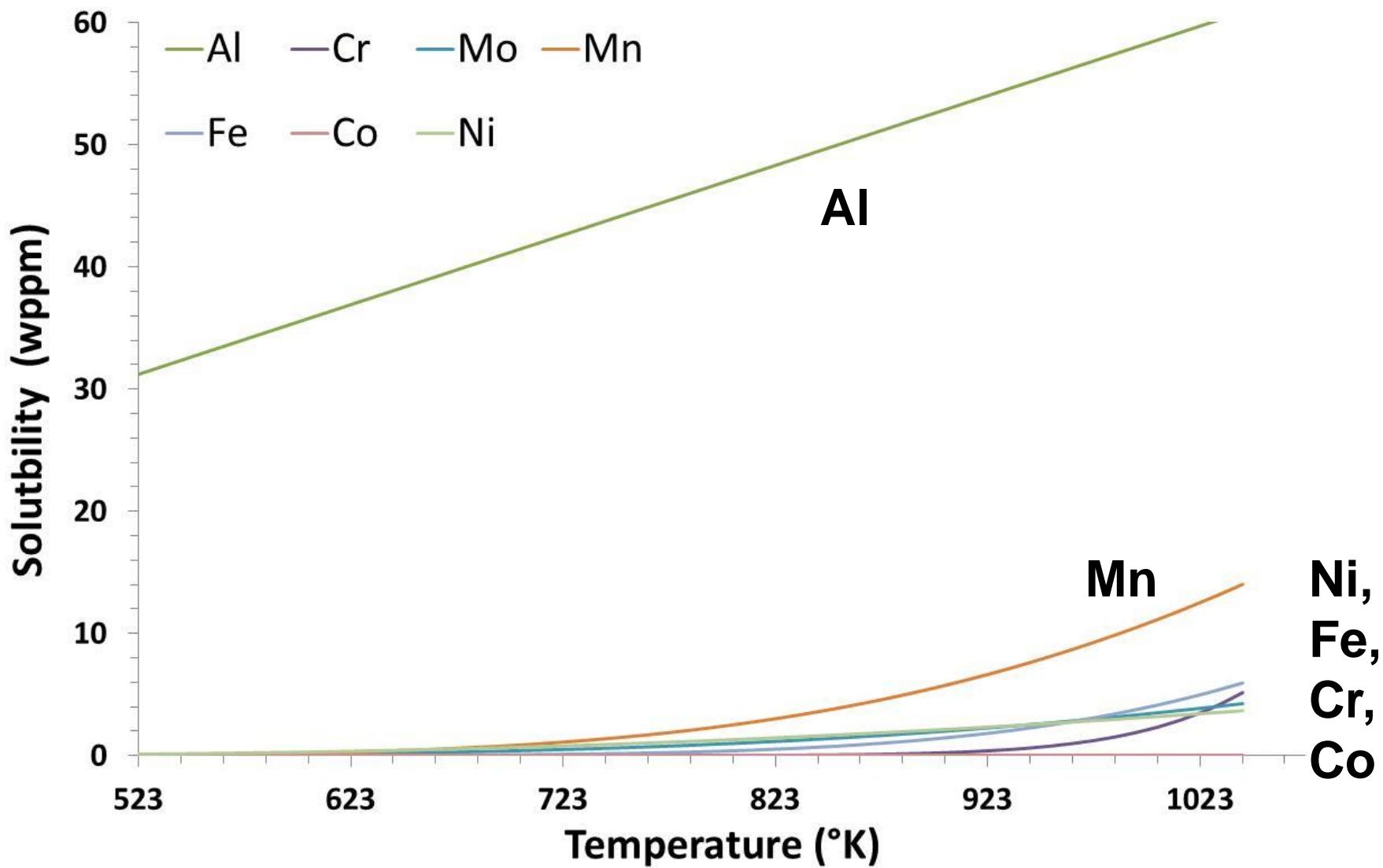
Oxidation

(governed by oxygen content in Na)

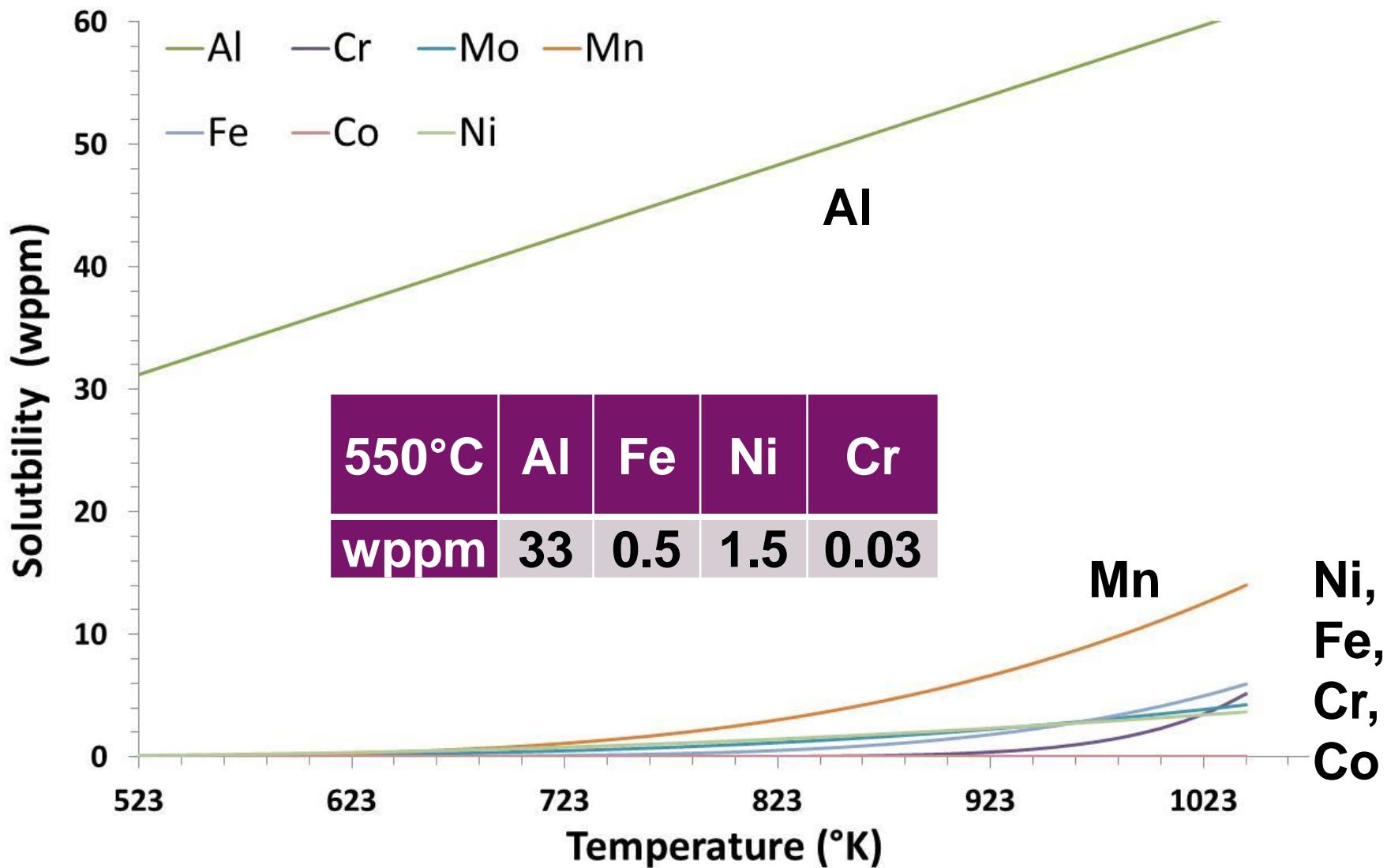
Dissolution

(governed by solubility)

DISSOLUTION IN NA

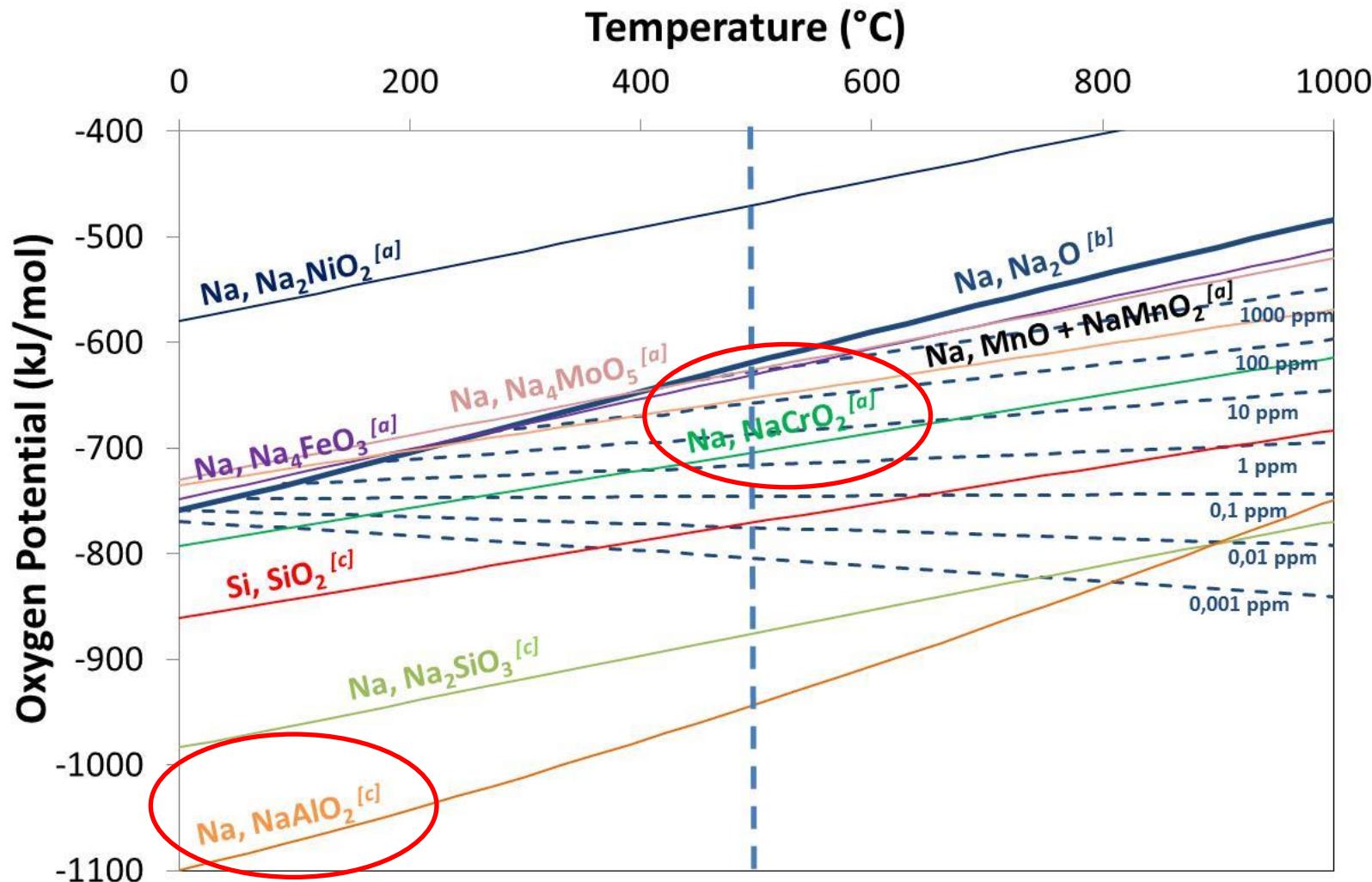


DISSOLUTION IN NA



OXIDATION IN NA

- All « usual » oxides are not stable in Na
- Formation of ternary oxides Na-M-O : $\text{Na}_2\text{O} + \text{oxide} = \text{Na}-\text{M}-\text{O}$

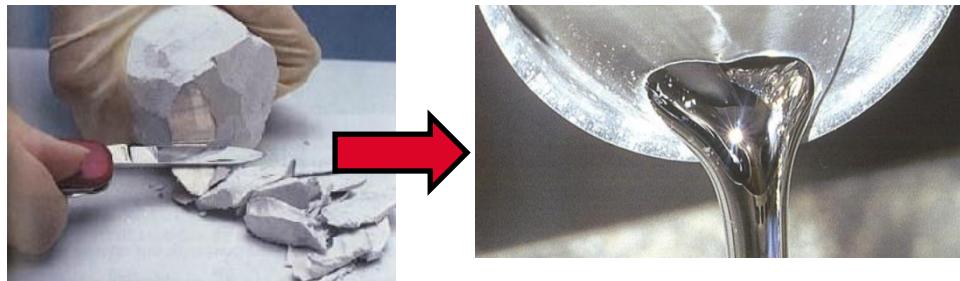


CORROSION TEST IN HT SODIUM

2.5 L static Na in Mo crucible

Temperature : **550 °C**

Exposure times : 850 h and 4250 h

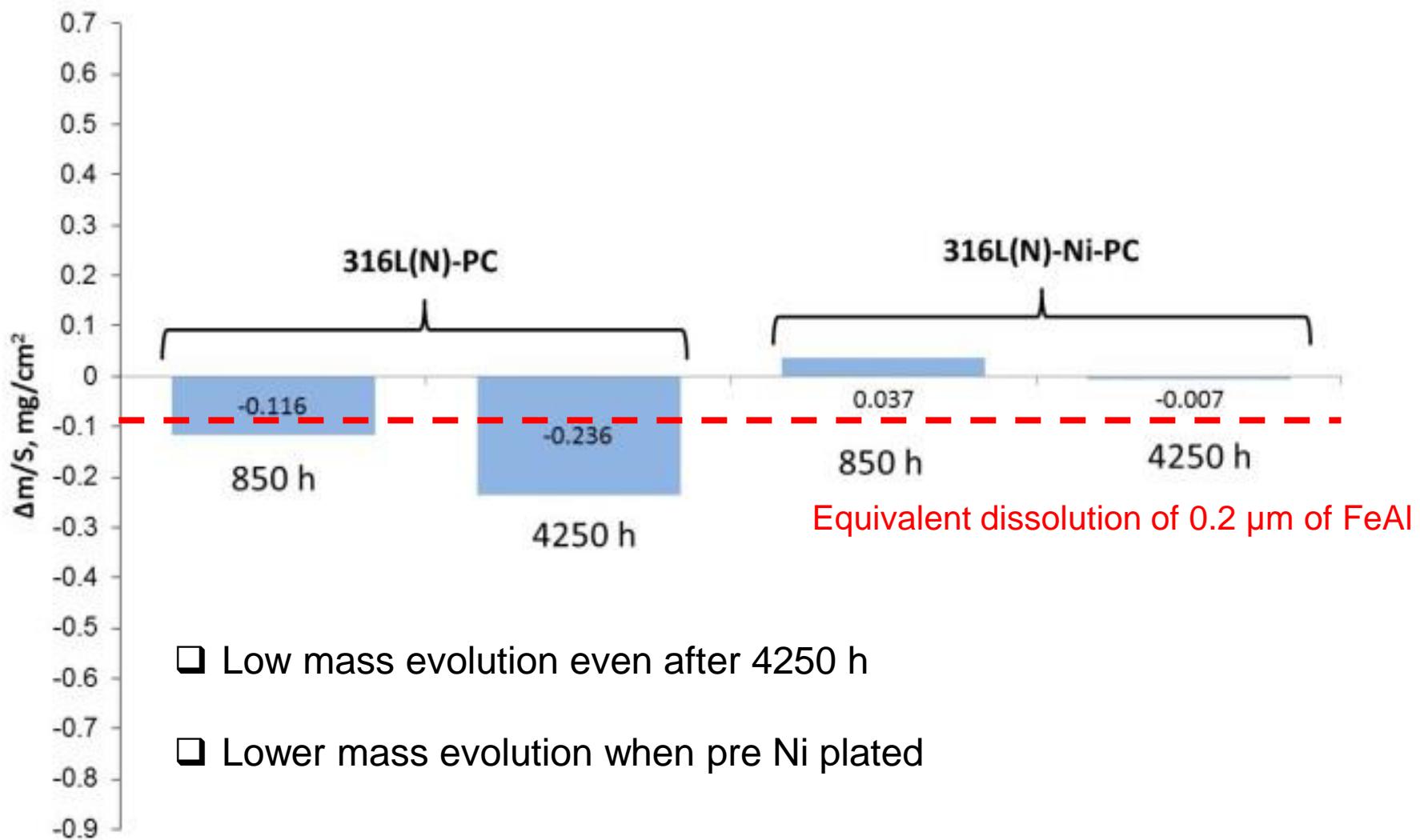


- Oxygen purified Na by Zr foil getter (600°C) before (3 days) and during sample exposure : $[O] < 5 \text{ ppm}$
- Cr sample to detect any Al dissolution

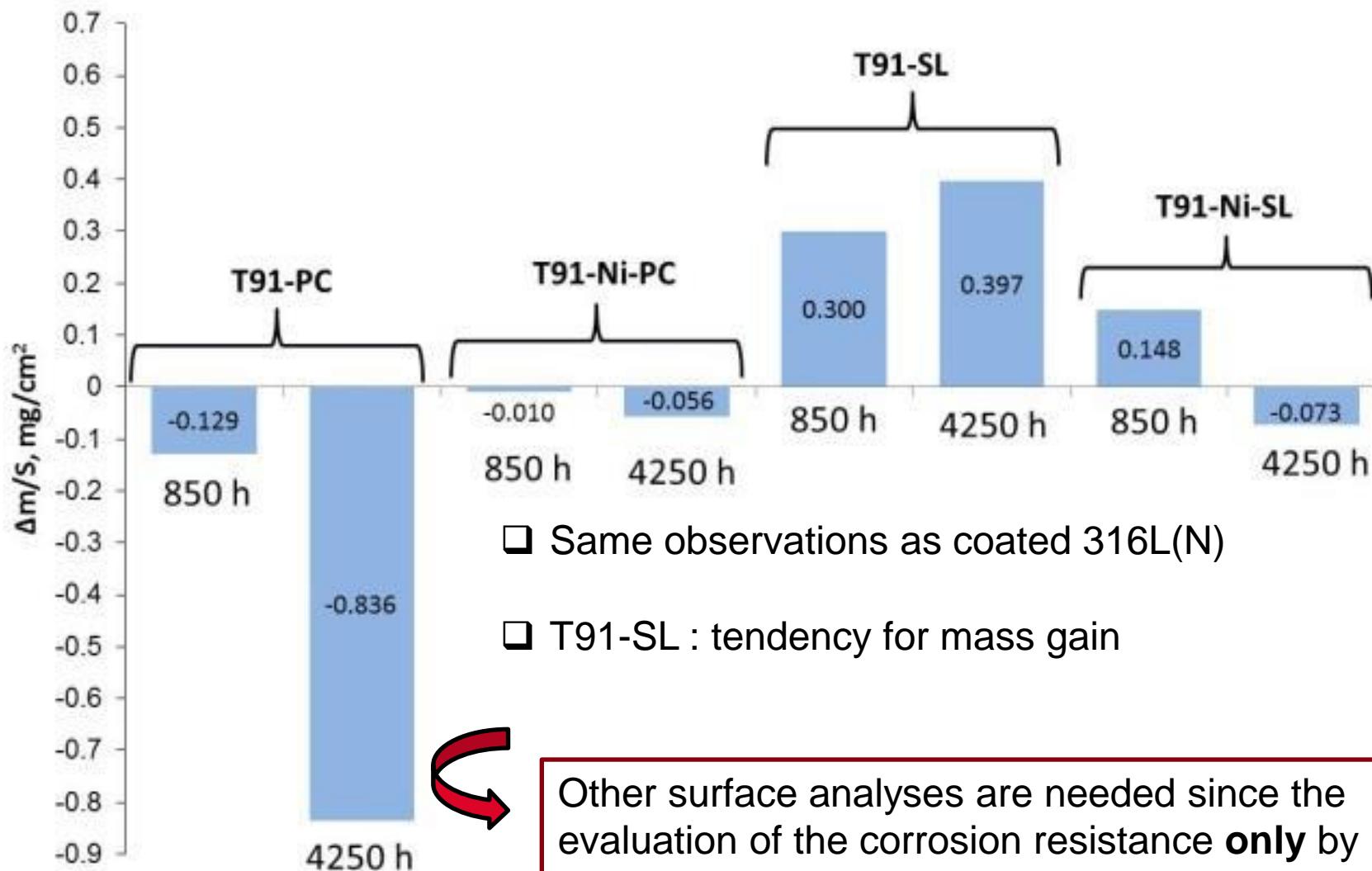


Specific washing procedure (ethanol+water) to remove residual surface sodium
Analyses : mass evolution, GDOES, SEM, Raman spectroscopy, XRD

PACK-CEMENTED 316L(N) SAMPLES



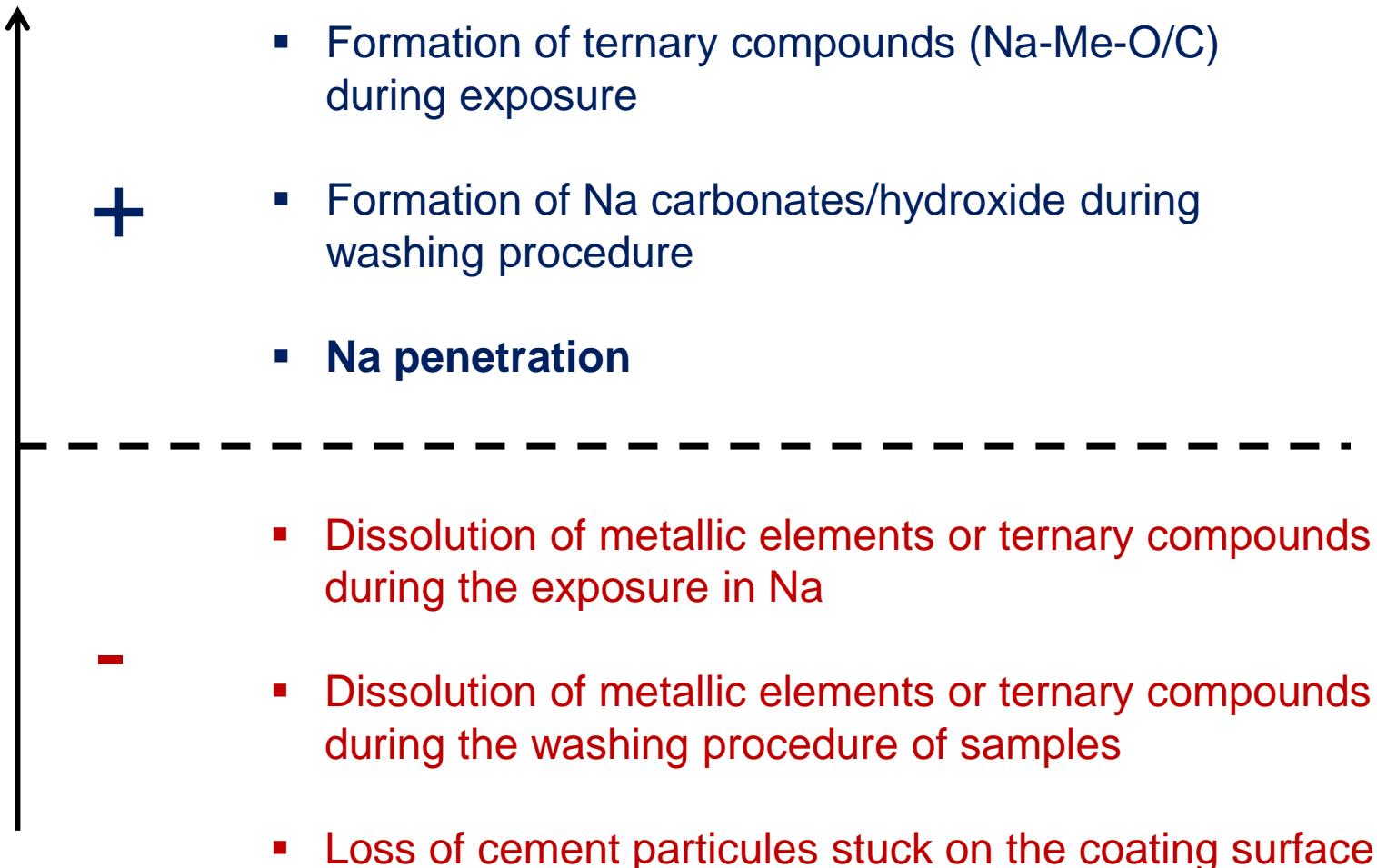
T91 SAMPLES COATED BY PC OR SL



Other surface analyses are needed since the evaluation of the corrosion resistance **only** by mass evolution interpretation can be very risky for Na

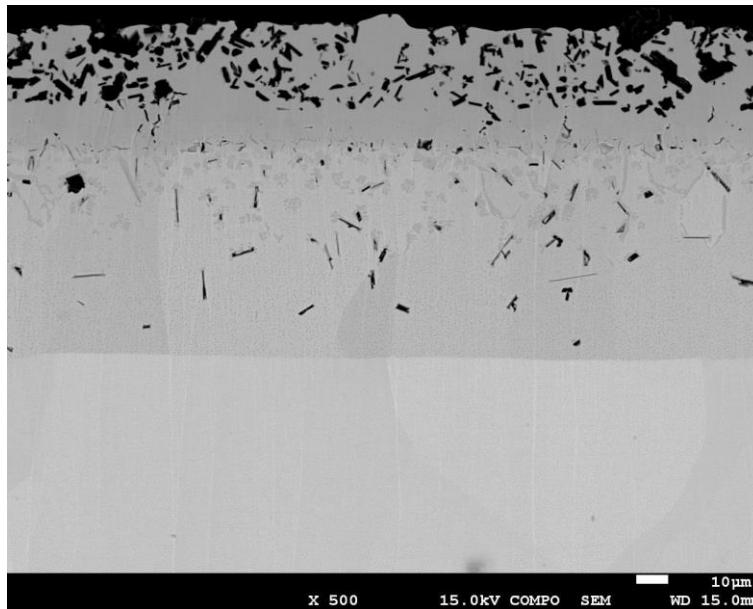
MASS EVOLUTION INTERPRETATION

Mass evolution

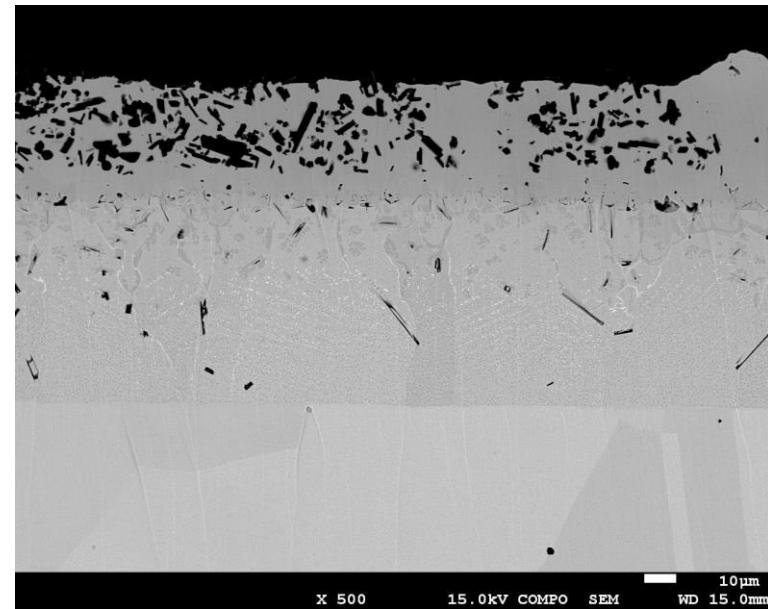
- 
- Formation of ternary compounds (Na-Me-O/C) during exposure
 - + ▪ Formation of Na carbonates/hydroxide during washing procedure
 - **Na penetration**
 - ▪ Dissolution of metallic elements or ternary compounds during the exposure in Na
 - Dissolution of metallic elements or ternary compounds during the washing procedure of samples
 - Loss of cement particules stuck on the coating surface

SEM IMAGES OF 316L(N) –PC BEFORE AND AFTER EXPOSURE

316L(N) **before** exposure

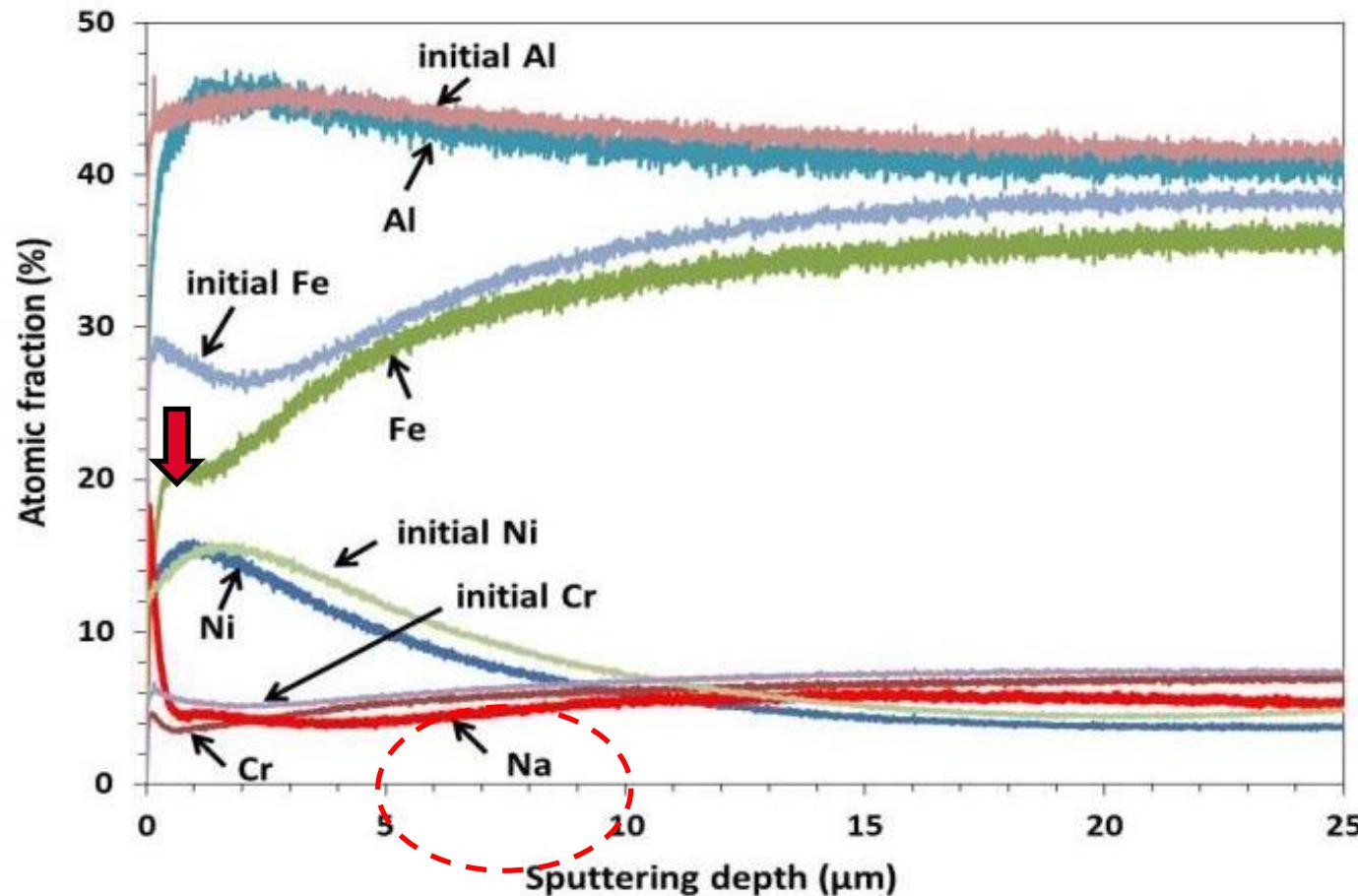


316L(N) **after** exposure in Na – 4250 h



- No clear degradation of the coating and the substrate-coating interface

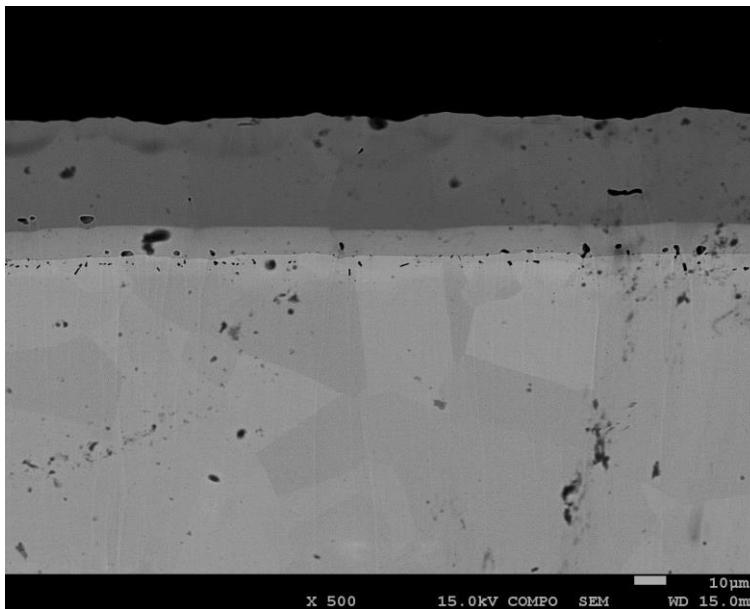
GDOES PROFILES FOR 316L(N)-PC



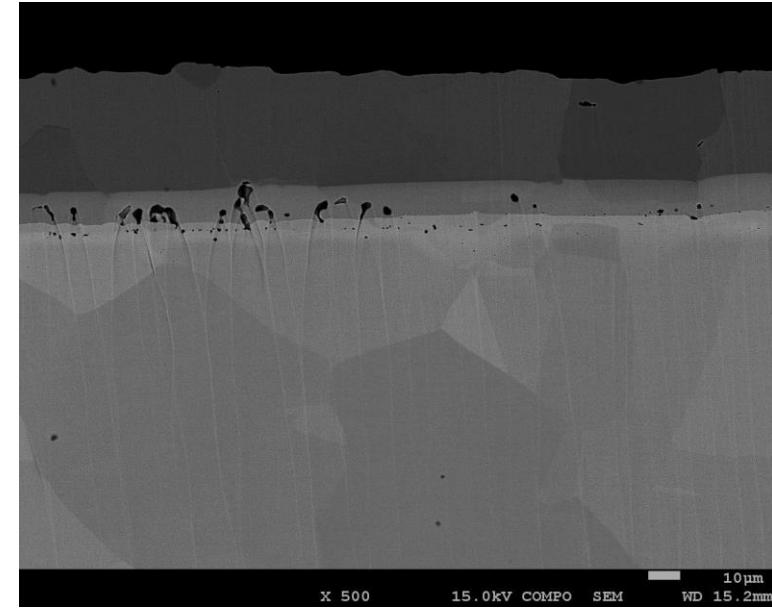
- No strong evolution of the surface coating composition as respect to its main elements : Al, Ni, Cr, Fe
- Deep penetration of Na > 25 μm in the Al_2O_3 particles enriched zone : interfaces = high diffusion paths ?

SEM IMAGES OF 316L(N) - N - PC BEFORE AND AFTER EXPOSURE

316L(N) **before** exposure

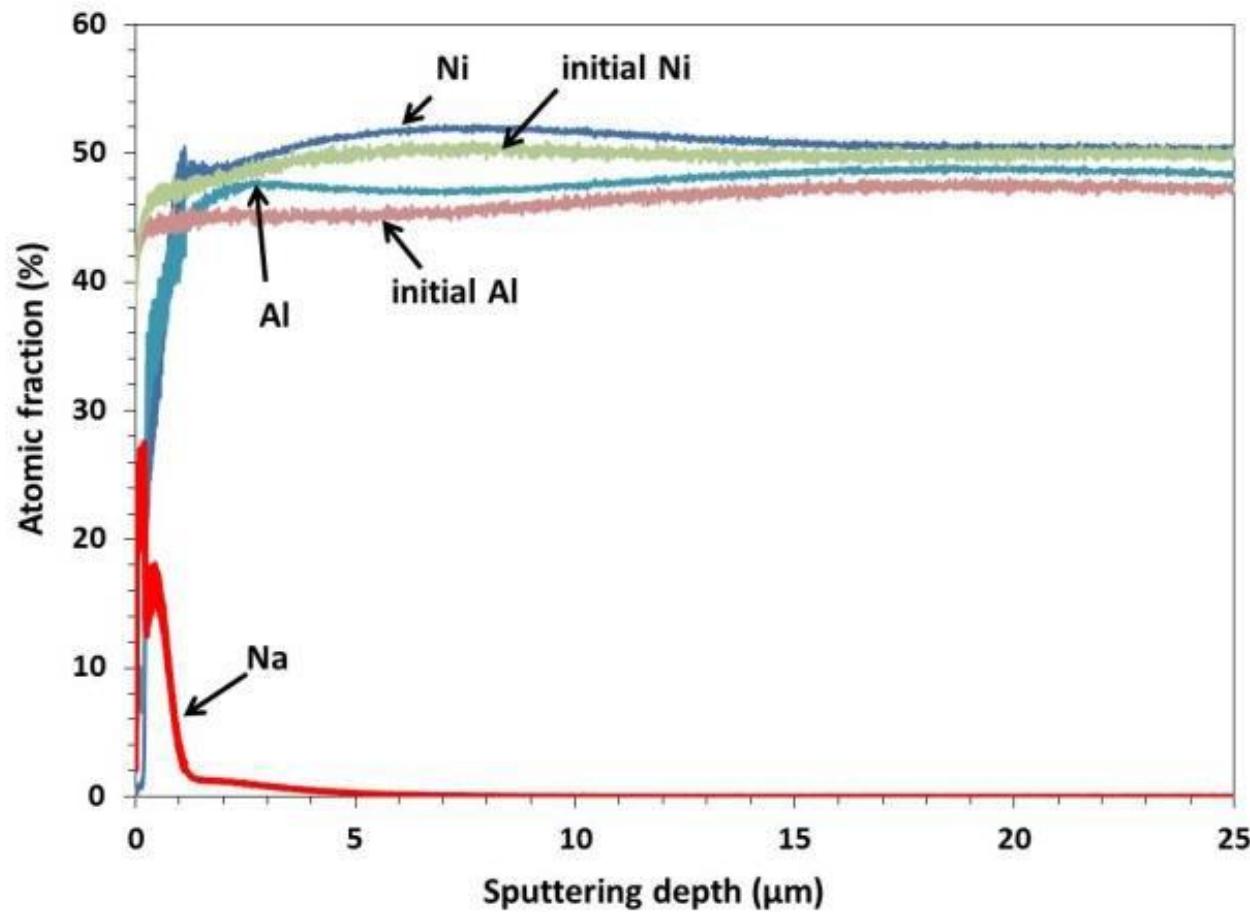


316L(N) **after** exposure in Na – 4250 h



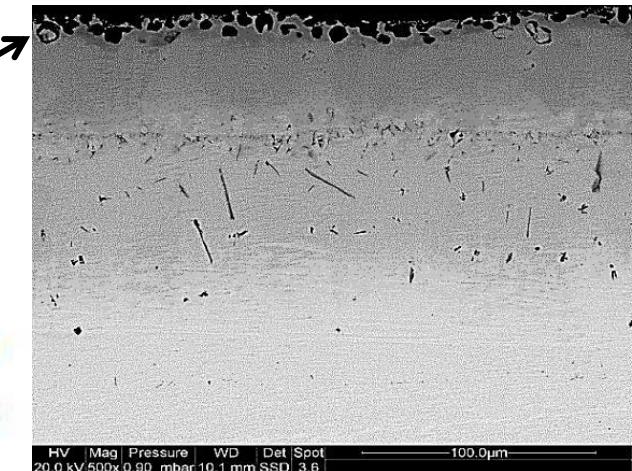
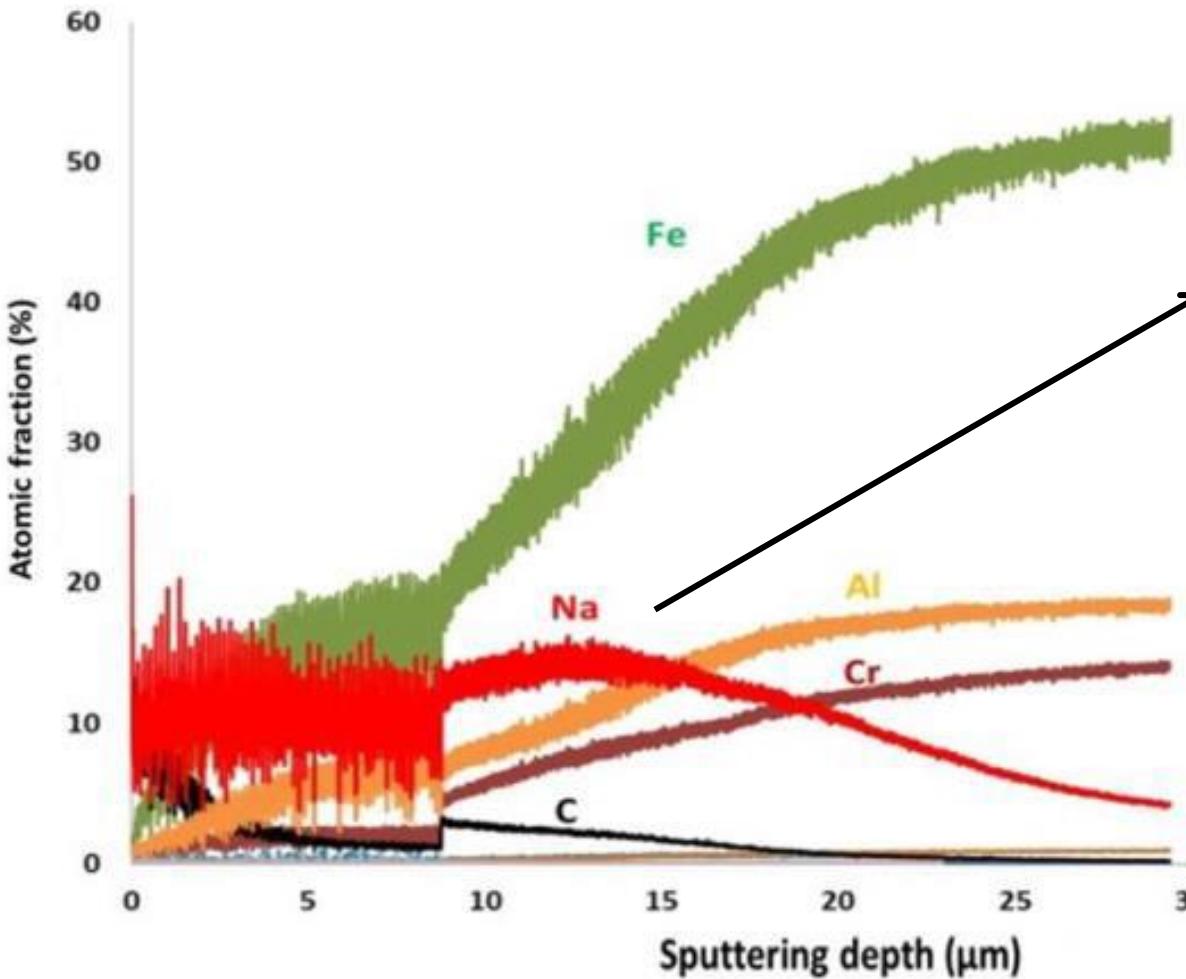
- No clear degradation of the coating surface
- Formation of voids in the Fe rich NiAl zone (Kirkendall effect)

GDOES PROFILES FOR 316L(N)-NI-PC



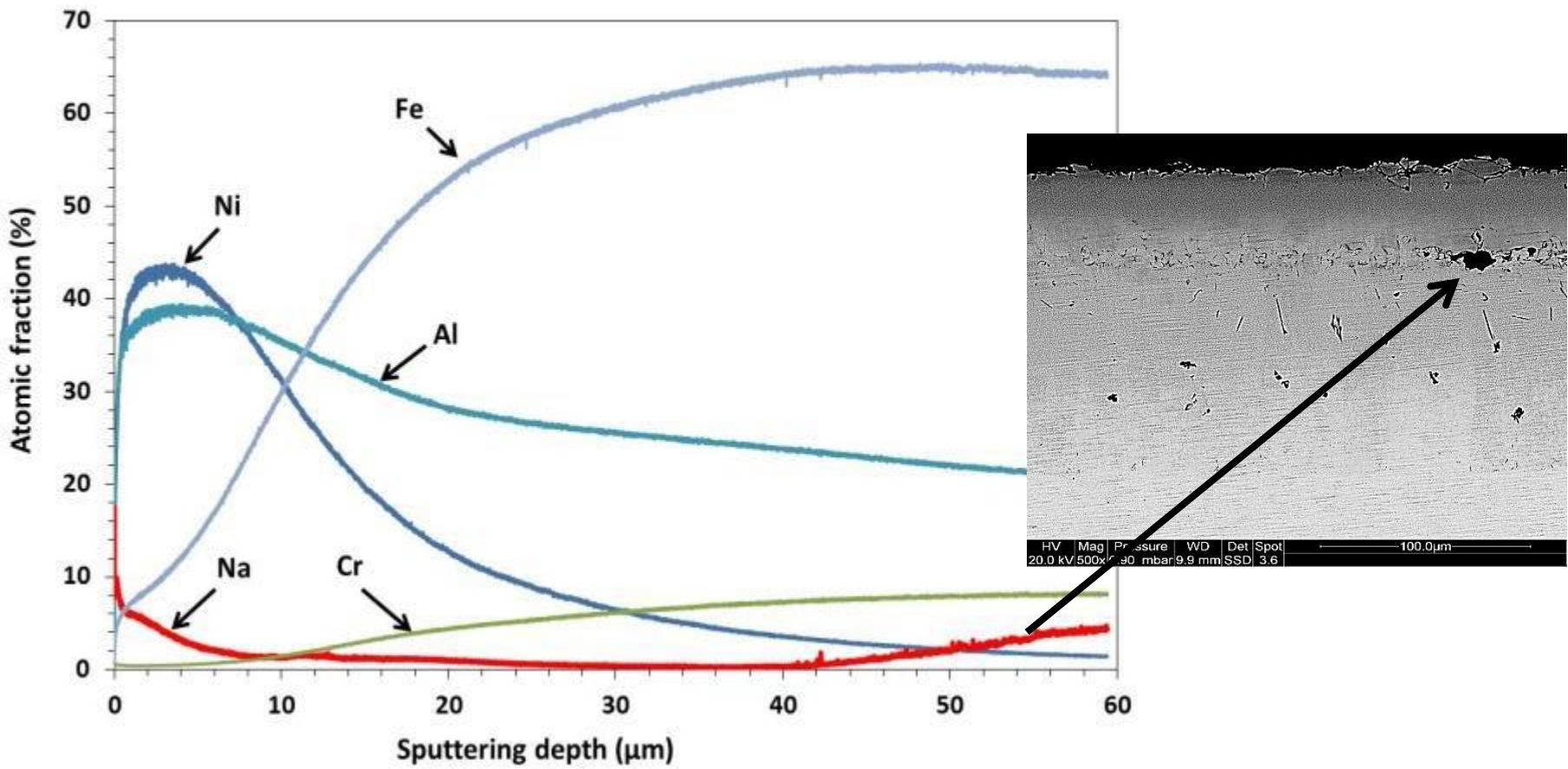
- Same observations as for 316L(N) – PC : no clear evolution of concentrations
- But lower penetration of Na $< 5 \mu\text{m}$: dense coating

T91 COATED BY SLURRY



- No formation of ternary compounds could be detected (Raman, XRD, SEM-EDX). Only aluminides and Al_2O_3
- Na accumulation in the porous region of the coating then penetration

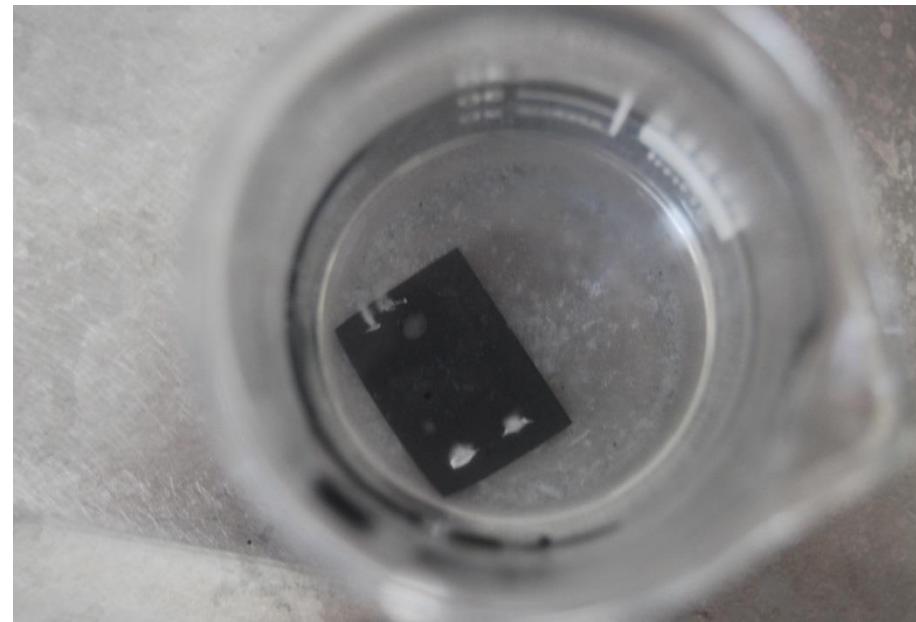
T91 – NI COATED BY SLURRY



Lower Na on the surface but penetration and accumulation detected deeper
(accumulation in voids ?)

AL DISSOLUTION ?

Yes !



EDX analysis on Cr sample surface
3 wt% of Al
[from Al_2O_3 , Fe-Ni aluminides]

CONCLUSIONS FROM EXPOSURE TEST

- No severe degradation of the aluminide coatings whatever the process used (PC or SL) : Aluminides are quite stable in HT Na



In good agreement with results from litterature :

- Maximum corrosion rate of NiAl on IN718 = $0.75 \mu\text{m/year}$ at 625°C
[Johnson et al., LIMET 1976]
- No evolution of the concentration of Al, Fe, Cr and Ni in the aluminide deposited on 304, 316 and IN718 alloys after exposure in flowing Na at 400°C for 3450 h completed by 350 h at 550°C
[Depierre et al., Materials Science And Engineering 88, 287 (1987)]

- But still :

- Slight Al dissolution
- Na penetration (function of the density of the microstructure of aluminides, particles, cracks, ...) :

CONCLUSIONS FROM EXPOSURE TEST

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[Johnson et al. / IMFT 1976]

What about the tribological behaviour of aluminides in Na ?

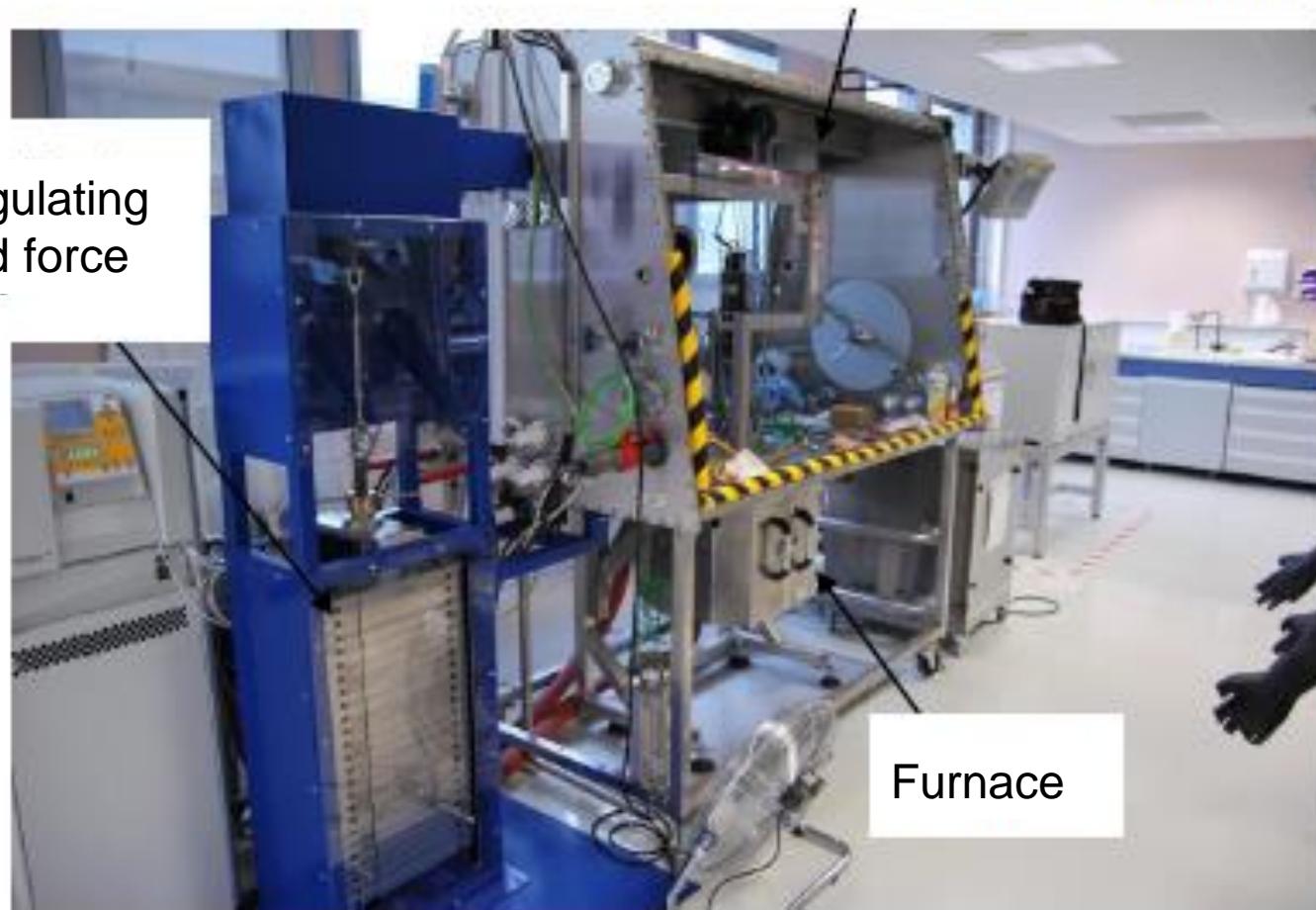
- But still :
 - Slight Al dissolution
 - Na penetration (function of the density of the microstructure of aluminides, particles, cracks, ...) : effect of tribological behaviour ?

SODIUM FACILITY FOR FRICTION-WEAR TESTS

Glove box

Weights regulating
the applied force

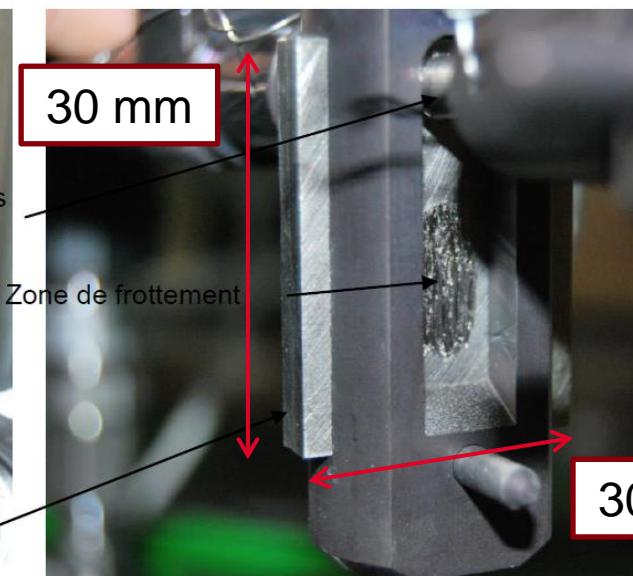
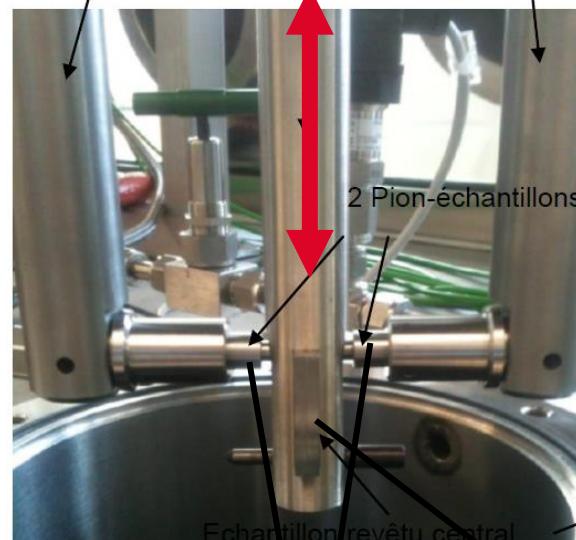
Furnace



SODIUM FACILITY FOR FRICTION-WEAR TESTS

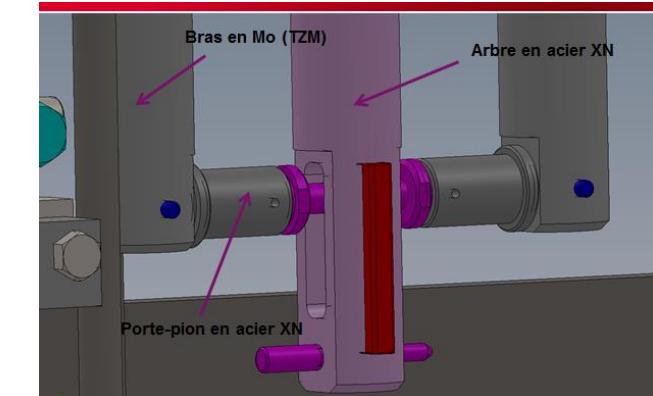
Arms applying normal force F_n

Oscillating
translation
movement



316L(N) pin

8 mm
16 mm



SolidWorks

WHAT CAN WE MEASURE ?

- Transversal force F_T needed for the oscillating translation movement :



FRICTION COEFFICIENT

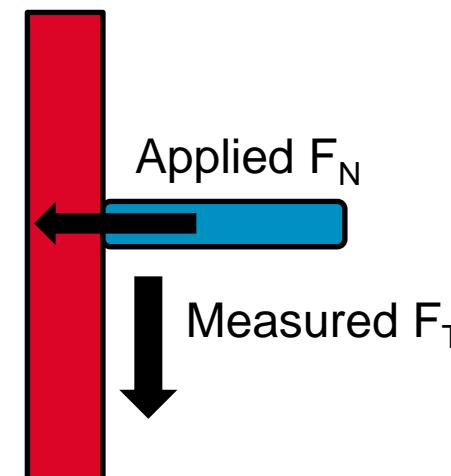
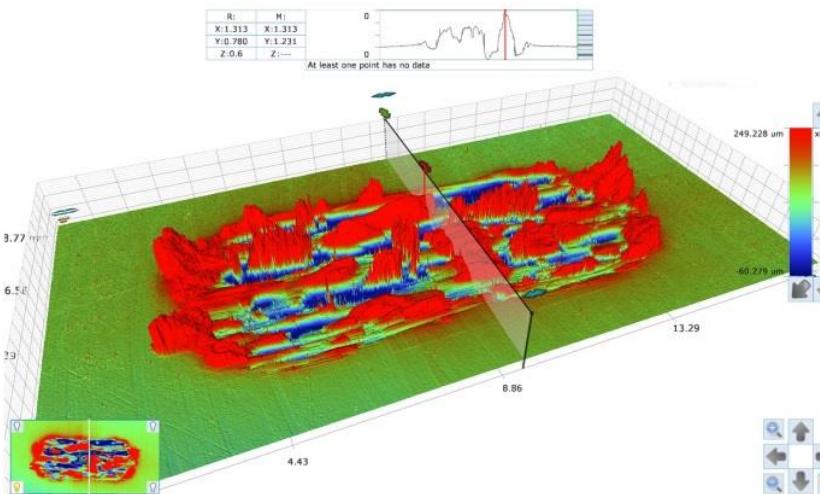
- Mass variation : pins and coated sample

- Macroscopic images



WEAR

- Profilometric measurements : lost, gained and displaced volume of material



$$f = F_T / F_N$$

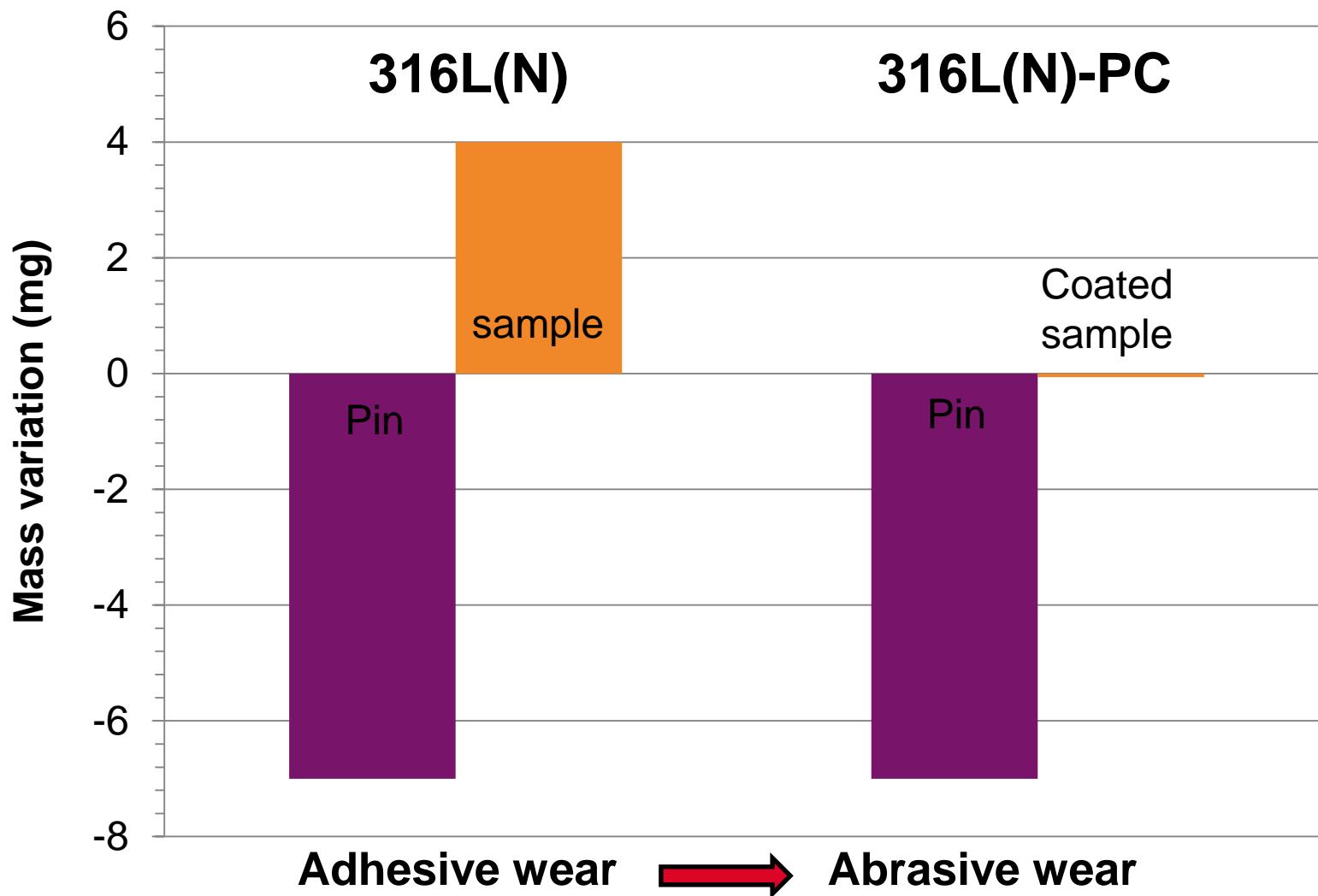
EXPERIMENTAL CONDITIONS

Na temperature	200°C
Translation rate	1 mm/s
Tracing length	10 mm
Total distance	4 m
Applied Stress	31 MPa

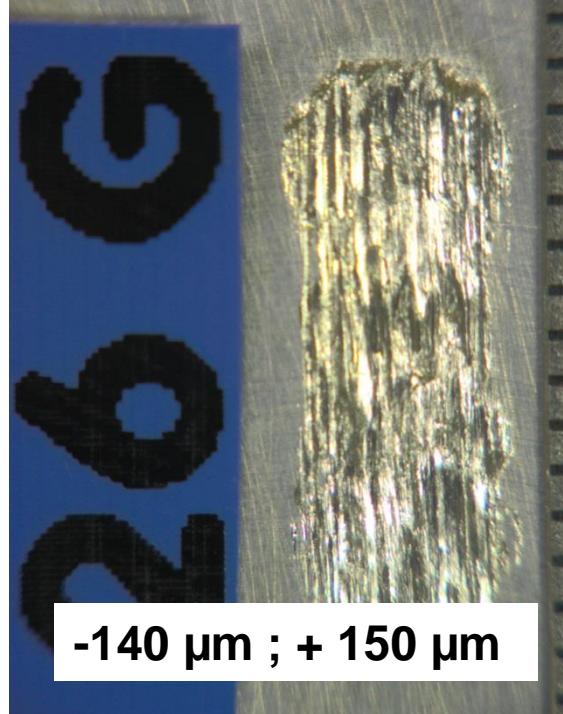
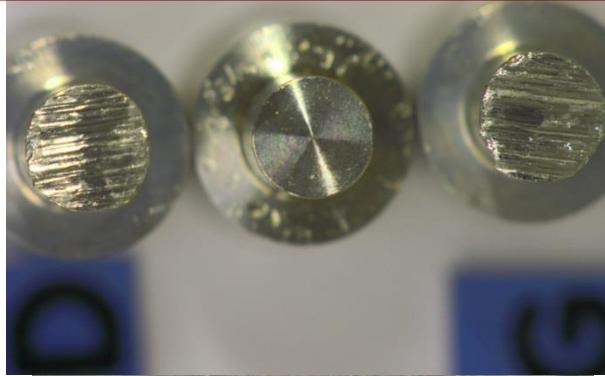
Pin material	316L(N)
Coated sample	316L(N) or 316L(N)-PC

2 friction tests : with or without coatings

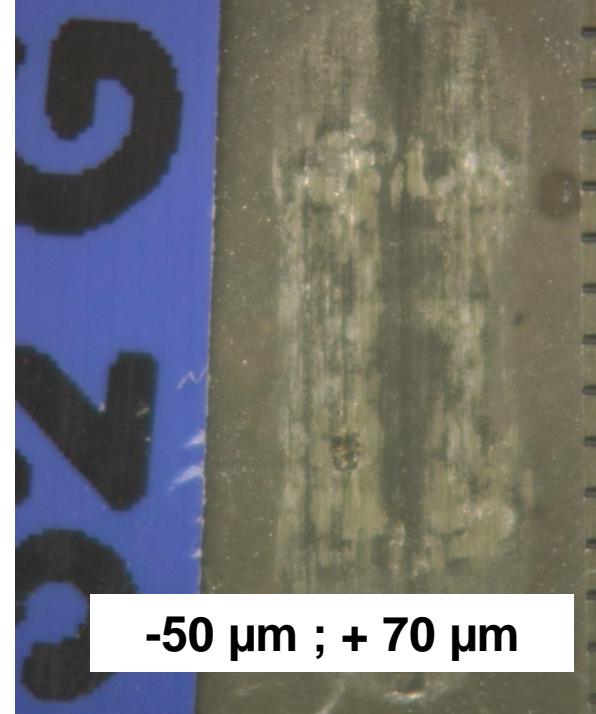
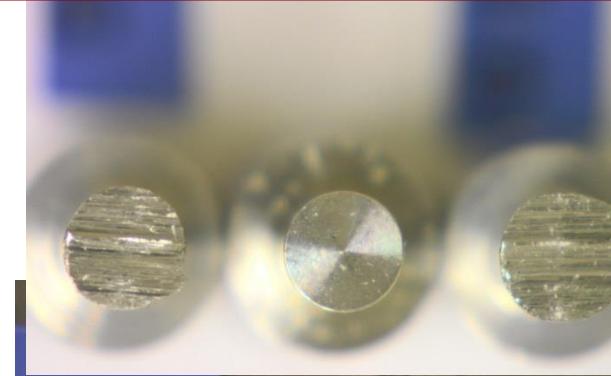
MASS EVOLUTION



MACROSCOPIC IMAGES

316L(N) - 316L(N)

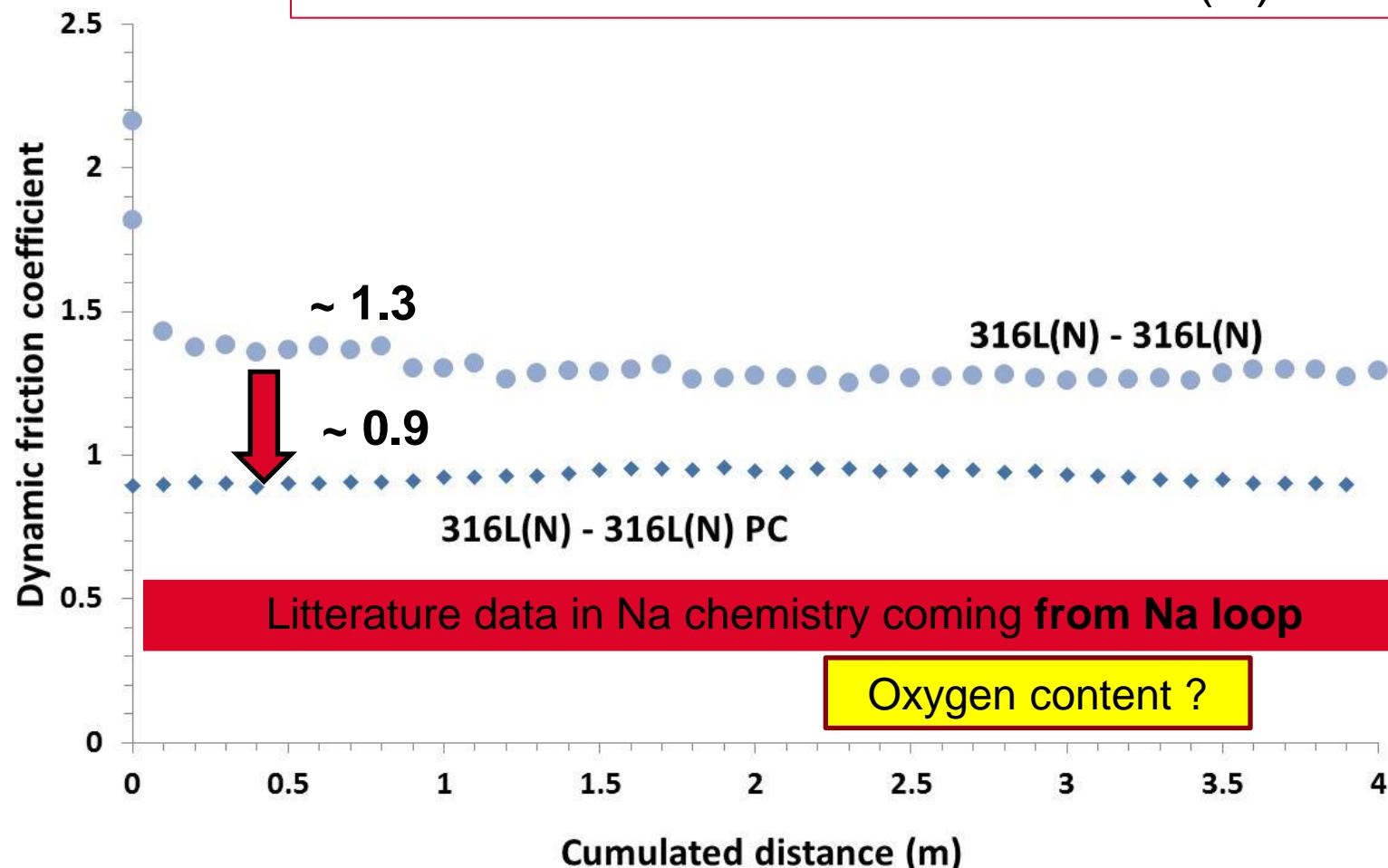
-140 µm ; + 150 µm

316L(N) - 316L(N) PC

-50 µm ; + 70 µm

Lower wear for
316L(N) PC

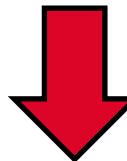
FRICTION COEFFICIENT MEASUREMENTS



CONCLUSIONS AND PROSPECTS

Conclusions

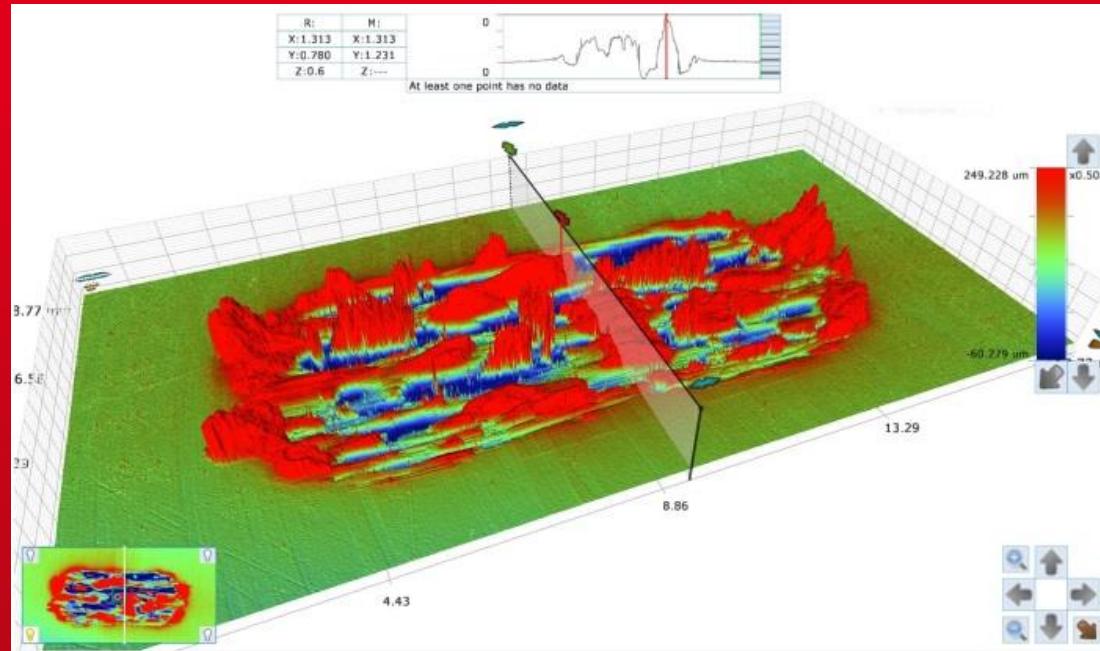
- Good compatibility of aluminides in Na (up to 550°C)
- Good wear resistance and lower friction coefficient than without coating



Prospects

- What are the influence of the aluminide coating on the wear/friction properties (nature, roughness) ?
- What is the influence of the [O] content in Na on the wear/friction properties ?
- Development of new coatings (additonal elements) favoring the formation of lubricating surface compounds in Na (reactor chemistry)

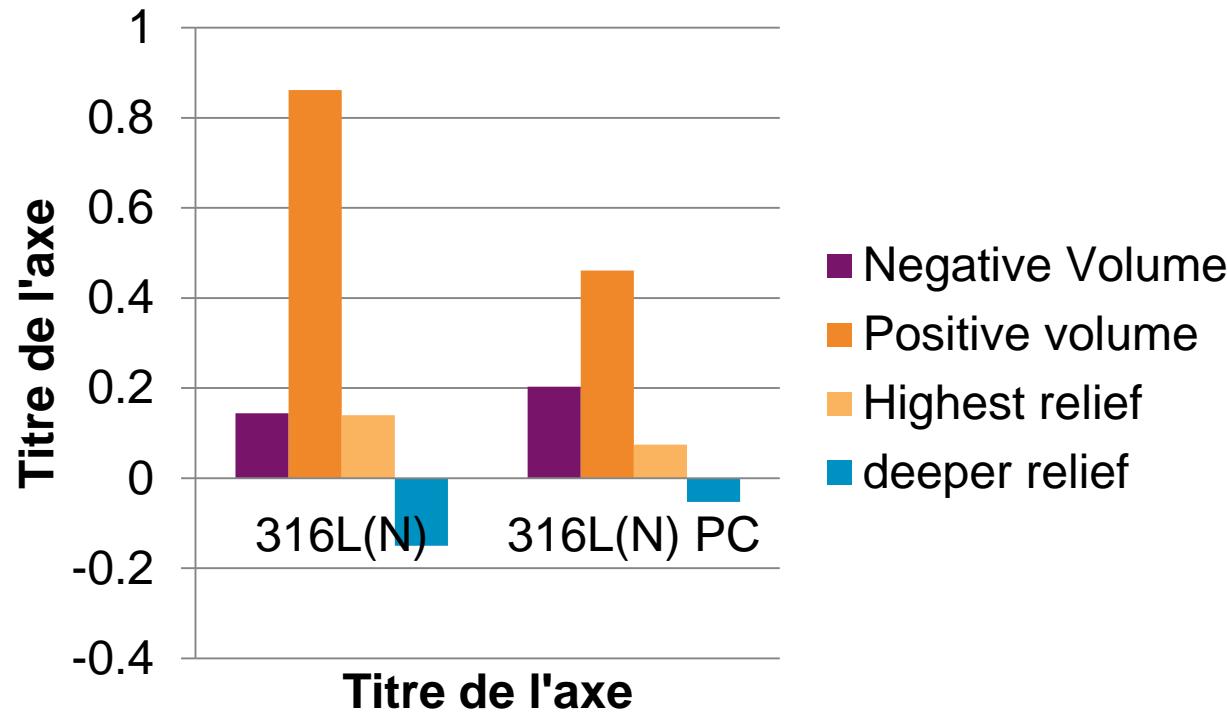
Thank You for your attention



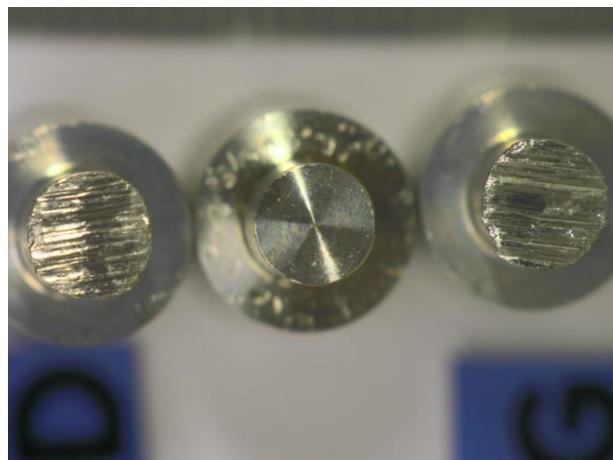
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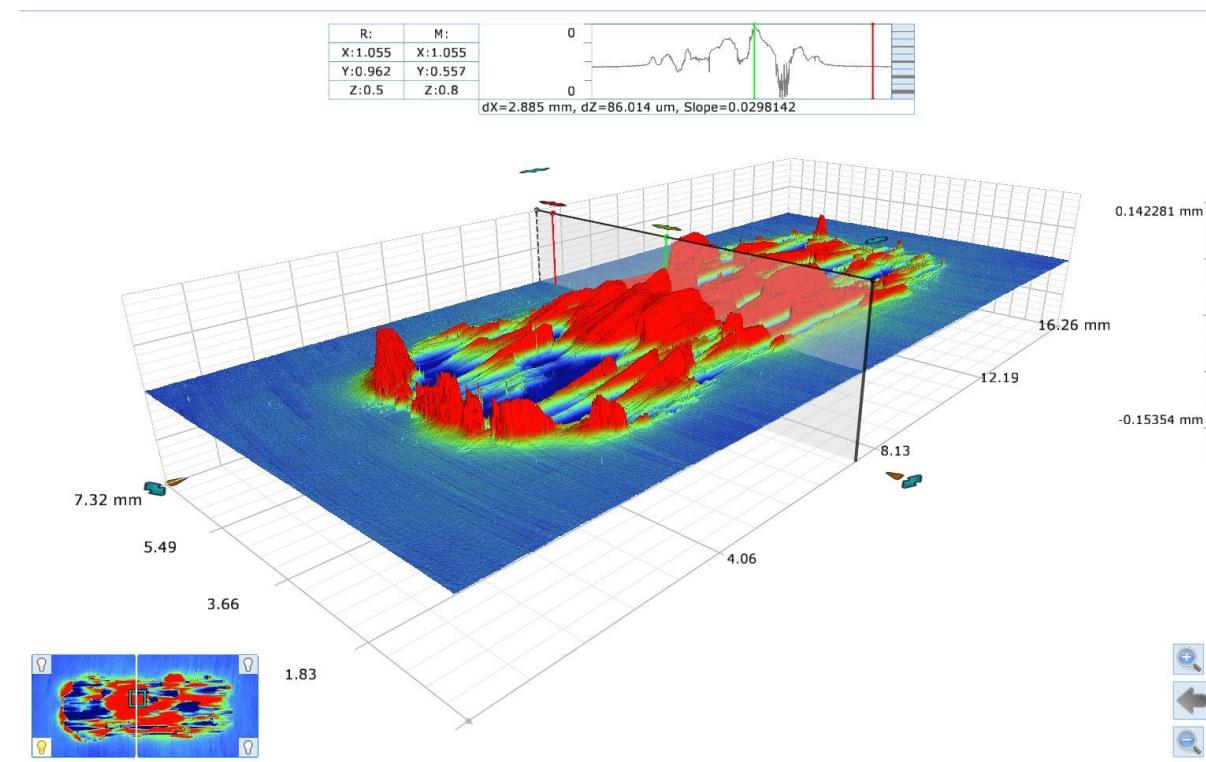
Titre du graphique

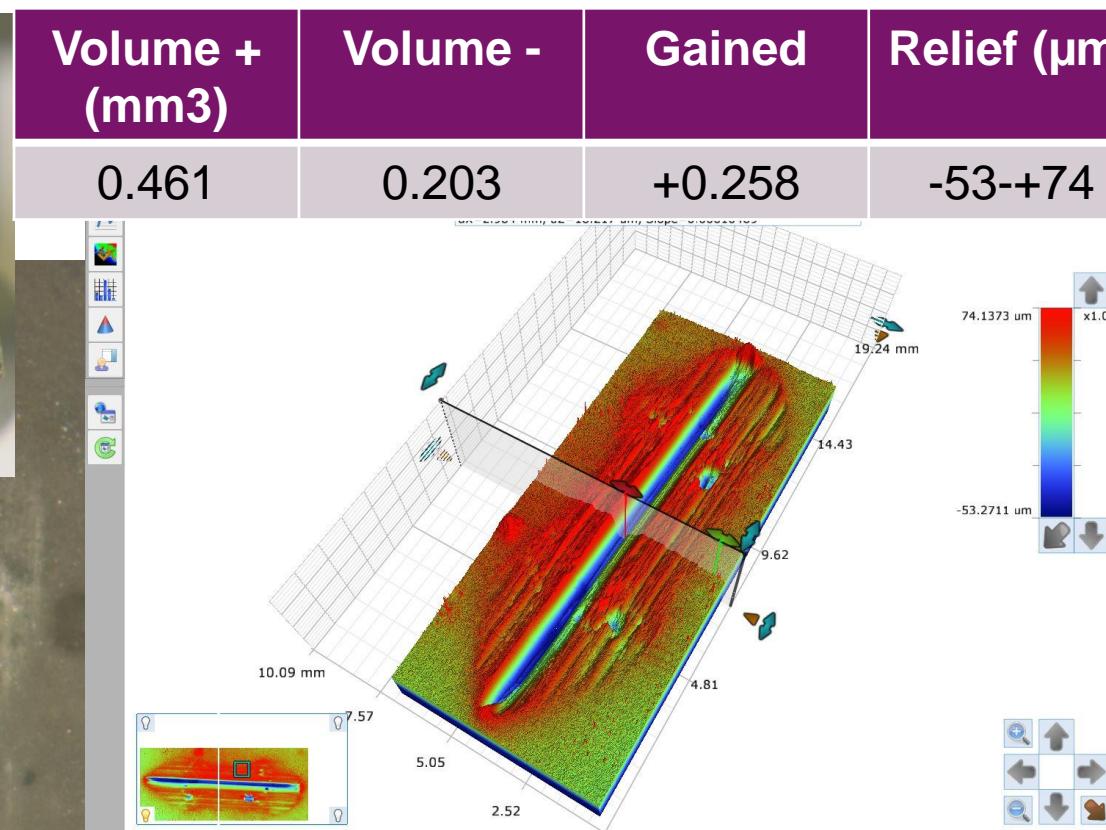
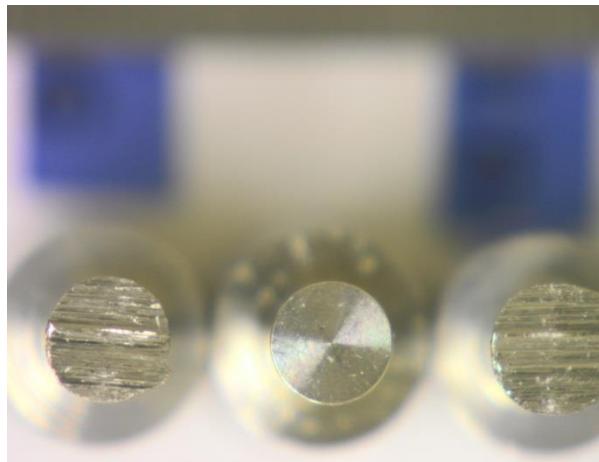


Test	Pin (mg)	Coated sample (mg)	Wear
316L(N) - 316L(N)	-7	+4	Adhesive wear
316L(N) - coated 316L(N) PC	-7	-0.06	Abrasive wear
316L(N) - coated 316L(N) Ni PC	-10	+4	Adhesive wear

316LN – 316LN 200°C

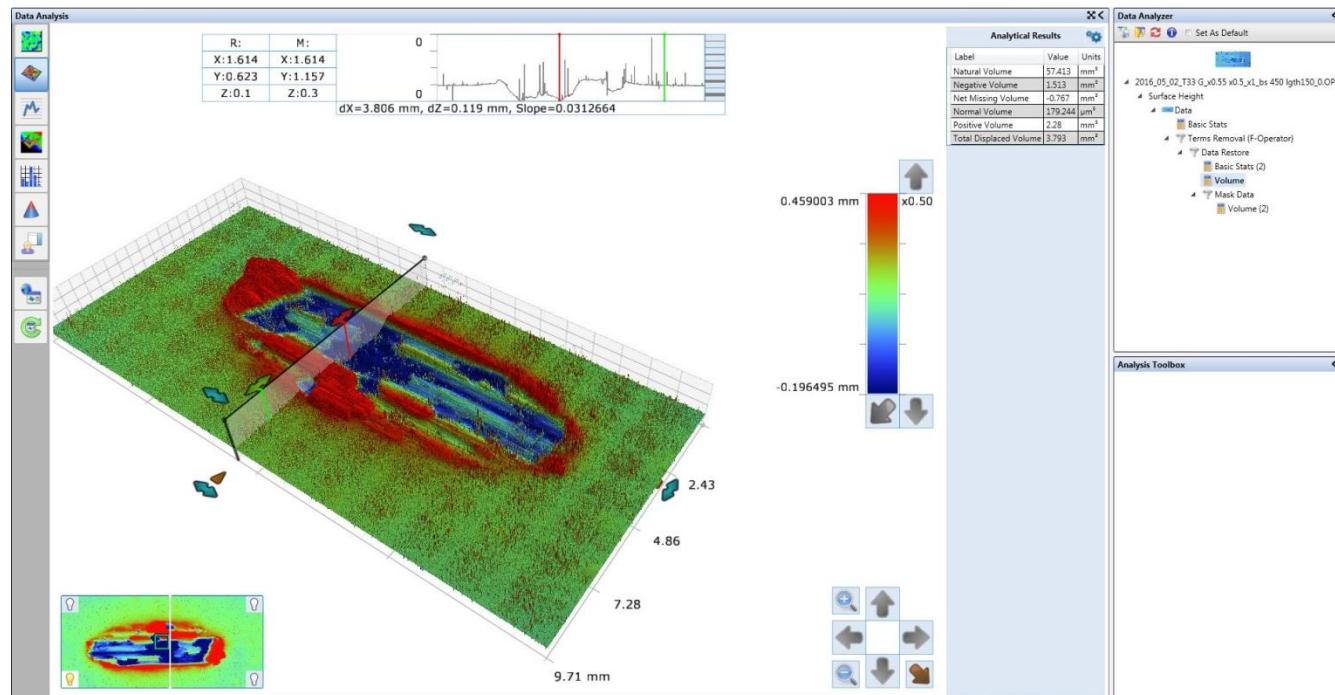
Volume + (mm³)	Volume -	Gained	Relief (µm)
0.862	0.144	+0.718	-150--+140



316LN – 316LN PC - 200°C AND 500°C

316LN – 316LN NI PC - 200°C AND 500°C

Volume + (mm³)	Volume -	Gained	Relief (µm)
2.28	1.51	+0.77	-196--+460



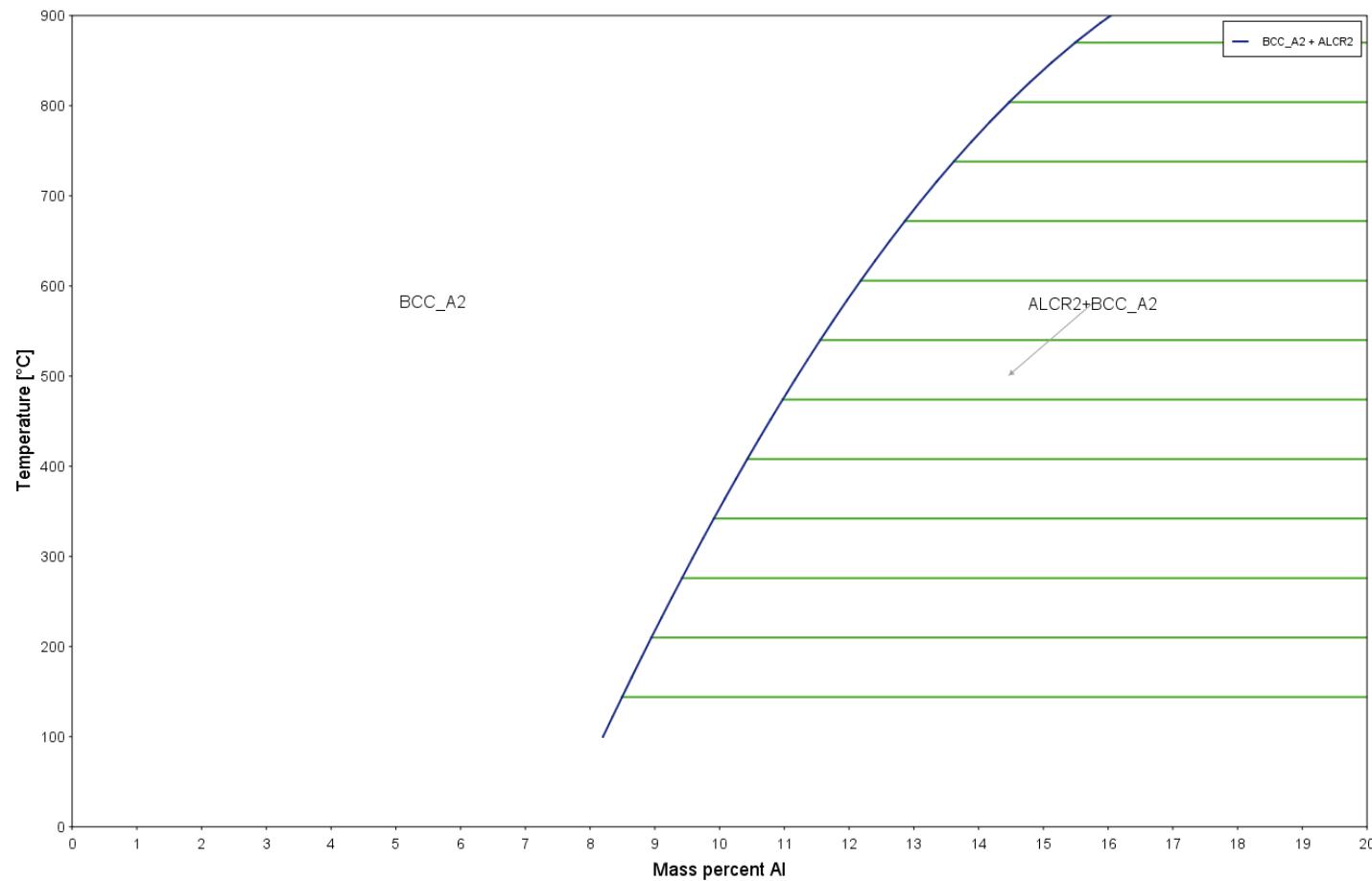
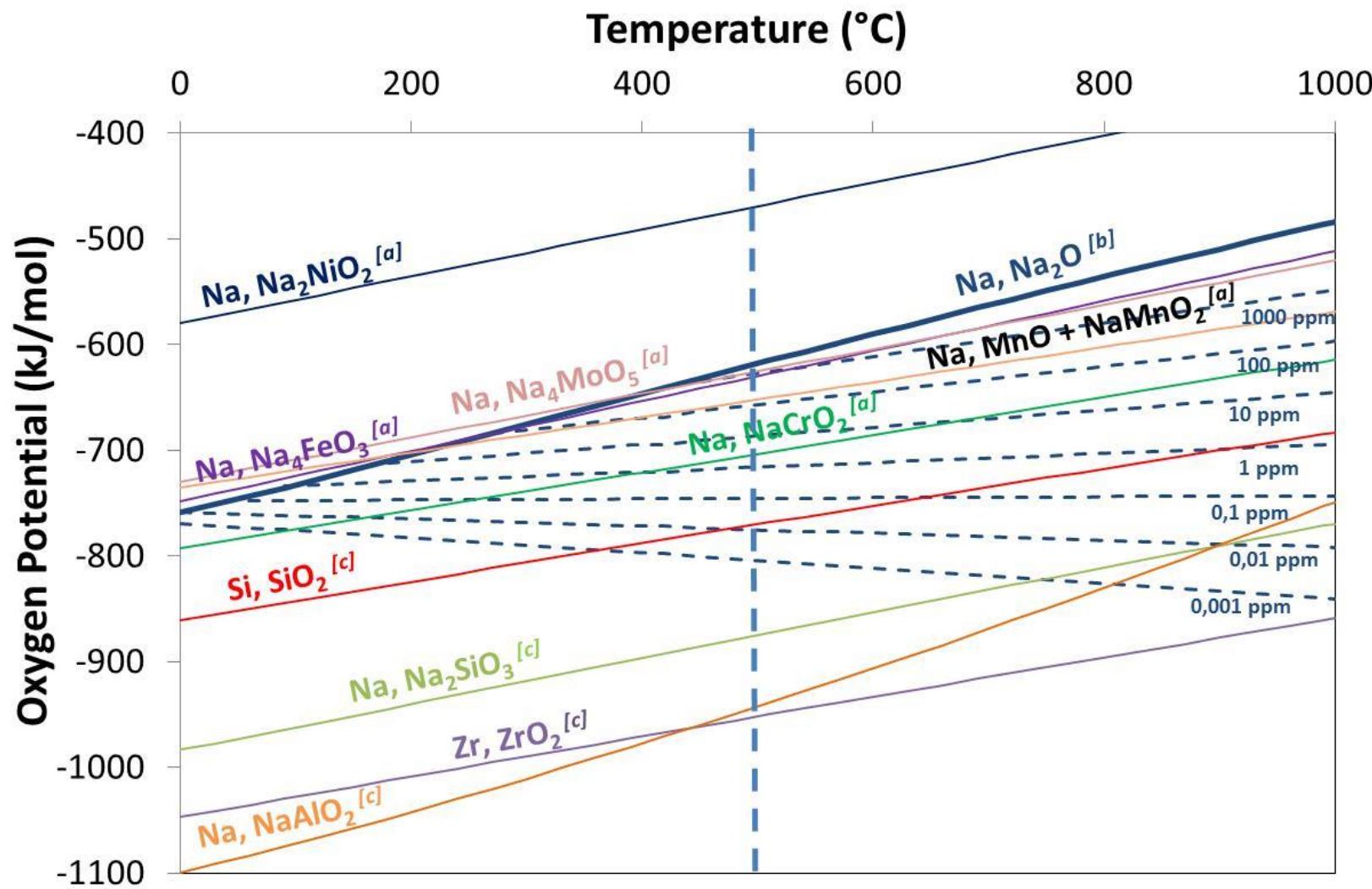
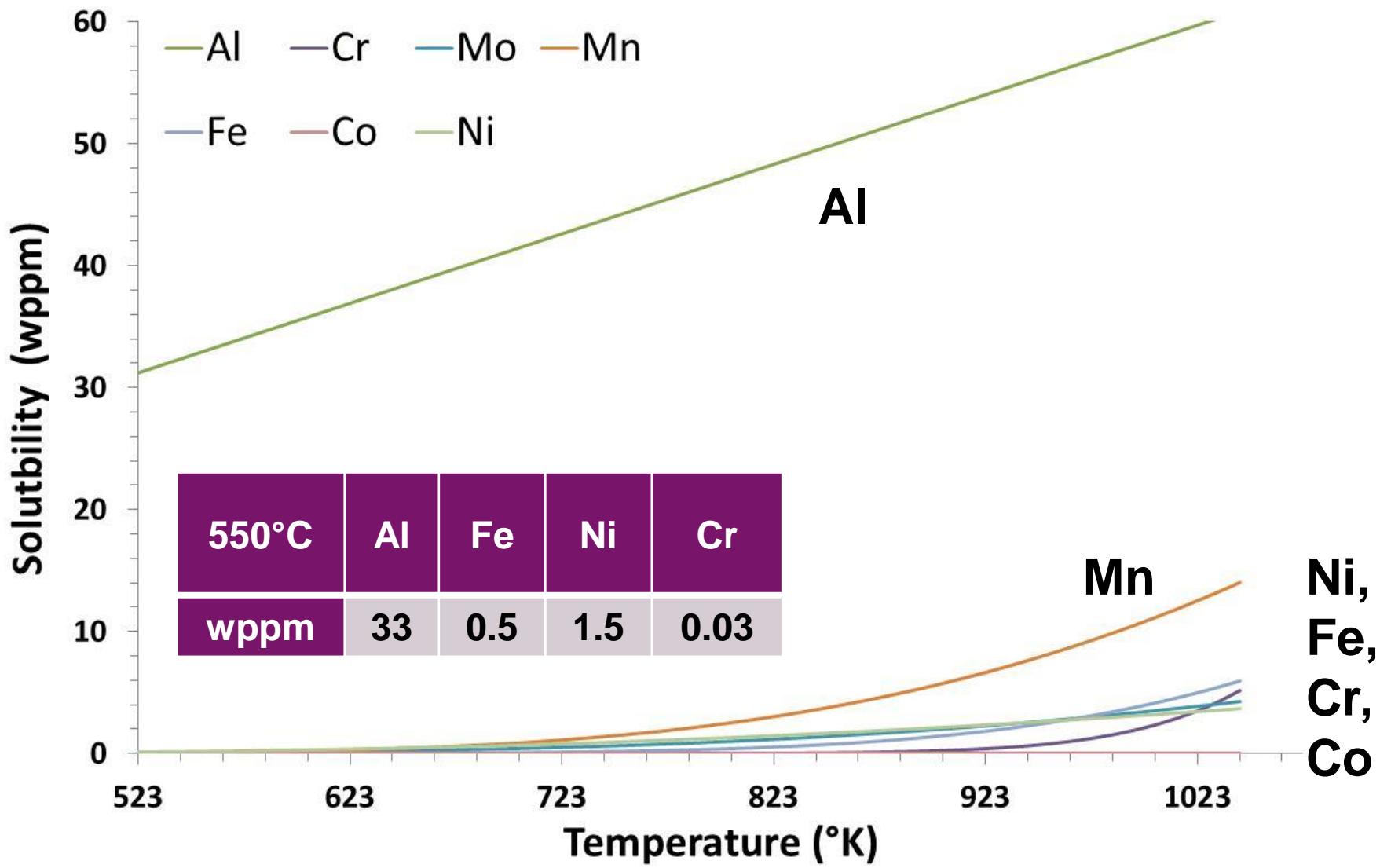
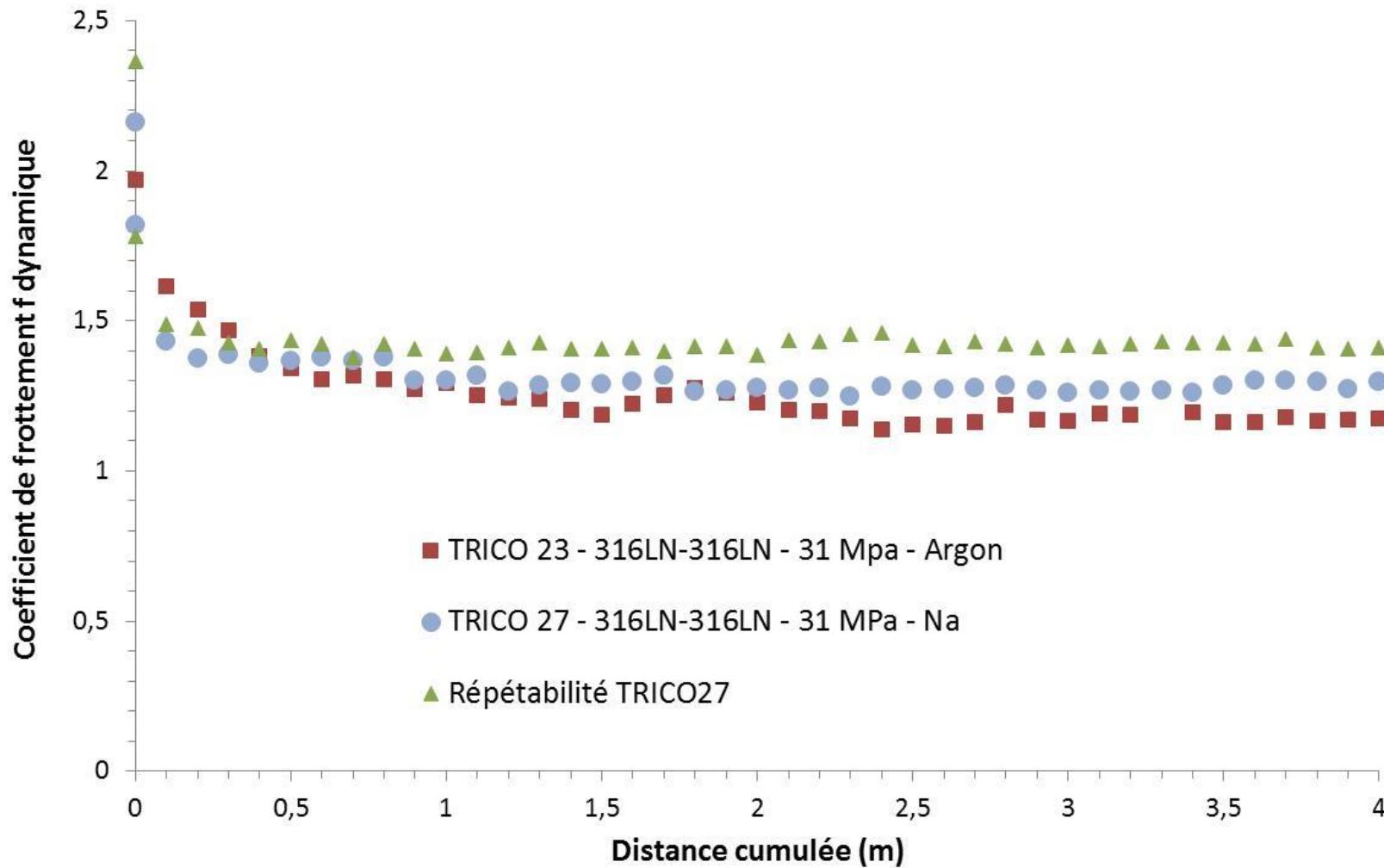


Diagramme thermos du binaire Cr-Al
A 550°C, solubilité de Al = 11.6 wt% puis formation de AlCr₂

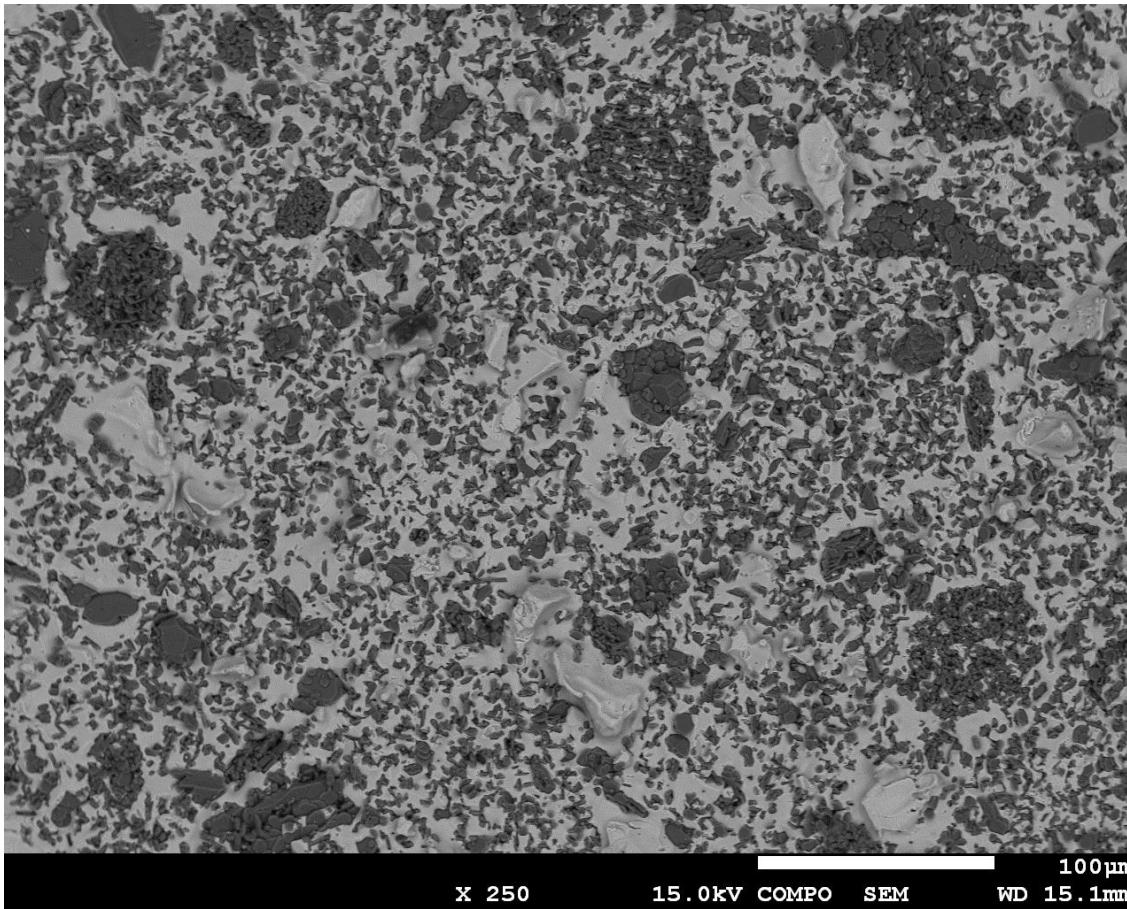




[Applied chemistry of the alkali metals, Borgstedt, 1987]



316L(N) – as received - PC



X 250

15.0kV COMPO SEM

100 μm

WD 15.1mm



Thank You for your attention



More information :
www.portail-fluides-supercritiques.com

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