ALFRED

FALCON

Qualification programme of candidate materials for ALFRED

TM on Structural Materials for Heavy Liquid Metal Cooled Fast Reactors

IAEA, Vienna , 15-17 October, 2019

ENEA

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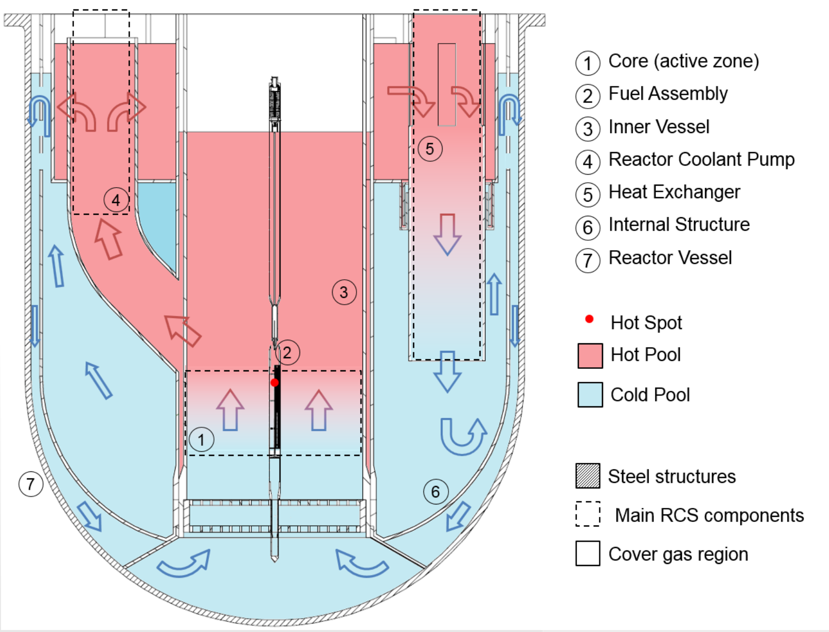
ALFRED "Staged Operation"

The operation of ALFRED will be based on a stepwise approach:

- 1st stage: operation at **low power** in **low-temperature** range
  - Currently existing proven materials (bare steels) working in **O-containing Pb coolant**
- nth stage: operation at **full power** in **high-temperature** range
  - Advanced materials or protective measures fully qualified during earlier stages

Parameter	Stage 1	Stage 2	Stage 3
Core inlet T (°C)	390	400	400
Core outlet T (°C)	430	480	520
Hot Spot T (°C)	450	535	600
Core Power (MWth)	100	200	300

Temperature and C_o influences the corrosion resistance of materials as well as mechanical properties in Pb environment



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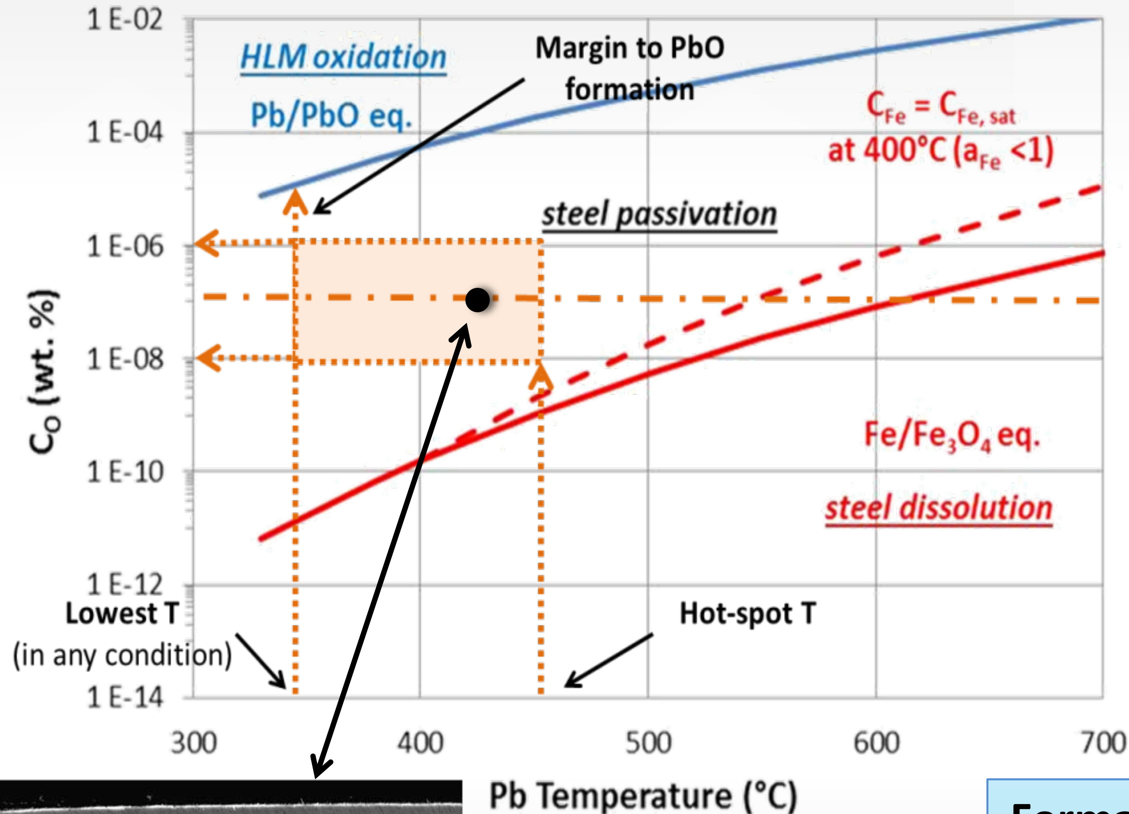
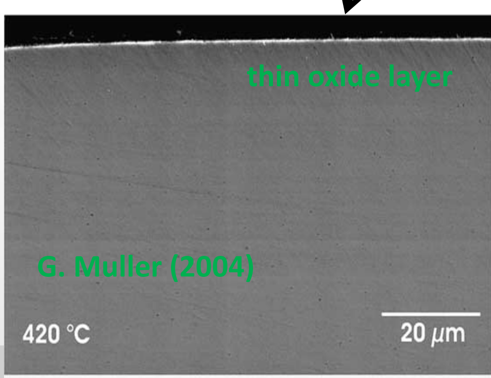
Impact of temperature (1st stage)





Thin protective oxide layer

316L steel in flowing LBE, 420°C, high C_O, 2000 h.

- ✓ Data in LBE considered;
- ✓ Lack of corrosion data in Pb;
- ✓ Need for steels qualification in Pb at high and low T.

Formation of oxide film on steels via Oxygen Control
T range = 390-430°C
T Hot Spot = 450°C
$C_O = 10^{-6} - 10^{-8}$ % wt.

Impact of temperature (n^{th} stage)

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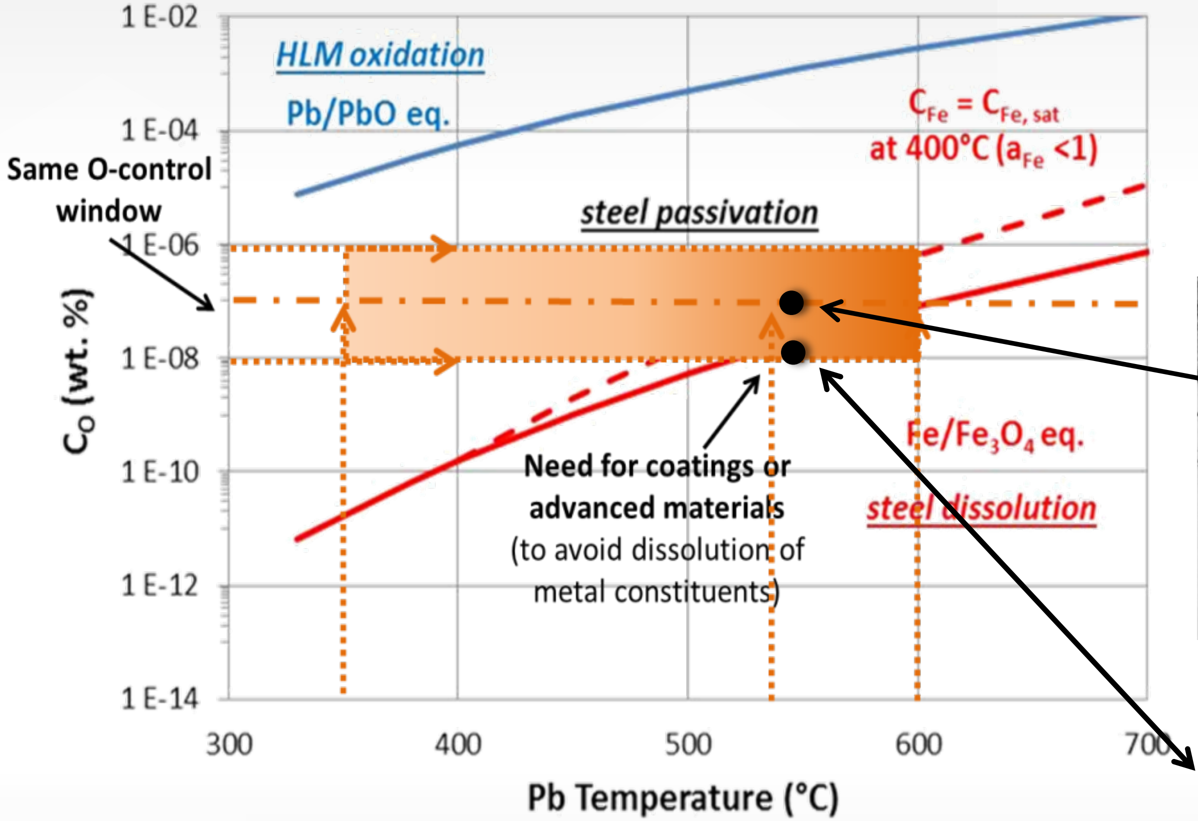
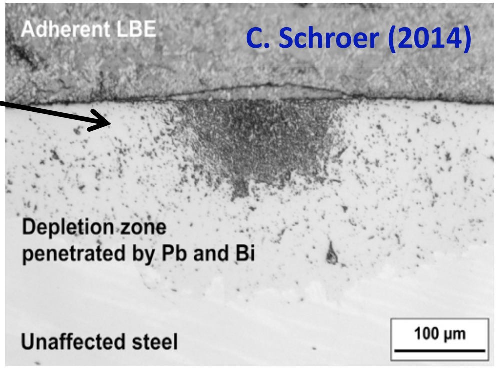


316L steel in flowing LBE, 550◦C, high C_O, 7500 h.

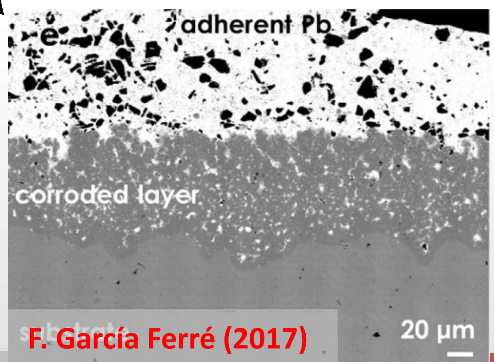
Oxidation + Ni/Cr dissolution (γ Fe → α Fe), Pb penetration



Oxide film via Oxygen Control not effective for T > 450-480°C
Need for coatings or advanced materials

15-15Ti steel in static Pb, 550◦C, low C_O, 4000 h.

Ni/Cr dissolution (γ Fe → α Fe), Pb penetration, no oxidation



Candidate materials ($C_O = 10^{-6} - 10^{-8}$ wt. %)

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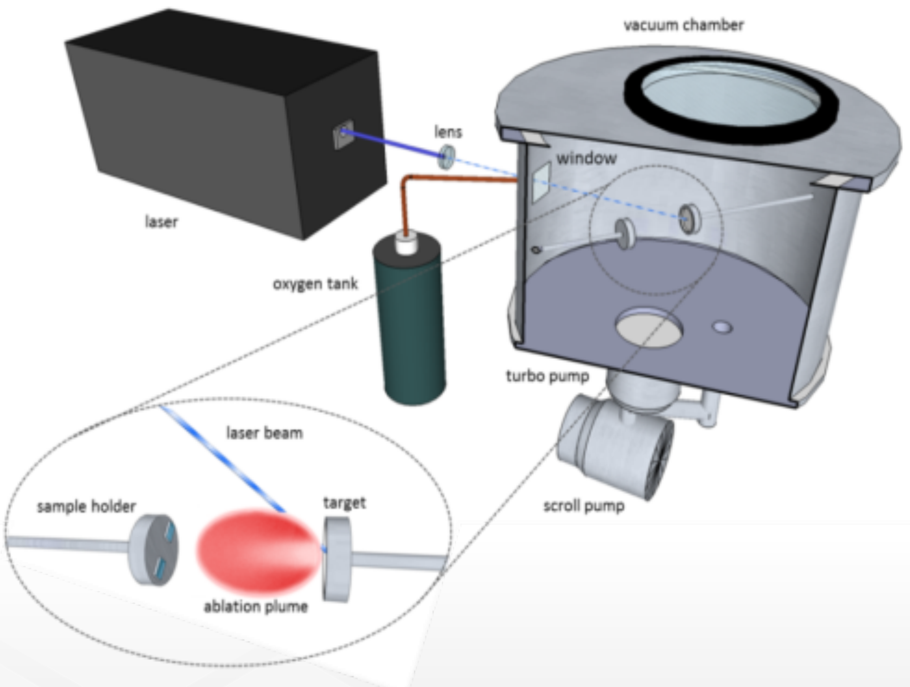
Component	N.O. Temp. (0) (°C)	Acc. Max Temp. (0) (°C)	Max. Lead vel. (m/s)	Max. Rad. damage (dpa)	Material (early stages with $C_O = 10^{-6}$-10^{-8} w%)	Material and/or Coating (later stages)
Fuel cladding	390-450/600	550/800	2	100	15-15Ti 20% CW (AIM1)	Al_2O_3 by PLD
FA Structures	390-430/540	500/700	2	100	15-15Ti 20% CW (AIM1)	Al_2O_3 by PLD/ALD
Internal structures	390-430/520	500/700	1.5	<2	AISI 316LN (ASTM)	FeCrAl diff. coating, or AFA steel
Steam Generator	390-430/520	500/700	0.9	0.01	AISI 316LN (ASTM)	FeCrAl diff. coating, or AFA steel
DHR Heat Exchanger	390-430/520	500/700	0.2	0.01	AISI 316L (ASTM) 15-15Ti (DIN 1.4970)	FeCrAl diff. coating, or AFA steel
Primary Pumps (impeller)	390-430/520	500/700	10÷20	0.01	AISI 300 series	FeCrAl diff. coating, or AlTiN coating
Reactor Vessel	390/400	420/430	0.1	<<2	AISI 316LN (ASTM)	O-control (or Al_2O_3 by D-Gun)

(0) Min. Temperature – Max. Temperature (early stages)/Max. Temperature (later stages)

PLD-Al_2O_3 coating

Capability to coat tubes and open surface (fuel cladding & fuel assembly)

amorphous alumina with nano-crystalline inclusions (by IIT)

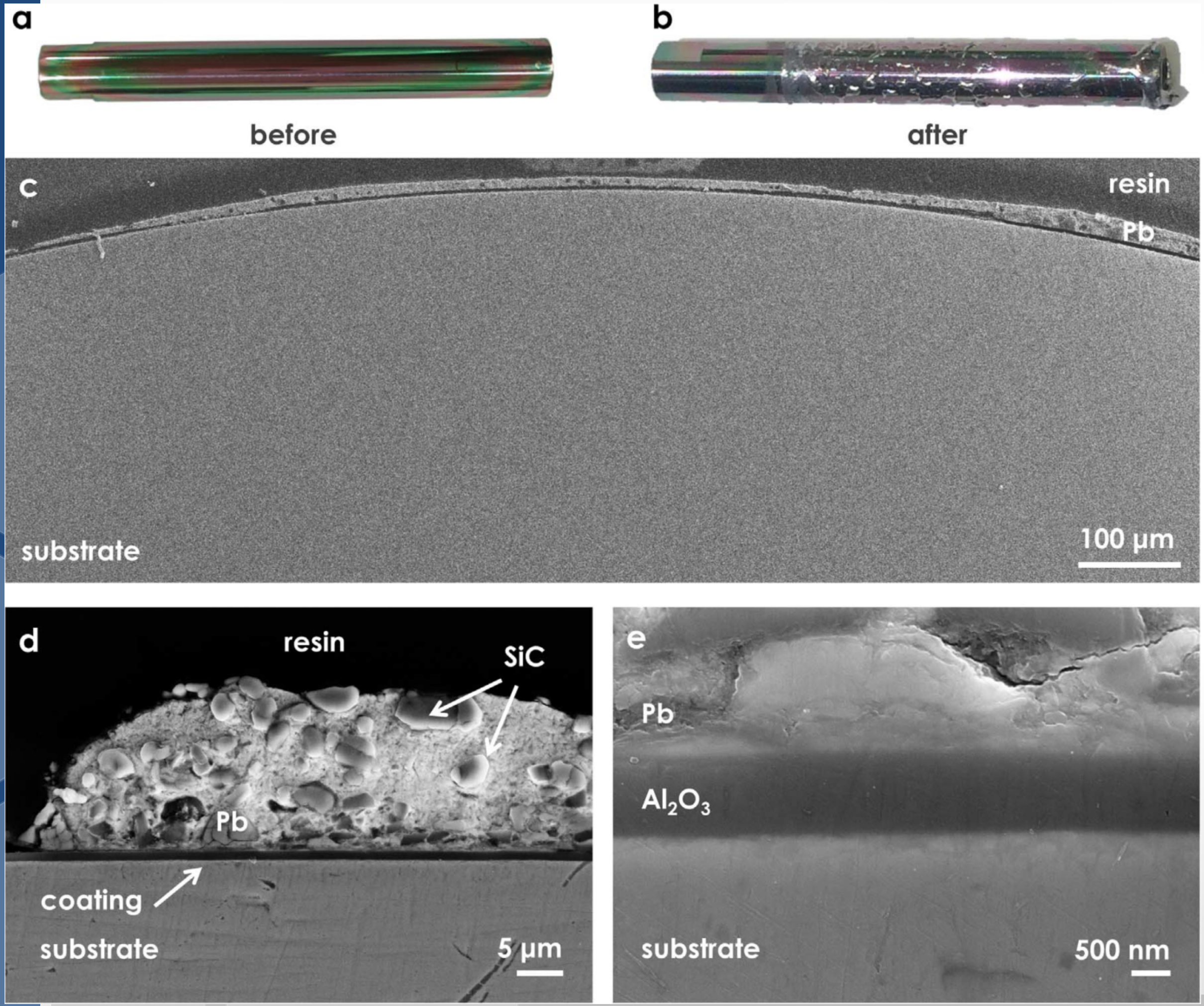


Pulsed Laser Deposition

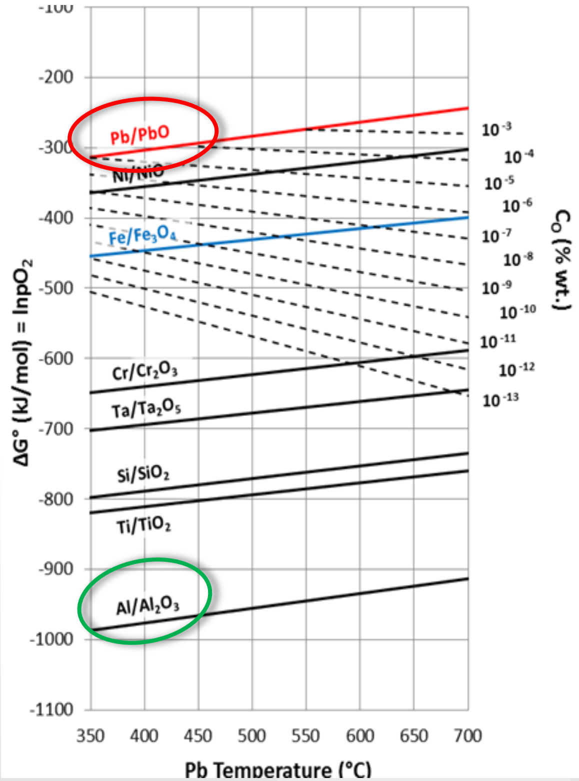
- ✓ quasi-metal mechanical behaviour: properties similar to that of steel substrate;
- ✓ room T process: possibility to coat cold-worked steels, i.e. AIM-1;
- ✓ anti-diffusion and anti-permeation capability against several gases (e.g. H_2 and D_2): particularly relevant for tritium confinement, for either fission- or fusion-based systems;
- ✓ high radiation tolerance: Al_2O_3 films have been irradiated with heavy ions up to 450 dpa, showing neither cracking, nor delamination.

PLD-Al_2O_3 coating – Pb corrosion

Test in static Pb, 550°C, 4000h, low oxygen 10^{-8} % wt.
(F. Garcia Ferré at al., 2017)



- ✓ No Pb corrosion, negligible wetting by Pb;
- ✓ Al_2O_3 stable in low oxygen Pb/LBE.



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FeCrAl diffusion layer

Capability to protect complex geometry of components such as HX and DHR

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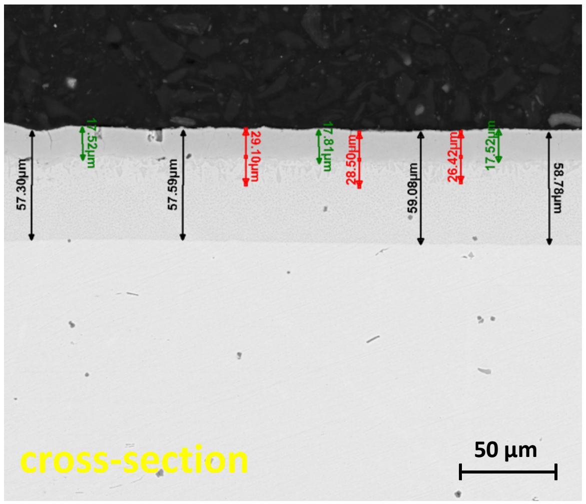
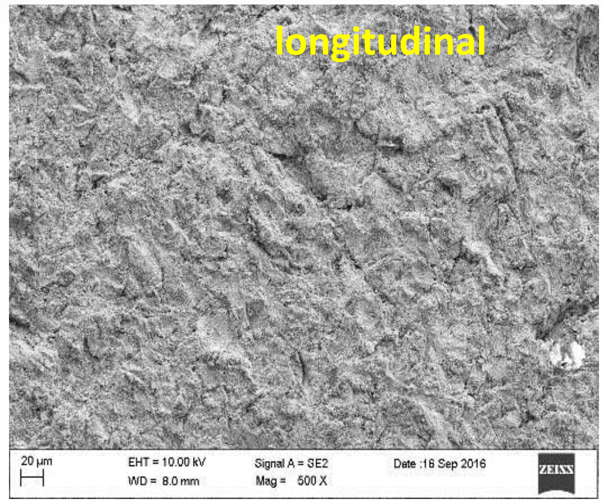
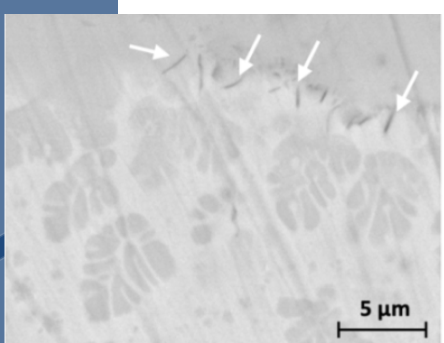
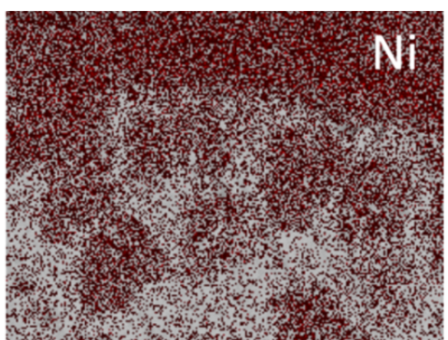
FeCrAl layer via Pack Cementation on 15-15Ti (by RINA-CSM, Rome)

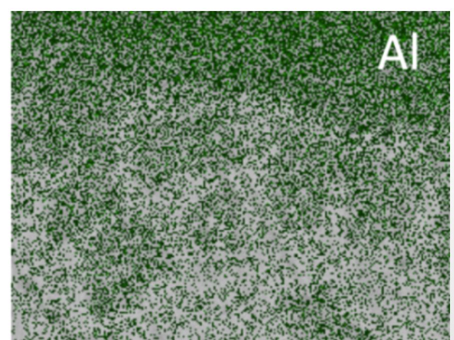




Two layer structure: internal layer 59 µm (11 % at. Al) + outer layer 17 µm (47 % at. Al, FeAl phase).
Between the two layer: Ni-rich areas.





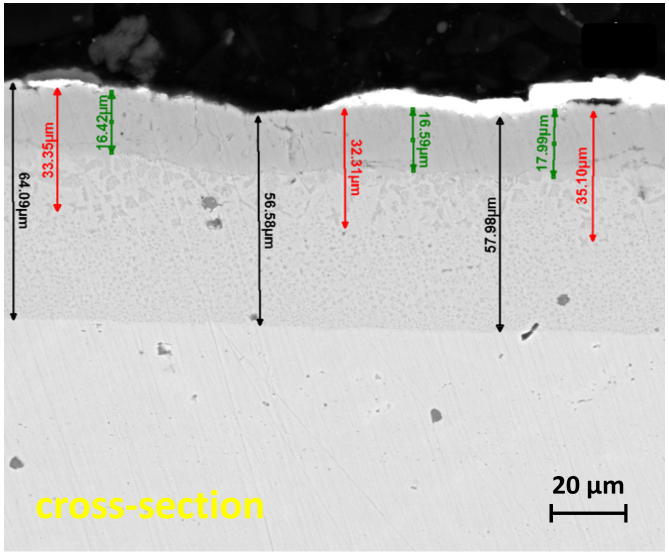


Presence of precipitates:

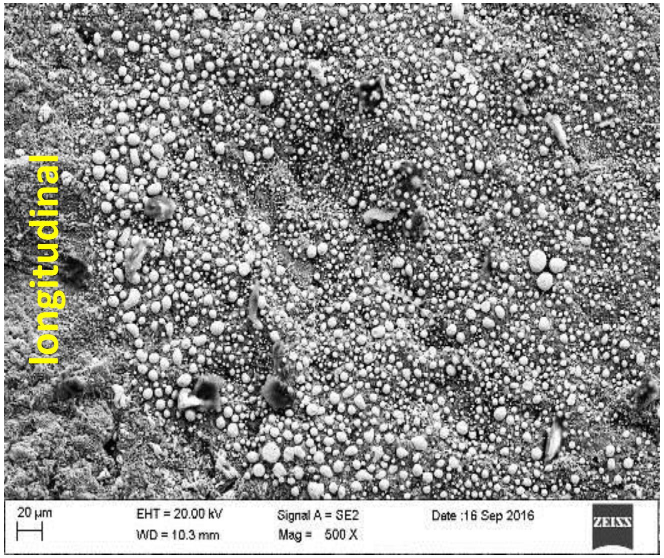
- ✓ black needles (white arrows) underneath the outer layer and ascribable to AlN;
- ✓ flower-like structures of AlNi, mainly concentrated near the interface between the two layers.

FeCrAl diffusion layer – Pb corrosion

FeCrAl layer by pack cementation after exposure in static Pb at 550°C, 1500h, low oxygen 10^{-8} % wt.



thickness of the layer conserved, no dissolution



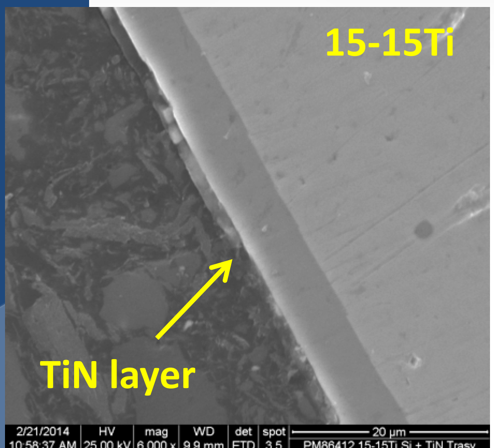
low wetting: Pb droplets isolated on the surface

Need for industrial methods to coat large components in large furnace

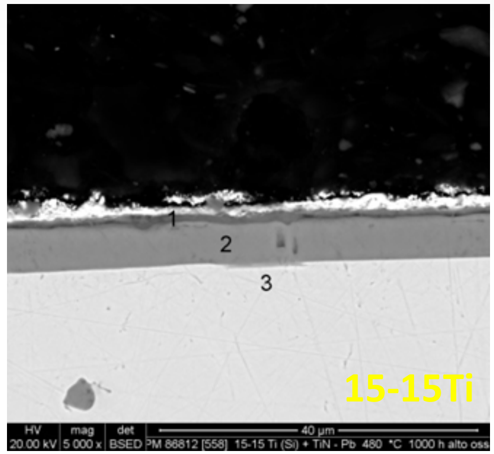
AlTiN coating by PVD

Considered for pump impellers protection for wear resistance and hardness

as-dep



after exposure in Pb at 480°C, 1000h, oxygen $3 \cdot 10^{-4}$ % wt.



% wt.	1	2	3
N	13.6	31.8	-
O	41.0	-	-
Si	-	-	0.8
Ti	42.1	67.8	1.9
Cr	-	-	14.1
Mn	-	-	1.6
Fe	0.4	0.4	64.8
Ni	-	-	15.1
Mo	-	-	2.0
Pb	2.9	-	-

oxidation of the layer (T_{ox} < 500°C in air from literature)

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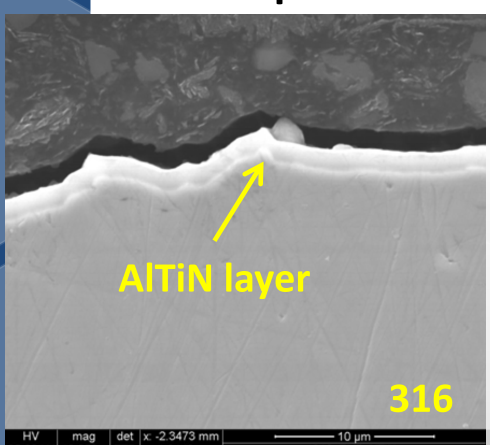
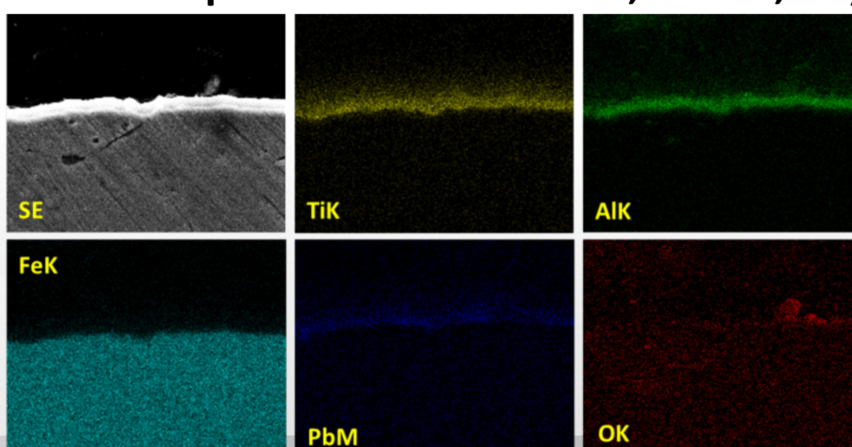
as-dep



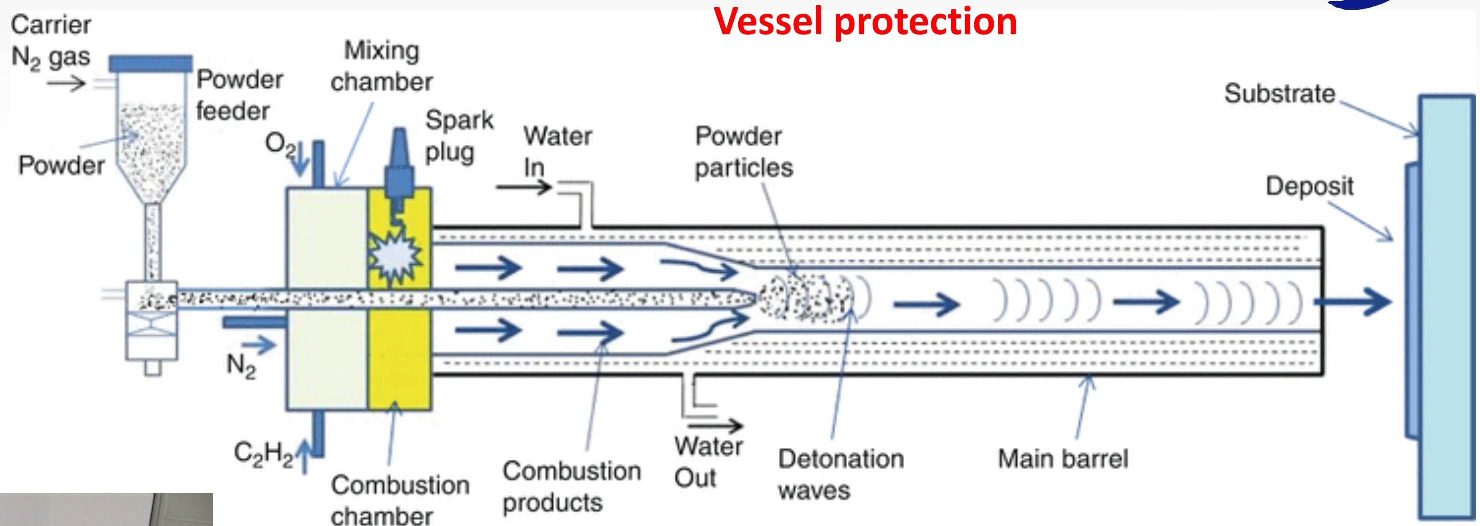
after exposure in Pb at 450°C, 1000h, oxygen $2 \cdot 10^{-4}$ % wt.



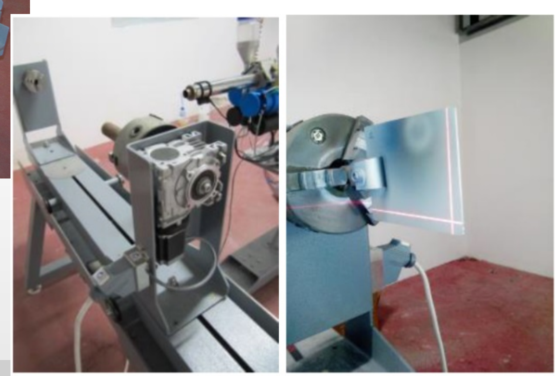
No oxidation observed (T_{ox} > 700°C in air from literature)

Al_2O_3 coating by D-Gun

Considered for Reatcor Vessel protection



D-Gun set-up in ENEA Brasimone



1) The combustion mixture O_2 + N_2 + C_2H_2 feed through tubular barrel.
2) Simultaneously, the coating powder is feed into combustion chamber.
3) Detonation reaction (ignited by spark plug) accelerates the coating particles up to 1200 m/s above the substrate.
4) The high pressure of the powders on the substrate produces a coating with good adhesive strength, low porosity and compressive residual stresses.

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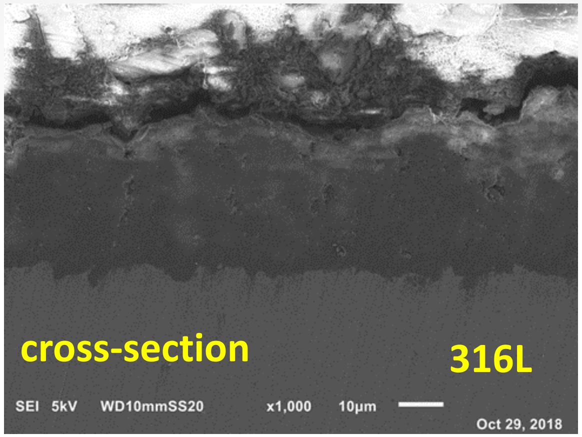
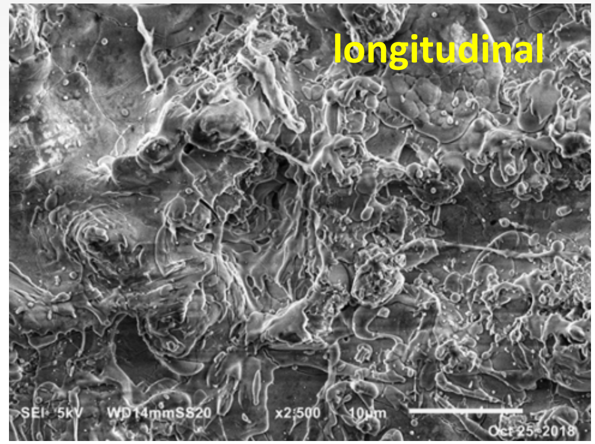
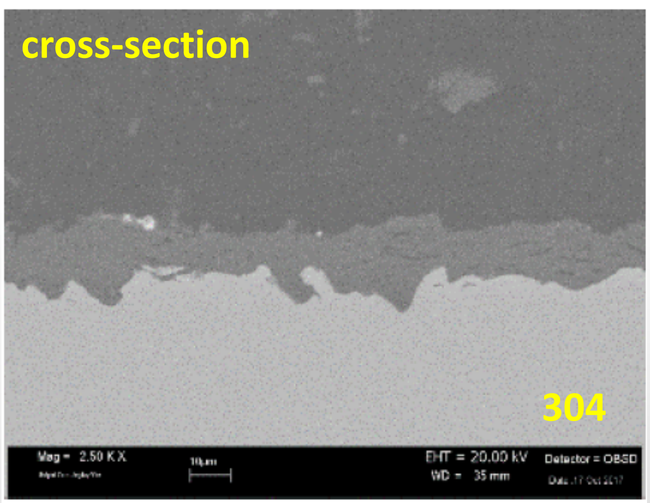
Al_2O_3 by D-Gun – Pb corrosion

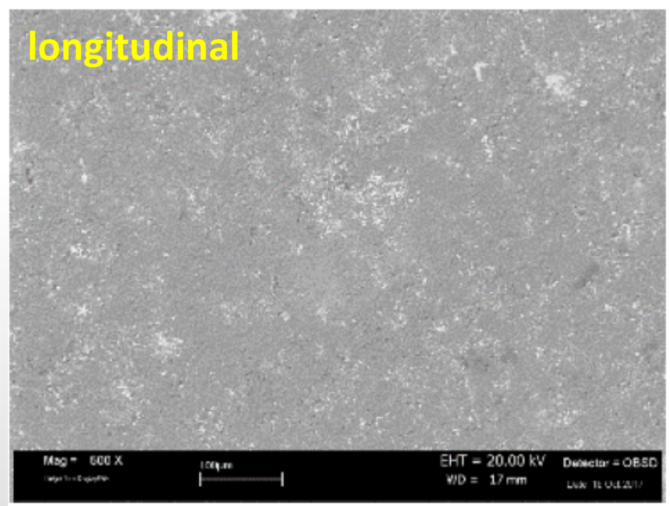




No wettability, no detachment of the layer, no damage

Preliminary exposure test: Al_2O_3 layer in Pb at 480°C, low oxygen, 190h





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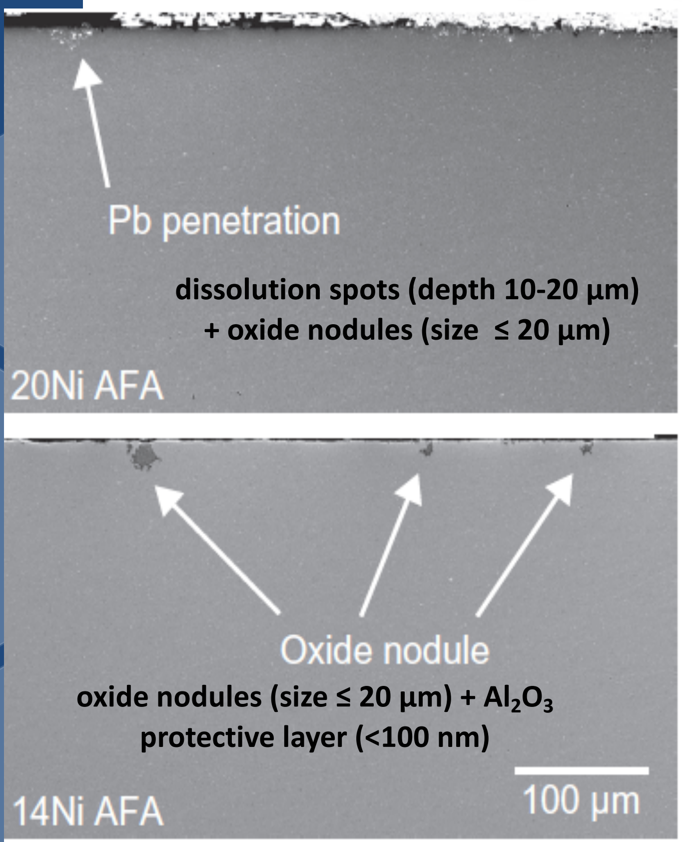
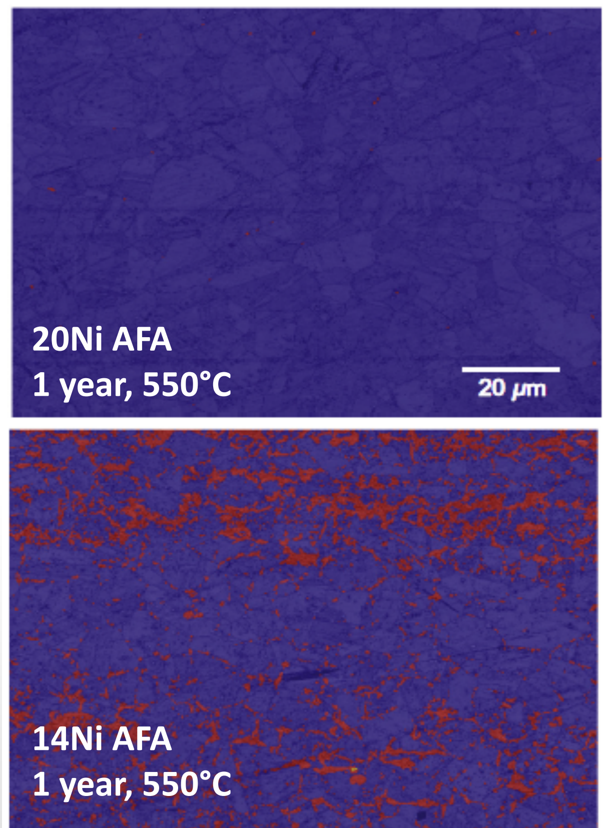
AFA steels – General consideration

Exposure in Pb, 550°C, 8760h, $C_o = 10^{-7}$ % wt, KTH data (J. Ejenstam et al., 2015)

Alloy	Fe	Cr	Ni	Al	Mn	Mo	Si	Nb	C
20Ni AFA	Bal.	14.2	19.7	2.47	1.64	2.43	0.18	0.87	0.08
14Ni AFA	Bal.	14.4	13.9	2.49	1.60	2.50	0.19	0.89	0.08





- ✓ Good creep properties;
- ✓ Potentiality as corrosion-resistant materials in Pb/LBE (formation of thin Al_2O_3 layer);
- ✓ Need for balance of Al, Cr, Ni % in the composition.

Austenite (FCC)
Ferrite (BCC)

Austenite (FCC) → Ferrite (BCC) (ageing effect)

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AFA low Ni

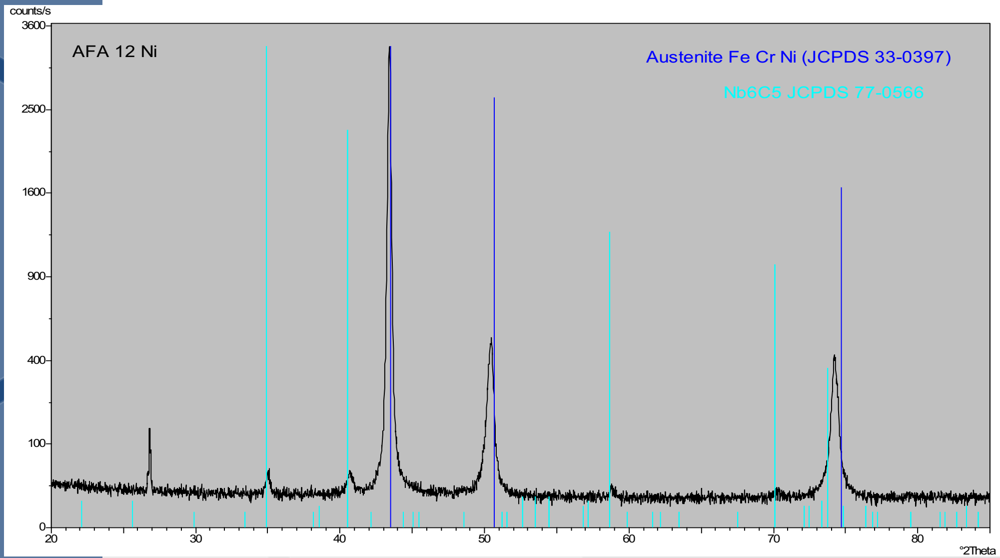
AFA considered as bulk material for component under low neutron flux such as HX, DHR

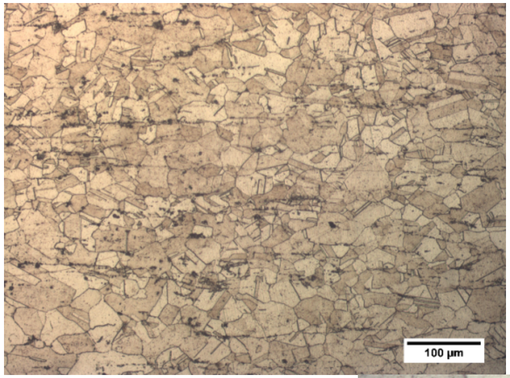
AFA	C	Cr	Ni	Mn	Al	Cu	Nb	Si	V	Ti	B
12Ni (OC-Q)	0.2	14	12	4	2.5	3	0.6	0.15	0.05	0.05	0.01
25NI (OC-E)	0.2	14	25	2	4	0.5	2.5	0.15	0.05	0.05	0.01

Plates from ORNL obtained by VIM-VAR followed by homogenization, hot-rolling and annealing; R_a= 0.02-0.04 µm

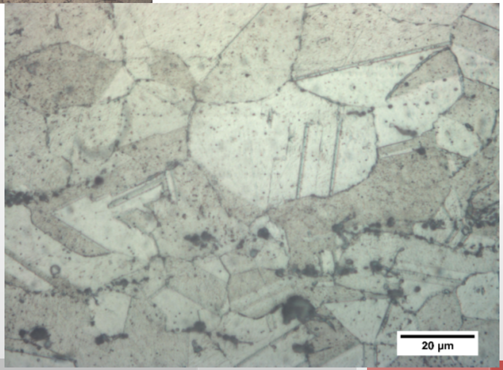
Pre-test characterization AFA 12Ni (OC-Q)

XRD: austenite + Nb carbides (0.6% Nb, 0.2% C)



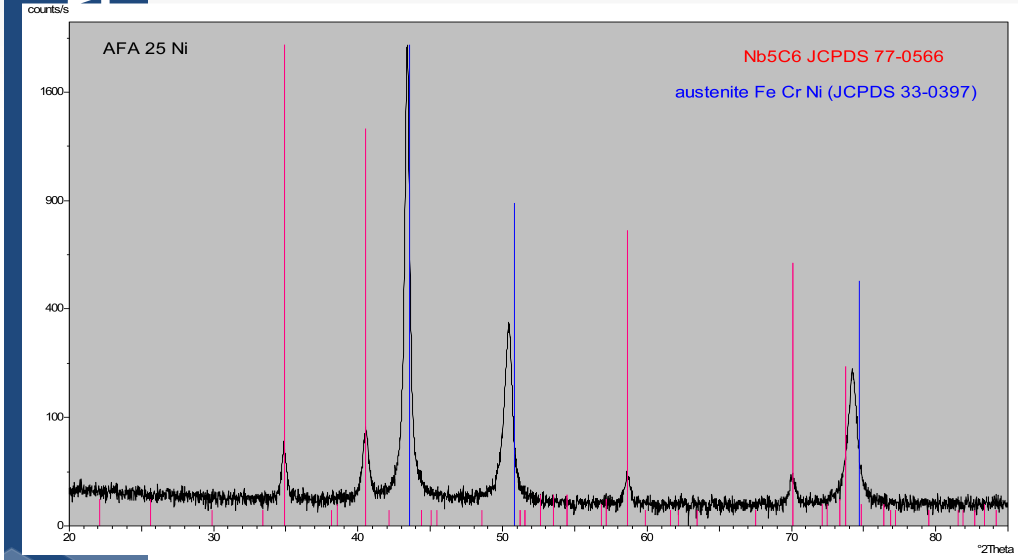


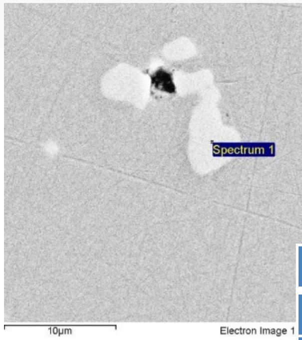
Etching + OM: austenite twins + Nb carbides (black spots)



AFA High Ni

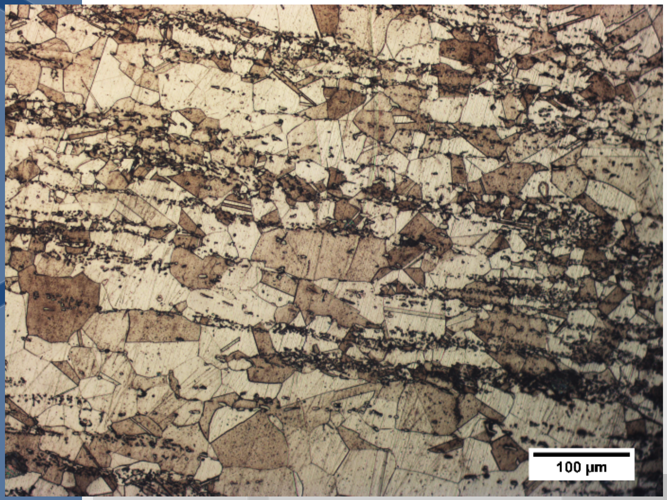
Pre-test characterization AFA 25Ni (OC-E)

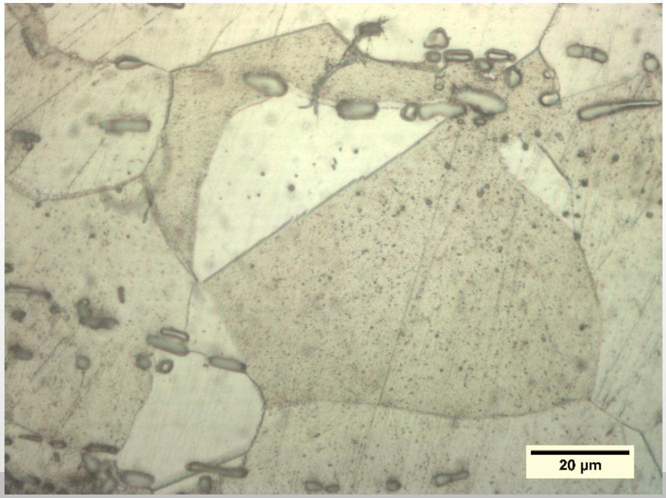




Element	Atomic %
C K	63.5
Ti K	0.9
Fe K	1.4
Nb L	34.2
Total	100

XRD: austenite + Nb carbides (2.5% Nb, 0.2% C)





Etching + OM: larger twin grains and greater amount of Nb carbides than in AFA 12Ni

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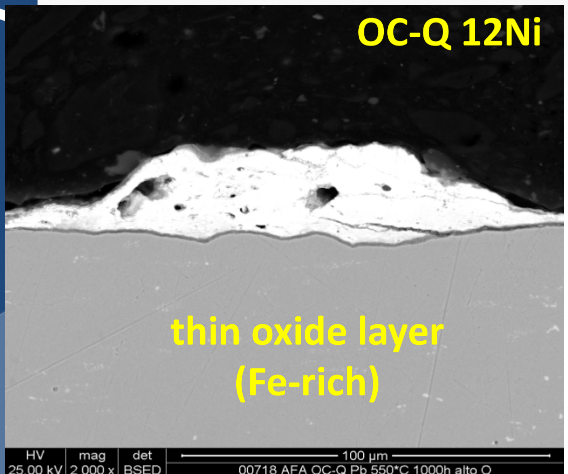
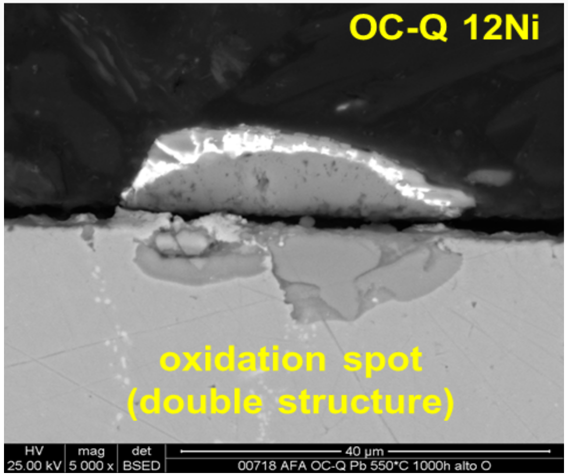
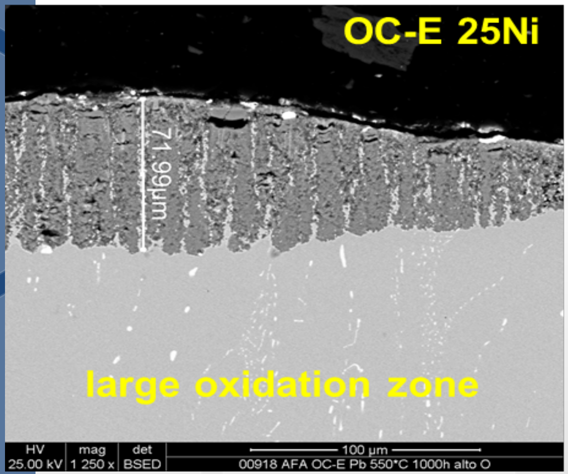
AFA – Pb corrosion (1)

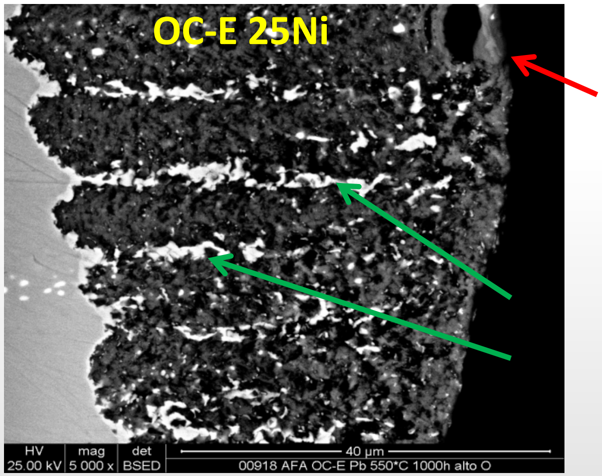
Test in static Pb at 550°C, 1000h, high oxygen 10^{-3} % wt.





- ✓ Thin and not protective oxide layer 0.5-1 µm thick on irregular surface
- ✓ Oxidation spots (depth up to 20 µm) with double structure (internal Fe-Cr-Ni-Mn-Al mixed oxide + external magnetite)

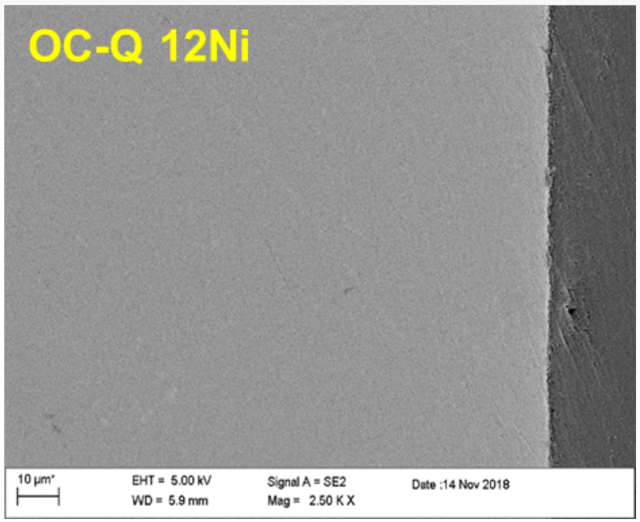


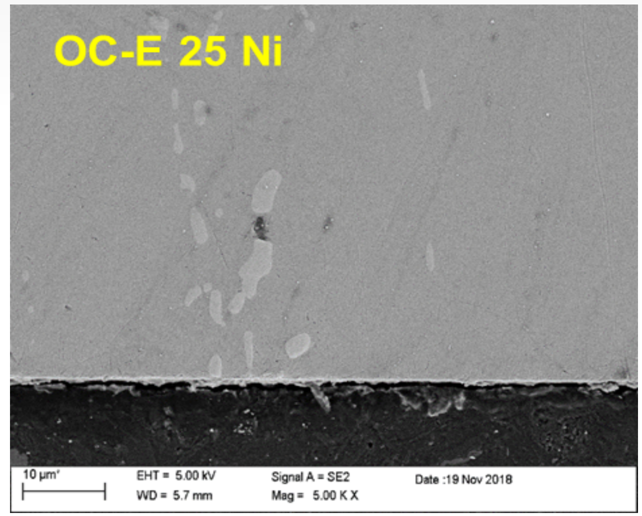


- ✓ Large and oxidation zones up to 70 µm depth: structure made of Fe-Cr-Ni-Mn-Al mixed oxide (dark area) and Ni-rich area (white zone)
- ✓ Presence also of oxidation spots with double strcuture

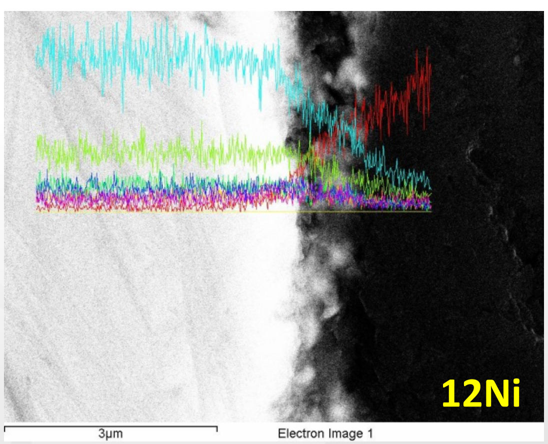
AFA – Pb corrosion (2)

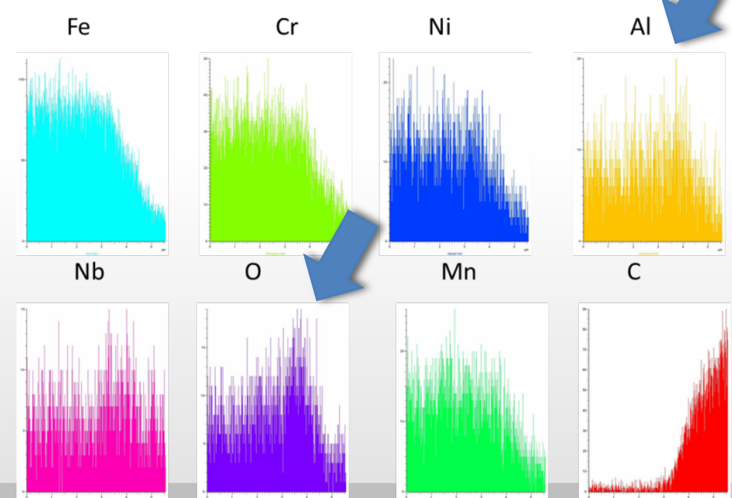
Test in static Pb at 550°C, 1000h, low oxygen 10^{-8} % wt.





- ✓ unaffected surface after test for AFA 12Ni and 25Ni
- ✓ Al_2O_3 layer not clearly visible (too thin) but Al, O peaks detected on EDX line-scan for 12Ni and 25Ni





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AFA – Ageing effect

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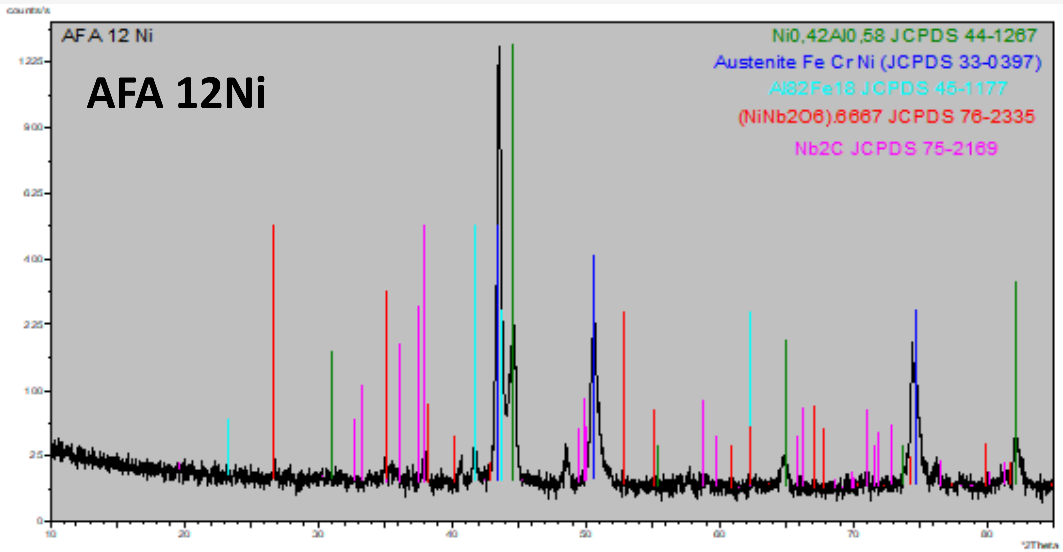
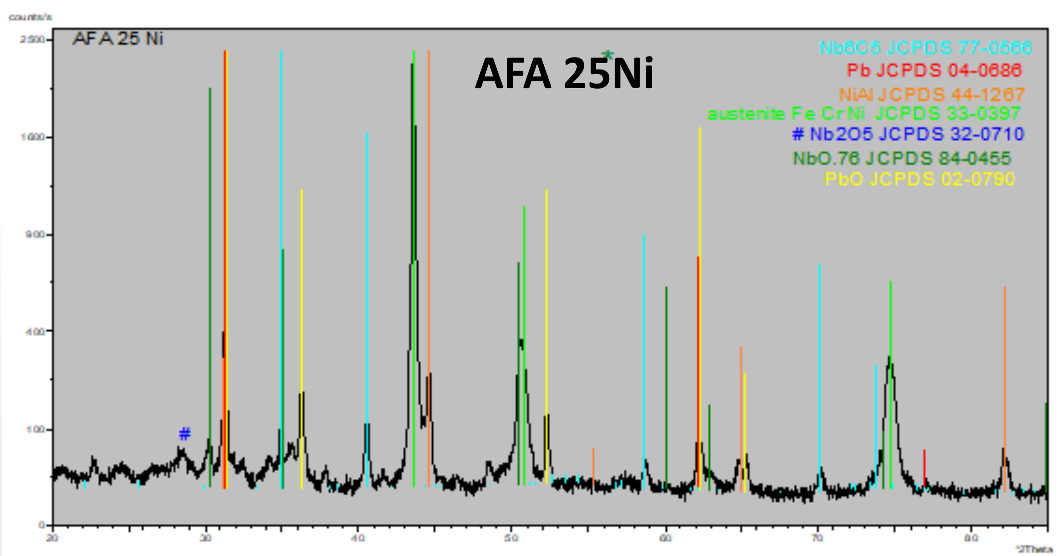
After exposure in Pb at 550°C, 1000h



- ✓ austenite phase predominant,
- ✓ formation of Ni_xAl_y
- ✓ formation Fe_xAl_y
- ✓ presence of Nb oxides

Austenite (FCC) → Ferrite (BCC)
(ageing effect)



- ✓ austenite phase predominant
- ✓ formation of Ni_xAl_y
- ✓ presence of Nb oxides

Conclusions

Steels, coatings and advanced materials were identified for the various stages of ALFRED operation. Development and qualification of coatings and advanced materials ongoing for the later stages:

- **Al_2O_3 layer by PLD** is the reference for **fuel cladding structures**, demonstrating excellent corrosion resistance in liquid Pb, good mechanical properties (similar to those of steel substrates) and irradiation resistance with heavy ions;
- **FeCrAl layer by aluminizing** such as pack cementation is considered to coat **HX, DHR and other complex components**, demonstrating very good corrosion resistance in liquid Pb;
- **Al_2O_3 layer by D-Gun** is potentially considered for **reactor vessel**, even if O-control should be sufficient to protect the component in all the operational stages;
- **AlTiN coating by PVD** is considered for the protection of **pump impellers** thanks to industrial availability and high wear/hardness properties. AlTiN is more resistant than TiN to high temperature oxidation in liquid Pb;
- **AFA steel** combines creep properties and corrosion resistance in liquid Pb, and could be used for components such as **HX and DHR** (low neutron flux).

R&D needs & future activities

Preliminary characterization was performed but long-term qualification in liquid Pb under relevant condition of T, Co and fluid-dynamic regime as well as investigation of the mechanical behavior should be carried out. Most of these activities will be performed in the frame of H2020 GEMMA project.

- **Al_2O_3 coating by PLD** will be qualified in static and flowing Pb in long-term exposures (H2020 GEMMA project);
- **FeCrAl layer by industrial company** is necessary to assess the suitability for complex components and it will be qualified in static and flowing Pb in long-term exposures (H2020 GEMMA project);
- **Al_2O_3 coating by D-Gun** will be qualified in static and flowing Pb in long-term exposures (H2020 GEMMA project);
- **AlTiN coating by PVD** needs to be tested with long-term tests and in relevant conditions of Pb fluid-dynamics (impeller prototypes to be tested);
- **AFA steels** from ORNL will be exposed at longer exposure times in Pb. Other tests on different AFA materials developed by KIT/KTH will be performed in static and flowing Pb in long-term exposures (H2020 GEMMA project);
- **Bare steels (15-15Ti AIM-1 and 316L with welds)** will be qualified at longer exposure times in static and flowing Pb (H2020 GEMMA project).

ALFRED

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