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Influence of deposition conditions on the microstructure of Al-based coatings for applications as corrosion and anti-permeation barrier

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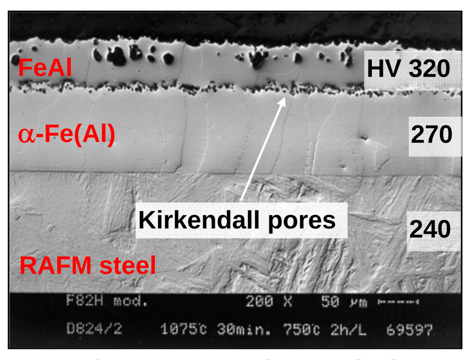
Motivation



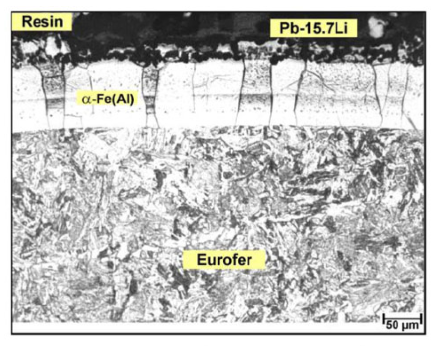
Reference scale made by HDA after heat treatment (1040°C/0.5 h + 750°C /1 h)

Previous research revealed that the application of aluminum-based barriers is suitable to minimize corrosion rates of Eurofer steel in Pb-15.7Li (picture to the right) and tritium-permeation from the liquid breeder into the cooling system (HCLL) in an envisaged future fusion reactor.

Developed deposition techniques to electroplate aluminum are based on water-free electrolytes. Either ECA or ECX process can provide several advantages compared to the established Hot-Dip Aluminization (HDA) process:

- Better thickness distribution
- Controllable coating thicknesses
- Reduced amounts of aluminum applicable (low activation)

However, the main challenge is to convert the electroplated Al-coatings during a subsequent heat treatment, to enable the formation of protective scales on Eurofer for applications as corrosion and anti-permeation barriers in Pb-15.7Li.

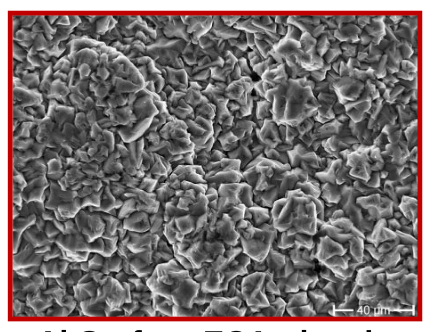


HDA coated sample after exposure in flowing Pb-15.7Li, taken from [1]

Electrodeposition of Aluminum

Aluminum is highly electronegative (E_0= -1,6 V vs. NHE)

- Al cannot be deposited from aqueous electrolytes
- Water-free / aprotic electrolytes are needed



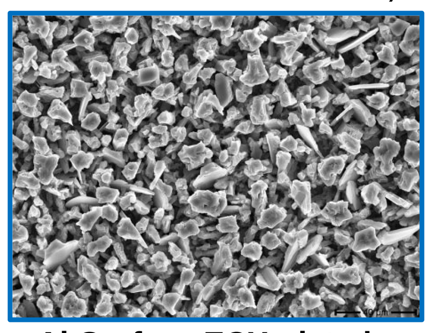
Al-Surface ECA-plated
10 mA/cm², 1 h, 100°C

Principle types of electrolytes to electrodeposit Al at moderate temperatures (RT-100°C)

1. Toluene-based electrolytes → ECA process
2. Ionic liquids → ECX process

Electrochemical processes have in common

- No reaction with the substrate material
- Adjustable Al thickness (time, current density)
- Good adherence to the substrate
- Industrial relevance is given

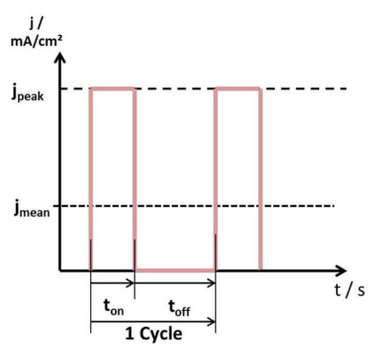


Al-Surface ECX-plated
20 mA/cm², 0.5 h, 100°C

Why ECX?

- Process is more flexible than ECA process
  - Deposition parameters adjustable in a wider range (T, j, pulse plating,…)
- Electrolytes not oxygen sensitive → non-inflammable

Electrodeposition of Al by ECX



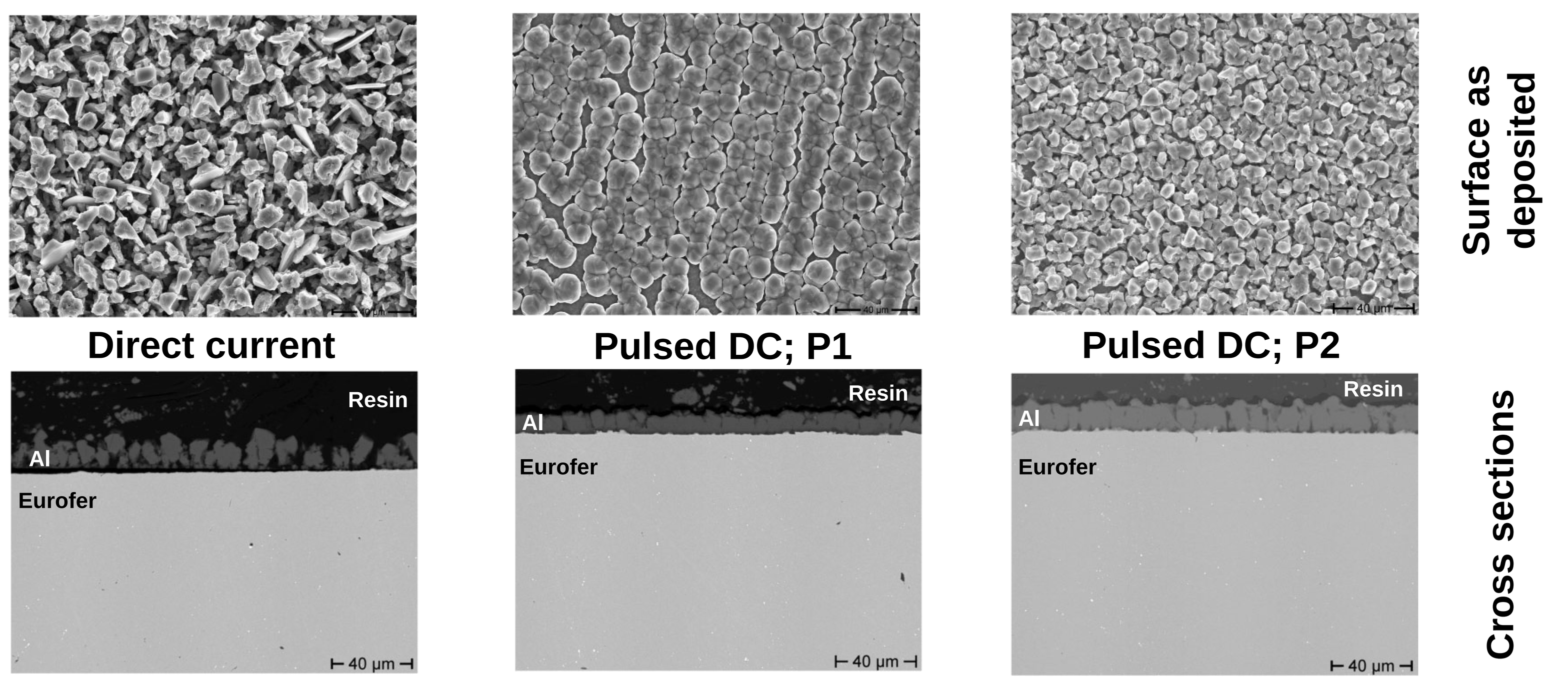
Scheme: Pulse plating

[Emim]Cl:AlCl$_3$;
T=100°C;
Agitation

- Electrodeposition of Al on Eurofer steel samples by ECX
  - From an imidazolium-based ionic liquid
- Direct current (DC) plated samples were compared to pulse plated (P) samples → j_{mean} and t were held constant
- Theoretical Al thickness should be about 12.5 µm

Parameter	DC	P1	P2
j_{mean}	20 mA/cm²		
j_{peak}	-	80 mA/cm²	25 mA/cm²
Duty Cycle	100%	25%	80%
t	30 minutes		

Influence of deposition parameters



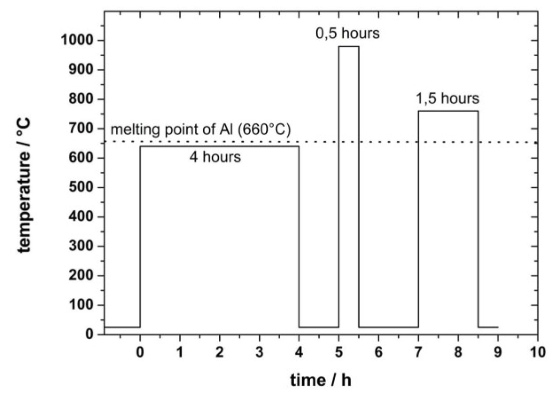
Surface morphology is strongly influenced by deposition conditions

- DC plated sample : Jagged and rough surface; crystallites not grown together
- Pulse plated samples: Dense, even and homogeneous morphology

Heat treatment behavior

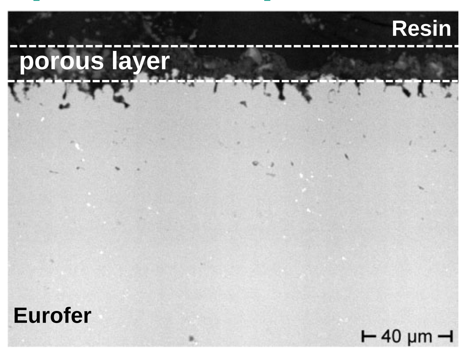
Experimental

- Heat treatment (HT) procedure according to [1]
  - 640°C, 4 h → 980°C, 0.5 h → 760°C, 1.5 h
- In flowing argon atmosphere
  - Reduction of oxidation effects
- Samples cooled down under flowing Ar

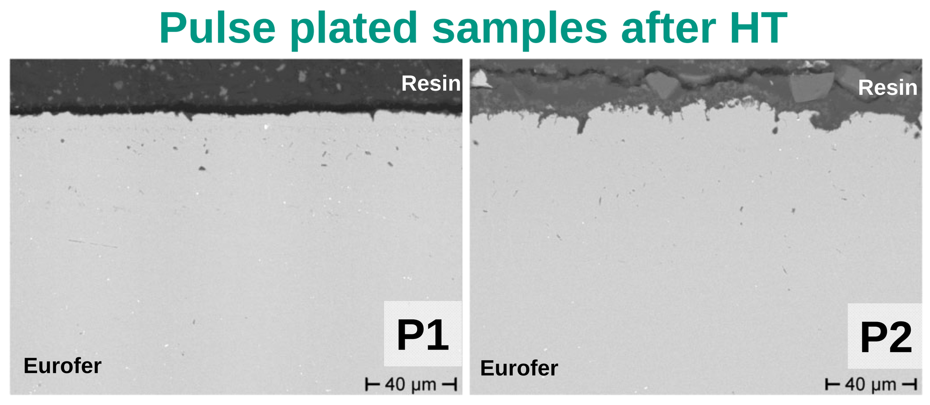


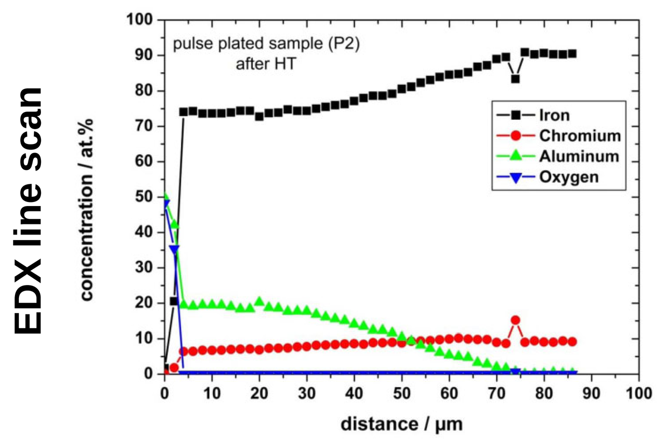
Scheme: Heat treatment

DC plated sample after HT



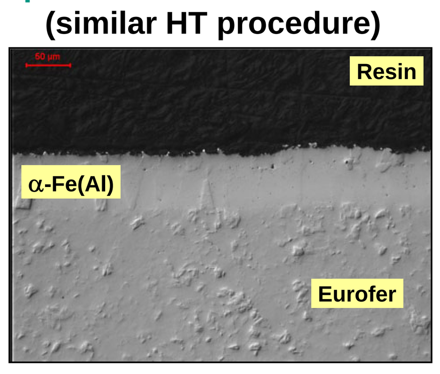
Pulse plated samples after HT





Pulse plated (P2) sample after HT

For comparison: heat treated ECA sample (similar HT procedure)



picture taken from [1]

Results

- Quality depends on surface morphology of Al-layers on Eurofer after electroplating
- Pulse plated samples by ECX: uniform formation of Al-based scales, comparable to scales produced by ECA (see [1])
- Rough and jagged surface structure (in this case DC plated): formation of relatively thick porous, oxygen containing layer on top of the sample
- Al amount in the reacting zone of ca. 20 at.% (decreasing with distance from surface)

Conclusions

ECX process show industrial relevance and deposit Al scales on Eurofer steel samples with controllable and reproducible thickness

- Morphologies of Al coatings made by ECX process depend on deposition parameters
- Al layers have to be converted into protective scales by heat treatments (HT)
- HT behavior depends on the morphology of the coating produced by ECX
  - Formation of Fe-Al scales during HT is influenced by morphology
  - Rough and jagged surface structures cannot be recommended for further HT
  - Even, dense and homogeneous morphologies are recommended for HT
- Heat treated samples with recommended morphology (pulse plated) were comparable to known ECA or HDA produced Al-based scales
- The more flexible ECX process has proven to be a promising alternative to ECA

References:
[1] Konys, J. et al.: Impact of heat treatment on surface chemistry of Al-coated Eurofer for application as anti-corrosion and T permeation barriers in flowing Pb-15.7 environment, Fus. Eng. Des., 87 (2012), 1483.