Impact of pretreatment conditions on defect formation during the fabrication of Al-based corrosion barriers by ECX process

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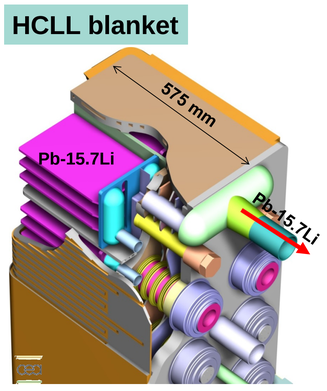
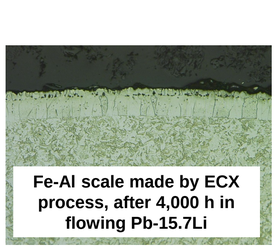
Motivation

HCLL blanket



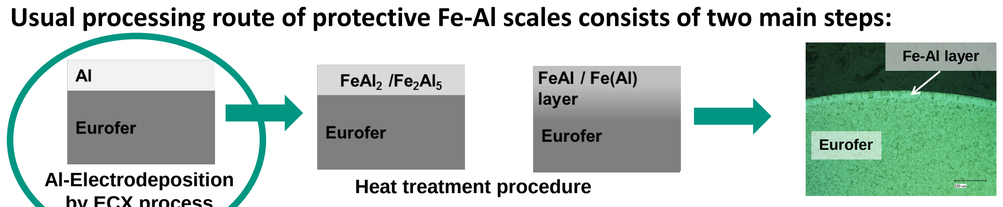
- In different blanket designs (HCLL, DCLL), reduced-activation ferritic-martensitic steels (RAFM) are supposed to be in direct contact with flowing Pb-15.7Li (breeder). Unfortunately, RAFM steels, e.g. Eurofer, suffer from strong corrosion attack in Pb-15.7Li due to dissolution.
- Fe-Al scales made by different coating techniques, e.g. hot-dipping aluminization (HDA), and electrochemical aluminization (ECA, ECX process) proved to protect Eurofer steel from corrosion for high exposure times in flowing Pb-15.7Li at temperatures up to 550°C.
- However, reliable pretreatment processes are needed; especially prior to electrodeposition of aluminum in non-aqueous electrolytes for electrochemical processes to ensure sufficient aluminum coating qualities and to obtain optimized protective Fe-Al coatings.
- Until now, only scarce data and experiences exist on the influence of pretreatment processes especially with respect to electroplating of aluminum on RAFM steels such as Eurofer.


Fe-Al scale made by ECX process, after 4,000 h in flowing Pb-15.7Li

Experimental procedure

General comments

Usual processing route of protective Fe-Al scales consists of two main steps:



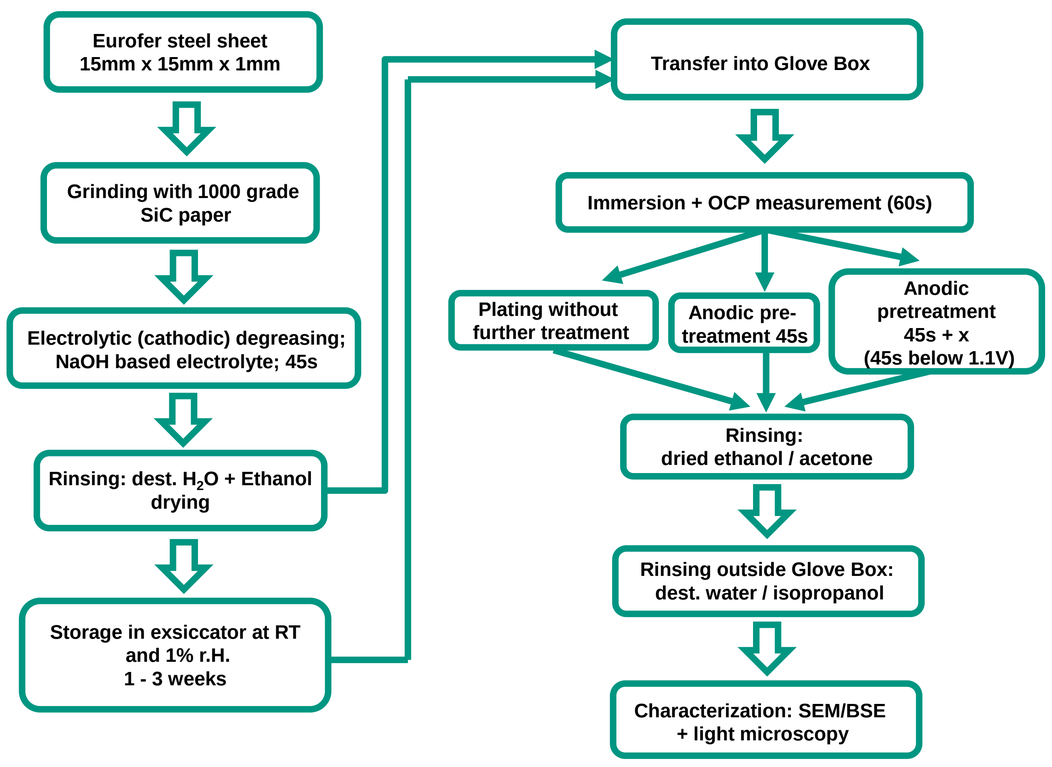
ECX process parameter

- [Emim]Cl:$AlCl_3$ as electrolyte for Al
- Deposition rates up to 25 µm/h
- Process temperature: <100°C
- Usually pulse plating is used

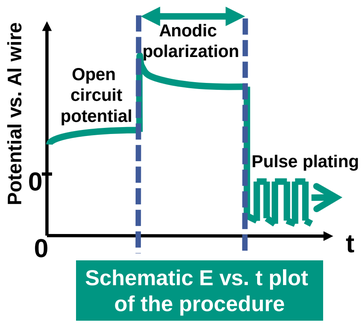
Reliable electrodeposition mandatory for subsequent production steps → Reliable substrate pretreatment needed

- Avoidance of failures/ coverage
- Good adhesion recommended

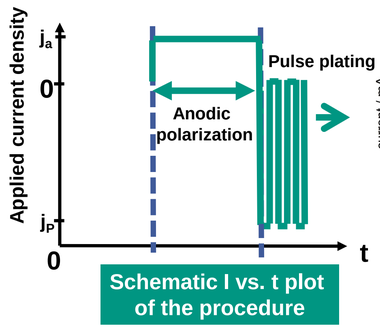
Procedure



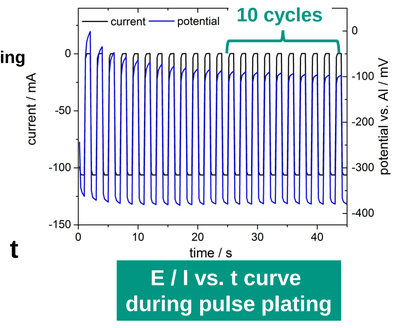
Electrochemical control and electrodeposition procedure



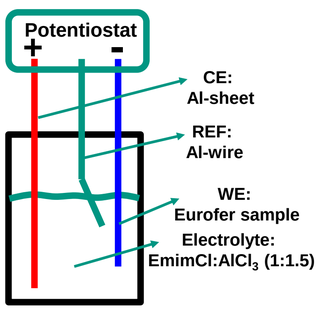
Schematic E vs. t plot of the procedure



Schematic I vs. t plot of the procedure



E / I vs. t curve during pulse plating

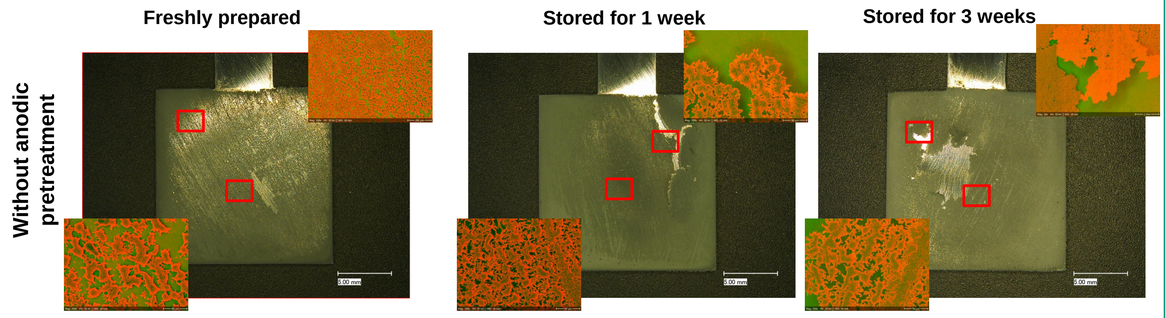


Experimental setup

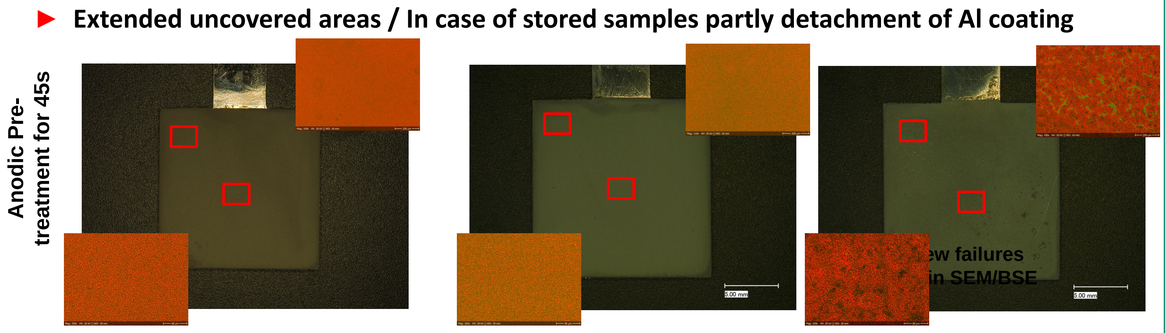
- Three electrode setup: Working electrode (WE), Counter electrode (CE), Quasi-reference electrode (REF)
- Control via IVIUM Potentiostat
- Volume of electrolyte: 500 ml
- Process temperature: 100°C +/- 1°C
- No agitation
- Pulse current density: 35 mA/cm²
- Deposition time: 34.5 min (2070 cycles)

Results

Impact of anodic pretreatment and storage duration

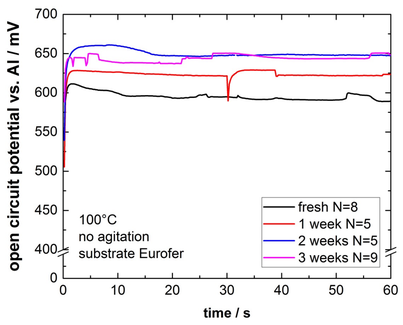


- Extended uncovered areas / In case of stored samples partly detachment of Al coating



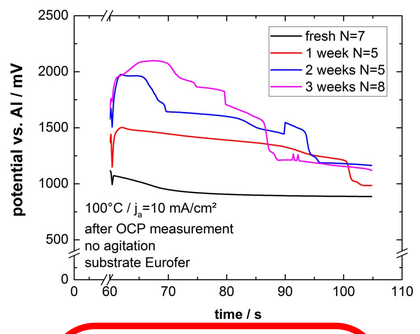
- Improved adhesion of the deposited Al coating on Eurofer / No failures in case of freshly prepared samples
- No / strongly reduced coating failures in case of stored samples (dependency on storage time)

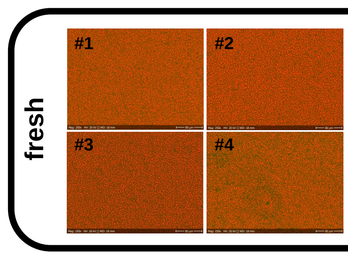
Influence on open circuit potential (OCP) vs. Al-wire reference



- OCP of Eurofer specimens vs. REF between approx. 580mV and 650 mV
- Dependency of measured OCP on the storage time
- Indication of at least partial natural surface passivation/oxidation with increasing time between preparation and experiment → Insufficient adhesion between Eurofer and electrodeposited Al layer

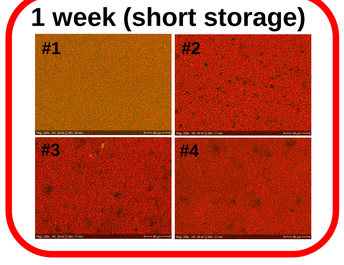
Electrochemical anodic behavior and coating properties





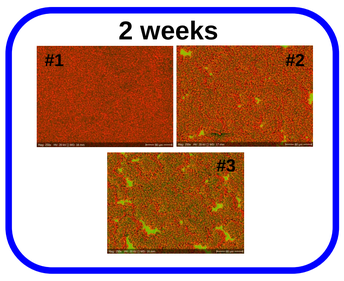
- Active surface
- → low potential
- Good coverage
- Good adhesion
- Good reproducibility

1 week (short storage)

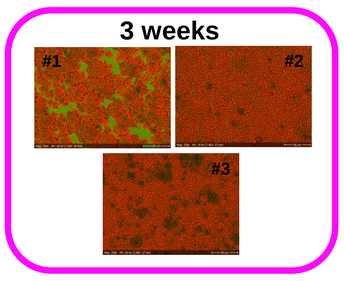


- Active surface after anodic pretreatment / good coverage
- Good adhesion
- Acceptable reproducibility

2 weeks



3 weeks



- Surface not totally active after anodic pretreatment
- No crystallization on passive sites → increasing formation of uncovered areas → failures
- Poor reproducibility

Conclusions

The investigations showed that a sufficient pretreatment of Eurofer substrate is needed to produce reliable quality of the electrodeposited Al layer by ECX process and later on of the heat-treated Fe-Al barriers.

- Only mechanical pretreatment (grinding) is not sufficient.
- Anodic polarization /dissolution as additional pretreatment step increased the reliability of the coating quality.
- Time span between sample preparation and deposition influences surface activation achieved by anodic polarization → Storage above 1 week decreases reliability.
- Control of potential during anodization shows good correlation with coating quality.

Acknowledgment

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