Karlsruhe Institute of Technology

Institute for Applied Materials
Material Process Technology (IAM-WPT)

Corrosion barriers processed by Al-electroplating and their resistance against flowing Pb-15.7Li

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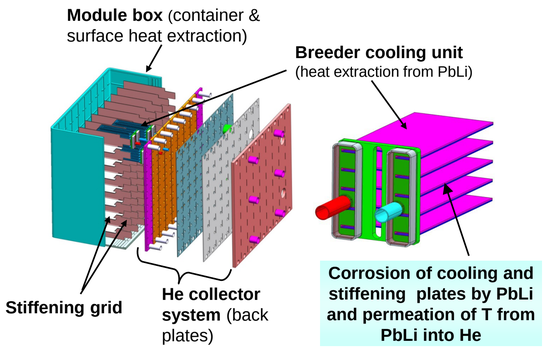
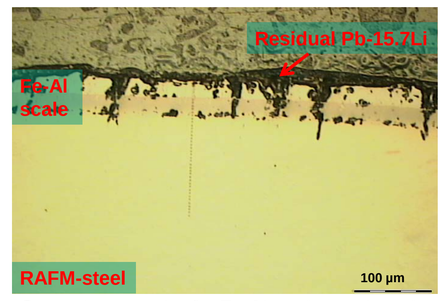
Motivation



- Reduced activation ferritic-martensitic steels (RAFM) are considered as main structural material in future fusion systems, e.g. envisaged in the He-cooled liquid lead (HCLL) design. Thereby, these steels will be in direct contact with the breeding material Pb-15.7Li, which lead to strong corrosion attack of the structural material.
- Aluminum rich Fe-Al scales on RAFM steels are considered as promising corrosion barriers, which also offer T permeation reduction properties.
- Scales produced by Hot-dipping aluminization (HDA) exhibited some disadvantages in the past, and therefore electrochemical processes (ECA, ECX) were introduced, but have lack concerning corrosion protection data in flowing Pb-15.7Li.



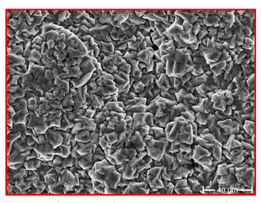
Corrosion attack of a Fe-Al scale produced by HDA after exposure to flowing Pb-15.7Li

Electrochemical Al-deposition

- Due to its high electronegativity (E_0 = -1.7 V vs. NHE) aluminum cannot be electrodeposited from common and well understood water-based electrolytes
  - Non-aqueous electrolytes are required for Al-deposition
- Two different deposition processes were used for the production of test samples for corrosion testing in PICOLO loop (1st series: ECA, currently started 2nd series: ECX)
  - Substrate: cylindrical Eurofer samples (diameter: 8 mm)

ECA

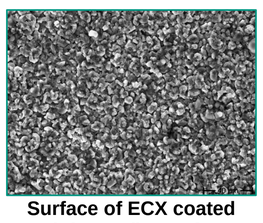
- Toluene based electrolytes
- Applied current density 10 mA/cm²
- Deposition rate: 10-12 µm/h
- Process temperature: 100°C
- Al thickness on Eurofer test samples: ca. 20 µm


Surface of ECA coated (20 µm Al) Eurofer

ECX

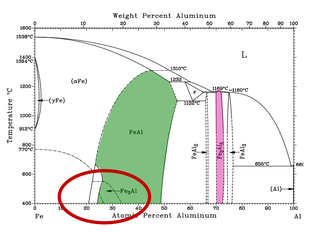
- Based on ionic liquids, in this study [Emim]Cl
- Pulse plating possible
- Mean current density 20 mA/cm²
- Deposition rate: 25 µm/h
- Process temperature: 100°C
- Al thickness on Eurofer test samples: ca. 13 µm
- Al thickness easily adjustable


Eurofer corrosion test sample coated by ECX

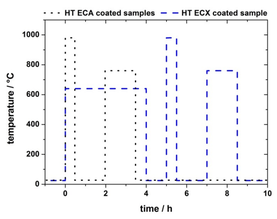

Surface of ECX coated (13 µm Al) Eurofer

Scale formation by heat treatment

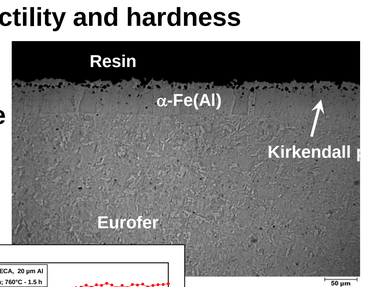
- Al-coated test samples need subsequent heat treating to convert the pure aluminum layers into the desired Fe-Al scales, responsible for the barrier properties
- FeAl and α-Fe(Al) are the preferred phases, due to their ductility and hardness comparable to Eurofer steel
- 1st series coated by ECA: Two-step heat treating procedure
- 2nd series coated by ECX: Optimized three-step procedure

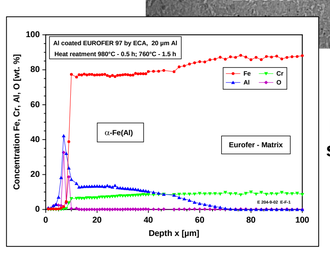


Fe-Al equilibrium phase diagram



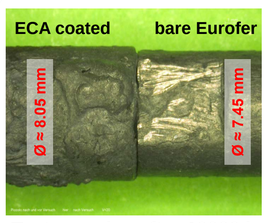
Heat treatment schemes



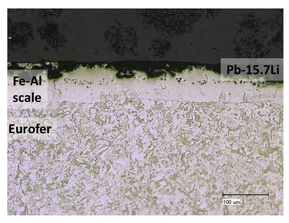


ECA coated Eurofer after two-step HT + EDX line scan after HT, taken from [1]

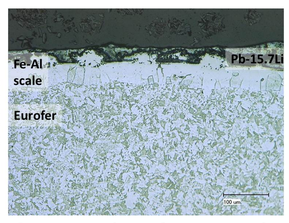
Corrosion resistance of ECA coated Eurofer samples



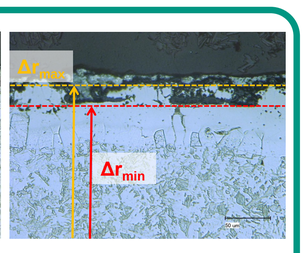
Test samples after 10000 h exposure to Pb-15.7Li

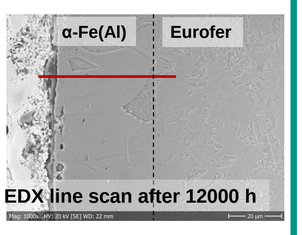


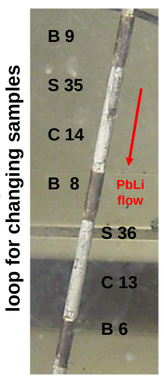
Cross section of test sample after 4000 hours



Cross section of test sample after 12000 hours

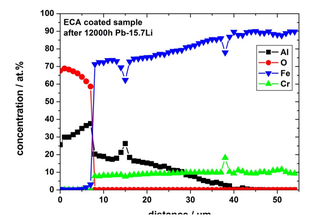




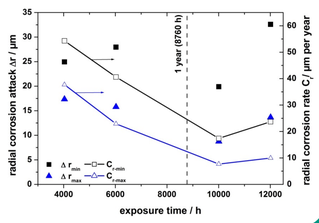


Stack of test samples removed from loop for changing samples

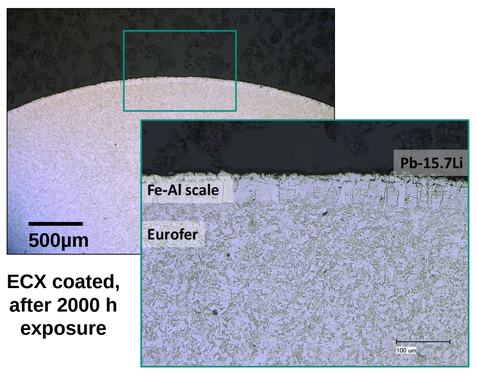
- Al based coatings made by ECA process protect Eurofer steel samples from corrosion attack in flowing Pb-15.7 Li for up to 12000 h
- Remaining scale thickness after 12000 h was above 35 µm
- Inhomogeneous corrosion attack Δr of the Fe-Al scale itself → "high" and "low" plateaus
  - Two values for radial material loss Δr_{max} / Δr_{min}
  - $\Delta r_{min/max} = r_{initial} - r_{min/max}$
- No clear dependency of material loss and exposure time was found (no control samples)



- Calculated radial corrosion rates were below 20 µm per year for long exposure times
  - At short times: Higher calculated corrosion rates
- Reduction of corrosion rate by a factor of >10 compared to bare Eurofer steel under the same conditions (Konys et al: 220 µm/a, reported at ICFRM-16, 2013)



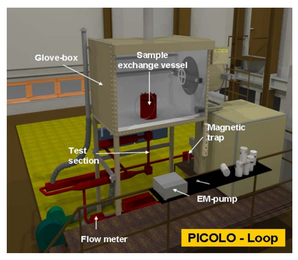
Outlook – First results from ECX coatings



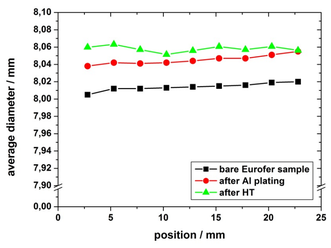
ECX coated, after 2000 h exposure

- First ECX coated Eurofer sample was removed from PICOLO loop after 2000 hours
- Sound, uniform Fe-Al scale of appr. 50 - 60 µm thickness observable in the cross section after 2000 hours of exposure to flowing Pb-15.7Li
- Radial material loss measured: <10 µm
- Corrosion resistance seems to be improved and a more uniform behavior exists compared to ECA coated Eurofer in flowing Pb-15.7Li

Corrosion testing



- Corrosion testing was performed in PICOLO loop
- Temperature in test section: 550°C
- Flow velocity of Pb-15.7Li: 0.1 m/s
- Exposure time: 1st series (ECA) up to 12000 hours
  2nd series (ECX) first results (2000 hours)
- Diameter of coated test samples were measured by a Laser scanning system prior to insertion of the samples in the loop
  → 9 vertical positions x 12 angles (0° to 330°)
- Average initial diameter at each position for corrosion attack calculation after exposure to Pb-15.7Li



Qualification of test samples before insertion to PICOLO loop

Conclusions

- ECA coated Eurofer samples showed high resistance against corrosion in fast flowing Pb-15.7Li (0.1m/s) at 550°C
- Corrosion protection proved for ECA samples for exposure times up to 12000 hours
- No accelerated corrosion attack observed up to 12000 hours
- Reduction of corrosion rate by a factor of >10 compared to bare Eurofer
- Nonuniform corrosion attack of the Fe-Al scale itself → not optimized surface structure
- 1st results for ECX (optimized surface, 2000 h in Pb-Li) showed improved properties
- Significant uniform corrosion attack of the Fe-Al scale itself compared to ECA samples
- Surface quality has significant impact on barrier stability

References

[1] J. Konys et al., Development of advanced Al coating processes for future application as anti-corrosion and T-permeation barriers, Fusion Engineering and Design 85 (2010) 2141-2145.