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

Sven-Erik Wulf, Wolfgang Krauss, Jürgen Konys
KIT, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

**Motivation**

- 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 electrodeplating of aluminum on RAFM steels such as Eurofer.

**Results**

<table>
<thead>
<tr>
<th>Impact of anodic pretreatment and storage duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freshly prepared</td>
</tr>
<tr>
<td>Without anodic pretreatment</td>
</tr>
<tr>
<td>Fe-Al scale made by ECX process process, after 4,000 h in flowing Pb-15.7Li</td>
</tr>
</tbody>
</table>

- 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)

**Experimental procedure**

**General comments**

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

- Electrolytic (cathodic) degreasing; Potential
- Electrodeposition of aluminum in non-aqueous electrolytes for Fe-Al scales made by ECX process

**Procedure**

1. Eurofer steel sheet 150mm x 150mm x 1mm
2. Grinding with 1000 grade SC paper
3. Electrolyte (cathodic) degreasing: NaOH based electrolyte, 4%a
5. Storage in exsicator at RT and 7% R.H. 1 - 3 weeks
6. Transfer into Glove Box
7. Immersion + OCP measurement (60s)
8. Plating without further treatment
9. Anodic pretreatment 45s
10. Anodic treatment for 45s (dc-swept 1.1V)
11. Rinsing: dried without / acetone
12. Rinsing outside Glove Box: dest. water / isopropanol
13. Characterization: SEM/BSE + light microscopy

**Electrochemical control and electrodeposition procedure**

**Schematic E-t plot of the procedure**

- Open circuit potential: Active surface
- Applied current density: low potential
- Pulse plating: Good coverage
- Anodic polarization: Good adhesion
- Characterization: SEM/BSE + light microscopy

**Experimental setup**

- Three electrode setup: Working electrode (WE), Counter electrode (CE), Quasi-reference electrode (REF)
- Control via IVUM Potentiostat
- Voltage 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)

**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.

**Acknowledgment**

This work has been partly carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

KIT – The Research University in the Helmholtz Association

www.kit.edu