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Optimisation of ECX permeation barriers towards thicker alumina

Carsten Schroer, Julia Lorenz, Olaf Wedemeyer, Aleksandr Skrypnik, Kateryna Khanchych

Karlsruhe Institute of Technology (KIT)
Institute for Applied Materials – Applied Materials Physics

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No tritium, no fusion

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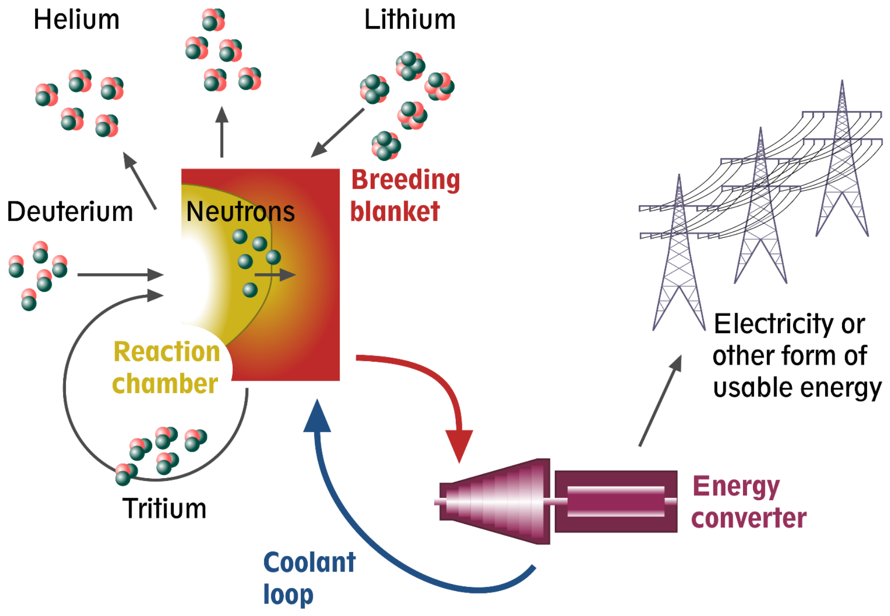
Tritium to maintain the fusion reaction best produced right in the fusion reactor (breeding blanket).

Small isotope that penetrates or permeates through structural materials, especially steel.

Hazardous substance that should arrive at the reactor unit for controlled tritium extraction.

In the breeding blanket, tritium-permeation barrier (TPB) on structural materials required, on the side of the tritium source.



Harvesting energy from magnetic-confinement fusion of deuterium and tritium.

TPB CANDIDATE MATERIALS

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Low hydrogen solubility and diffusivity, paired to high hydrogen retention.

In the breeding blanket:

- Tolerant of unprecedented neutron irradiation, minimum of neutron absorption.
- Chemically resistant against the breeding material/atmosphere.
- Thermo-mechanically compatible with the substrate material, e.g., thermal cycling.

Appropriate deposition process for producing a defect-free coating must exist.

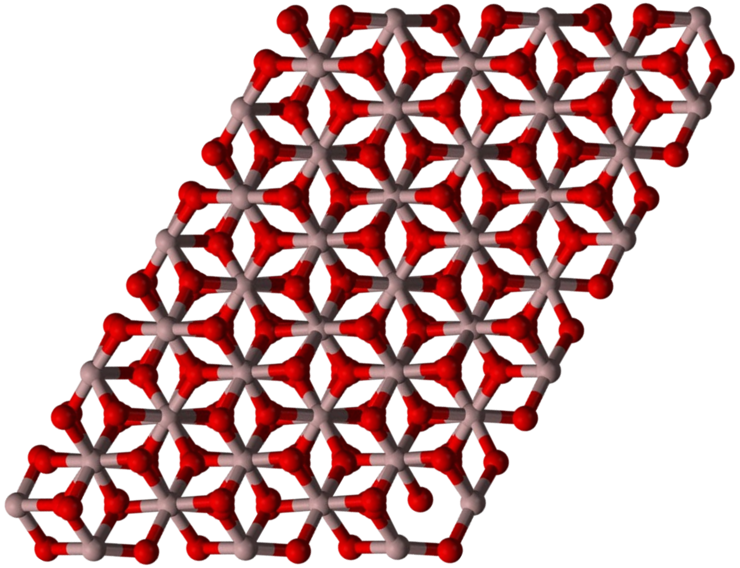
Oxides,
Nitrides,
Carbides,
Some metals,
e.g., tungsten.

ALUMINA

Favourable properties as to hydrogen permeation, especially for α–Al_2O_3.

Issues as to service in the breeding blanket:

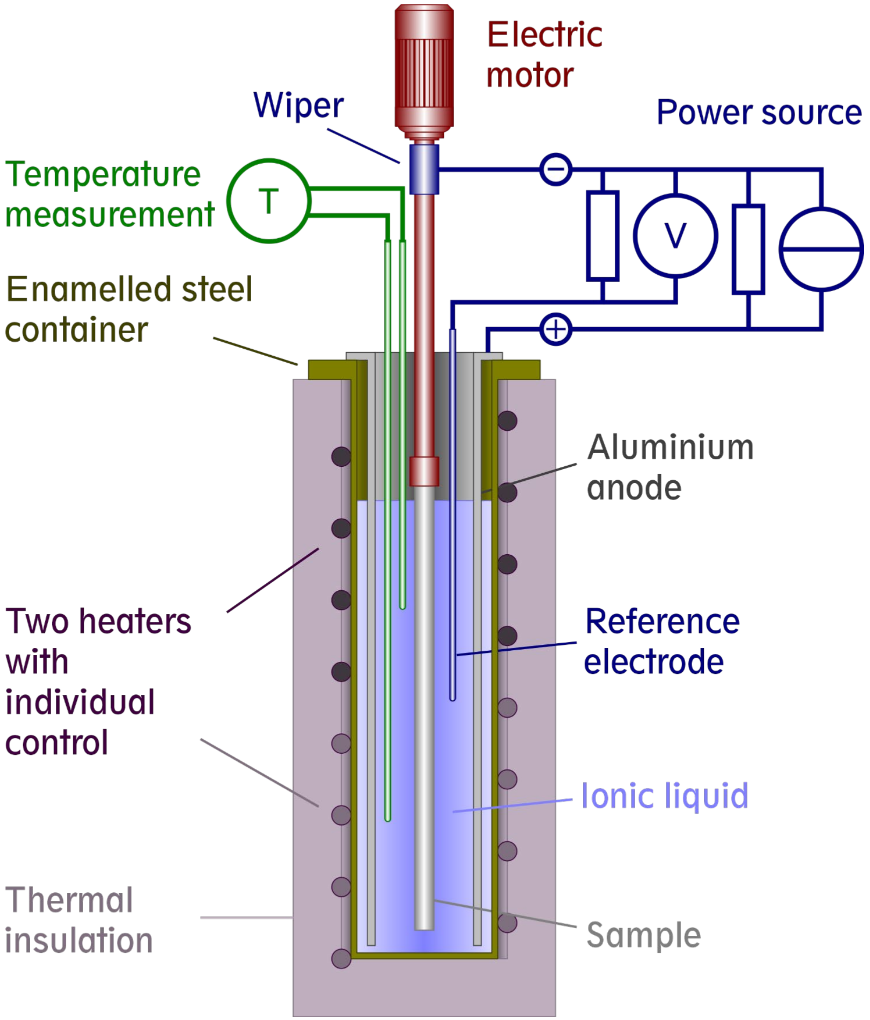
- Anisotropic swelling of hexagonal α–Al_2O_3, of less concern for cubic γ–Al_2O_3 or amorphous Al_2O_3.
- Aluminium tends to be activated, needs to be removed from the components after service.
- Lithium uptake from the breeding material, especially in the case of eutectic lithium–lead.



Ball-and-stick model of α–Al_2O_3.
By Ben Mills - Own work, Public Domain, https://commons.wikimedia.org/w/index.php?curid=4222743

Unsolved issues relating to neutron irradiation irrelevant to the tritium-extraction and removal sub-system (TER).

ECX process: (1) Electrochemical X (Al) deposition



Set-up for electroplating.

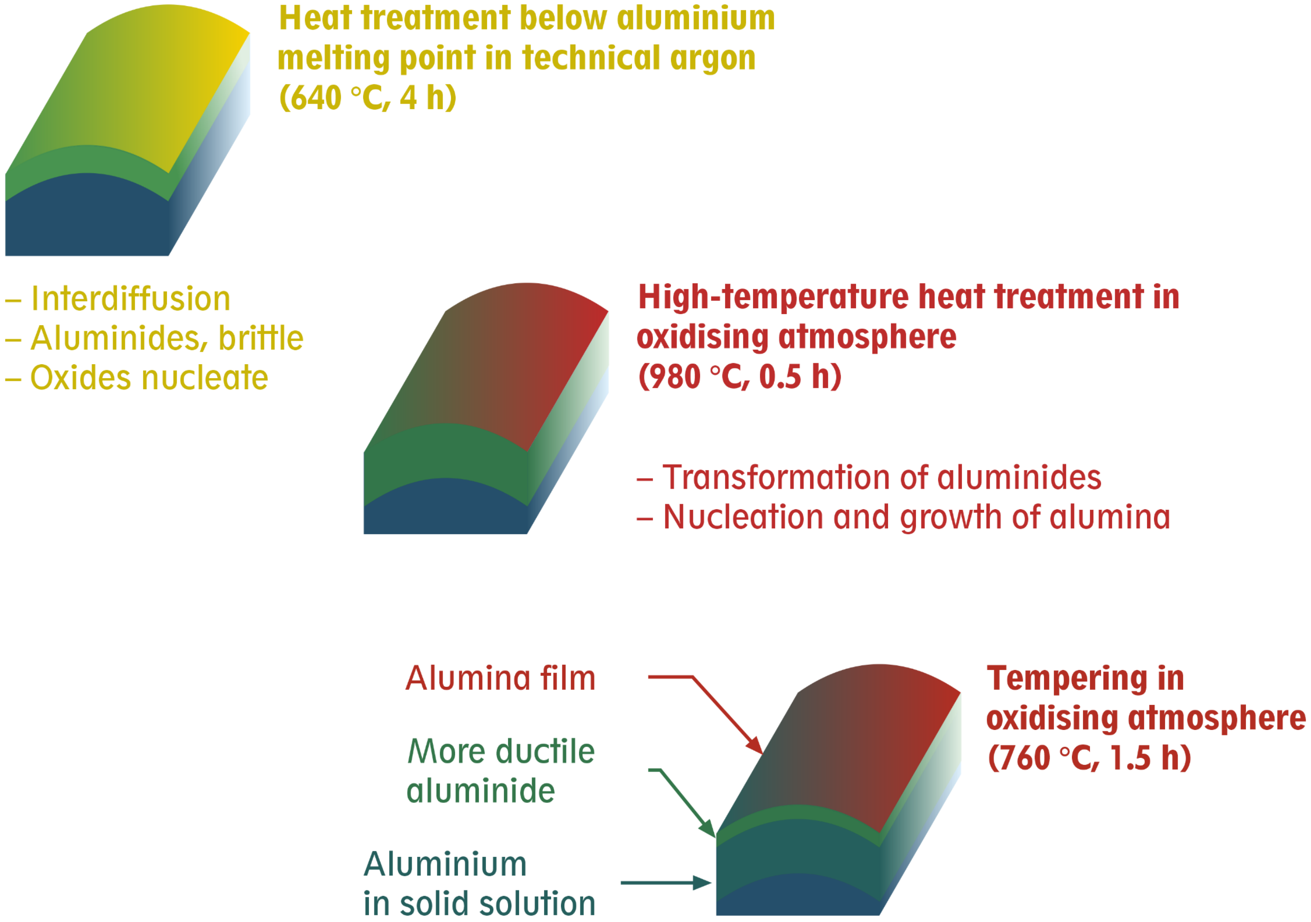
Aluminium transfer from a dissolving anode sheet onto the (EUROFER) substrate.

Ionic liquid used as an electrolyte (1-Ethyl-3-methyl-imidazolium-chloride + $AlCl_3$,1:1.5).

15 mA per cm² of substrate surface area (deposition rate ~20 µm Al/h), at 110 °C, in dry argon atmosphere (< 1 ppm O_2 or H_2O).

Pulse plating with 1 Hz.

ECX PROCESS: (2) THREE-STAGE THERMAL TREATMENT



To produce aluminium-diffusion coating with alumina surface film.

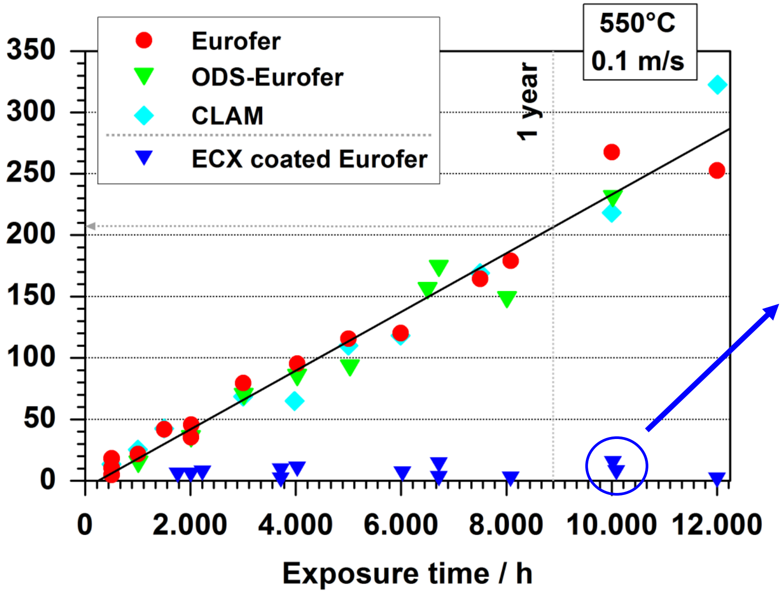
Standard oxidising atmosphere in stage 2 and 3: Technical argon.

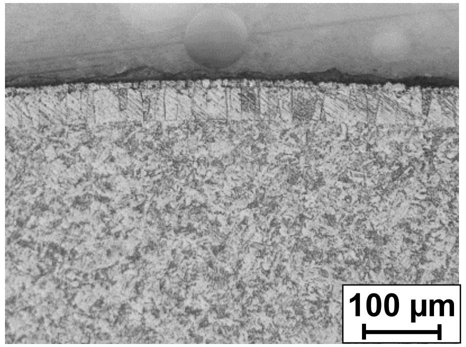
Heat treatment to produce an aluminium-diffusion coating with alumina surface film.

Raising thicker alumina: Why and how?

Standard heat treatment in argon:

- Corrosion resistance in flowing eutectic lithium–lead suggests thin alumina surface layer, but
- Too thin for satisfactory (deuterium) permeation reduction.





Surface recession (µm) after exposure to flowing Pb–16Li at 550 °C (PICOLO) of ECX coated EUROFER in contrast to RAFM steels and cross section of ECX–coated EUROFER after exposure for 10,000 h.

(S.-E- Wulf et al., Workshop on LM technology, Nov. 6th–7th 2018, Madrid, Spain).

Approach to thicker alumina:

- Change heat-treatment atmosphere during stage 2 and 3 to air.
- Prolong holding at 980 °C.

	640 °C	980 °C	760 °C
Standard	4 h, Ar, C1	0.5 h, Ar, C1	1.5 h, Ar, C1
Mod. 1	4 h, Ar, C1	0.5 h, **air**, C2	1.5 h, **air**, C2
Mod. 2	4 h, Ar, C1	1 h, **air**, C2	1.5 h, **air**, C2
Mod. 3	4 h, Ar, C1	2 h, **air**, C2	1.5 h, **air**, C2

Time, atmosphere, cooling (C1: in cold furnace section, Ar stream; C2: air cooling outside furnace).

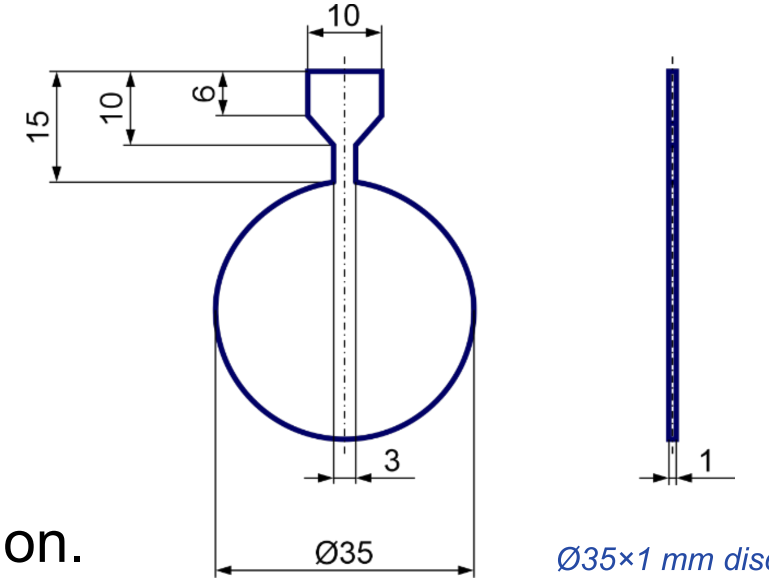
SAMPLES AND CHARACTERISATION

- EUROFER 97 electroplated with ~5 µm Al.
- Standard heat-treatment in argon and three modified heat-treatments.

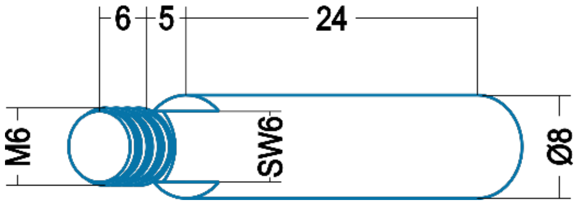
- Ø35×1 mm discs for later tests on deuterium permeation (at CIEMAT), X-ray diffraction spectroscopy (XRD) and metallographic examination.

- Ø8×24 mm cylinders for exposure to eutectic lithium–lead in the PICOLO loop (550 °C, low flow velocity to static).

+ Additional flat sample for XRD before and after each stage of the standard heat treatment



Ø35×1 mm disc, one side masked during electroplating.



Ø8×24 mm cylinder for tests in PICOLO loop.

METALLOGRAPHIC ANALYSIS OF THE COATINGS

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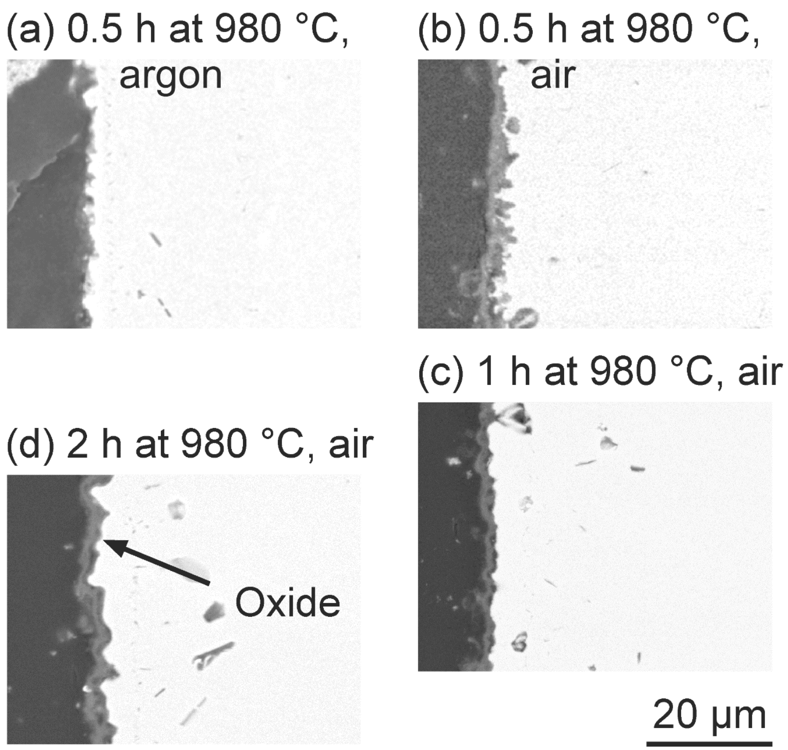
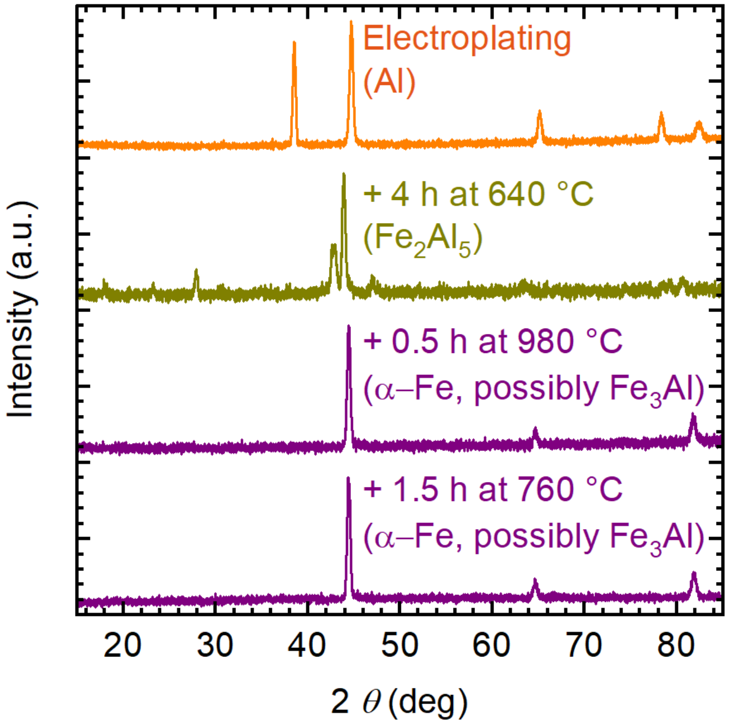
Scanning electron microscopy (SEM) on cross sections, at medium magnification, combined with energy-dispersive X-ray spectroscopy (EDS)

- 1–2 µm oxide for second and third stage of heat treatment in air, << 1 µm for standard treatment in argon.
- Oxide clearly aluminium-rich.
- Oxide protrusions interlock the surface layer with the underlying metal, with benefit to oxide adherence.
- 40 and 70 µm aluminium penetration into the substrate steel after 0.5 and 2 h holding at 980 °C, respectively.



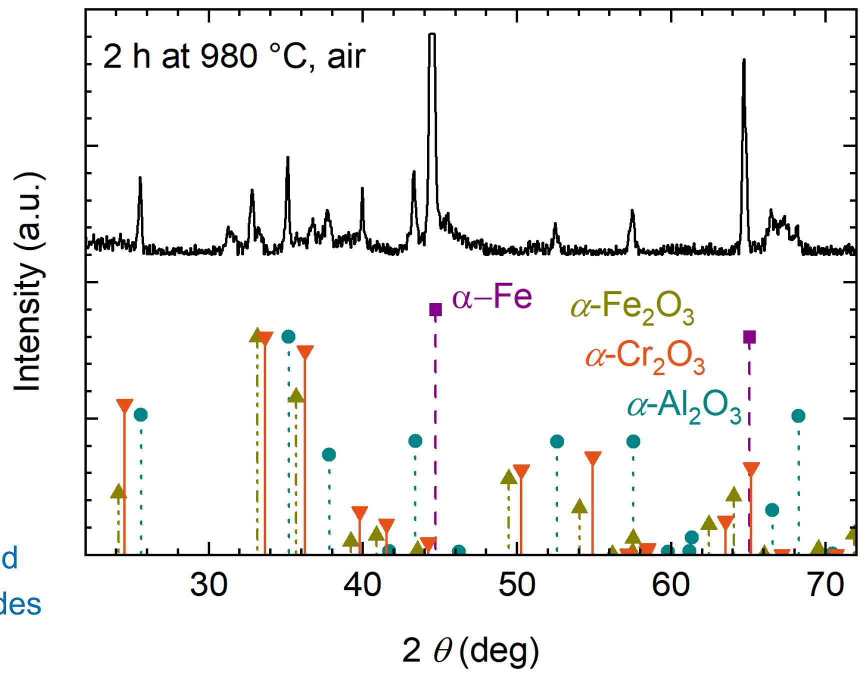
Surface oxide on ECX-coated EUROFER with (a) standard heat treatment in argon and (b–d) modified heat treatment.

XRD ANALYSES



Grazing-incidence XRD (3 °) before and after each stage of standard heat treatment in argon.

XRD spectrum recorded from sample with modified heat treatment that includes 2 h at 980 °C in air.



- Phase transitions in the metallic portion of the ECX coating.
- After standard heat treatment, any oxide possibly formed too thin for assessment with XRD.
- Oxide signals appear after modified heat treatments, especially α–Al_2O_3.
- Intensity of peaks associated to oxides increases with time of holding at 980 °C.

Exposure to Pb–16Li at 550 °C

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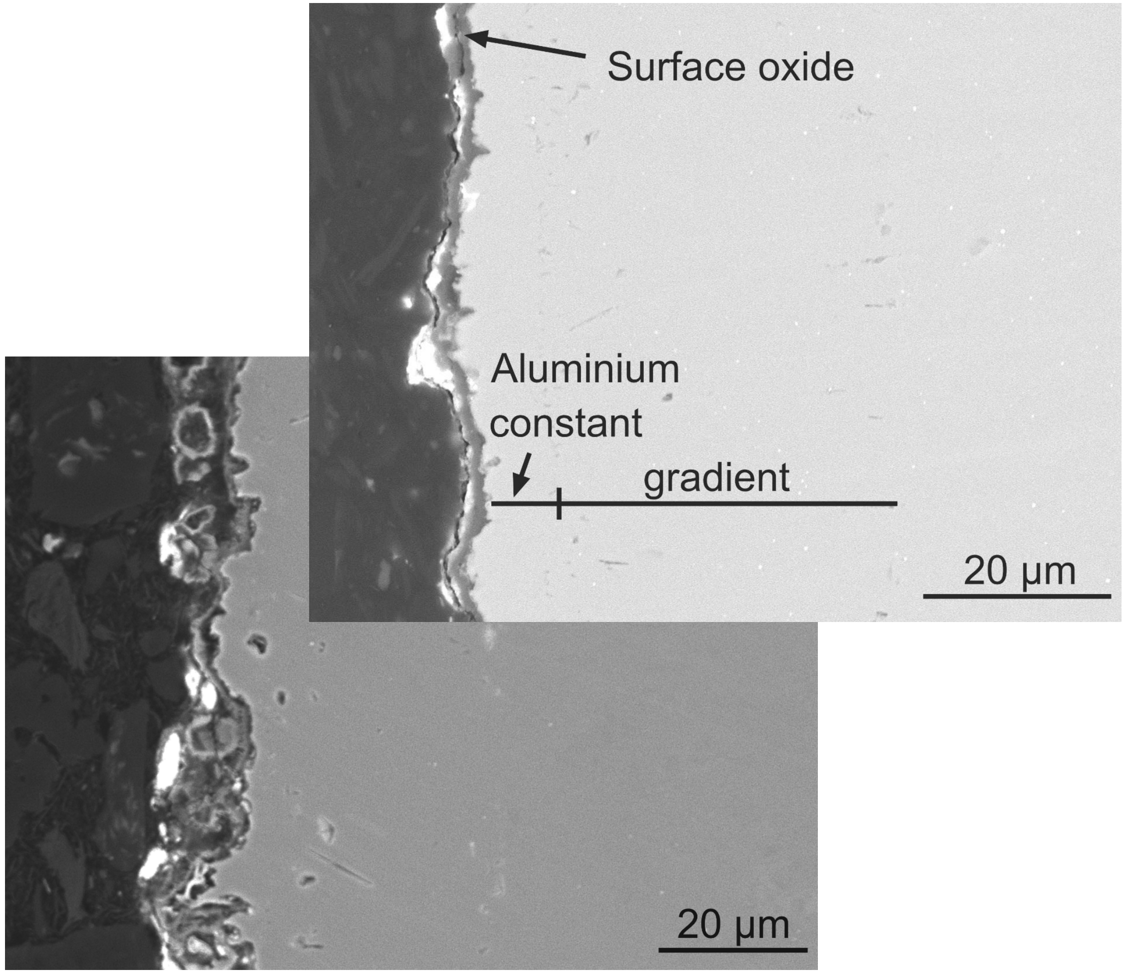
After 3000 h in the PICOLO loop, under low-flow to stagnant conditions

- Metallic part of the coating largely protected from contact with the liquid metal.
- Number of weak spots the smaller the longer the time of holding at 980 °C in air.
- Oxide layer heterogeneous, locally detached from the substrate (?).
- Around 50 µm aluminium penetration into the substrate irrespective of the heat treatment.



Electron-optical micrographs of ECX coating with heat treatment Mod. 2 (Table 1) after exposure for 3000 h to eutectic Pb–16Li.

Conclusions

- Anticipated effect of air as to promoting alumina growth, but optimisation for continuity or minimisation of defects still required.
- Prolong holding at 980 °C? – Probably not an option for EUROFER substrate.
- Especially for ECX, other selective changes with regard to the heat-treatment rather than the deposited aluminium (e.g., doping with other elements).
- Why not grow γ–Al_2O_3 then?

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