

COMPAS_sCO₂

EXTERNAL WALL INVESTIGATIONS RESULTS

COMPAS_sCO₂ Second Stakeholders Workshop

*Next generation advanced materials for
particle/supercritical CO₂ heat exchangers*

Hybrid- physical venue: Hotel Anker,
Kolpingstraße 7, 97828 Marktheidenfeld,
Germany
Online: Zoom

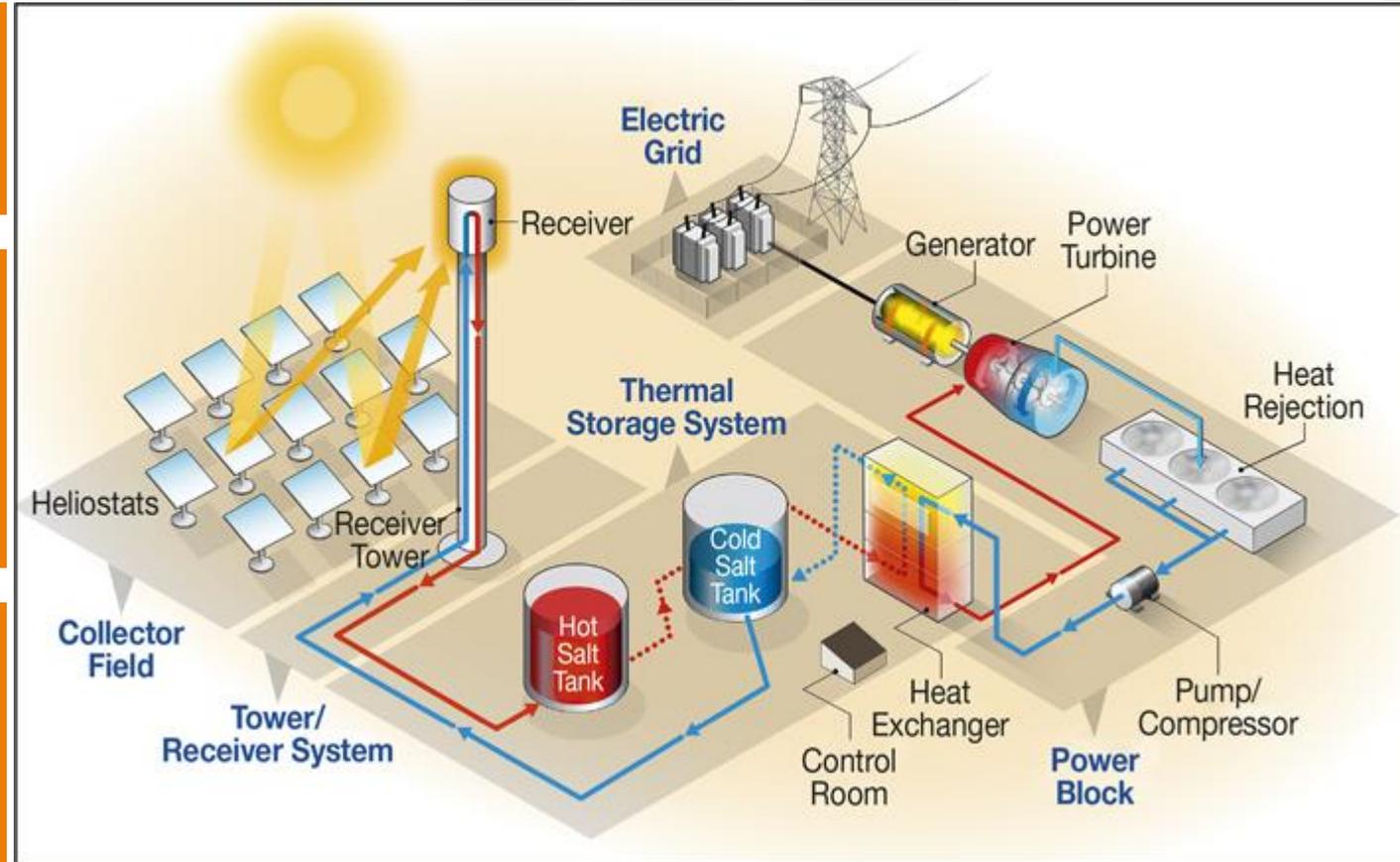
September 25th, 2023
11h00 – 15h00 CET
No registration fees

AIMS – DEVELOPMENT OF NEW MATERIALS / ALLOYS

▶ **Replacing molten nitrates (solar salts):** ceramic (bauxite) particles as environmentally friendly heat transfer medium; no freezing issues as molten salts

▶ **Interactions between substrate steels and heat transfer medium:** Sanicro 25 (austen.), IN 740 (Ni-base), Alloy 617 (Ni-base), Haynes 282 (Ni-base), and P 92 (ferritic); understanding microstructure, phase composition, and chemical stability

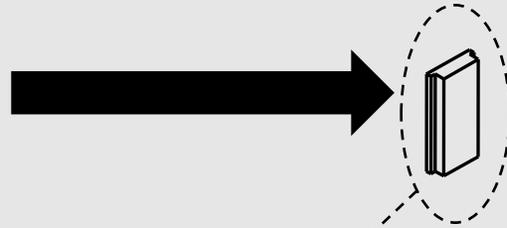
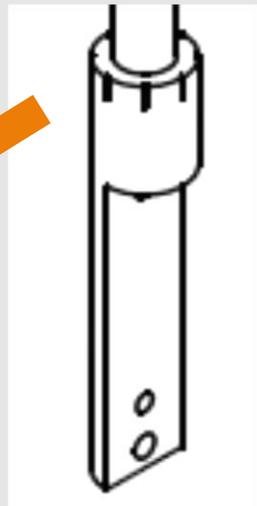
▶ **Lifetime estimation:** investigations of developed materials (monolithic and/or coated) in regard to their performance at high temperatures under the CSP conditions



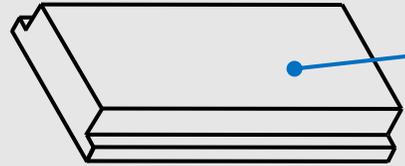
(Credit: Al Hicks, National Renewable Energy Laboratory, US)

TEST SET-UP FOR EROSION TESTING

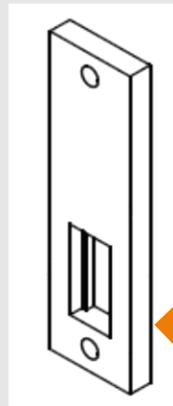
Erosion Testing Facilities at FZJ



sample specimen



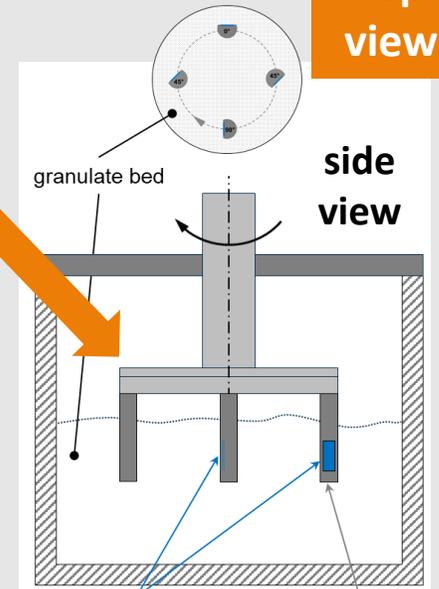
surface in contact with granulate (20 mm x 8 mm)



sample holder

particle flow rate = holder rotation speed

top view



side view

granulate bed

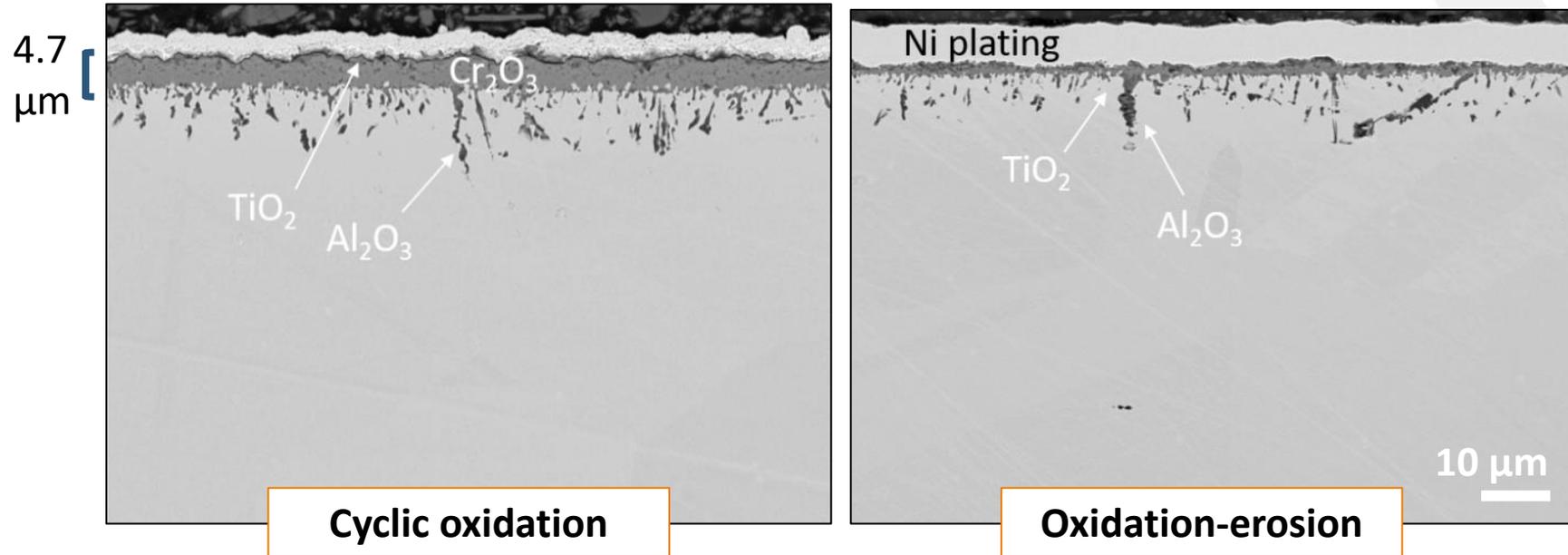
specimens

specimen holder

Method: impact of granulate (bauxite) on specimens manufactured for erosion testing
Analysis: mass change, metallographic cross-sections (SEM / EDX), optical metallography

EROSION WEAR BEHAVIOUR

Back scattered electron images of cross-sectioned IN740



Erosion testing performed
at 600 °C (P 92), 700 °C
and 900 °C

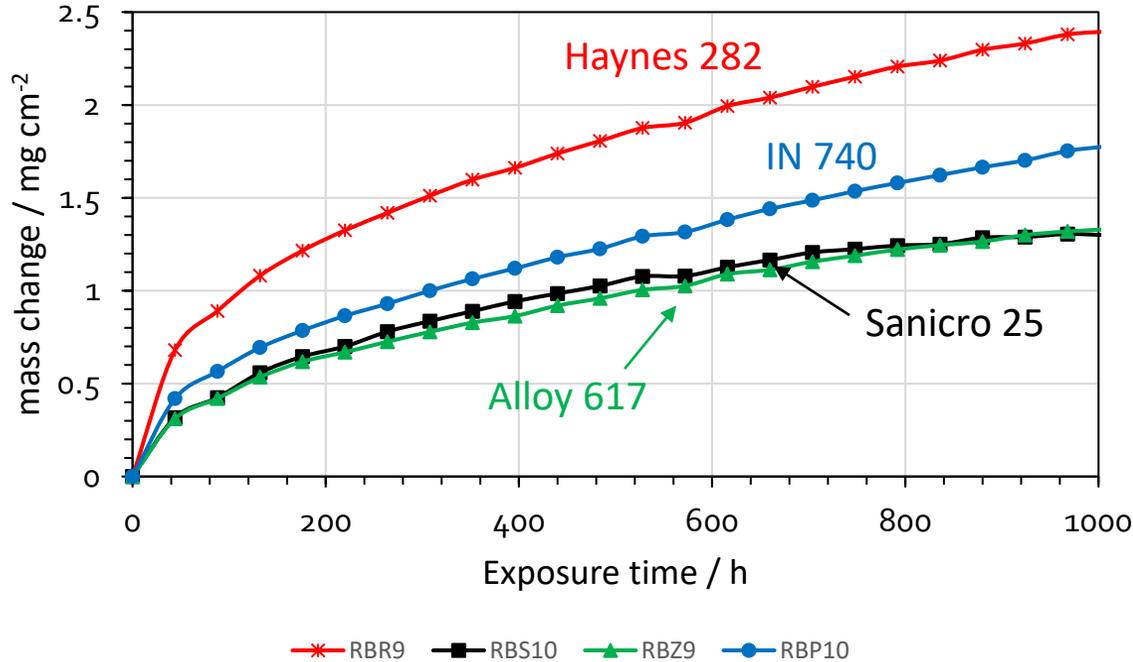
The images clearly indicate the rate of material degradation; chromia and titania-rich oxide layers are typically formed, creating a hard barrier on the surface of the material substrate that may enhance corrosion resistance

Exposures for 250 h at 900°C in air

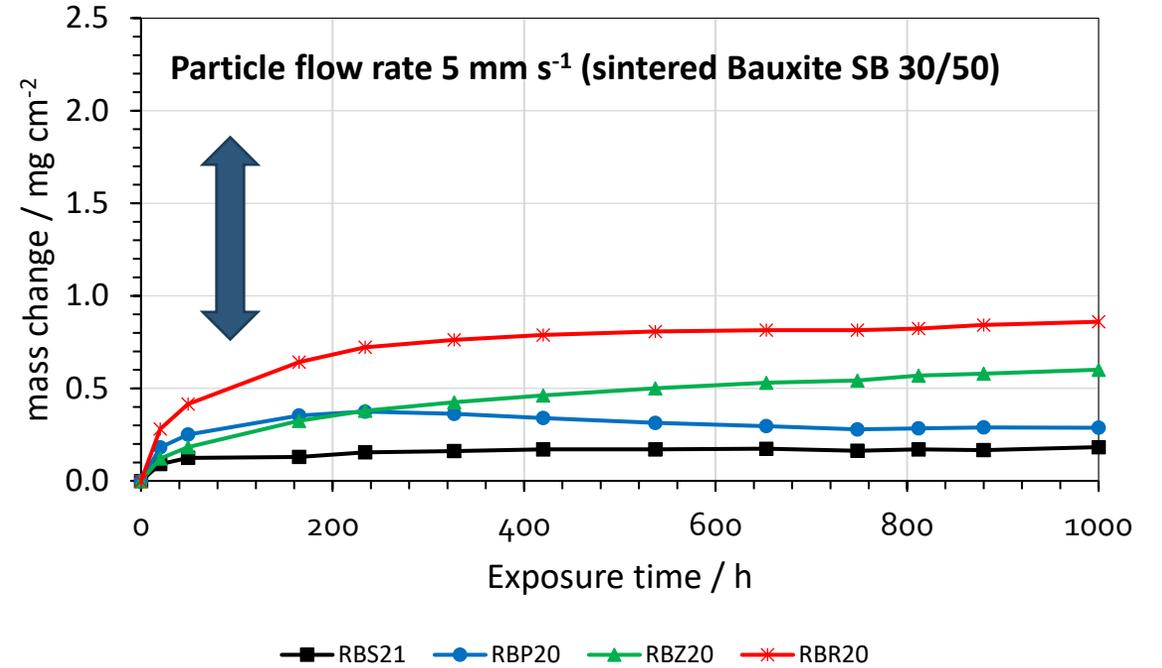
Oxidation-erosion: *sintered bauxite (particle size 300 to 600 μm), provided by Saint-Gobain*

EROSION TESTING – WEIGHT CHANGES

Cyclic oxidation exposure at 900 °C in air



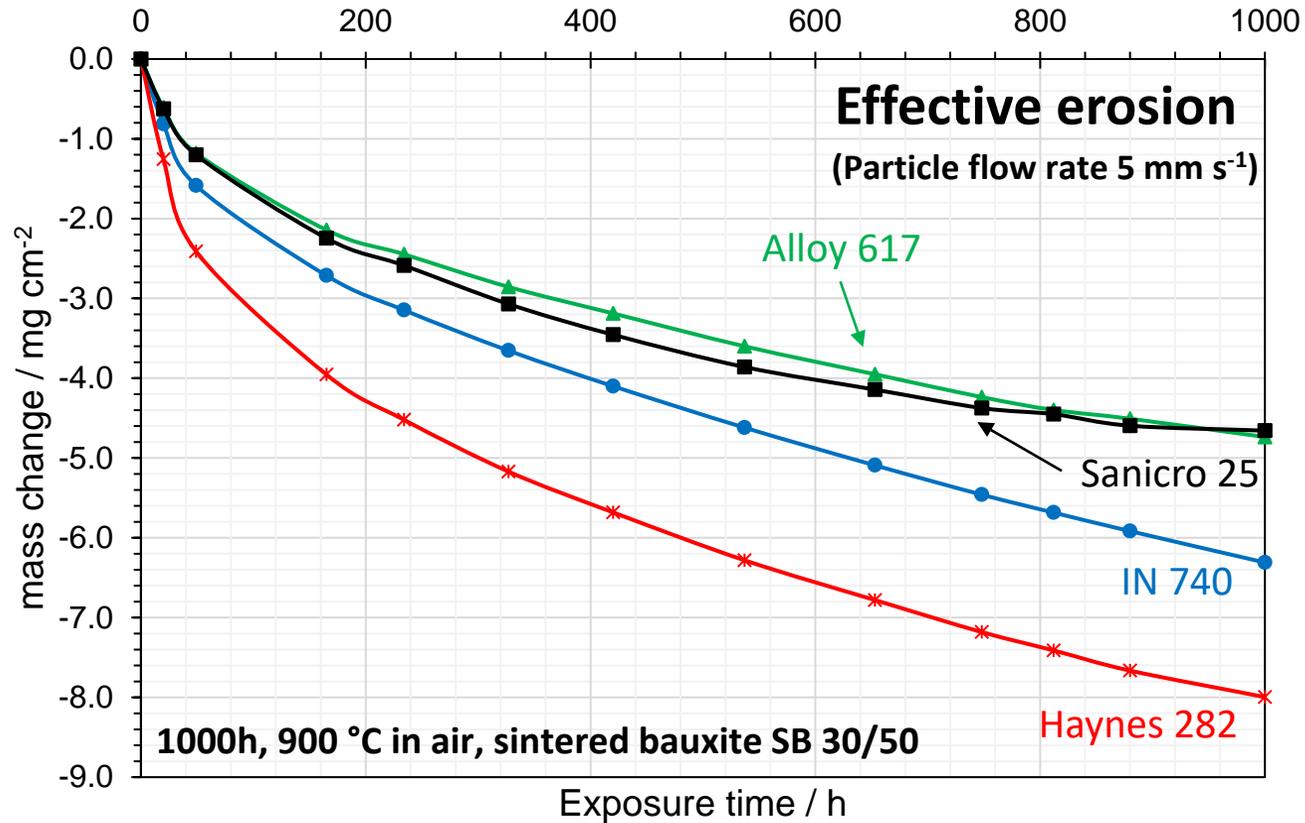
Oxidation and erosion exposure at 900 °C in air



The change in thickness of the oxide layers corresponds with the mass change data.

Mass changes relate to total specimen surface area (including sides not exposed to erosion)

EROSION TESTING – WEIGHT CHANGES

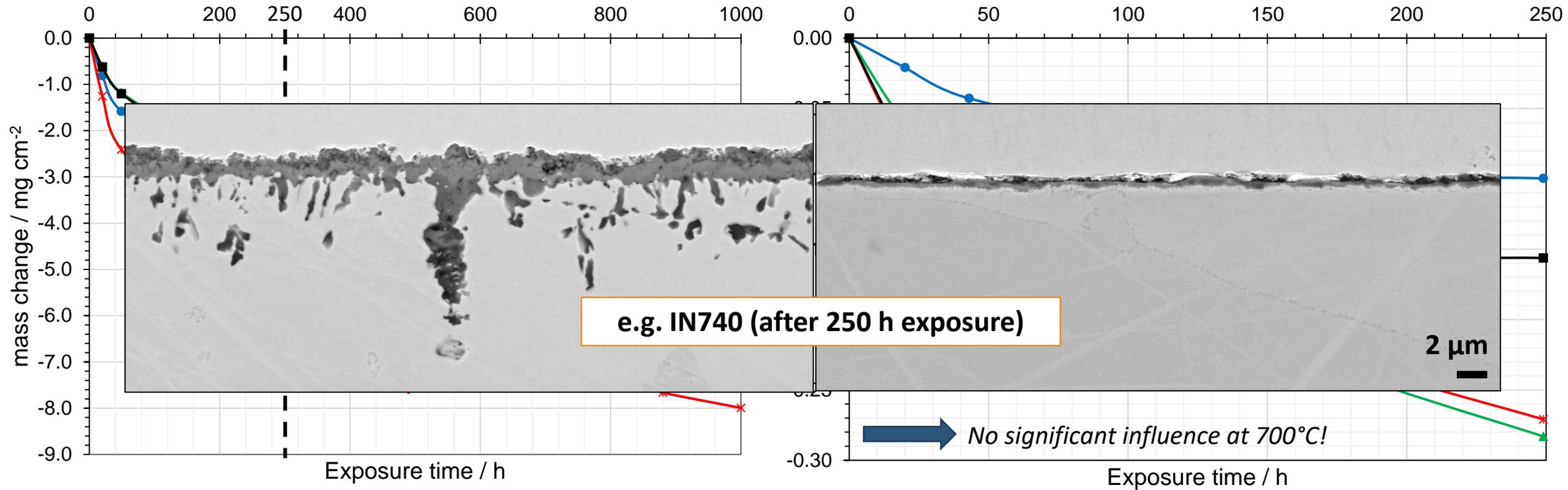


Mass changes relate to front specimen surface area:

$$\frac{(A_{\text{erosion}}^{\text{whole}} \cdot \Delta m_{\text{erosion}}) - (A_{\text{cycl.oxidation}}^{\text{whole}} \cdot \Delta m_{\text{cycl.oxidation}})}{A_{\text{erosion}}^{\text{exposed}}}$$

The highest mass loss is observed in Haynes 282 (–8 mg cm⁻² after 1000h exposure), whereas the lowest is observed in Alloy 617 and Sanicro 25 (<4 mg cm⁻²); both Alloy 617 / Sanicro 25 exhibit similar changes in mass after 1000h exposure)

EROSION TESTING – WEIGHT CHANGES

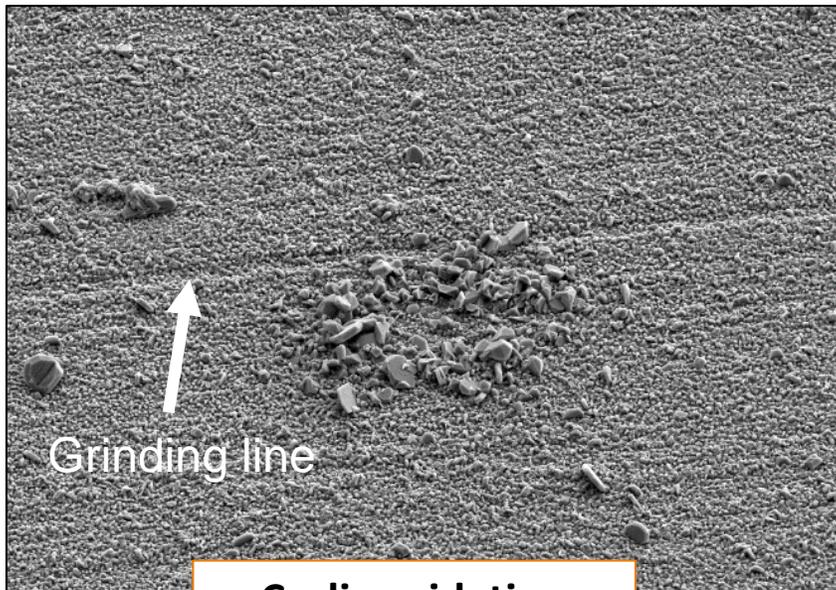


1000h, 900°C in air, sintered bauxite SB 30/50 (5 mm s⁻¹)

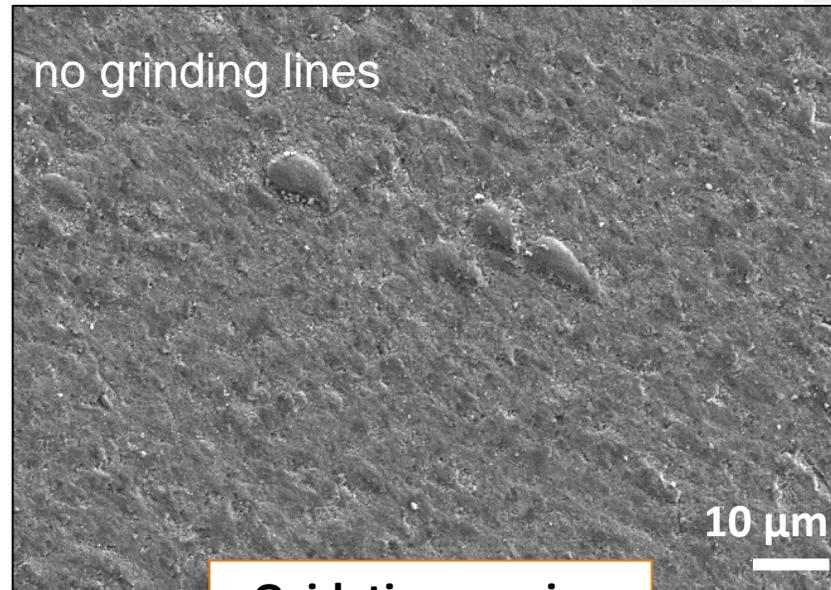
250h, 700°C in air, sintered bauxite SB 30/50 (5 mm s⁻¹)

EROSION WEAR BEHAVIOUR

Back scattered electron images of sample specimen surfaces (Alloy 617)



Cyclic oxidation



Oxidation-erosion

Impact of erosion rate (5 mm s^{-1}) sufficient to erode whole surface area of the sample, as polishing lines (1200 Grit) could not be observed after erosion-oxidation testing anymore

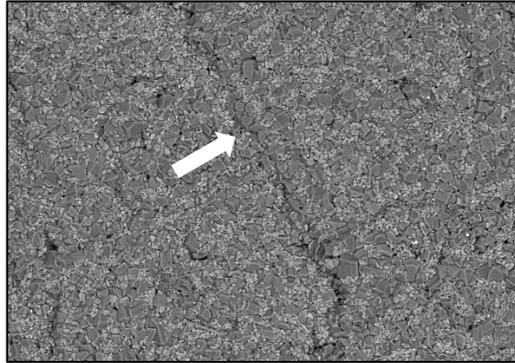
Exposures for 250 h at 700°C in air

Oxidation-erosion: *sintered bauxite (particle size 0.3 to 0.6 mm), provided by Saint-Gobain*

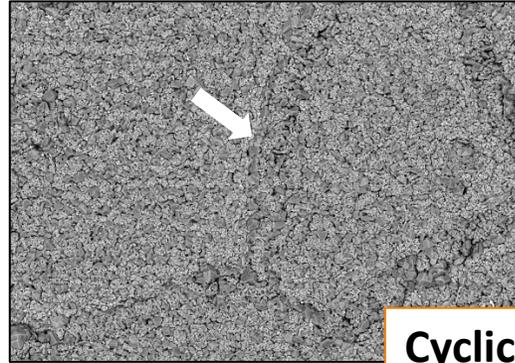
EROSION WEAR BEHAVIOUR

Sample matrix exhibits grain boundary structures

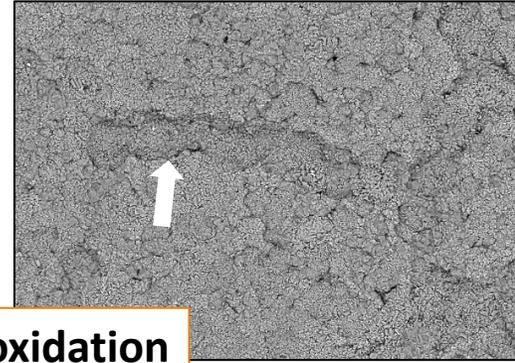
Haynes 282



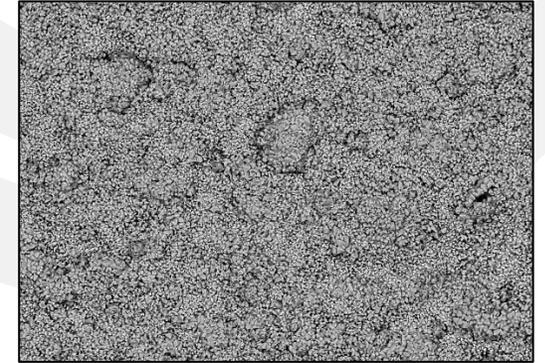
IN740



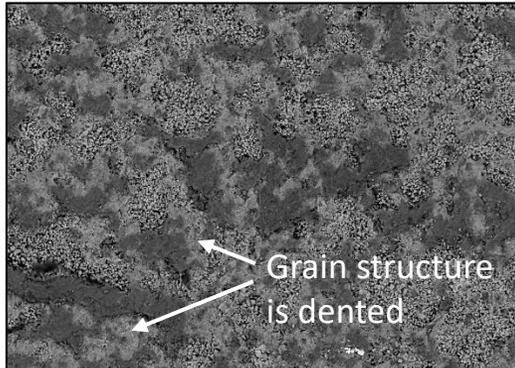
Alloy 617



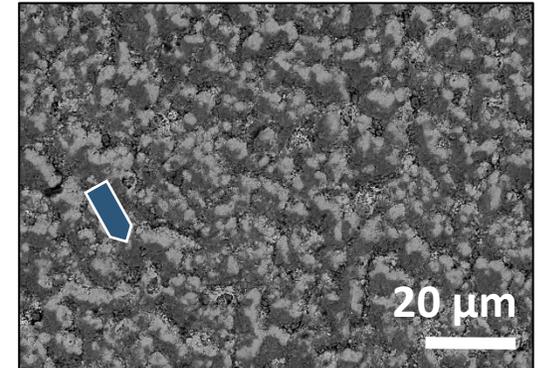
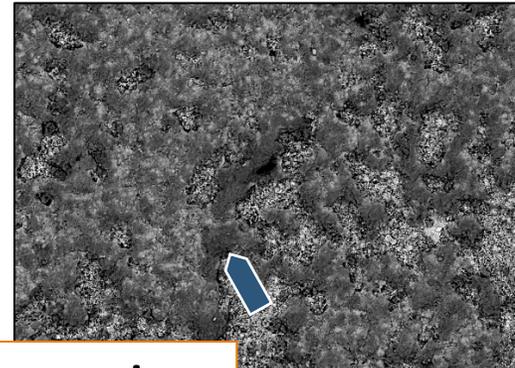
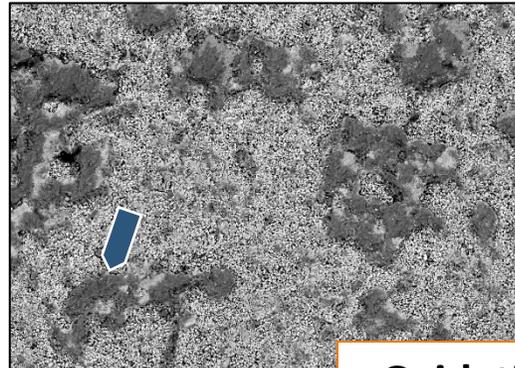
Sanicro 25



Cyclic oxidation



Grain structure is dented



Oxidation-erosion

Granulate impacted on the surface morphology of the alloys

250 h / 900°C

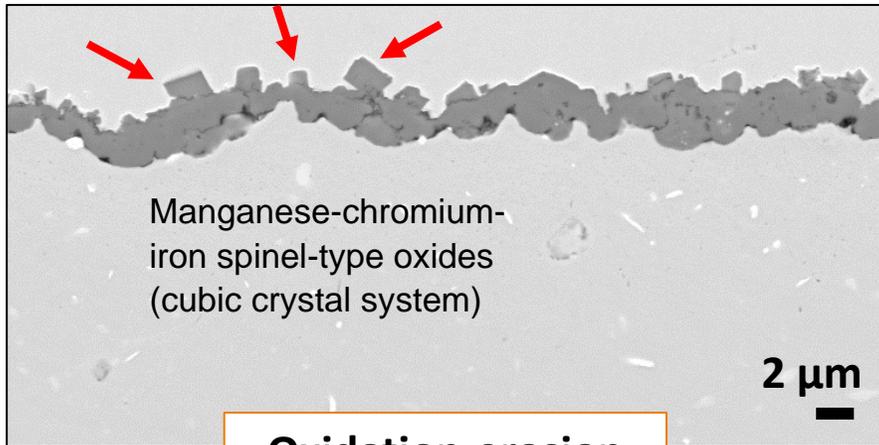
Black discoloration within the sample matrix implies granulate deposition of the bauxite material

EROSION WEAR BEHAVIOUR

- **How can we explain the coherences observed (weight changes and SEM images)?**
- Granulate deposition is a sign of less erosion degradation (mass change data)
- **1st assumption:** possible reason for less erosion degradation might be related to the sample roughness, which is attributed to the chemical composition of the sample
- **2nd assumption:** mechanical properties of oxide scale; specifically, a high Ti-content promotes oxide scale formation which can be susceptible to erosion

EROSION WEAR BEHAVIOUR – SAMPLE ROUGHNESS

Sanicro 25



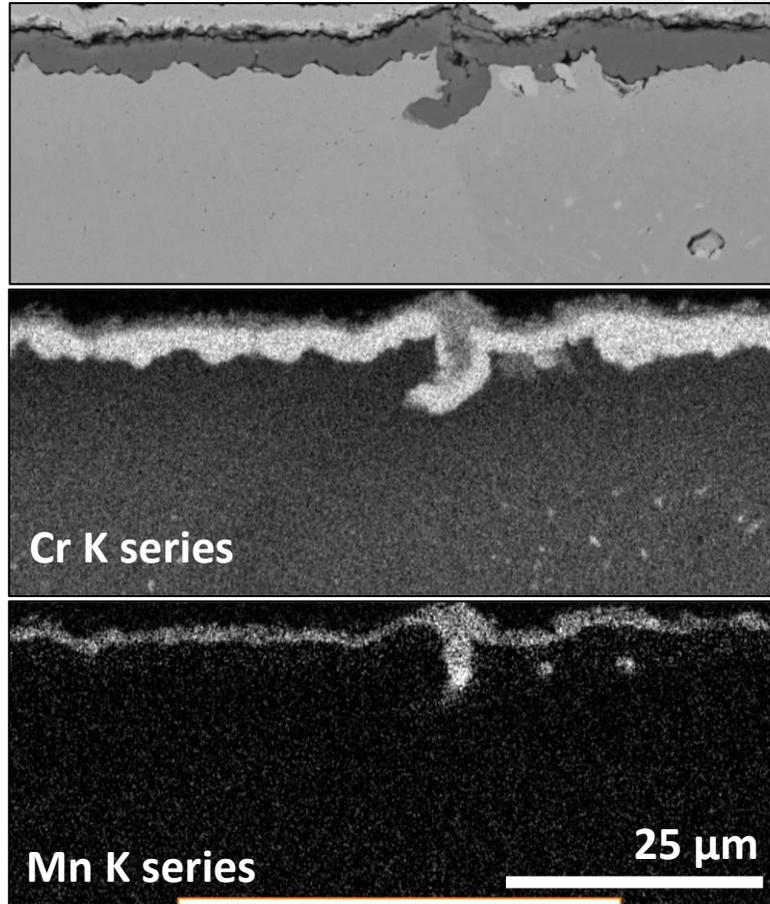
Manganese-chromium-iron spinel-type oxides (cubic crystal system)

2 µm

Oxidation-erosion

Exposures for 250 h at 900°C in air

Oxidation-erosion: sample back side, not exposed to bauxite granulate



Cr K series

Mn K series

25 µm

Cyclic oxidation

EDX mapping of the scale formed on Sanicro 25

Mn has accumulated on the surface of the outer layer, suggesting that it has dissolved in the chromia and diffused onto the alloy's surface → it has clearly reacted with the chromia to create **manganese chromia spinell**

ROUNDUP ON EROSION WEAR BEHAVIOUR

- ▶ **We determined erosion rates at 900°C, and degradation mainly occurred at these conditions (the erosion rate seems being strongly dependent on the temperature)**
- ▶ **Highest mass loss observed (effective erosion) for Haynes282, and lowest mass loss observed for Sanicro 25 and Alloy 617**
- ▶ **Less erosion showed less granulate deposition and could not be attributed to mechanical properties, which means that the oxide scale determines the erosion properties**
- ▶ **Manganese chromia spinel formed on the outer layer surface of Sanicro 25 – moreover, the sample demonstrated the highest granulate deposition and the lowest wear compared to all the other materials examined using bauxite granulate (SB 30/50)**



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THANK YOU



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