Characterisation of oxide scales resulting from the exposure of various steels to liquid lead-bismuth eutectic

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Introduction

Structural steels degrade in contact with heavy liquid metals (HLMs), such as the lead-alloy coolants (Pb, LBE) used in lead-cooled fast reactors (LFRs). A way to mitigate the undesirable effects of liquid metal corrosion (LMC) during LFR operation is by controlling the amount of dissolved oxygen in the liquid metal, so as to form oxide scales on the steel surface that will protect it from further corrosion. Knowing the mechanisms of formation and degradation of these oxides as a function of the conditions of steel exposure to the HLM environment (i.e. temperature, HLM oxygen content, HLM flow velocity, etc.) is crucial for the assessment of the long-term LMC behavior of structural steels in LFR nuclear systems.

Objectives

Characterize by means of scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) oxide scales formed on the surface of various steels exposed to liquid LBE under different test conditions, so as to better understand the steel oxidation mechanisms as a function of the exposure conditions.

Materials and exposure conditions

- Tests in static liquid LBE – all steels: T ≈ 490°C, ~5016 h, oxygen-saturated LBE } Vessel with static LBE, SCK•CEN
- Tests in flowing liquid LBE – Eurofer steels: T ≈ 550°C, 1007-7511 h, LBE C_O[§] ≈ 10^{-6} wt%, LBE flow velocity: 2 m/s
- Tests in flowing liquid LBE – EF-ODS steels: T ≈ 550°C, 5012-20039 h, LBE C_O ≈ 10^{-6} wt%, LBE flow velocity: 2 m/s } CORRIDA loop, KIT

Table 1. Composition of studied steels: concentration of various alloying elements (in mass %); the balance is Fe.

Steels exposed to static liquid LBE									Steels exposed to flowing liquid LBE *(CORRIDA loop)*									
	Cr	Mo	W	V	Mn	Ni	Si	C		Cr	Mo	W	V	Mn	Ni	Si	C	Y[†]
T91	8.99	0.89	-	0.21	0.38	0.11	0.22	0.1	**Eurofer**	8.82	<0.001	1.09	0.2	0.47	0.02	0.04	0.11	-
EP-823	11.7	0.74	0.6	0.3	0.55	0.66	1.09	0.16	**EF-ODS-A***	9.4	0.004	1.1	0.185	0.418	0.067	0.115	0.072	0.297
S2439	11.58	1.03	0.48	0.31	0.37	0.48	2.75	0.19	**EF-ODS-B**	8.92	0.0037	1.11	0.185	0.408	0.0544	0.111	0.067	0.192

[†] In the form of dispersed yttria (Y_2O_3) particles * ODS = oxide dispersion strengthened [§] C_O = HLM oxygen content

Results

Tests in static liquid LBE

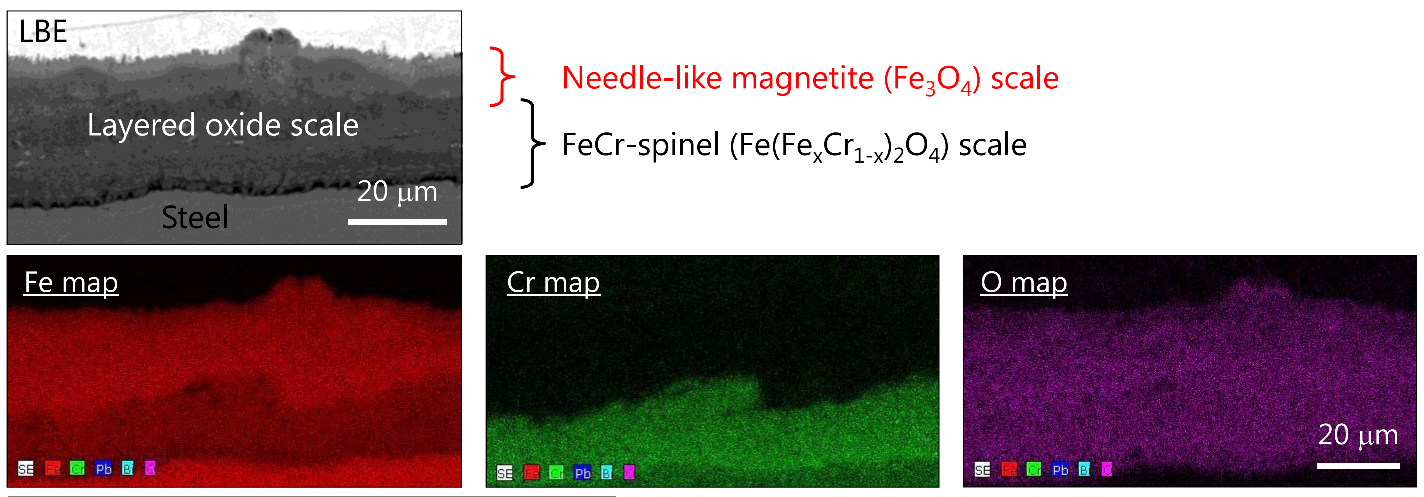

Figure 1. Oxide scales on T91 steel.

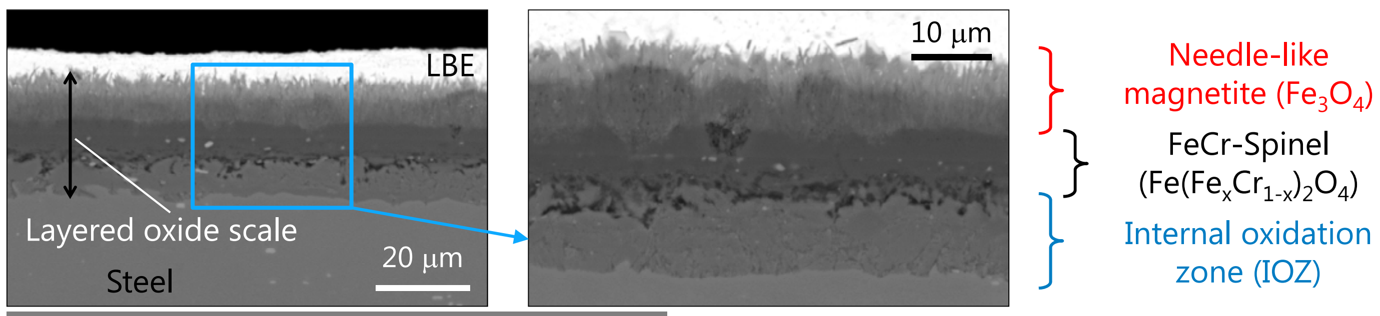

Figure 2. Oxide scales on EP-823 steel.

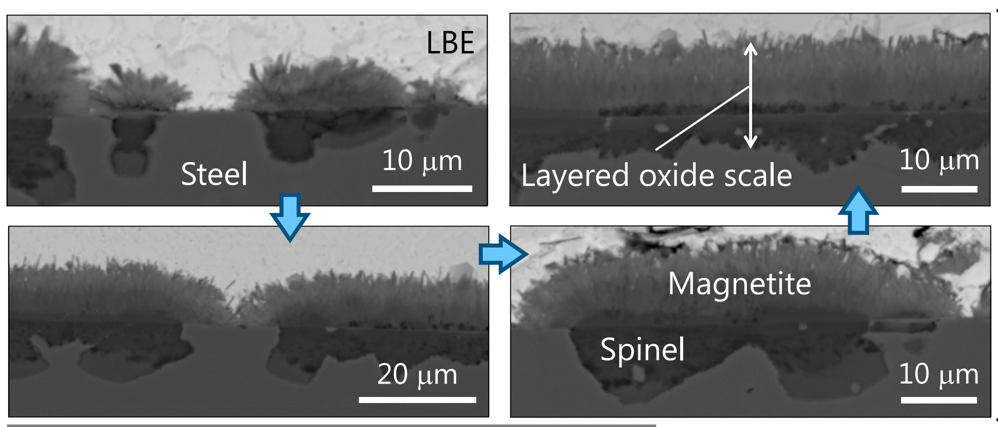

Figure 3. Oxide scales on S2439 steel.

Oxidation mechanism:

- Simultaneous nucleation of magnetite & spinel
- Lateral expansion of magnetite/spinel pairs
- Lateral coalescence of magnetite/spinel pairs
- Formation of continuous magnetite/spinel scale

Tests in flowing liquid LBE

Oxidation behavior of Eurofer steel in LBE flow:

- Single FeCr-spinel oxide (no magnetite) is observed
- Disruption of oxide scale continuity leads to LBE penetration and steel dissolution underneath the oxide scale
- Steel dissolution starts intergranularly

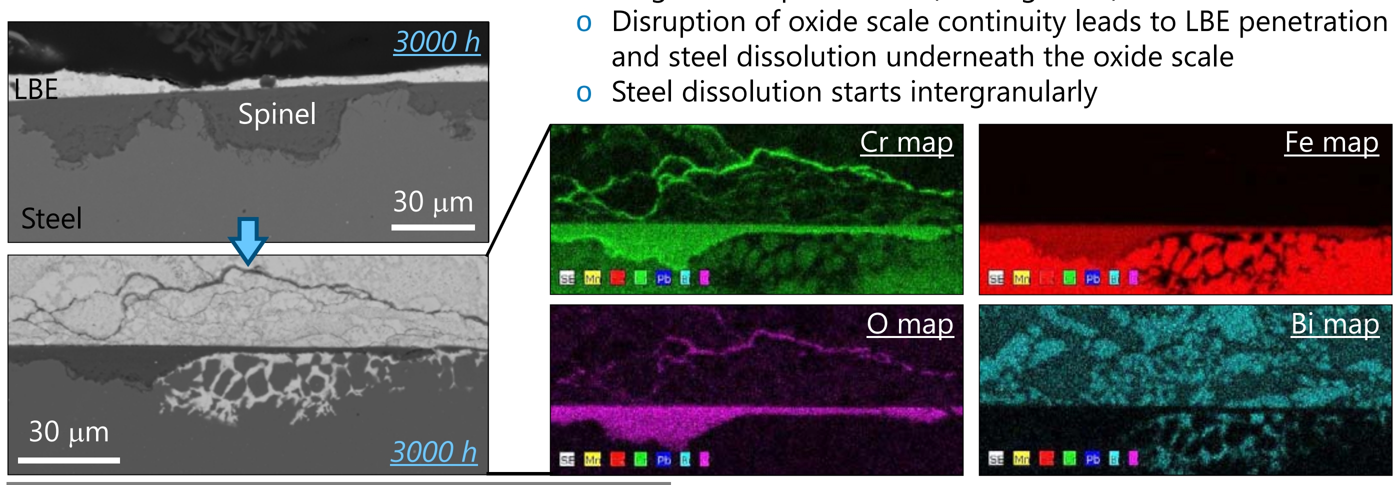

Figure 4. Oxide scales on Eurofer steel.

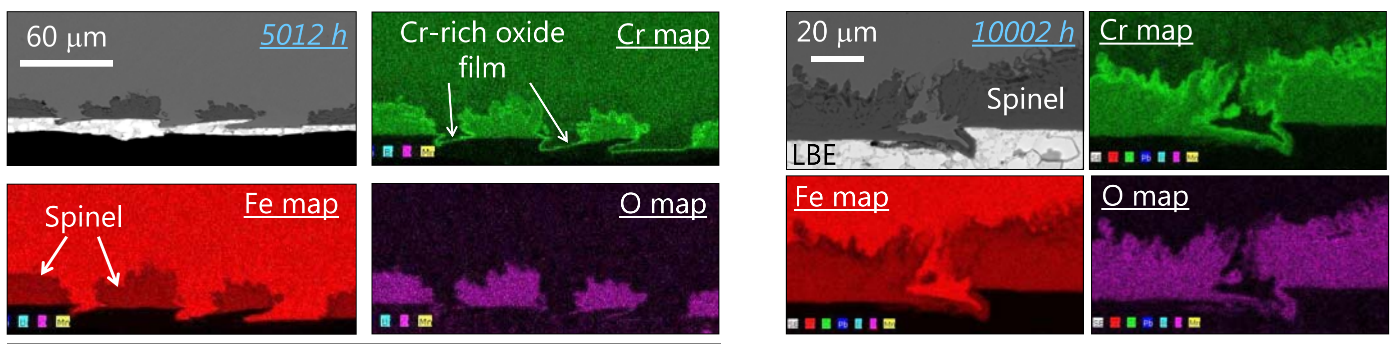

Figure 5. Oxide scales on EF-ODS-A steel.

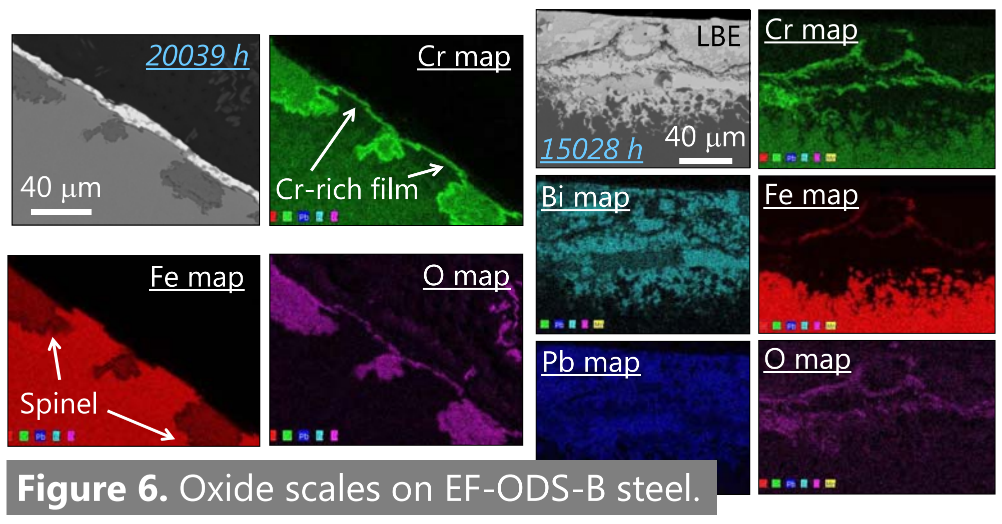

Figure 6. Oxide scales on EF-ODS-B steel.

Oxidation of EF-ODS steels:

- Cr-rich oxide film covers initially the steel surface
- Sporadic nucleation and locally accelerated growth of FeCr-spinel oxide
- Disruption of the oxide scale continuity may lead to severe steel dissolution underneath the superficial oxide scale
- ➢ Questionable protectiveness of steel oxide scales!

Discussion

Exposure of T91, EP-823 and S2439 steels to static LBE produced multilayered oxide scales with a needle-like magnetite outer layer and an FeCr-spinel inner layer. Magnetite formation was not observed in Eurofer and EF-ODS steels exposed to flowing LBE, and oxidation started with the formation of Cr-rich oxide films. The oxidation behavior of all steels was highly non-uniform with no clear reason other than potential inhomogeneities/flaws in the steel and the oxide scales.

Conclusions

The oxidation behavior of all studied steels was non-uniform, irrespective of the conditions of their exposure to liquid LBE. The protectiveness of the oxide scales is questionable as they demonstrate vulnerability to LBE penetration, which subsequently leads to severe dissolution of the bulk steel underneath the oxide scales.

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