

Microstructural changes in freeze linings during zinc fuming processes

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ABSTRACT

Freeze linings are often used as furnace protection in pyrometallurgical processes. An example is the zinc fuming process in which zinc is recovered and slag is valorised. Due to changing furnace conditions and bath compositions, it is challenging to obtain a stable freeze lining throughout the process. To investigate the evolution in freeze linings by a change in bath composition, experiments were performed in which an air cooled probe is submerged in a liquid slag bath without ZnO and PbO resembling the composition at the end of the fuming phase. By adding ZnO and PbO, the composition during the charging phase was approached. The formed freeze lining samples are microstructurally investigated with EPMA to characterise the changes and to identify if the freeze lining stays stable during the process.

INTRODUCTION

Earlier research investigated the formation and microstructure of freeze linings in zinc fuming processes and with Zn-rich slags¹⁻⁷ but dynamic bath compositions were not yet considered. In this research, the possible microstructural changes were investigated when an $\text{Al}_2\text{O}_3\text{-CaO-FeO-SiO}_2$ slag is changed to an $\text{Al}_2\text{O}_3\text{-CaO-FeO-PbO-SiO}_2\text{-ZnO}$ slag during the freeze lining formation.

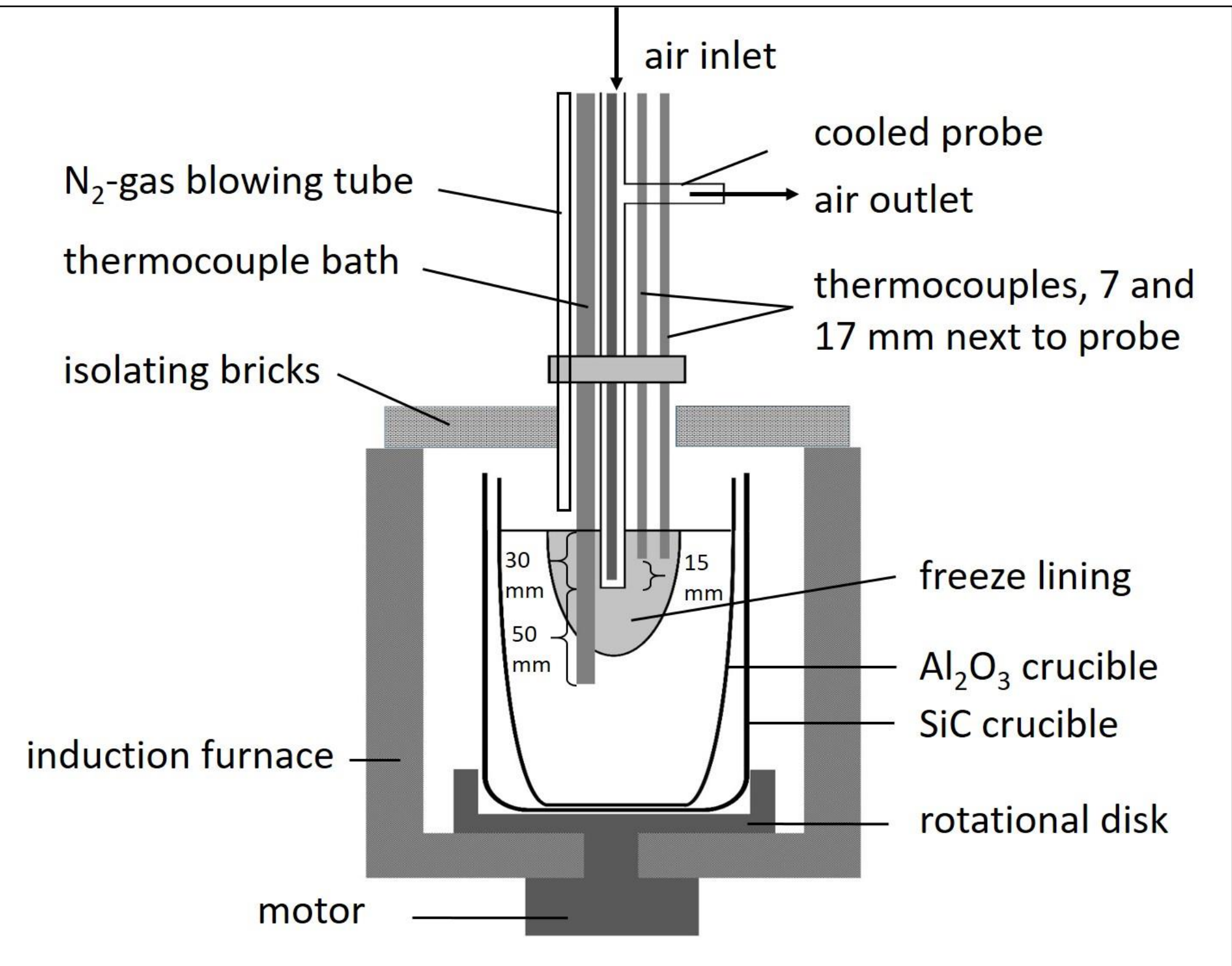


Figure 1: Freeze lining setup

METHODOLOGY

A cooled probe setup was used similar as in earlier freeze lining research² (Figure 1). Air (20 m³/h) was used as cooling agent. Powder (3000 g, Table 1) was molten at 1250 °C. 100 l/h N₂ gas was blown above the crucible. Cokes (30 g, diameter: 3 mm) were placed on the bath surface. Once the slag was molten, the crucible was rotated (25 rpm) and the probe was placed in the slag. A freeze lining was formed during 90 min. Next, ZnO and PbO were added to obtain the final bath composition (Table 1). The probe stayed submerged for another 90 min. Afterwards, it was removed and quenched in water. The freeze lining was analysed with an electron micro-probe. Initial and final slag samples were analysed with XRF.

Table 1: Targeted slag composition

(wt%)	total Fe	SiO ₂	CaO	Al ₂ O ₃	ZnO	PbO
initial	25.8	39.4	24.7	0.6	0	0
final	23.2	35.5	22.3	0.5	8.0	2.0

RESULTS

The XRF analyses show that the target compositions of the slag bath are well approached. Only the Al_2O_3 content is +/- 5 wt% higher due to the dissolution of the crucible and protective sheaths.

In the freeze lining six zones are distinguished from cold to hot face (Figure 2): a glassy layer with small crystals, a mixed glassy and microcrystalline layer, a closed crystalline layer, two open crystalline layers and a bath material layer with few crystals. Zone 4 and 5 can be seen as a whole, but in zone 5, fewer crystals are present and Pb and Zn were found.

The microstructural pictures of zones 4, 5 and 6 (Figure 3) show the phases melilite, spinel and quenched liquid. Only in zone 5 and 6 Zn and Pb are present (Table 2) indicating that in this region the original freeze lining is altered. The measured Zn and Pb concentrations are however lower than in the final slag.

The freeze lining alteration has two possible causes: decay of the initial outer zones which are rebuilt from the final slag, or the diffusion of elements into the existing freeze lining. Diffusion is expected since the Zn and Pb concentration are lower than in the final slag.

According to these results it is expected that the freeze lining thickness in a zinc fuming furnace will not change significantly by changing bath compositions.

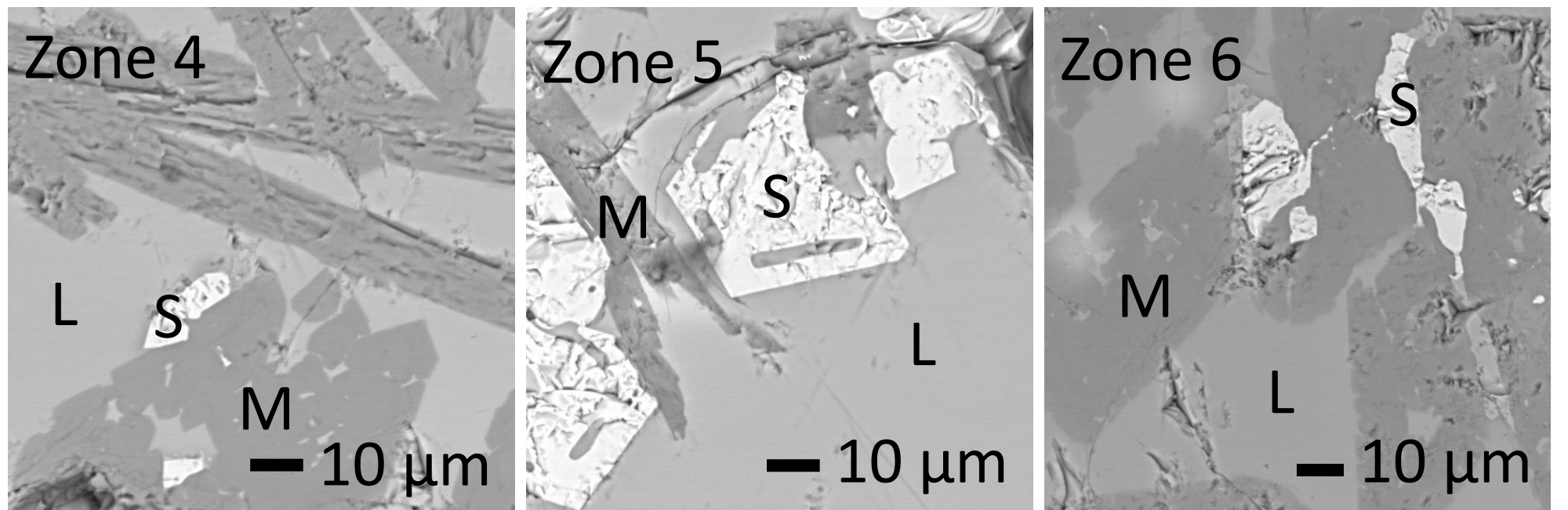


Figure 3: Microstructures with the phases M (melilite), L (quenched liquid) and S (spinel)

Table 2: The Zn and Pb content increases when moving to the outer zones of the freeze lining

phase	Zn content (wt%)			Pb content (wt%)		
	zone 4	zone 5	zone 6	zone 4	zone 5	zone 6
melilite	0	0.1	0.5	0	0	0.1
liquid	0	2.2	4.2	0	1.2	2.4
spinel	0	1.8	4.5	0	0	0.2

CONCLUSIONS

- Six different zones are distinguished
- Zn and Pb are only observed in the two outer zones: the open crystalline layer and the bath material with formed crystals
- The changes are probably caused by diffusion
- The freeze lining thickness did not change significantly by the changing bath composition

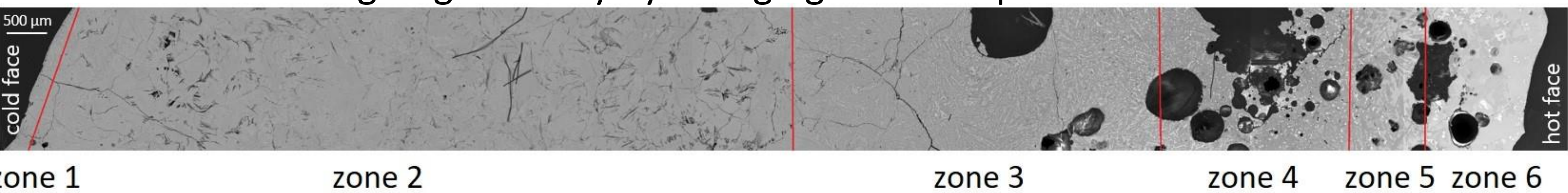


Figure 2: Cross section of the freeze lining

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