Supporting Information

Investigating the Interaction between Ilmenite and Zinc for Chemical Looping

Ivana Staničić[1*], Emil Ola Lidman Olsson[2,3], Hao Wu[2], Peter Glarborg[2], Iñaki Adánez-Rubio[4], Henrik Leion[5], Tobias Mattisson[1]

[1] Department of Space, Earth and Environment, Division of Energy Technology, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden
[2] CHEC Research Centre, Department of Chemical and Biochemical Engineering, Technical University of Denmark, Kongens Lyngby 2800, Denmark
[3] Sino-Danish College, University of Chinese Academy of Science, Eastern Yanqihu campus, 380 Huaibeizhuang, Huairou district, Beijing, 101400, China
[4] Instituto de Carboquímica (ICB-CSIC), Miguel Luesma Castán 4, 50018, Zaragoza, Spain
[5] Chemistry and Chemical Engineering, Chalmers University of Technology, 412 93 Göteborg, Sweden
* stanicic@chalmers.se

1. X-ray Diffractograms

1.1. Synthetic ilmenite

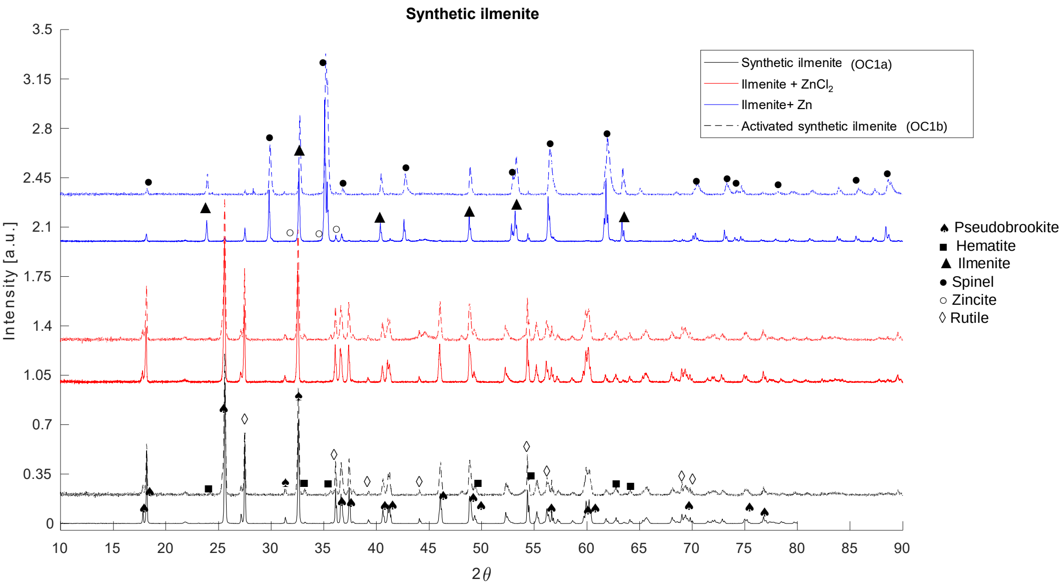


Figure S 1 Diffractograms for the six samples with synthetic ilmenite. Normalized intensity against two theta angle. Dashed lines represent diffractograms of the activated sample OC1b.

1.2. Rock Ilmenite

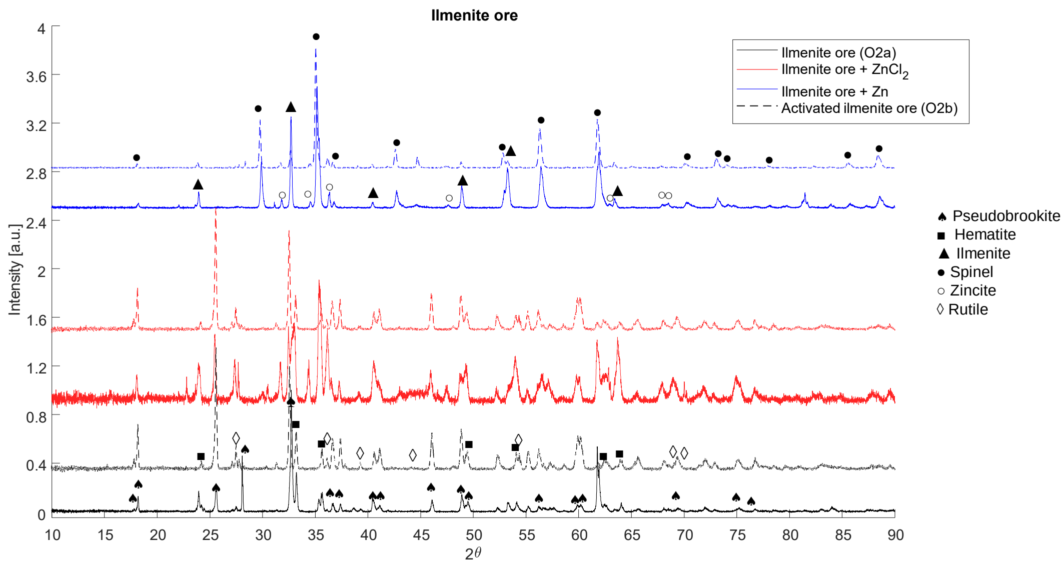


Figure S 2 Diffractograms for the six rock ilmenite samples. Normalized intensity against two theta angle. Dashed lines represent diffractograms of the activated sample OC2b.

1.3. Used ilmenite

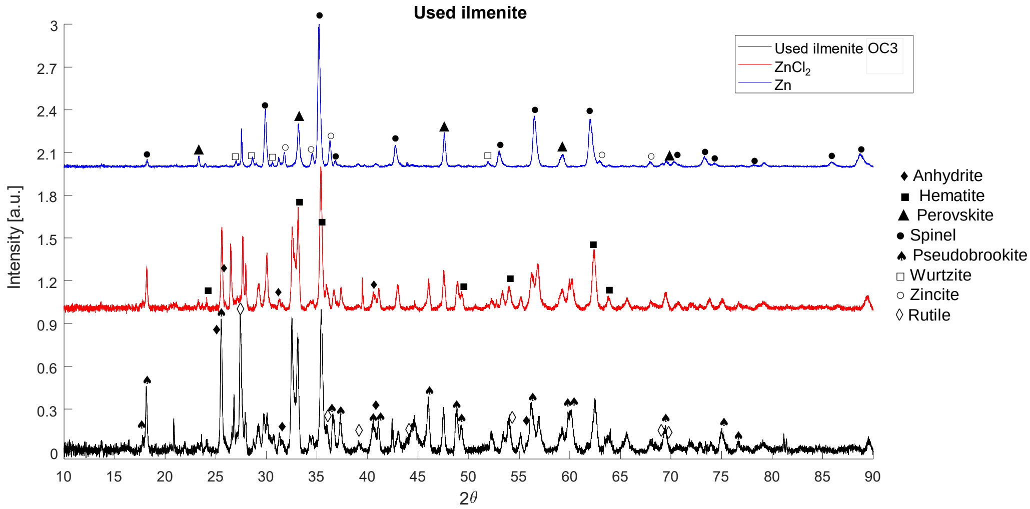


Figure S 3 Diffractograms of used ilmenite (OC3). Normalized intensity against two theta angle. Phases are indicated in the figure.

2. Thermodynamic Equilibrium Calculations

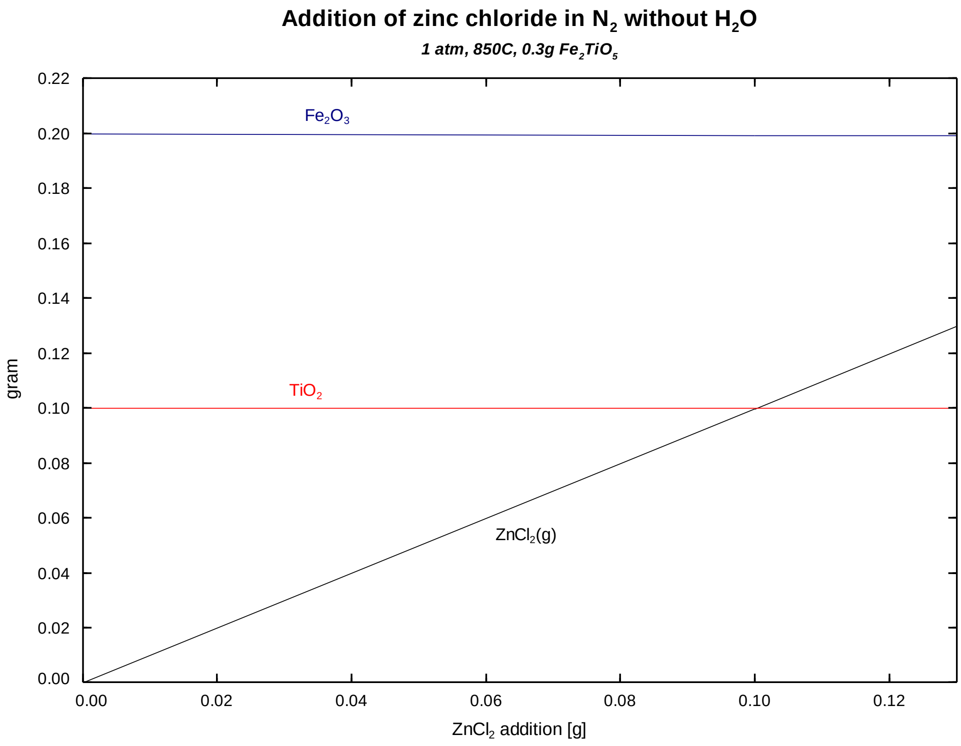


Figure S 4 Evolution of phases during stepwise addition of 0.13 g zinc chloride to 0.3 g oxidized ilmenite at 1 atm, 850°C in the absence of steam. As observed in the figure, there is no interaction between zinc and ilmenite and only Fe_2O_3 (s), TiO_2 (s), and $ZnCl_2$ (g) are stable.

3. Temperature Profile

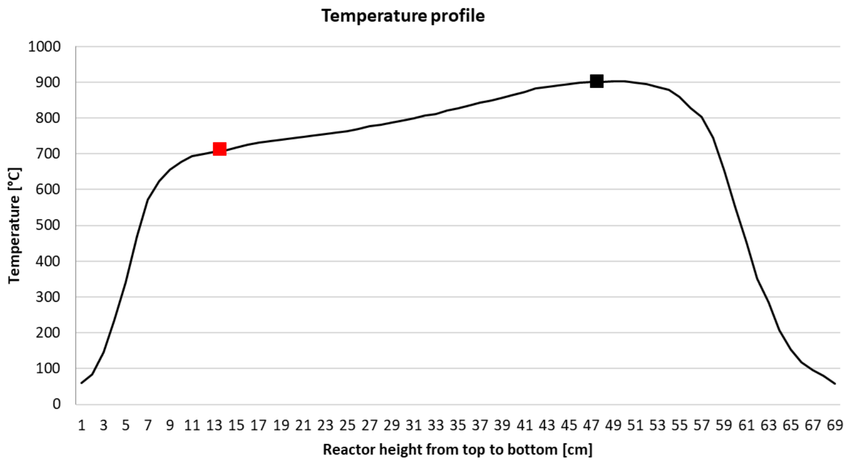


Figure S 5 Temperature profile for the zinc experiments using the setpoint temperatures 715°C -800°C-900°C. The position for Zn (red square) and ilmenite (black square) is indicated in the figure.

4. Rock Ilmenite– Morphology and Chemical Distribution

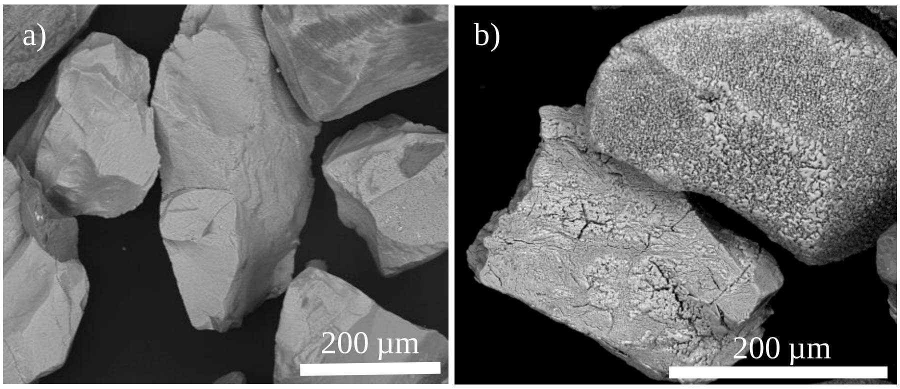


Figure S 6 a) Surface micrograph (20 kV) of rock ilmenite (OC2a) and b) surface micrograph of activated ilmenite (OC2b) showing the structural changes due to activation.

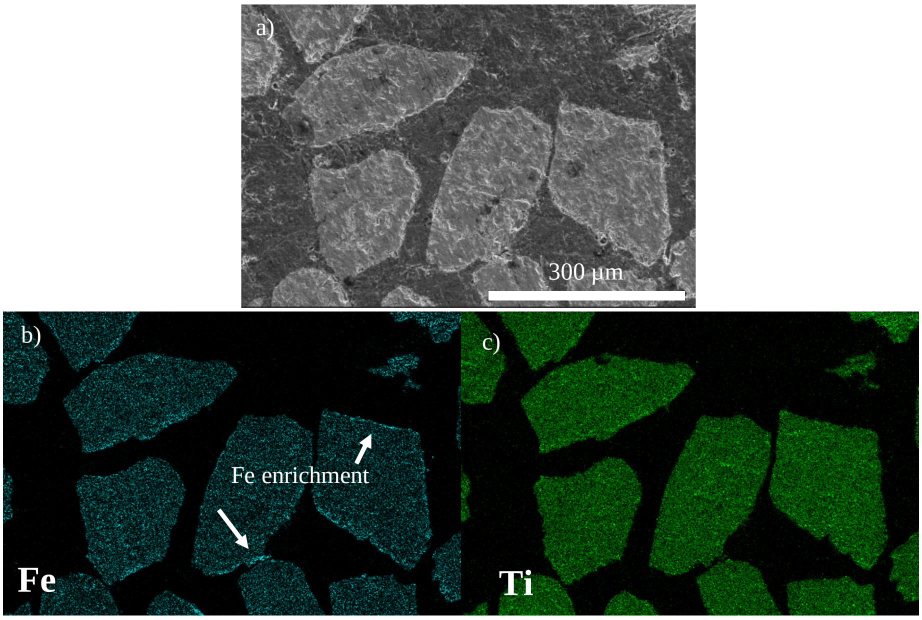


Figure S 7 a) Cross-section micrograph of rock ilmenite (OC2b) (SE-detector) along with chemical maps of iron b) and titanium c). The iron enrichment is indicated in the figure.

5. Synthesized Ilmenite – Chemical Distribution

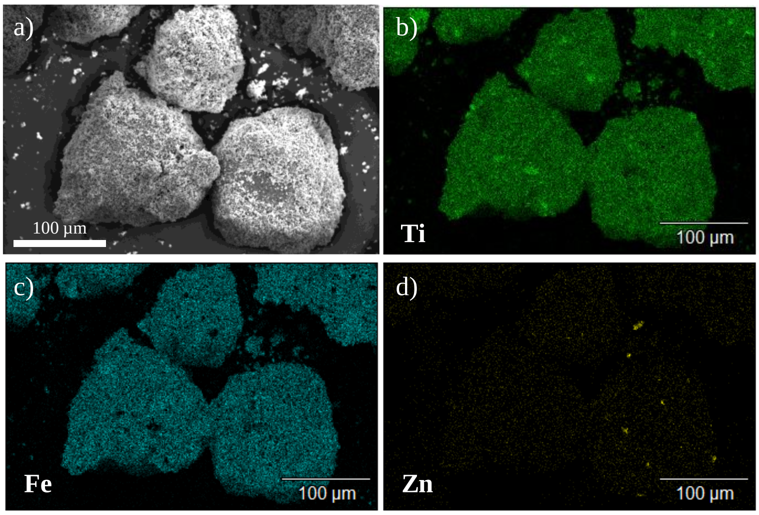


Figure S 8 a) Surface micrograph of synthesized ilmenite (OC1a) after experiments with $ZnCl_2$ along with chemical maps of titanium (b), iron (c), and zinc (d).

6. Used Ilmenite – Chemical Distribution

6.1. Experiments with Zinc Chloride

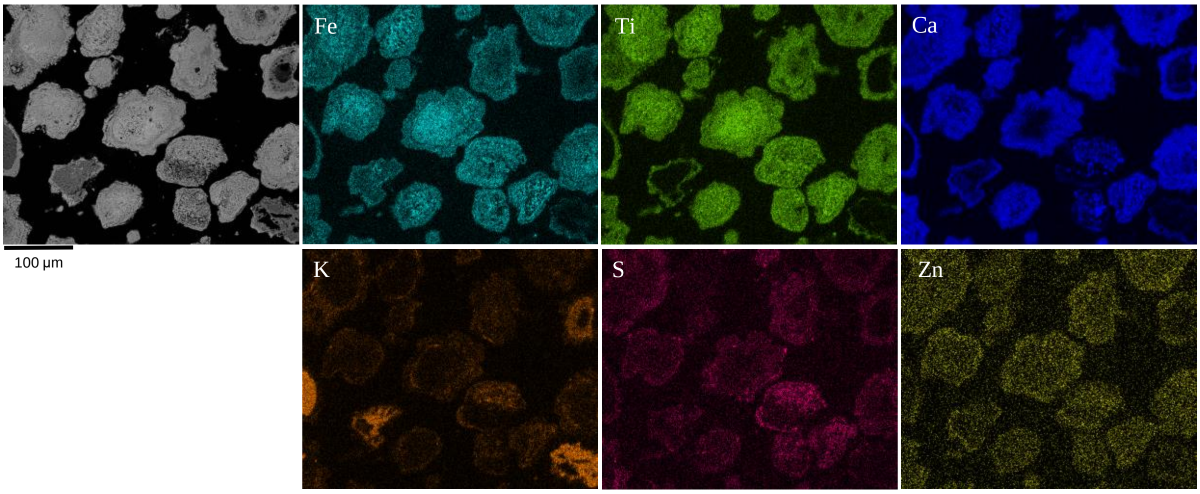
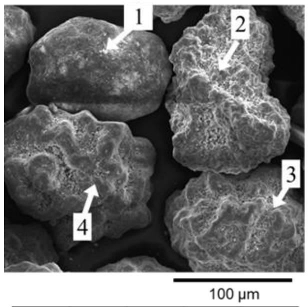


Figure S 9 Micrograph of the cross-section of used ilmenite (sample OC3) before experiments along with the chemical maps of the elements iron, titanium, zinc, potassium, sulfur and calcium. The saturation fraction is 9.5%.



At%	1	2	3	4
Fe	7.7	7.6	25.8	16.9
Ti	8.8	3.2	15.8	16.0
Ca	2.5	7.9	17.6	5.7
K	5.3	0.6	0.4	0.1
Cl	9.7	0.0	0.0	0.0
Zn	8.3	2.6	8.5	5.9

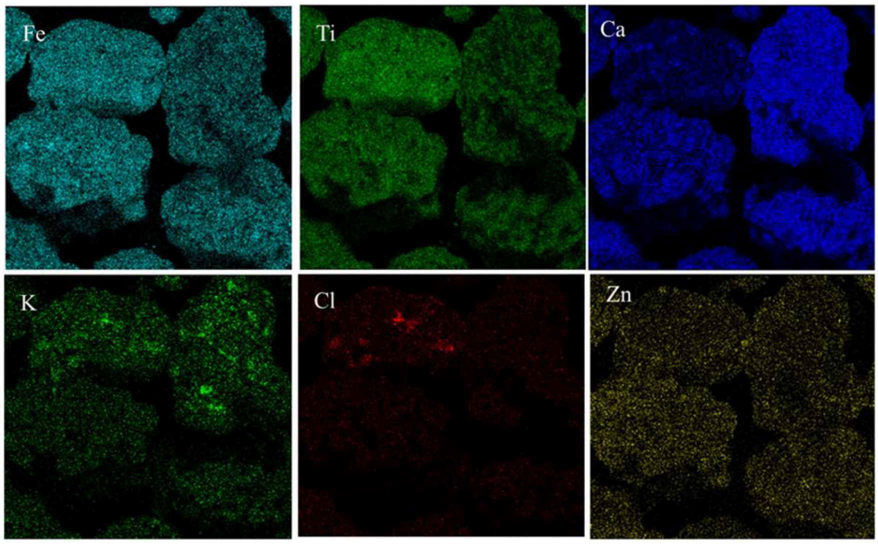


Figure S 10 Surface micrograph of used ilmenite (sample OC3) after gas-solid interaction with zinc chloride along with the chemical maps of existing ash elements potassium and calcium as well as iron, titanium, zinc and chlorine. Point analyses are presented in the table.

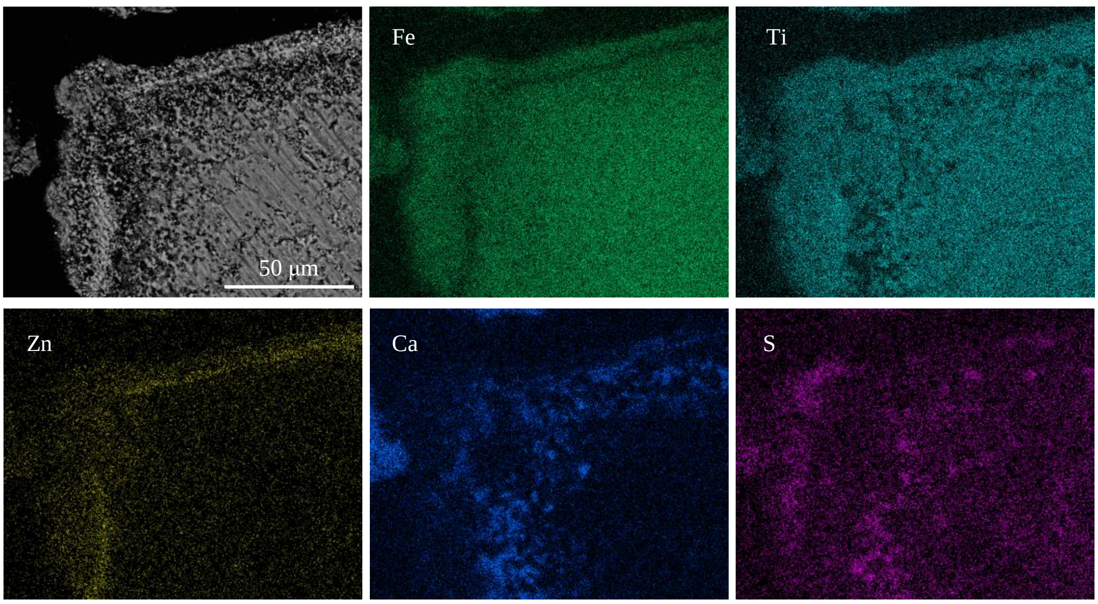


Figure S 11 Micrograph of the cross-section of used ilmenite (OC3) after experiments with zinc chloride. Chemical mappings of iron, titanium, calcium, sulfur, and zinc are presented. The saturation fraction is 19%.

6.2. Experiments with Metallic Zinc

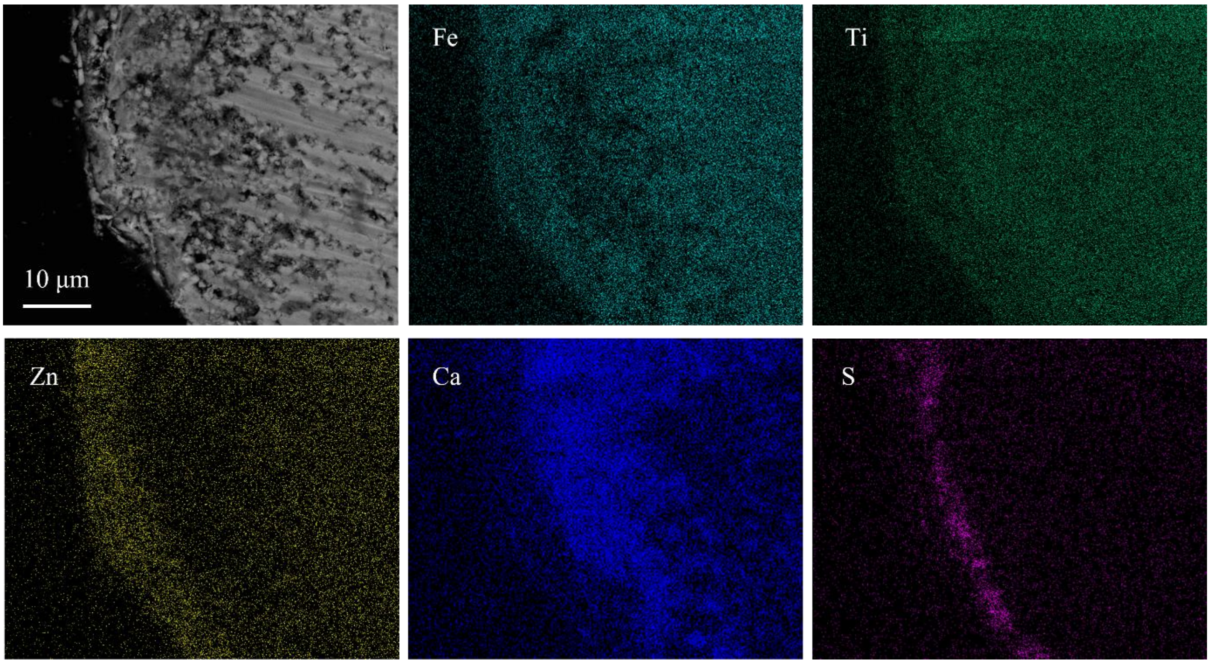


Figure S 12 Micrograph and chemical maps of used ilmenite (OC3) after experiments with metallic zinc. Chemical maps of iron, titanium and the ash elements calcium, sulfur and zinc. The saturation fraction is 118%.

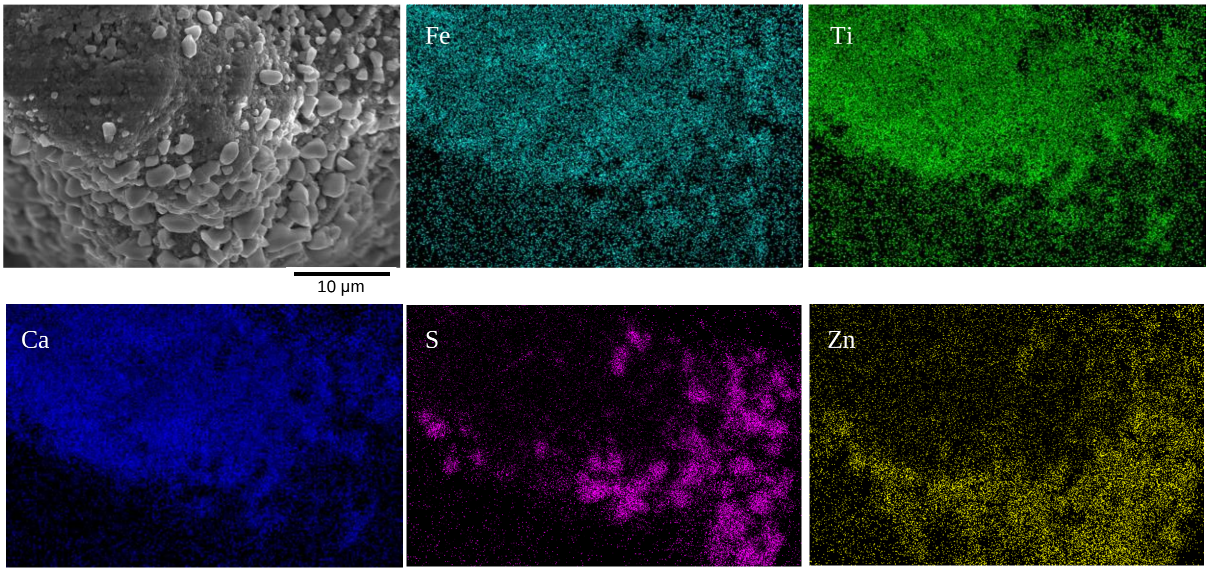


Figure S 13 Surface micrograph of used ilmenite (sample OC3) after experiments with metallic zinc along with the chemical mappings of the ash elements calcium and sulfur as well as iron, titanium and zinc.

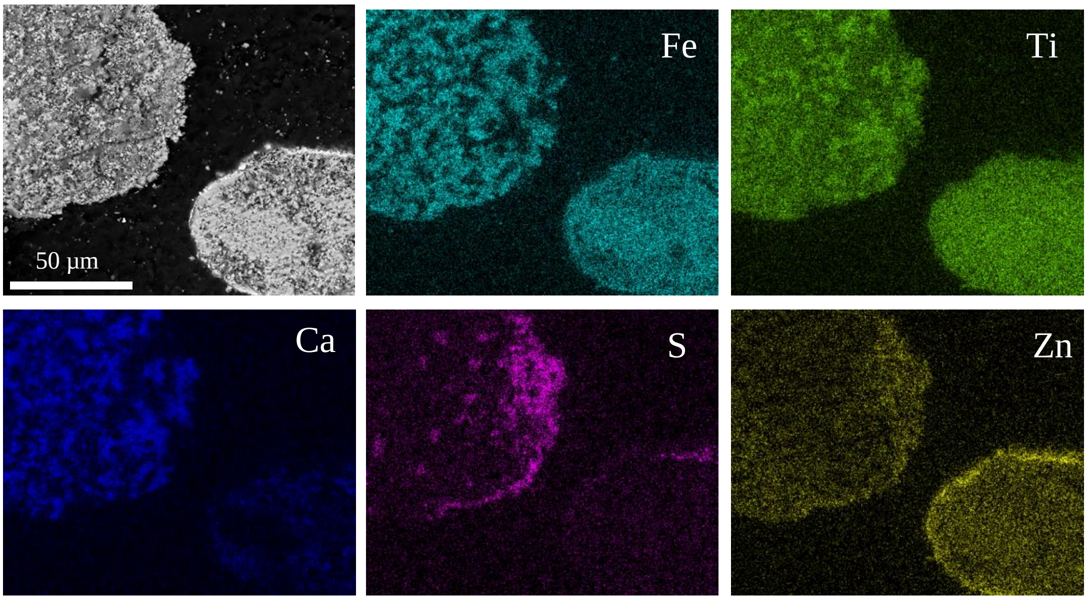


Figure S 14 Micrograph and chemical maps of used ilmenite (OC3) after experiments with metallic zinc. Chemical maps of iron, titanium and the ash elements calcium, sulfur and zinc. The figure illustrates the interaction with an Ca-rich particle (left) and an Ca-lean particle (right).