Low-temperature kinetics of the oxidation of iron powders

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Motivation and summary

The kinetics of solid-state oxidation of iron dominates the ignition step [1], hence strongly influences the combustion behavior of the powder. However, a detailed description of the oxidation process is currently missing.
To this end, we carried out a thorough kinetic investigation of the oxidation of micron-sized iron particles based on thermogravimetric analysis (TGA), product characterization by SEM imaging and XRD analysis, and particle-based models.

TGA test campaign

Approximately 100 individual experiments have been conducted with two TGA instruments to ensure reproducibility of the results.

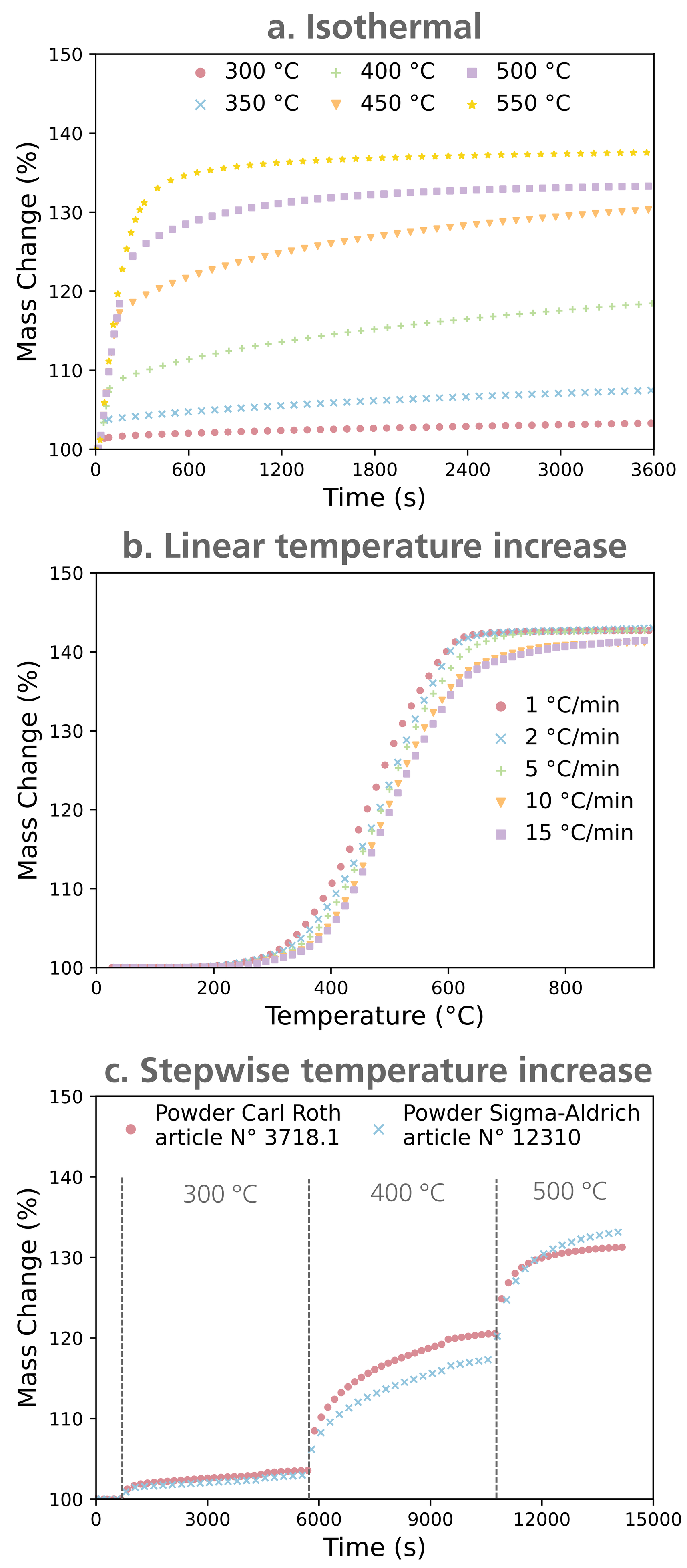
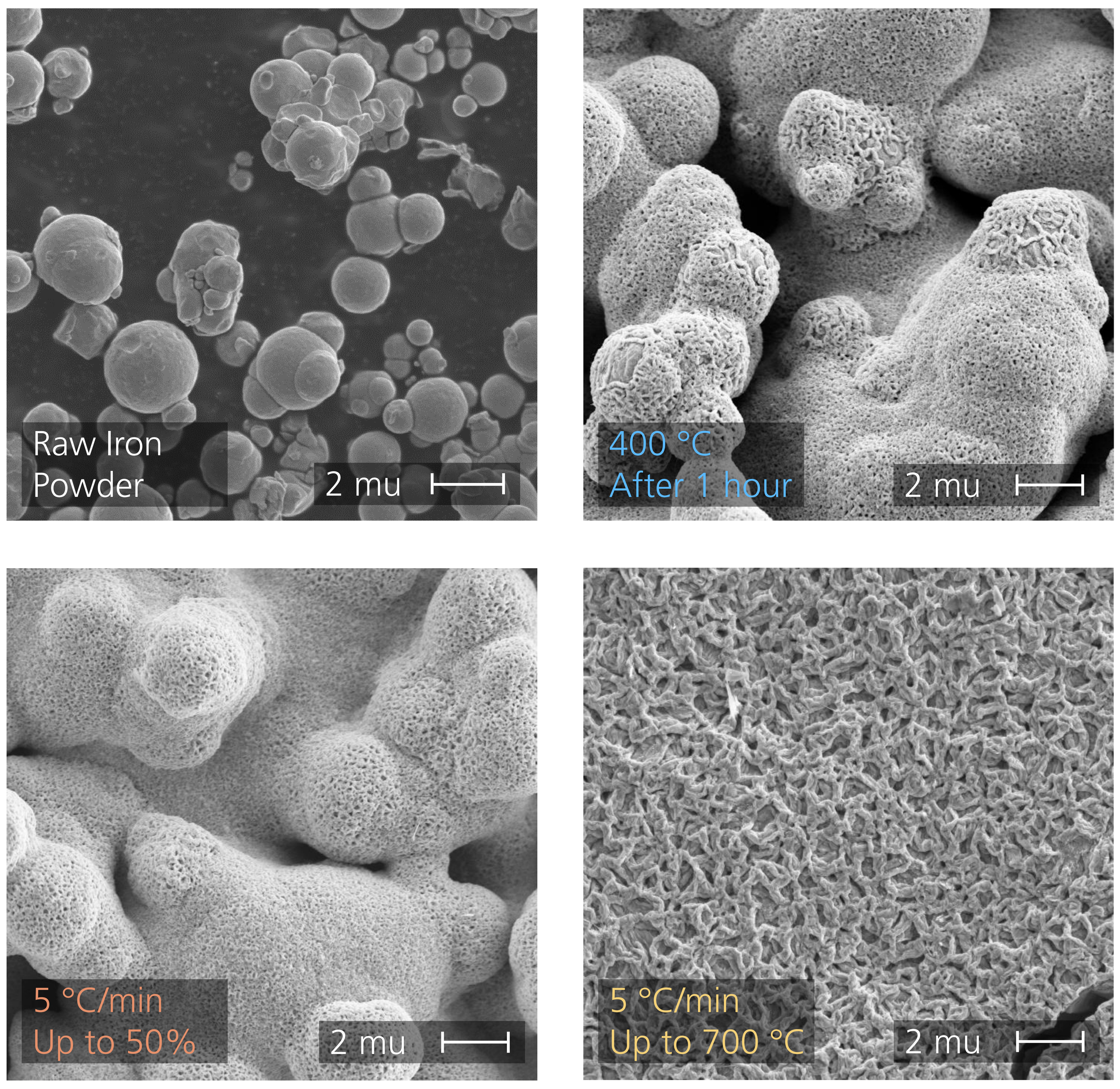


Figure 1: Few results from the TGA test campaign performed by UC3M and DLR: a. under isothermal conditions; b. with linear increases of the temperature and c. stepwise increases of the temperature 300-400-500 °C

Product characterization



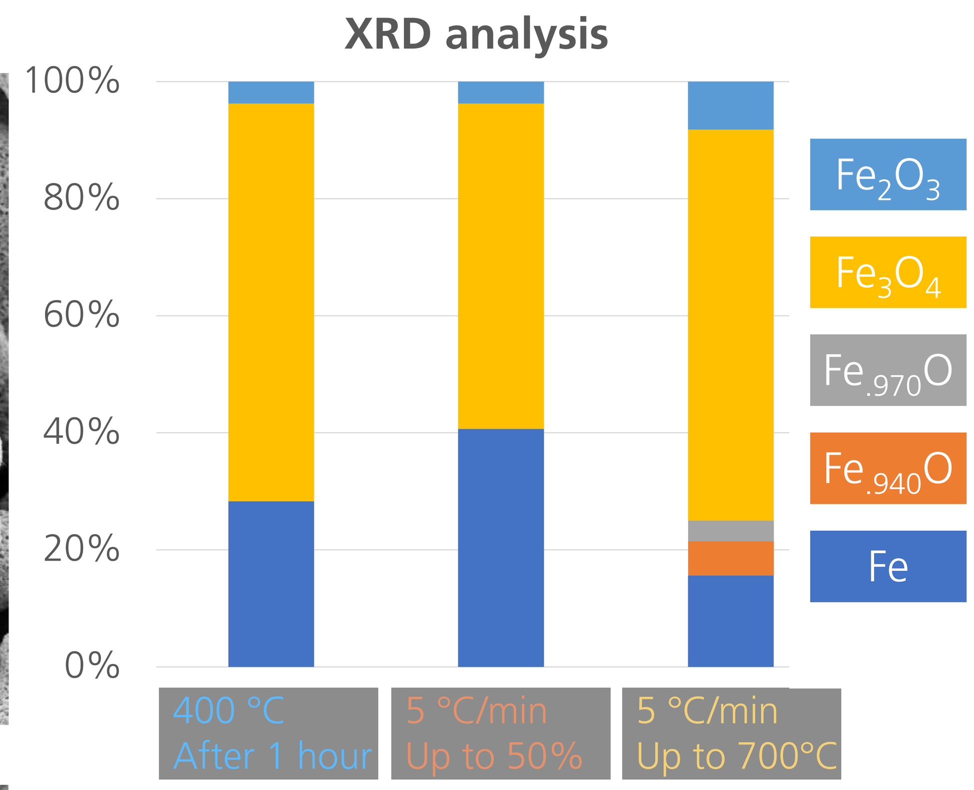


Figure 2: SEM images of raw iron and oxidized products under various conditions and corresponding XRD analysis - conducted by UC Louvain

Main results

Under the tested conditions, mostly magnetite (Fe_3O_4) forms during the oxidation.
Though single particle are still identifiable for partly-oxidized samples, sintering causes the formation of a continuous surface on the analyzed samples between 500 and 700 °C.

Interpretation

From the TGA data, the microstructural characterization, and literature on oxide scales [2,3], the following oxide growth mechanism for the oxidation of iron particles under 570 °C is suggested:

- Formation of a duplex magnetite layer from
  - outward migration of cations to Fe_3O_4/Fe_2O_3 interface, forming a columnar magnetite outer layer
  - inward transport of oxygen to the metal/oxide interface, forming an equi-axed inner layer
- Formation of a thin hematite layer by cation outward diffusion to the oxide/air interface

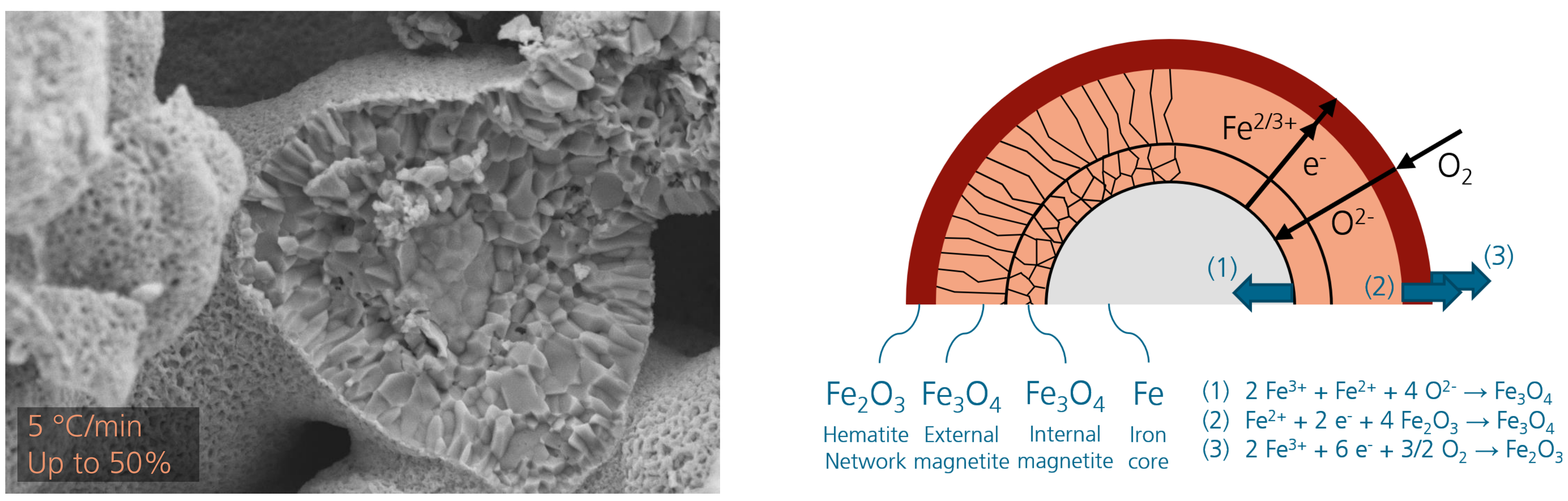


Figure 3: (left) SEM image of a fractured iron/iron oxide particle from a 50%-oxidized sample and (right) scheme of the oxidation process; not to scale

Outlook

- Determination of the rate-limiting steps by combining microstructural analysis and the TGA curves
- Integration of a mathematical description of the sintering phenomenon into a kinetic model [4] based on lattice diffusion of ions following Wagner's theory for isolated iron particles [5]
- Comprehensive model validation with the TGA experiments to determine accurate kinetic parameters

References

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