Manufacturing of Fine Spherical Iron Powder and the Influence of the Powder Morphology on the Sintering Behaviour

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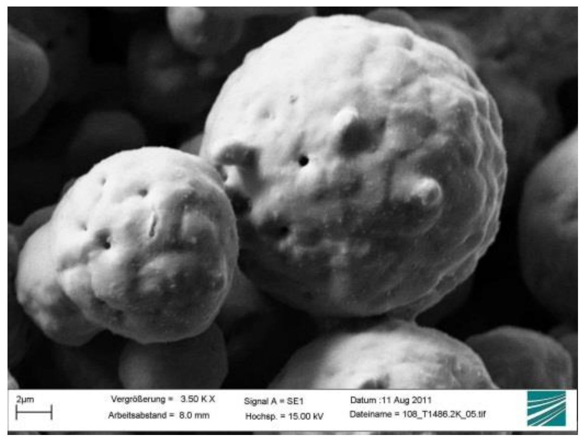

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Content

- Motivation and objectives
- Manufacturing technology
  - Hydrogen reduction process
  - Powder processing
- Powder properties
- Sintering behavior
- Summary and outlook

Motivation and Objective

- Application of spherical iron powders < 20 µm
  - magnetorheological liquids
  - materials absorbing microwaves
  - MIM
  - slurry technologies (direct typing, hollow spheres and metal foam manufacturing)
  - Diamond tools
- high price level of iron carbonyl or atomized powders (~7-10 €/kg) is a limiting fact for many applications (competition to alternative production technologies)
- → alternative production route for fine powders?
- iron oxide powder (hematite) - waste product from pickling slurry of the steel industry (d_{50} < 1 µm)
- current application in color pigments and ferrite industry
- question: possibility to use iron oxide for powder production?

Motivation and Objective

- Powder properties required for MIM
  - **Low particle size** (< 20 µm) to ensure low surface roughness and high part precision
  - **High sinter activity** to obtain sinter densities > 95%
  - **Spherical particle size** to get an injectable feedstock with high powder loading and low tool wear

- target: → development of a cost efficient powder manufacturing process to reduce iron oxide to spherical iron powder < 20 µm

Manufacturing Technology – Idea and approach

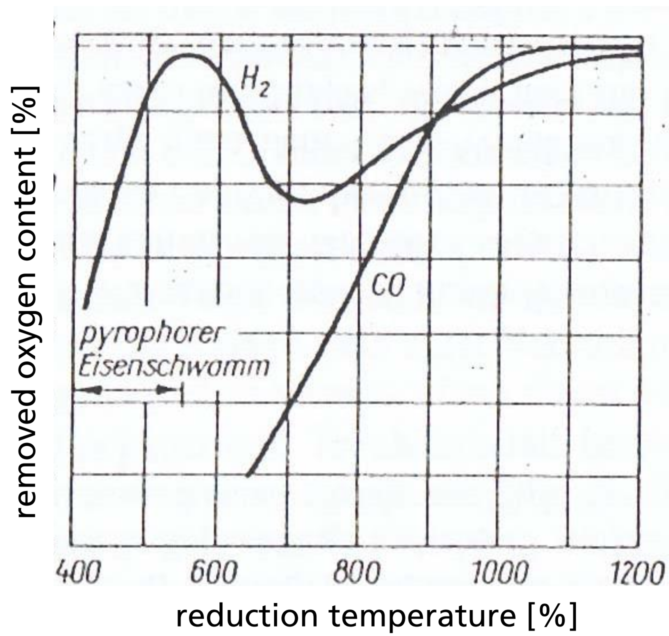
- **CO** → full reduction of iron oxide only by high temperatures (>1000 °C) → formation of a strong sinter cake

- **H2**-reduction

  <u>1st temperature step</u>
  full reduction between 500°C and 600 °C

  →pyrophoric powder (specific surface >> 1 m²/g)

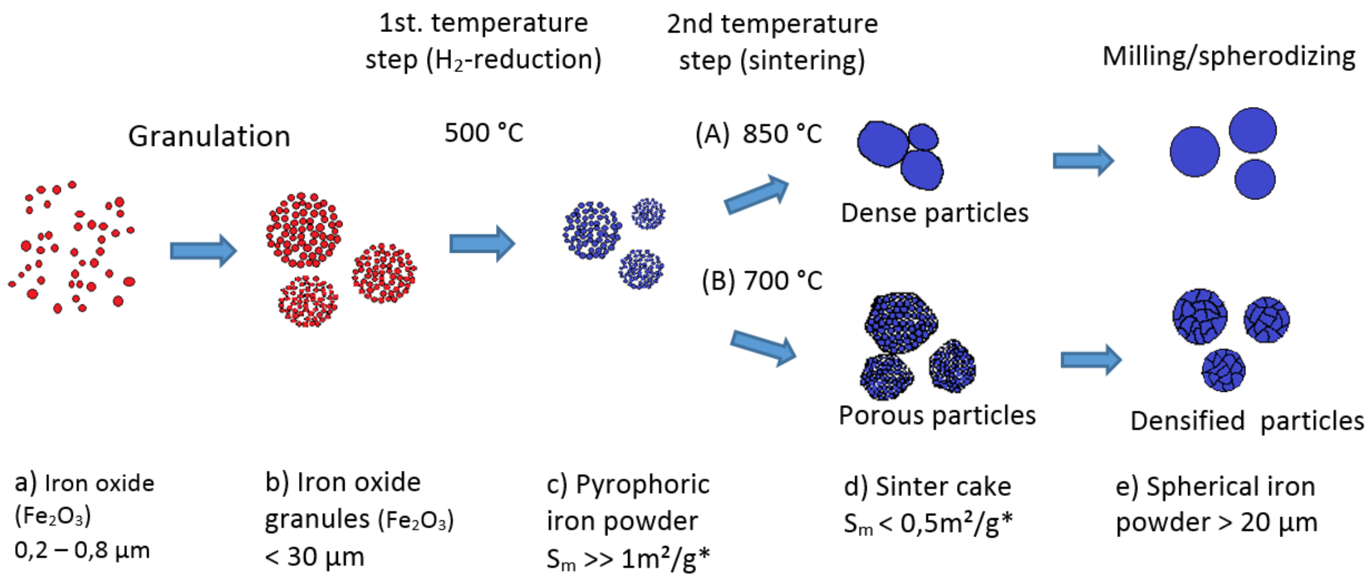
  <u>2nd temperature step</u> → 2nd temperature step is needed to reduce the specific surface, simultaneous the sinter cake has to be easily processed to a fine powder



temperature dependence of the reduction of iron oxide under H_2 and CO [M. Wiberg]

Manufacturing– Idea and approach

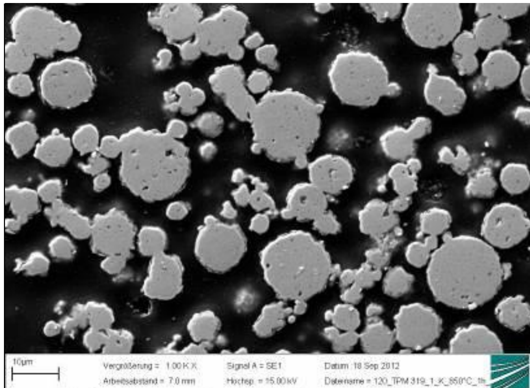
- sintering activity raises with increasing surface curvature (high surface energy)
  - granulation (spray drying) → high sinter activity of the primary particles → densification of the granules to spherical particles
  - granules among themselves → less sinter activity (lower surface curvature) → processable sinter cake



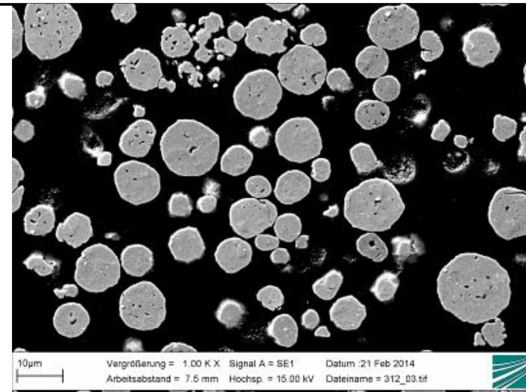
*S_m – specific surface

Results – Reduction and powder processing

reduction at 500°C, 1h + 850°, 1h under hydrogen

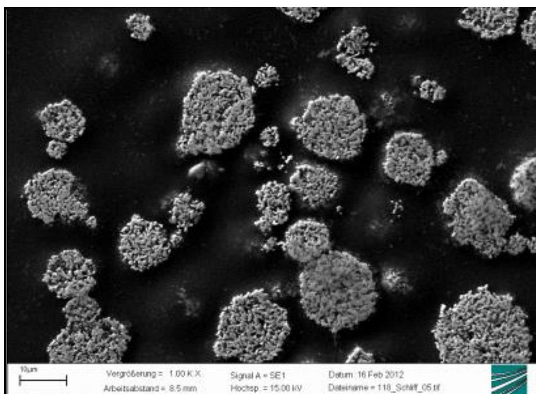


Sinter cake after reduction

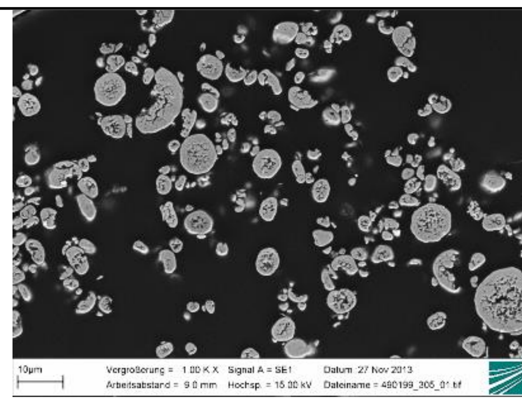


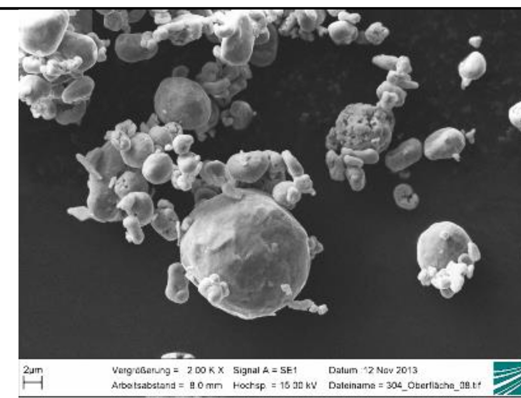
cross section and topography after milling in the Nara Hybridizer (NH) for 4 min, 16000 rpm

reduction at 500°C, 1h + 700°, 24h under hydrogen



Sinter cake after reduction





cross section and topography after milling in the Nara Hybridizer (NH) for 4 min, 16000 rpm

Results - Reduction

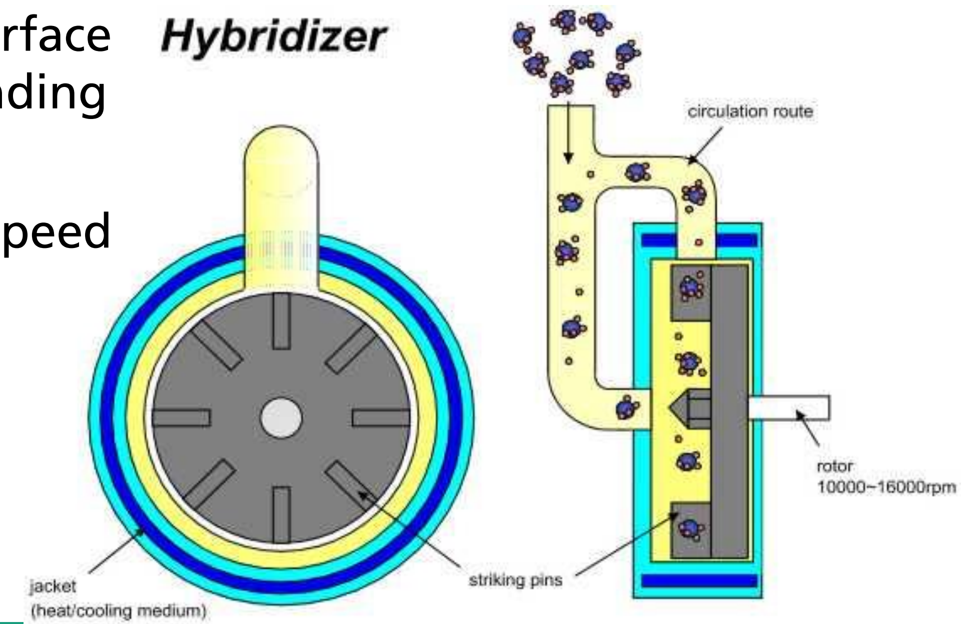
Properties of the main different reduced and milled powders in comparison to state of the art carbonyl and atomized iron powders

powder qualities	oxygen [%]	carbon [%]	particle size [µm]			apparent density [g/cm³]
			d_{10}	d_{50}	d_{90}	
carbonyl-Fe	0,240	0,760	2,1	4,5	8,6	4,2
atomized Fe	0,615	0,001	6,0	13,7	22,0	3,7
reduced 500°C, 1h + 850°C, 1h	0,368	0,042	13,2	22,1	35,6	2,7
milled 240 s, 16000 U/min NH	-	-	7,3	11,4	16,9	3,3
reduced 500°C, 1h + 700°C, 24h	0,741	0,132	11,6	18,6	29,4	2,2
milled 240 s, 16000 U/min NH	-	-	3,4	5,1	7,6	2,8

Powder processing

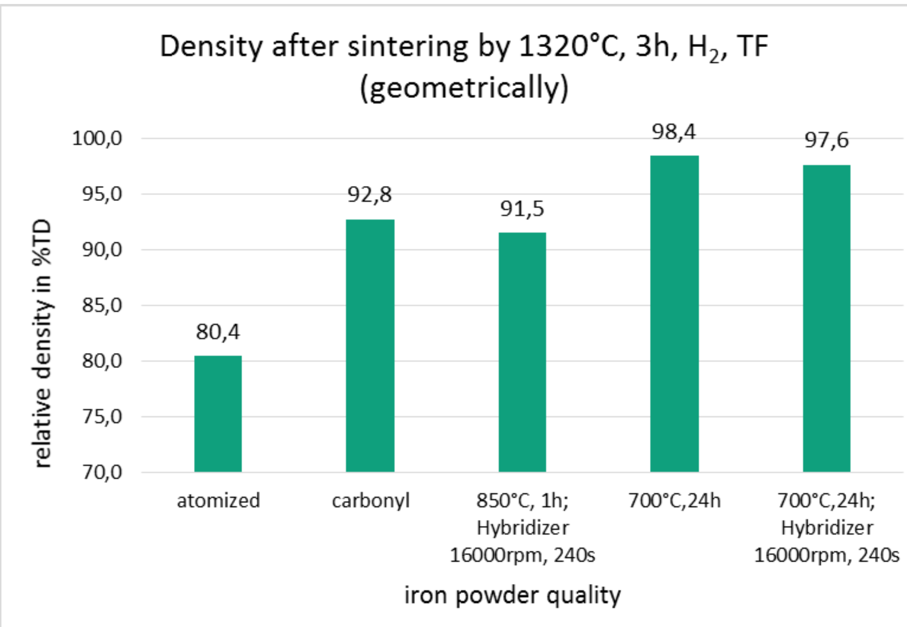
- Aim:
  - separate sinter cake (agglomerates) to smallest possible particle size
  - spherical particles → high shear stresses without plastic deformation

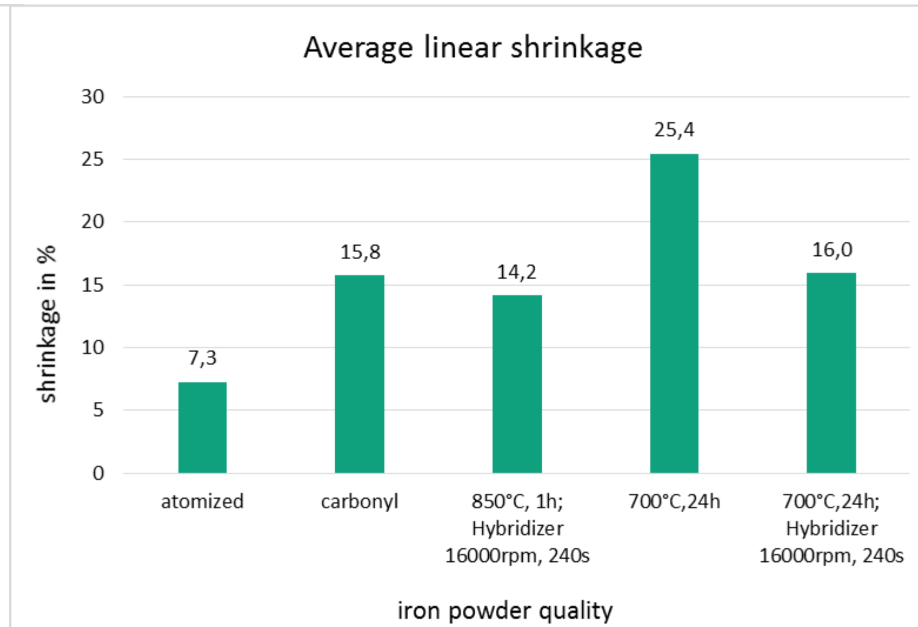
- Milling unit:
  - Nara Hybridizer (NH): 4 min with 16000 rpm
    - Innovative milling system for surface modification especially for rounding of particles
    - Powder is circulating in a high speed gas flow (Ar) through rotating bucket wheel → crushing and rounding by impact and shear forces



Sintering behavior

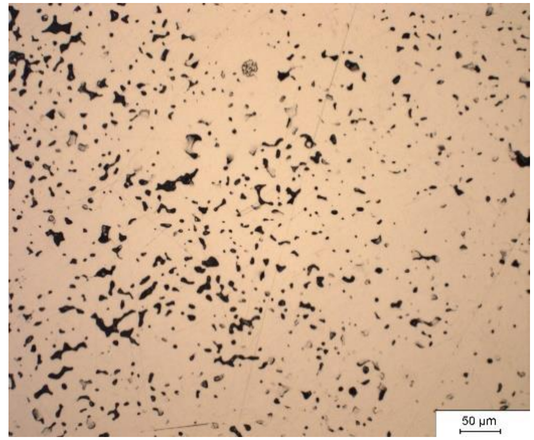
- Aim: evaluation of shrinkage and sinter density
- Cylindrical samples Ø8 mm pressed with 100 MPa and 0.5 % binder
- Sintered at 1320 °C for 3h under H_2 in a tube furnace (TF)



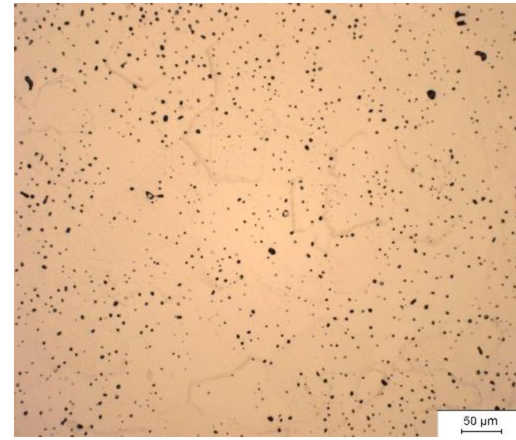


Sintering behavior – cross section

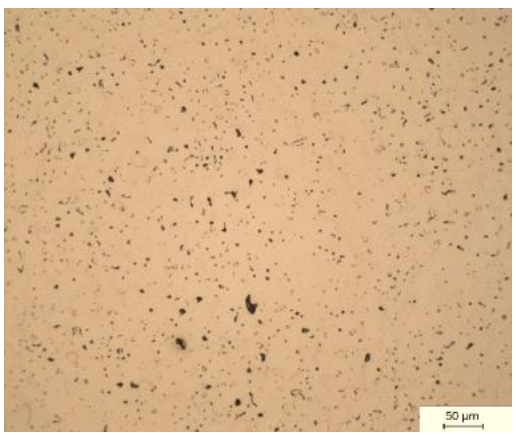
- Highest porosity for sinter part using atomized powder
- Carbonyl Fe and reduced powders – fine distributed pores
- Lowest porosity for sinter part using powder reduced at 700°C,24h



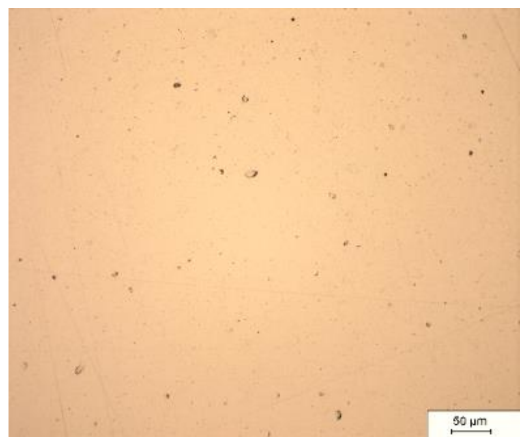
atomized Fe



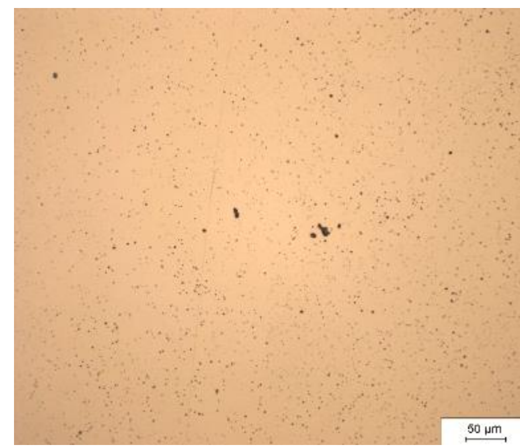
carbonyl Fe



reduced 850°C,1h and Hybridizer milled for 240s, 16000 rpm



reduced 700°C, 24h, not milled



reduced 700°C, 24h and Hybridizer milled for 240s, 16000 rpm

Summary and Outlook

- Process is suitable to produce fine spherical iron powder
- Two potential powder qualities:
  - Spherical dense powder (850°C) → sinter density and shrinkage comparable to carbonyl iron, but particle size higher
  - Near spherical/potato-shaped porous powder (700°C) → particle size and shrinkage comparable to carbonyl iron, sinter density is higher;
    impact of not real spherical morphology to MIM-processing must be proved
- New process:
  - Environmentally friendly process chain
  - Low-cost raw material (from recycling process)
  - Production of a cost efficient powder
  - → High potential for MIM market by powder cost reduction (especially with increasing MIM parts dimensions)
- Further development:
  - Process optimization and upscaling for industrial applications

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Thank you for your Attention!

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