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

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Motivation and Objective

- Application of spherical iron powders < 20 µm
  - magnetorheological liquids
  - materials absorbing microwaves
  - MIM

- 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
  1st temperature step
  full reduction between 500°C and 600 °C

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

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

![Graph showing the temperature dependence of the reduction of iron oxide under H₂ 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

Diagram:

1. Granulation
2. 1st. temperature step (H₂-reduction) 500 °C
3. 2nd temperature step (sintering)
   - (A) 850 °C
   - (B) 700 °C
4. Milling/spherodizing

- a) Iron oxide (Fe₂O₃) 0.2 – 0.8 µm
- b) Iron oxide granules (Fe₂O₃) < 30 µm
- c) Pyrophoric iron powder Sₘ > 1m²/g*
- d) Sinter cake Sₘ < 0.5m²/g*
- e) Spherical iron powder > 20 µm

*Sₘ – 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

<table>
<thead>
<tr>
<th>powder qualities</th>
<th>oxygen [%]</th>
<th>carbon [%]</th>
<th>particle size [µm]</th>
<th>apparent density [g/cm³]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>$d_{10}$</td>
<td>$d_{50}$</td>
</tr>
<tr>
<td>carbonyl-Fe</td>
<td>0,240</td>
<td>0,760</td>
<td>2,1</td>
<td>4,5</td>
</tr>
<tr>
<td>atomized Fe</td>
<td>0,615</td>
<td>0,001</td>
<td>6,0</td>
<td>13,7</td>
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<tr>
<td>reduced 500°C, 1h + 850°C, 1h</td>
<td>0,368</td>
<td>0,042</td>
<td>13,2</td>
<td>22,1</td>
</tr>
<tr>
<td>milled 240 s, 16000 U/min NH</td>
<td>-</td>
<td>-</td>
<td>7,3</td>
<td>11,4</td>
</tr>
<tr>
<td>reduced 500°C, 1h + 700°C, 24h</td>
<td>0,741</td>
<td>0,132</td>
<td>11,6</td>
<td>18,6</td>
</tr>
<tr>
<td>milled 240 s, 16000 U/min NH</td>
<td>-</td>
<td>-</td>
<td>3,4</td>
<td>5,1</td>
</tr>
</tbody>
</table>
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 H₂ in a tube furnace (TF)

![Density after sintering by 1320°C, 3h, H₂, TF (geometrically)](chart1)

![Average linear shrinkage](chart2)
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

- 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
Acknowledgements

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Thank you for your Attention!

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