Powder Manufacturing & Characterization

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Outline I

**Powder Production Methods:**

- Mechanical
  - Disintegration without phase change
    - Machining
    - Mechanical comminution (milling)
  - Disintegration with phase change
    - Atomization techniques
- Chemical & electrochemical methods
  - Chemical reduction
    - Ore reduction
    - Thermal decomposition (carbonyl powders)
    - Hydrometallurgy
    - ...
  - Electrolytic reduction
    - Precipitation from aqueous solutions
    - Melt electrolysis
Outline II

**Powder Characterization:**

- Physical properties
  - Size
  - Shape
- Chemical properties
  - Chemical composition
  - Phase distribution
- Processing properties
  - Bulk:
    - Density (Tap, Apparent), Flow, Fillability, Segregation
  - Pressed:
    - Density (Green), Strength (Green), Compressibility
  - Sintered
    - Mechanical properties
    - Chemical properties
    - Tolerances
    - Appearance
Powder Production Methods – Mechanical

Disintegration without phase change
Machining

Al 6061-T6 particulate produced by Modulated Assisted Machining

J. B. Mann et al. / Scripta Materialia 57 (2007) 909–912
Mechanical comminution

- Roll crushing
- Ball mill
- Hammer milling

- Frequency of rotation
- Diameter of the mill
- Size of the mill balls
- Proportion of material/mill balls
- Degree of filling of the mill

Powder Production Methods – Mechanical

Disintegration with phase change
Powder Production Methods – Mechanical

Disintegration with phase change - Atomization

Gas atomization

Water atomization

Atomization with a rotating consumable electrode

Centrifugal atomization


Water atomization

Atomization process variables:
- Temperature and amount of superheat of the molten metal (composition)
- Water/metal ratio (10-15 liter per 1 kg of produced powder)
- Diameter of the molten metal stream
- Geometry of the nozzle (amount of water jets and angle of incidence between water jet and molten metal stream)
- Water pressure
Water atomization

Lubanska $d_{50} = kD \left[ \left( \frac{\eta_m}{\nu Y_D} \right) \left( 1 - \frac{f_m}{f_l} \right) \right]^X = kD \left[ \left( \frac{\eta_m \rho \sigma}{\nu Y_D} \right) \left( 1 - \frac{f_m}{f_l} \right) \right]^X$

- $k$=constant
- $D$=diameter of the tundish nozzle, mm
- $f_m$ = metal flow, kg/min
- $f_l$ = water flow, $\frac{\text{liters}}{\text{min}}$
- $\eta_m$ = kinematic viscosity of metal, $\frac{m^2}{s}$
- $\rho$ = density of metal, $\frac{g}{\text{cm}^3}$
- $\sigma$ = surface tension of metal, $\frac{N}{m}$
- $V$ = water velocity at the impact point, $\frac{m}{s}$

Figure 5.4 Water jet configurations: (a) annular jet; (b) open V-jets; (c) closed V-jets.
The Water-Atomising Process

1. Selected scrap
2. Arc furnace
3. Liquid steel
4. Injection
5. Atomising
6. Dewatering
7. Drying
8. Magnetic separation
9. Screening
10. Equalizing
11. Transportation to the powder plant

5. Atomising
   A. Tundish
   B. Steel stream
   C. High-pressure water
   D. Nozzle
   E. Atomised iron powder
Scrap selection & Electric Arc Furnace Melting

Selected steel scrap

Raw material melting and addition of alloying elements (if needed)
Molten metal transfer

• Transfer of the molten metal to a ladle furnace and on the atomizing station
• Continuous control of oxygen levels and adjustment of the alloying elements content (if needed)
• Temperature control and adjustment if needed
Atomization
Annealing

The dried atomized powder is superficially oxidized and extremely hard.

Oxide and residual C reduction via soft annealing in belt furnaces.
Water atomized powder

ASC100.29
(pure Fe powder)

Astaloy CrM
(Fe-3Cr-0.5Mo)
pre-alloyed

Distaloy HP
(Fe-4Ni-2Cu-1.4Mo)
Diffusion alloyed with Ni and Cu
Gas atomization

• Used for the production of more oxidation sensitive materials that would be very difficult to produce via water atomization
• Production of high purity powders for special applications
• Spherical powders
Gas atomized powder

316L stainless steel powder
Powder Production Methods – Chemical

Ore reduction
Non-ferrous

Mo-powder: reduction of oxide or ammonium molybdate by hydrogen

W-powder: reduction of ore concentrates (wolframite or scheelite) by hydrogen

Re-powder: reduction of compound by hydrogen
The Sponge Iron Process
Direct reduction of iron ore (magnetite) $\text{Fe}_3\text{O}_4$ (Höganäs process)

- Reduction mix (coke breeze+limestone+coal)
- Drying
- Charging the tubes
- SiC tube
- Reduction mix

Reduction in tunnel klins - 1200°C-260m long

- Fe-ore
- Annealing
- Crushing, mag. separation, grinding & screening
Sponge Fe-powder

Due to the reduction processes the powder has a «spongy» appearance – high internal porosity
Powder Production Methods – Chemical

Carbonyl method

Production of high quality powders esp. Ni and Fe (possible also for Co, Cr, W, Mo, V, Mn etc)

1st step: formation of (penta-for Fe or tetra- for Ni)carbonyl: \( \text{Me} + x\text{CO} \rightarrow \text{Me}[\text{CO}]_x \)

2nd step: decomposition of the carbonyl: \( \text{Me}[\text{CO}]_x (g) \rightarrow \text{Me} + x\text{CO} \)
Powder Production Methods – Chemical

Hydride decomposition (HDH)

Used for precious metals. Hydrides are binary compounds of metals and hydrogen.

Main steps:

• Hydride Formation: Metals with high affinity to hydrogen (e.g. Ti) if heated (400°C for Ti) in the presence of hydrogen atmosphere they form hydrides

• Milling: the formed hydrides are brittle in nature and thus can be easily crushed and ground to fine powder

• Dehydridation: the fine hydride powder is heated (700-800°C) under dynamic vacuum at elevated temperature to eliminate hydrogen from metal, and consequently a fine metal powder can be obtained

Typical alloys consist of Ti-6Al-4V and Ti-6Al-6V-2Sn
Powder Production Methods – Chemical Electrolysis

Using aqueous solutions:
- direct deposition of a loosely adhering powdery or spongy deposit that can easily be disintegrated mechanically into fine particles (e.g. Cu and Ag)
- deposition of a dense, smooth, brittle layer of refined metal that can be ground into powder (e.g. Fe and Mn).

Following steps include washing, drying, screening, annealing etc.
Powder Production Methods – Chemical

The FFC Cambridge process

- Reduction of metal oxides to metals and mixtures of metal oxides to alloys in a solid state process using electrolysis.

- The reduction of the oxide(s) is taking place in a bath of electrolyte, typically molten calcium chloride at a temperature between 800°C and 1000°C and applying a cathodic potential to the oxide via a suitable immersed anode.

- The reduced metal is subsequently washed to remove salt, dried and further post processed as required (i.e. milling).

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Outline II

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  - Sintered
    - Mechanical properties
    - Chemical properties
    - Tolerances
    - Appearance
Physical Properties

Shape

- Acicular (chemical decomposition)
- Irregular rodlike (chemical decomposition, mechanical comminution)
- Flake (mechanical comminution)
- Dendritic (electrolytic)

i) 1-D

- Spherical (atomization, carbonyl (Fe), precipitation from liquid)
- Irregular (atomization, chemical decomposition)
- Rounded (atomization, chemical decomposition)
- Porous (reduction of oxides)

ii) 2-D

- Angular (mechanical disintegration, carbonyl (Ni))

iii) 3-D
Physical Properties

Shape

Atomized

Sponge

SEM

Cross-section
Physical Properties

Size

**Particle size**

Different definitions:

- **The sieve diameter** is the width of the minimum square aperture through which the particle will pass.

- **Projected area diameter** is the diameter of a circle having the same projected area as the particle in random orientation.
Physical Properties

Size

Particle size distribution

Laser diffraction (projected area diameter)

Decreasing sieve aperture

Sieve analysis (sieve diameter)
Chemical Properties
Chemical composition

Analysis Techniques

Surface analysis
- X-ray Photoelectron Spectroscopy
- Augen Electron Spectroscopy
- Secondary Ion Mass Spectroscopy

Bulk analysis
- Inductively Coupled Plasma
- Atomic Absorption Spectrometry
- Optical emission (High T combustion)
- Infra-red
- X-ray Fluorescence
- X-ray powder diffraction

Microanalysis
- Scanning Electron Microscopy
- Energy Dispersive X-ray Spectroscopy
- Nanoprobe

Chemical Properties

Phase distribution

Electron BackScatter Diffraction
Phase identification/orientation

Impurities and inclusions

- Chemical analysis (C, O, N, S, Ca, Al, Si, etc)
- Microscopy techniques

Processing Properties
Bulk: density, flow

**Apparent (or Bulk) Density (AD)** is the mass of particles divided by the volume of a loosely filled powder

Influenced by the lubricant/mix and is an indicator for the tool filling depth

**Flow rate (s/50g)** is the time required for 50g of dry powder to pass the aperture of a standardized funnel

Influenced by the lubricant/mix and is an indicator for the tool filling rate and consequently the productivity of the press
Processing Properties

Bulk: density, flow

Spherical (stainless steel)
AD: 4-4.5 g/cm$^3$
Flow: 13-15 s/50g

Sponge (irregular and porous)
AD: 2.3-2.65 g/cm$^3$
Flow: 29-32 s/50g

Atomized (irregular)
AD: ~3 g/cm$^3$
Flow: ~25 s/50g
Processing Properties

Bulk: fillability

Measuring the fill (apparent) density of each cavity as a function of the fill shoe speed

\[
\text{Fill index} = \frac{(\delta_{13\text{mm}} - \delta_{2\text{mm}})}{\delta_{13\text{mm}}} \times 100\%
\]

Die filling simulator
Processing Properties

Bulk: segregation

In a particulate system there are **no motions equivalent to the molecular diffusion** of gases and liquids.

Factors influencing segregation:

a) Particle shape
b) Particle size
c) Induced movement
d) Density of additives (organic material, graphite, alloying elements)
Processing Properties
Pressed: compressibility – green density

Factors influencing compressibility:

- a) Compaction pressure
- b) Particle shape/size
- c) Particle porosity
- d) Lubricant/organics content/type
- e) Powder composition

Increasing pressure

\[ \Phi 25 \text{mm} \]
Processing Properties
Pressed: compressibility – green density

ASC100.29
Processing Properties

Pressed: spring back

The elastic expansion of a compact after ejection from the compacting die

\[
\frac{\text{Outer diameter of the compact} - \text{Inner diameter of the die}}{\text{Inner diameter of the die}} \times 100
\]

Factors influencing spring back:

a) Compaction pressure
b) lubricant
c) Particle porosity
d) Chemistry
The bending strength of a green rectangular test bar

Factors influencing green strength:
- Powder surface area (shape and size)
- Compaction pressure
- Surface-to-volume ratio
- Chemistry
- Organics content (and type)
- Impurities or oxidation


Green strength at 600 MPa

(1) ABC 100.30
(2) ASC 100.29
(3) SC 100.26
(4) NC 100.24
(5) MH 80.23
Thank you