Additive Manufacturing of Metals and Alloys
Powder fabrication and characterization

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Demands for AM powder

- Question: How do we make a metal powder for AM?

Demands for AM powder

- «Good» flowability, no agglomeration
- High packing density (for LBPF and EBM)
- High permeablity (during LPBF or EBM, gas must be able to escape from the cavities inbetween powder particles)

/www.beam-machines.com/

Demands for AM powder

- «Good» flowability, no agglomeration
- High packing density
- High permeablity (gas must be able to escape from the cavities inbetween powder particles)

→ Spherical

→ Usual size distribution

  → 15 µm < d < 50 µm (SLM)

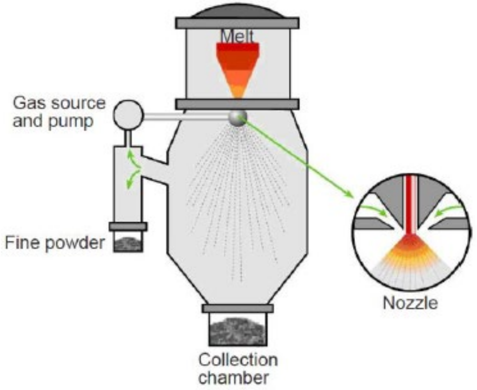
  → 40 µm < d < 150 µm (EBM, DMD)

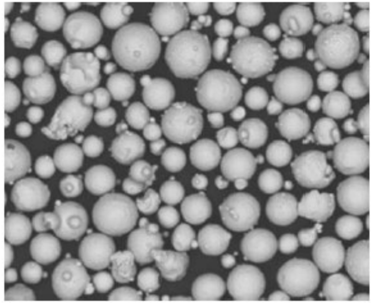
→ Narrow size distribution, for SLM/EBM sometimes addition of finer powder fraction for filling gaps

→ Not oxidized, low humidity

Gas atomization

- Alloy is molten in a crucible
- Atomization with inert gases (Ar, N2) under high pressure
- Solidification during flight in vessel
- Particle geometry, microstructure and composition of powder determined by process gas
- Spherical particles, relatively broad particle size distribution

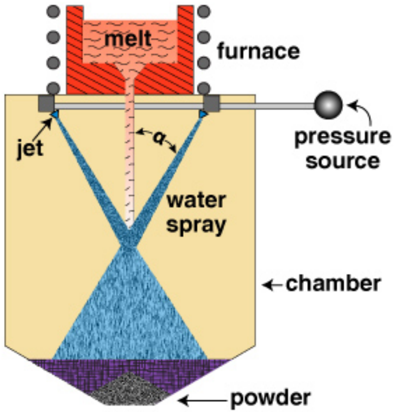




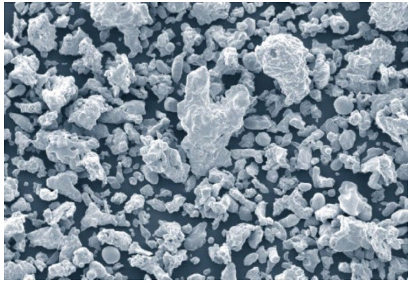
http://www.ggp-metal.com/en/products-water-atomized-bronze-powders.php
Jason Dawes, Robert Bowerman and Ross Trepleton: http://dx.doi.org/10.1595/205651315X688686 *Johnson Matthey Technol. Rev.*, 2015, 59, (3), 243–256

Water atomization

- Alloy is molten in a crucible
- Atomization with several high-pressure water hets
- Relatively simple, fast and cost efficient method for alloy powder production
- Disadvantage: irregular and facetted powder grains, large size distribution Pulverkörner



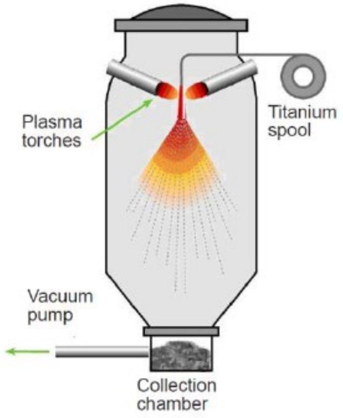
The water atomization process, where a molten metal stream is disintegrated by multiple water jets. The angle determines the atomization efficiency.
(Source: *Fundamentals of Powder Metallurgy*)

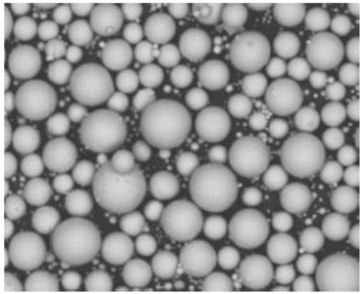


/www.mpif.org/
/http://www.ggp-metal.com/

EIGA/PIGA

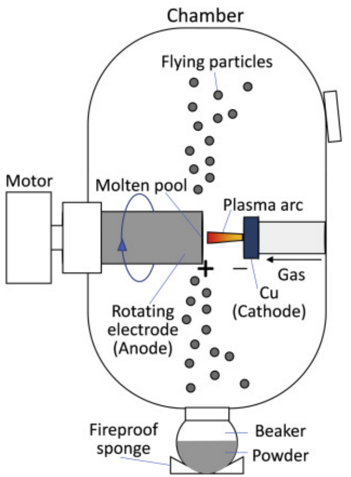
- EIGA = Electrode Induction Melting Gas Atomization
- PIGA = Plasma-melting Induction-guiding Gas Atomization
- Contact-free melting of a sharpened rod using a plasma or an induction coil
- Suitable for producing powder from reactive metals such as Ti
- Spherical powders with very low amounts of impurities (no reaction with crucible)

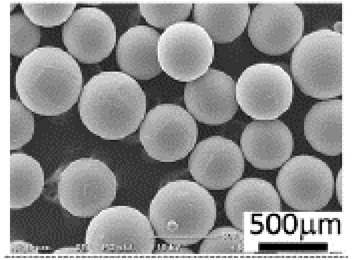




Rotating electrode (centrifugal) atomization

- Material in the form of a rod electrode is rotated at about 15,000 rpm
- The tip of the rod is melten by an arc
- Molten metal is ejected centrifugally as droplets that solidify before hitting the walls of the inert-gas-filled chamber
- Suitable for producing powder from reactive metals such as Ti
- Spherical powders with very low amounts of impurities (no reaction with crucible)





/Y. Cui et al., Powder Technology 376 (2020) 363–372/

/C. Leinenbach, AM of Metals, EPFL/

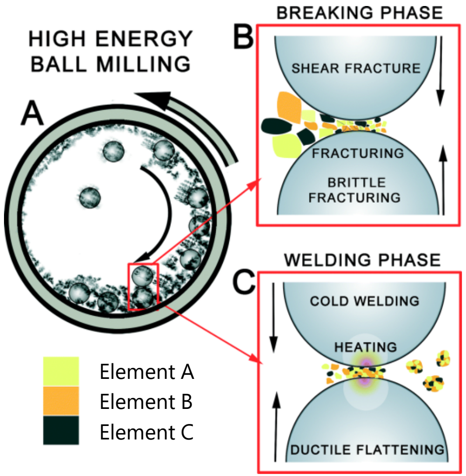
Atomization processes - comparison

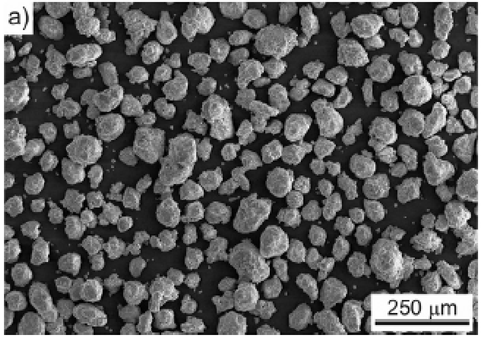
Manufacturing process	Particle size, μm	Advantages	Disadvantages	Common uses
Water atomization	0–500	High throughput Range of particle sizes Only requires feedstock in ingot form	Post processing required to remove water Irregular particle morphology Satellites present Wide PSD Low yield of powder between 20–150 μm	Non-reactive
Gas atomization (Inc. EIGA)	0–500	Wide range of alloys available Suitable for reactive alloys Only requires feedstock in ingot form High throughput Range of particle sizes Use of EIGA allows for reactive powders to be processed Spherical particles	Satellites present Wide PSD Low yield of powder between 20–150 μm	Ni, Co, Fe, Ti (EIGA), Al
Plasma atomization	0–200	Extremely spherical particles	Requires feedstock to either be in wire form or powder form High cost	Ti (Ti64 most common)
Plasma rotating electrode process	0–100	High purity powders Highly spherical powder	Low productivity High cost	Ti Exotics
Centrifugal atomization	0–600	Wide range of particle sizes with very narrow PSD	Difficult to make extremely fine powder unless very high speed can be achieved	Solder pastes, Zinc of alkaline batteries, Ti and steel shot

/I.E. Anderson et al., Current Opinion in Solid State and Materials Science 22 (2018) 8–15/

High energy ball milling (HEBM)

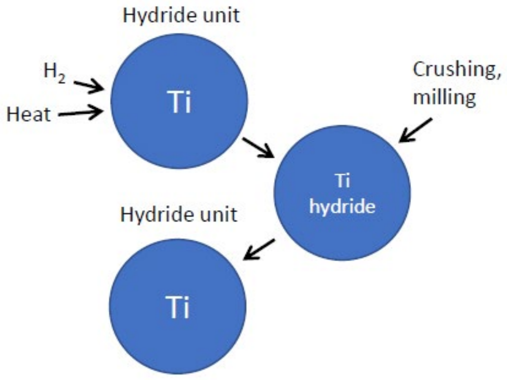
- Mechanical alloying from elemental powders and milling in a planetary mill
- Particle size is a function of milling time (up to 24 h); very fine powders (<1 µm) possible
- Powder morphology influenced by use of additives
- Suitable for producing powders from hard and brittle materials, composites

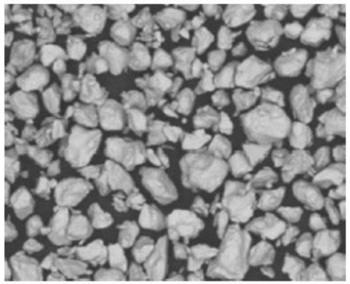




Hydration -Dehydration

- Reaction of Ti with H2 and formation of brittle Ti hydride
- Mechanical milling of the Ti hydride to powder of the required size
- Dehydration in vacuum furnace at elevated temperatures
- Cost efficient method for the production of Ti powder
- Disadvantage: irregular and facetted powder grains

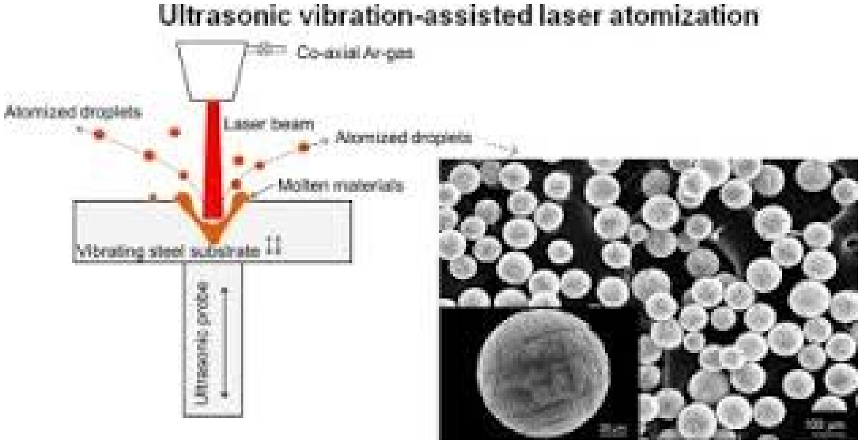




Ultrasonic atomization

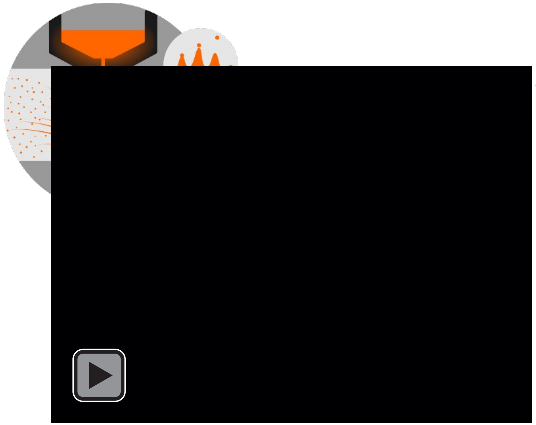
EPFL

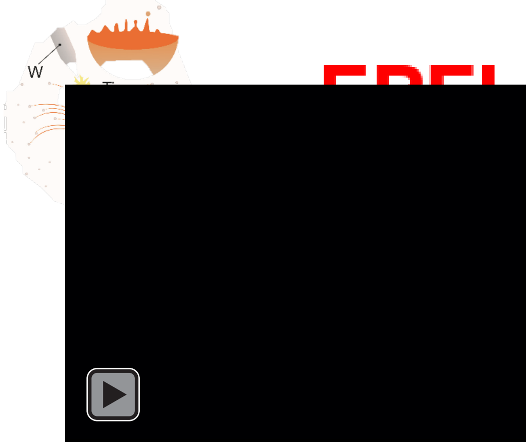
- Alloy is molten (arc, induction, laser) under shielding gas in a Cu crucible, which is attached to a sonotrode (20-60 kHz)
- Small alloy droplets are ejected from the crucible and solidify subsequently (spherical particles)
- Particle size is a function of sonotrode frequency; sizes 20 µm – 150 µm possible
- Suitable for producing small amounts (~1kg/day) of customized powders



/www.x-mol.com/

rePowder

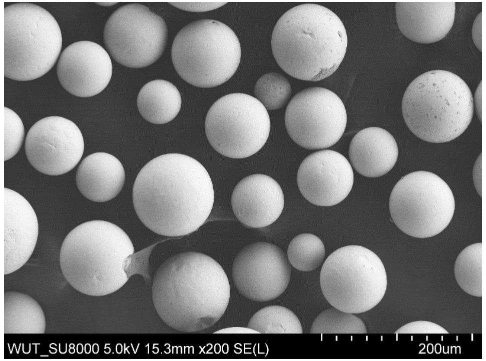


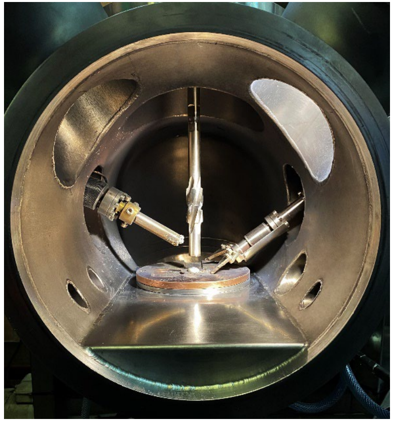


R&D POWDER PRODUCTION
- Arc-melting system 340 A
- Ultrasonic frequency: 20 kHz
- Ultrasonic maximum power: 2 kW
- Universal tooling for atomization and alloying
- Replaceable sonotrode tips

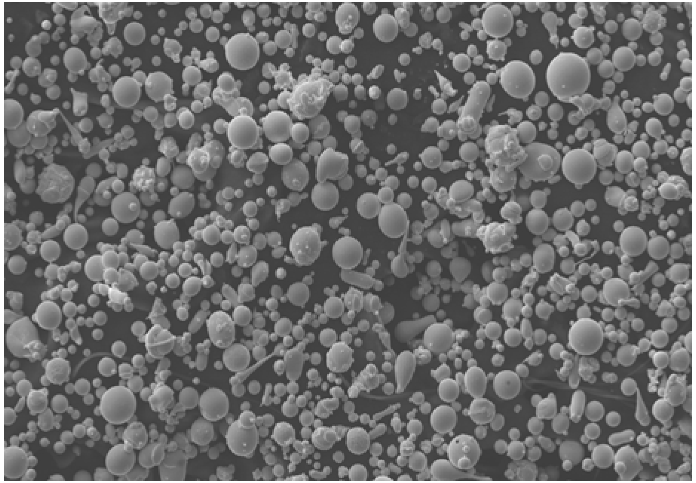
AVAILABLE OPTIONS
- Inductionsystem: 10 kW power
- Electron Beam Unit up to 60 kV
- Multifrequency generator 20-70 kHz
- 40 kHz ultrasonic transducer and sonotrodes
- Ultrasonic assisted forging up to 500J
- High vacuum unit up to 5x10^{-5} mbar
- 10 kW chiller

PROCESSED ALLOYS
- Zr-, Cu- and Fe-based BMGs
- Various HEAs including WTaVTi
- Ag- and Au-based precious alloys
- High speed steels
- Ti alloys - forging and atomisation
- AlSi12, AlSiMg, AlFe-intermetalics
- NiMnGa magnetocalloric alloys
- AZ31 magnesium alloy



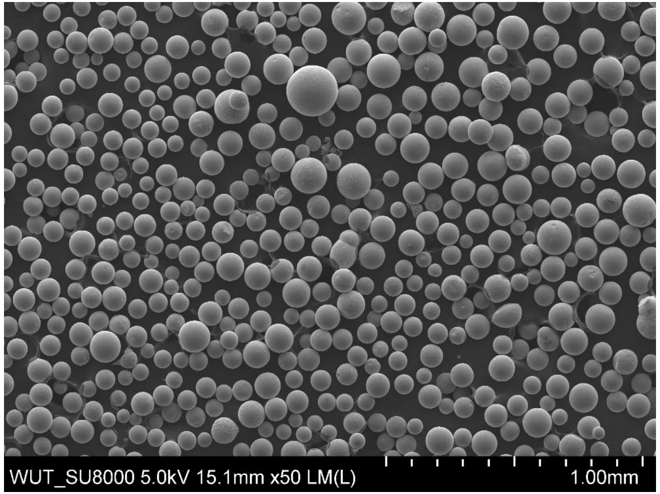


EIGA

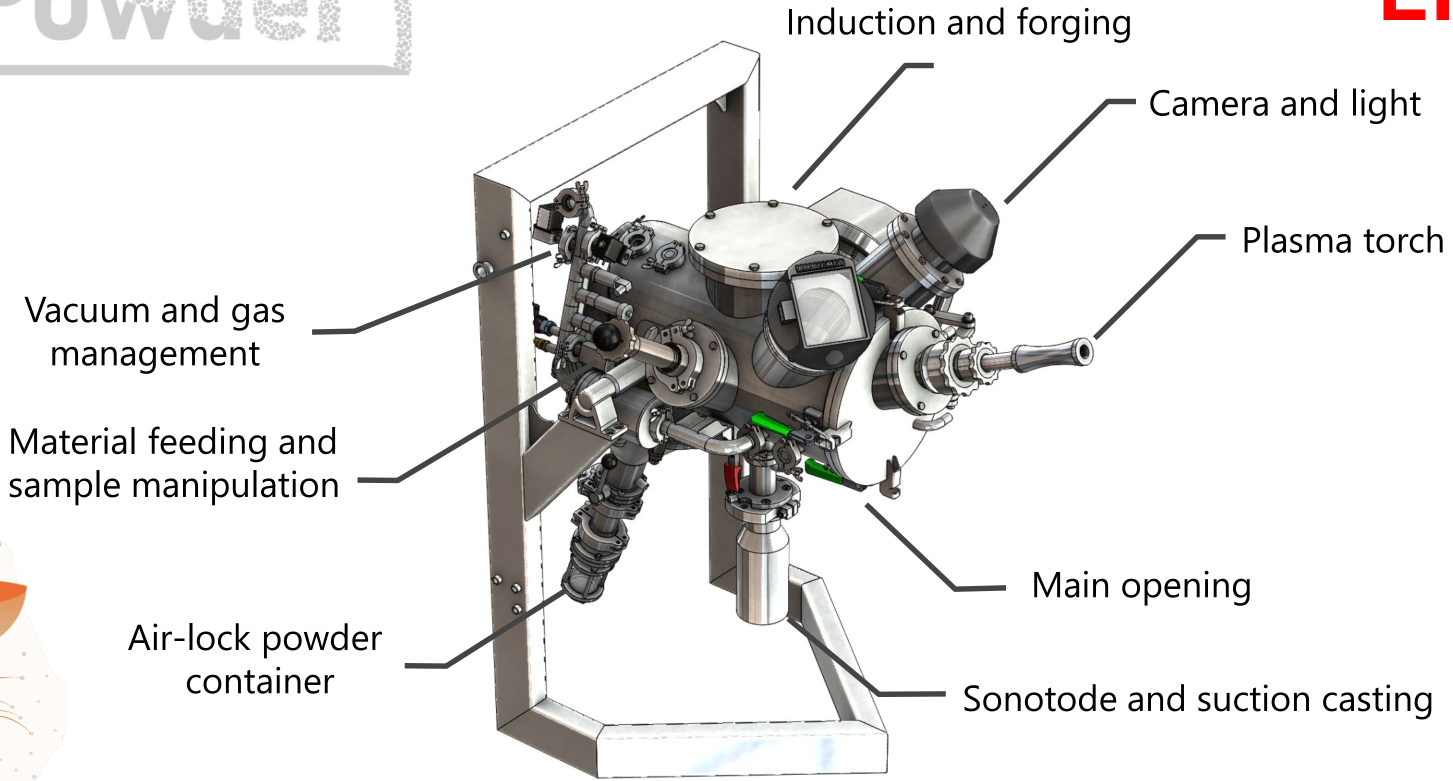


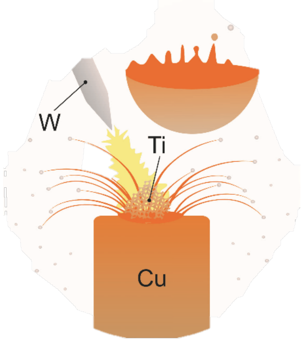
+ finer particles (up to 5μm)
- Splats and fines
- Minimum quantity

Ultrasonics



+ perfect sphericity
+ no minimum quantity
+/- no fines



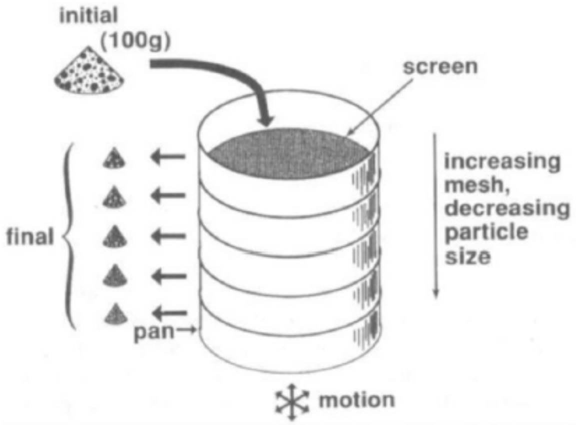


/C. Leinenbach, AM of Metals, EPFL/

Powder separation and classification

Sieving

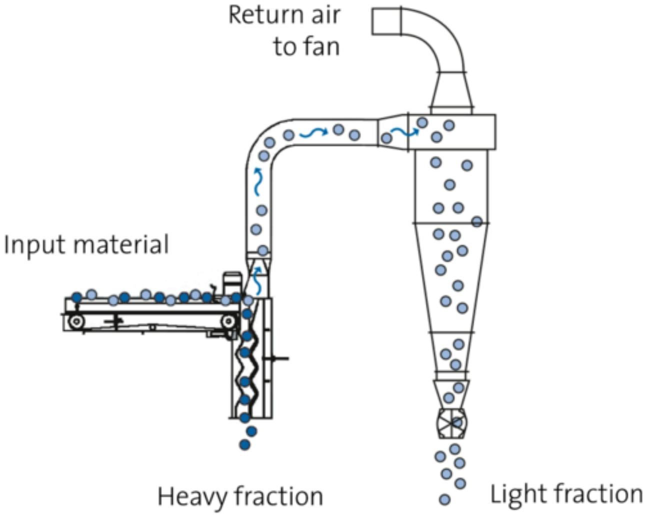
- Particles of different sizes are separated in sieving towers by using sieves with decreasing mesh sizes
- Simple and cost efficient method for powder size classification; standardized in ISO4497
- Can be used for powder 30 µm – 1000 µm;
- No precise size information for irregularly shpae powders



Powder separation and classification

Air separation

- Powder partikel are separated based on the ration between inertia and/or gravitation and flow resistance in a gas stream
- Different designs available (horizontal gas stream, rising pipe, zigzag, levitation)
- Used for separation of fine powder fractions (<25 µm)



/www.nihot.co.uk/

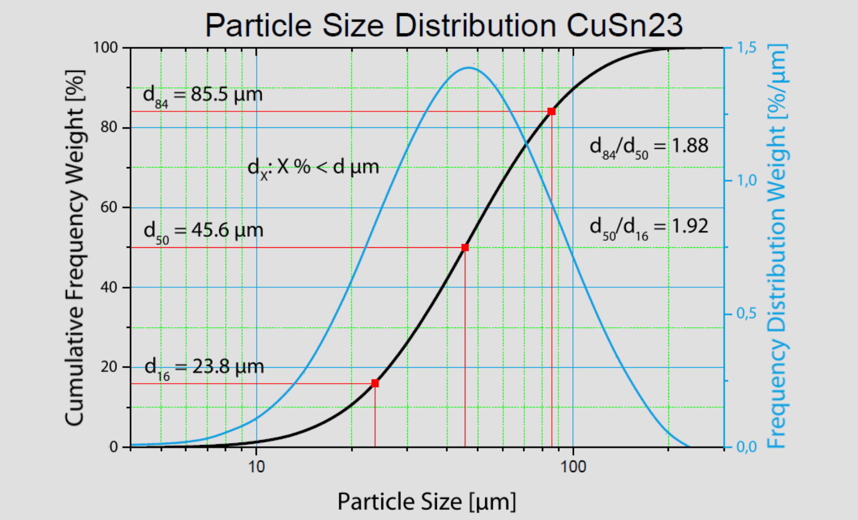
Characterization of powder sizes

- Sifting
  - d<mesh size
- Optical
  - Light-/Scanning Electron Microscopy
- Laser diffraction
  - Scattering of laser light is a function of particle size
- BET (Brunauer-Emmett-Teller) method
  - Measurment of the specific surface using adsorption/desorption of inert gas

Powder separation and classification

Particle size distribution

- Presentation of cumulated of weigthed particle size distribution
- Typical parameters for the powder characterization
  - $d_{16/50/84}$: diameter of 16/50/84% of the powder particles < $d_{16/50/84}$
  - +xx µm/-yy µm: d_{16}>xx; d_{84}<yy
  - d_{84}/d_{16}: distribution width

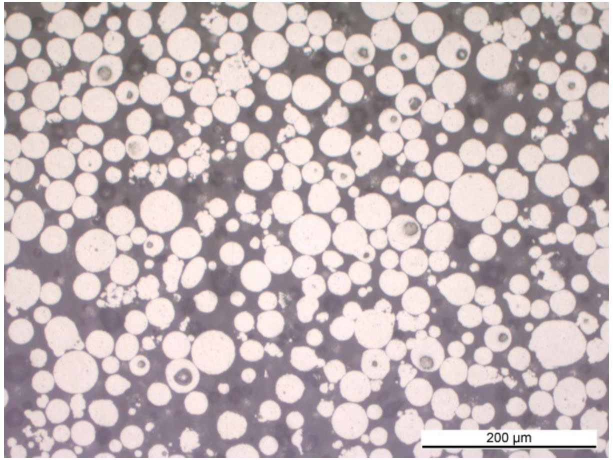


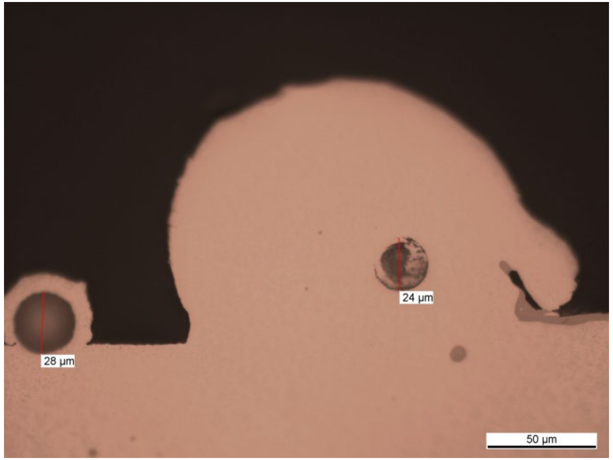
/M. Stobik, Alloys for Additive Manufacturing Workshop, MPI Düsseldorf, 04.07.2016/

Powder quality – porosity

- During the gas atomization process, pores can form in the powder grains because of entrapped gas
- During AM processing, these pores cannot escape from the melt because of the rapid solidification and viscosity of the liquid metal

→ Pores will remain in the consolidated part





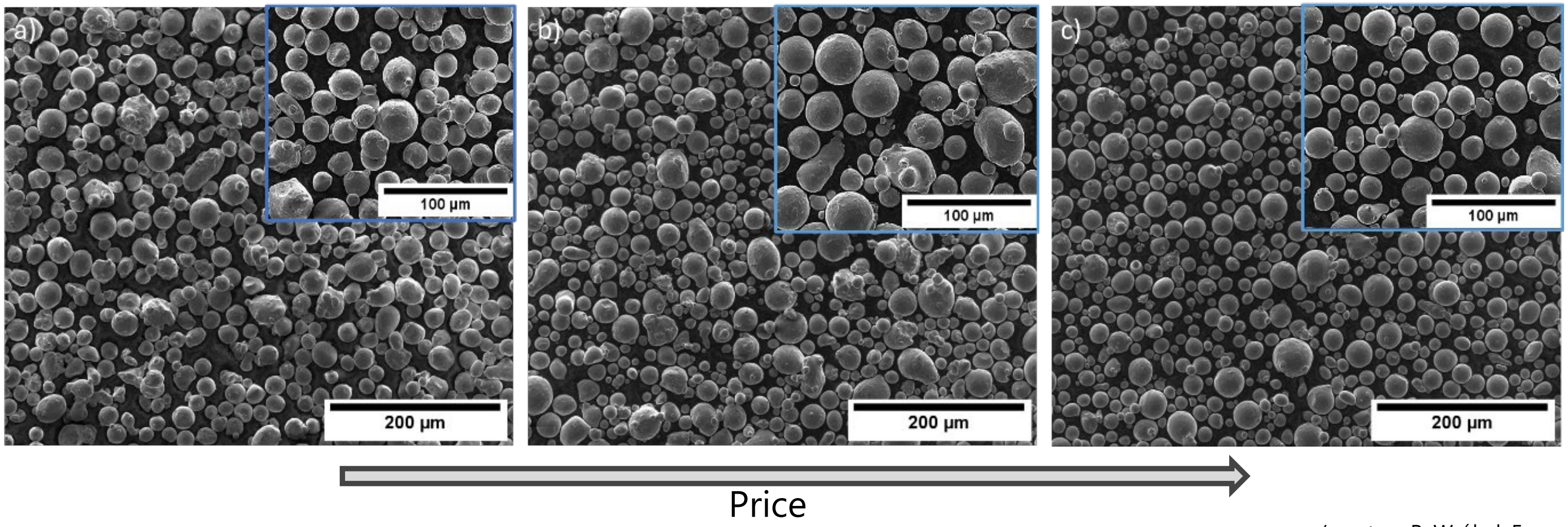
/C. Leinenbach, AM of Metals, EPFL/

Powder quality – sphericity, satellites

- Powders of the same material produced with the same technology can reveal differences in their shape and in the amount of satellites

→ These differences can affect flowability and thus

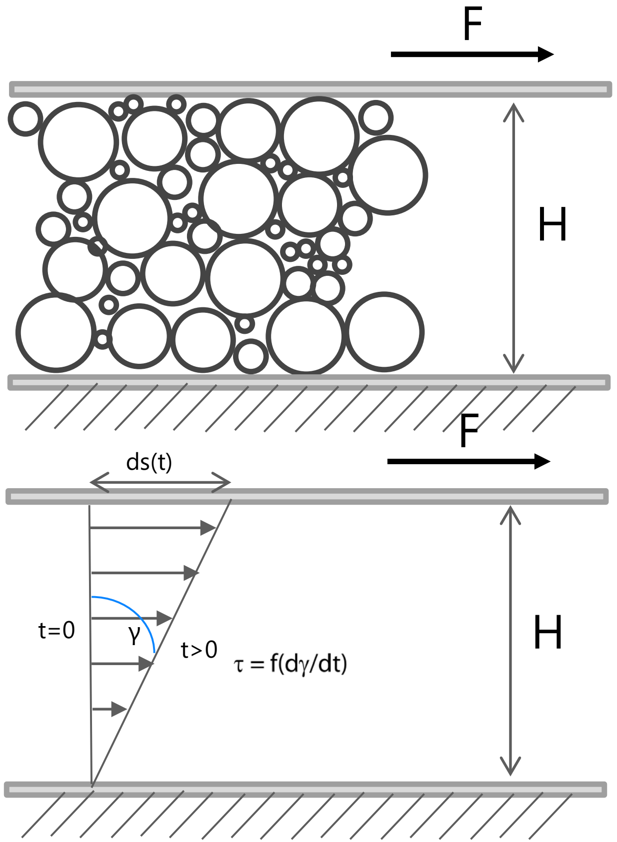
SS316L, GA, Vendor A SS316L, GA, Vendor B SS316L, GA, Vendor C



/courtesy R. Wróbel, Empa/

Powder flowability

- The flow of a powder can be considered similar to the flow of a liquid
- Flowability is the resistance of the powder against flow; it describes the friction between the powder particles
- Flowability is not a measurable physical quantity
- What does «good» flowability mean then?

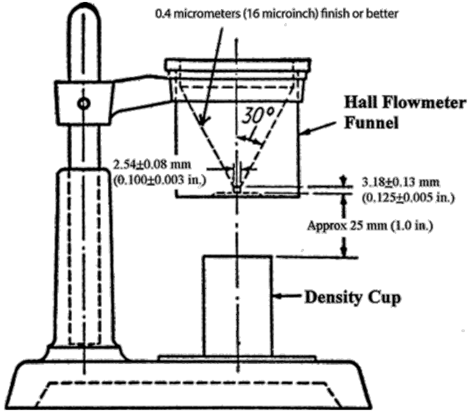


Characterization of powder flowability

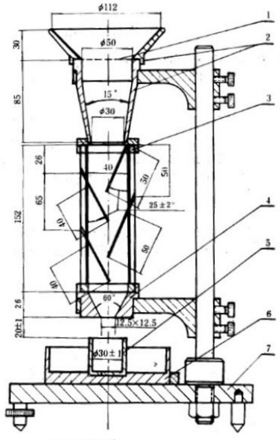
Flow meter

- Flow rate, v_{flow}: time required for a powder sample of a defined quantity (50 g) to run through a funnel of a defined geometry
- Different setups (Hall, Scott) used for different powder sizes

Hall flowmeter



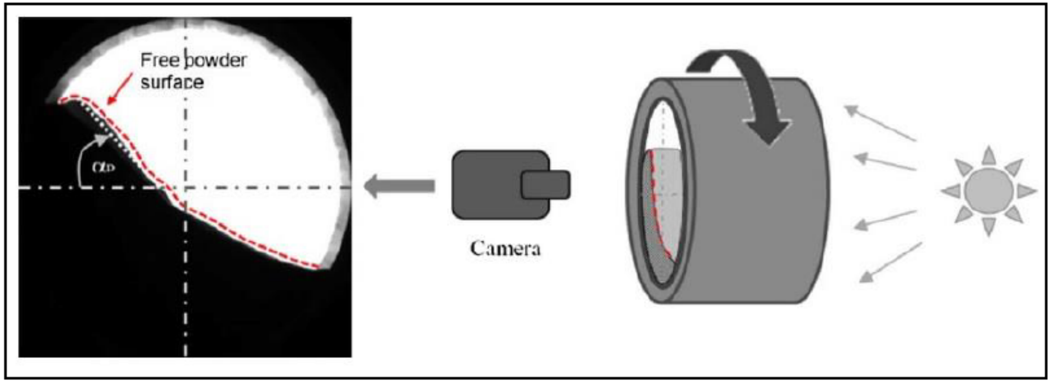
Scott flowmeter



Characterization of powder flowability

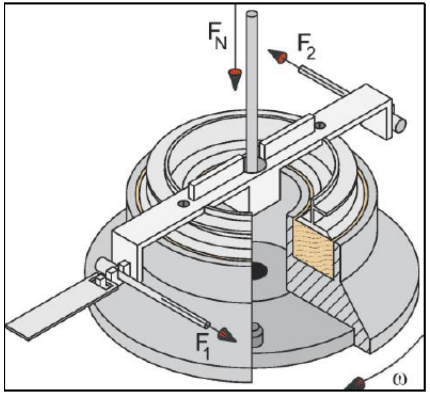
- Revolution powder analyzer
  - Rotating drum with inner diameter of 100 mm and width of 35 mm; measurement of avalanche angle (α) with camera
- Ring shear tester
  - Powder contained in annular shear cell ("ring") and loaded from the top with vertically acting force N; measurement of shear forces F_1 and F_2

Revolution powder analyzer



Mercury Scientific Inc.

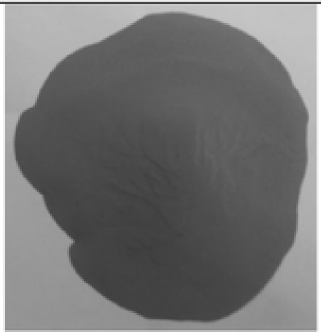
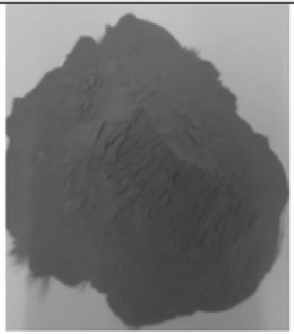
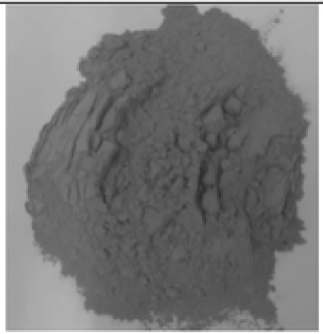
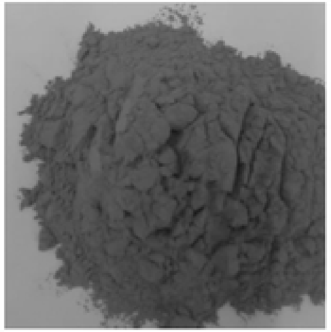
Ring-shear-tester



Dr. Dietmar Schulze Schüttgutmesstechnik

Characterization of powder flowability

- A first qualification of a powder can be done by dumping a defined powder quantity on a plate and subsequent optical evaluation of powder agglomerates
- Spierings et al. have introduced 4 powder quality classes

φ = 1 – very good flowability no agglomerations	φ = 2 – sufficient flowability Very loose agglomerations	φ = 3 – critical flowability loose agglomerations	φ = 4 – unsufficient flowability severe agglomerations
			

/ Spierings, A.B., Voegtlin, M., Bauer, T. et al. Prog Addit Manuf (2016) 1: 9./

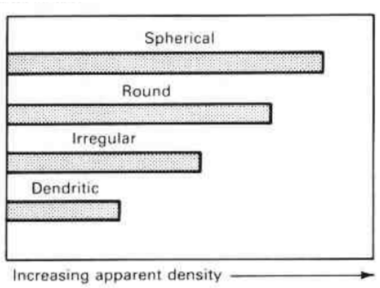
Powder flowability

Influencing factors

- Particle morphology (spherical, angular or edged)
- Particle size distribution
- Roughness/surface topography
- Moisture
- Short-range attracting forces (van der Waals)
- Chemical composition (surface oxides/nitrides)

Powder density

- Bulk density: ratio of the mass of an untapped powder sample and its volume including the contribution of the interparticulate void volume.
- Measured usually by pouring a powder into a vessel of a defined size (25 cm^3) → Hall flowmeter



- Tapped density: increased bulk density attained after mechanically tapping a container containing the powder sample (without additional pressure)

Where can I get my powder?

- For SLM and EBM, powder can usually be obtained at a (relatively high) price directly from the machine manufacturer
- However, there are a number of powder manufacturers from whom AM powder can be purchased directly
  - Oerlikon Metco
  - Carpenter-LPW
  - Sandvik
  - Höganäs
  - Heraeus
  - …

Powder – Safety aspects

EPFL

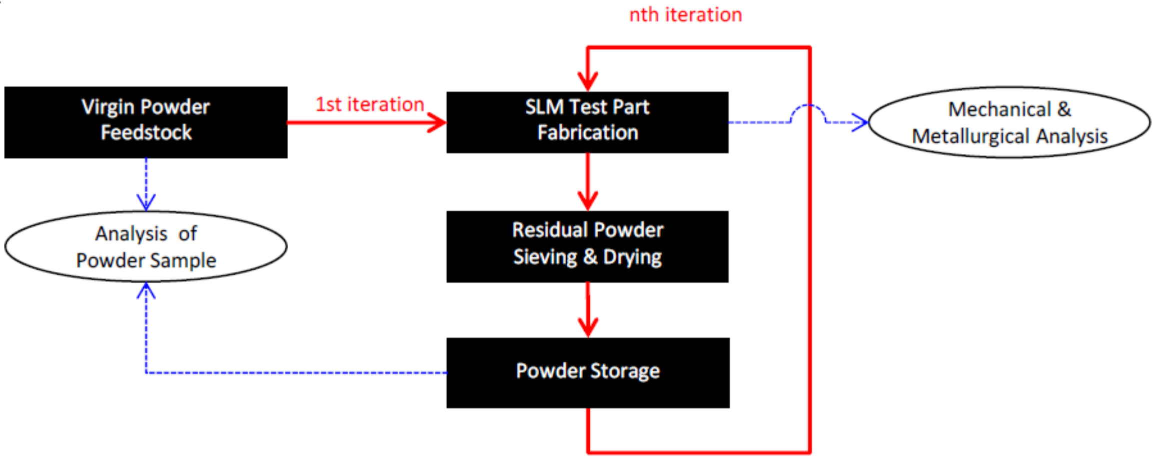
- Powder has a very high surface in comparison with the bulk material
- Example: consider 1 kg Titan
  - Sphere, radius 3.756 cm → surface A = 177 cm^2
  - Cube, side length ≈6.06 cm → surface A = 220 cm^2
  - Powder (Ø 30 µm), not compacted → A = 12'112 cm^2

  → factor: 68
- Powders from reactive materials such as Ti can start burning even at relatively low heat inputs
  - → The temperatures can reach very high values >2000°C
  - → Extinction with water not possible (decomposition into H_2 and O_2)

Powder – Safety aspects

Influence of powder recycling

- The powder storage conditions might lead to uptake of moisture in the powder, affecting the flowability
- How does recycling of already used powder affect the powder and part properties?

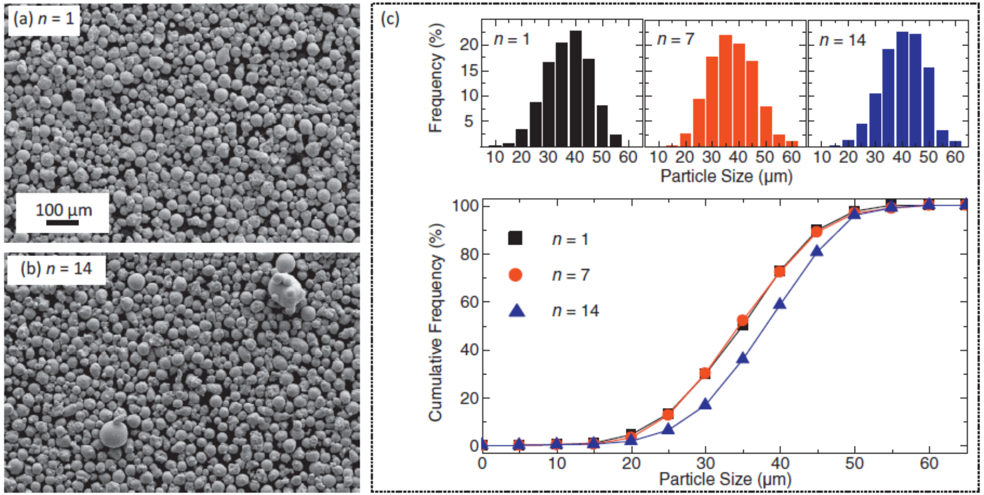


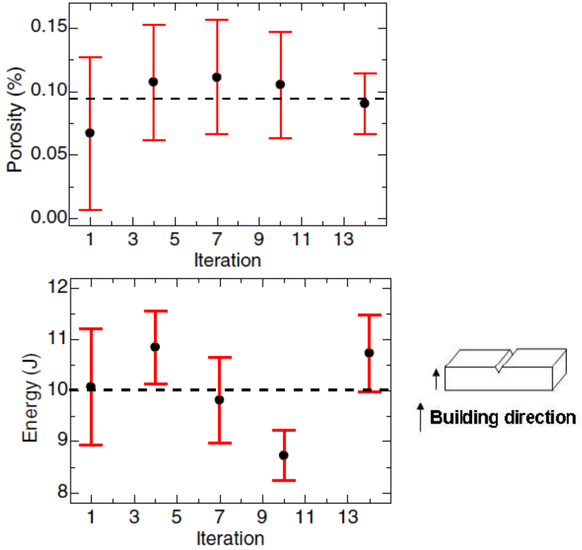
/L.C. Ardilla et al., Physics Procedia 56 (2014) 99-107/

Influence of powder recycling

Inconel 718 – LPBF

- The powder storage conditions might lead to uptake of moisture in the powder, affecting the flowability
- How does recycling of already used powder affect the powder and LPBF part properties?





/C. Leinenbach, AM of Metals, EPFL/

/L.C. Ardilla et al., Physics Procedia 56 (2014) 99-107/

Influence of powder recycling

Inconel 718 – EBM

- During EBM, powder is at elevated temperature over a long time
- Uptake and accumulation of oxigen over several build jobs
- Formation of Al-rich oxides on powder surface

Table II. Build Cycle Information with Powder Oxygen Levels, Obtained from Combustion Analysis

Sample Designation	Average Powder Oxygen Level, ppm	Individual Powder Oxygen Measurements, ppm	Build Cycle No.	Accumulated Process Time, Hours
B1	146	131, 148, 159	1	0
B6	195	185, 196, 204	6	220
B14	266	257, 264, 277	14	500
B30	312	282, 297, 357	30	≥1000

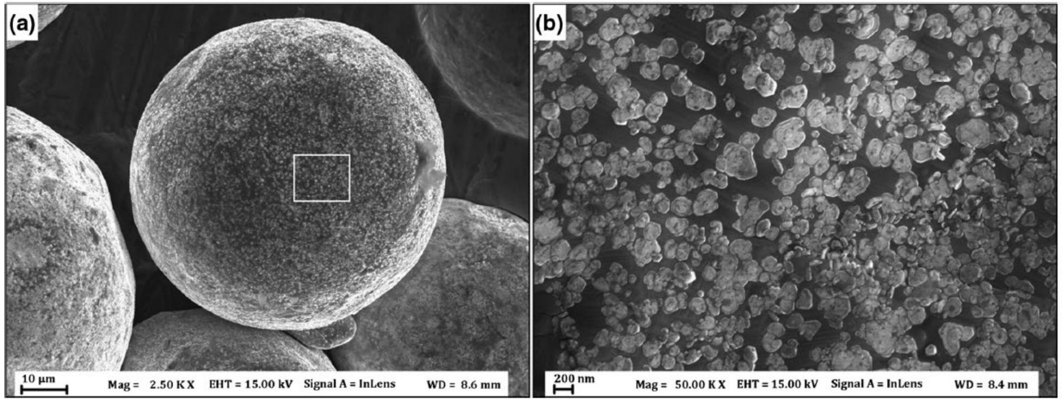


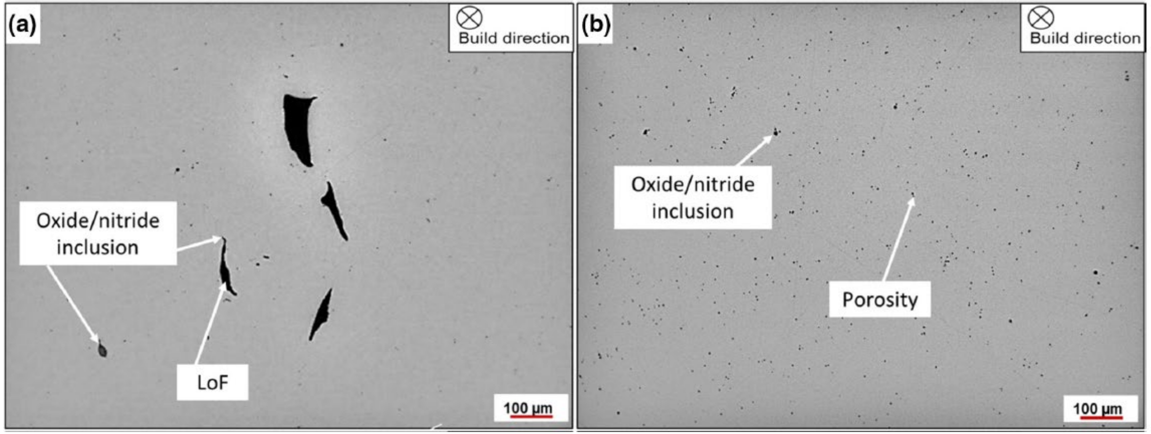
Fig. 3—(*a*) Secondary scanning electron image of the powder before build cycle 30; (*b*) Al-rich oxide particulates on the surface at higher magnification.

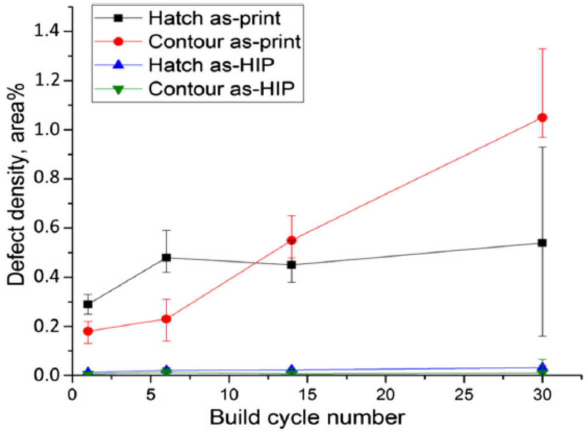
/H. Gruber et al., Metall. Mater. Trans. A, 51 (2020) 2430-2443/

Influence of powder recycling

Inconel 718 – EBM

- Al-rich oxides are included in the build part,
- Defect density in as-build parts increases over number of build cycles





/H. Gruber et al., Metall. Mater. Trans. A, 51 (2020) 2430-2443/

Influence of powder recycling

AlSi10Mg – LPBF

- The particle size distribution of AlSi10Mg powder is shifted to the left (smaller powder grains)

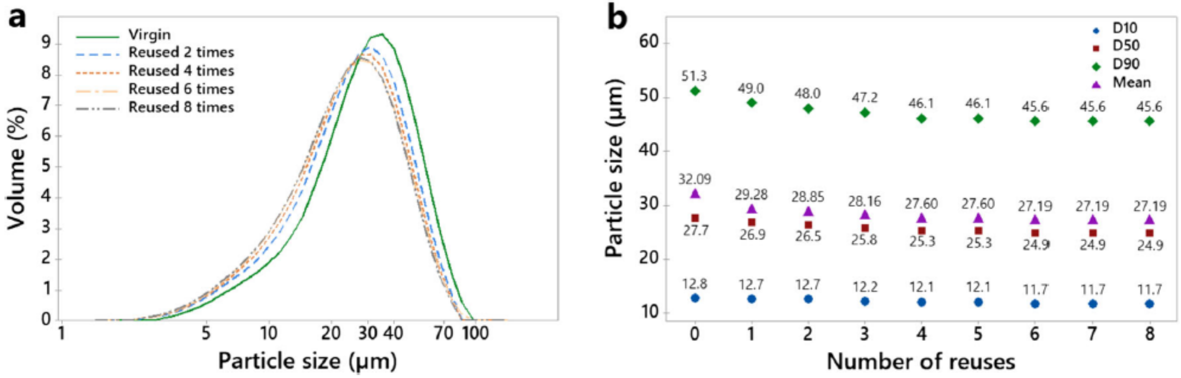


Fig. 5 Particle size distribution of the AlSi10Mg powder versus reuse times. **a** Probability density size distributions. **b** Mean value, 10th ($D10$), 50th ($D50$) and 90th ($D90$) percentiles of the particle diameter under the assumption of gamma distribution

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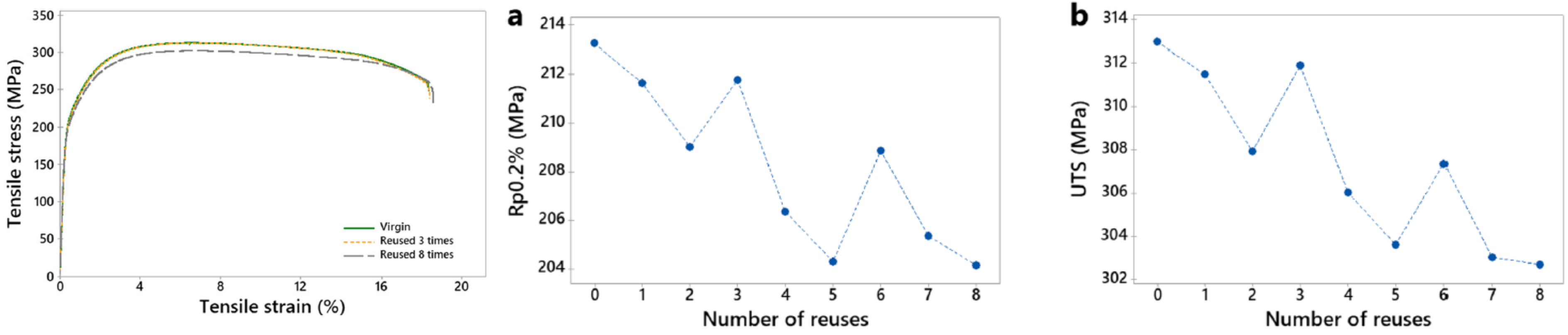
Influence of powder recycling

AlSi10Mg – LPBF

- YS and UTS exhibited a systematic decrease of 10 MPa over number of reuses
- High cycle fatigue strength exhibited a systematic decrease, from 160 to 145 MPa, over number of reuses



/C. Leinenbach, AM of Metals, EPFL/

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