



# Foundry Institute RWTH Aachen University

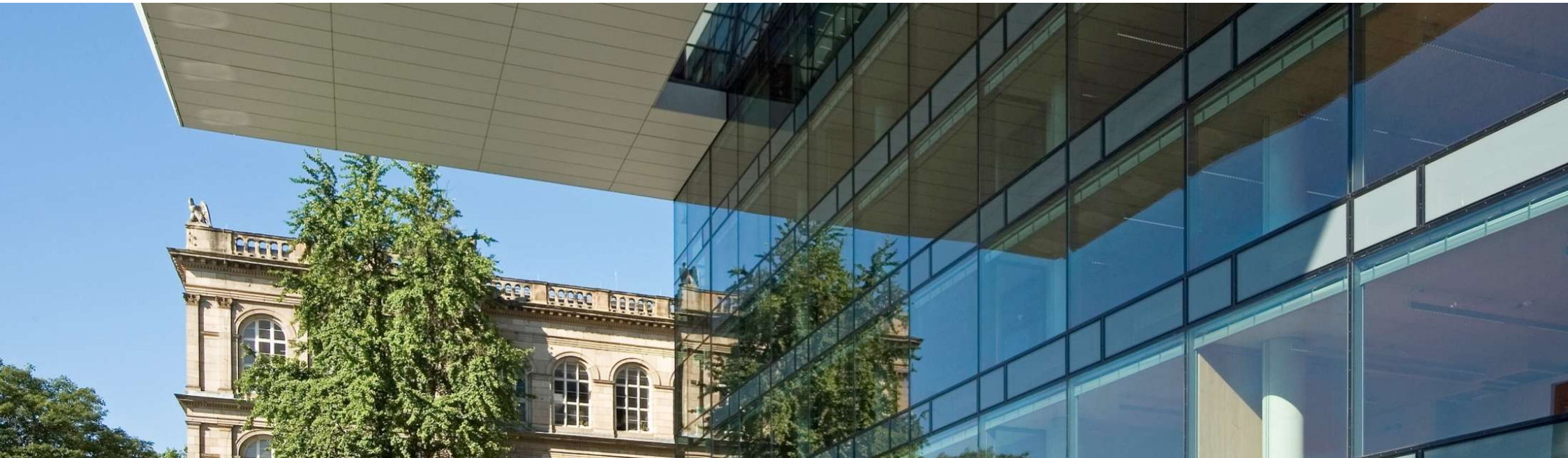
## Alloy Design from Powder to Printing: The Complete AM-Process Cycle

Iris Raffeis, Frank Adjei-Kyeremeh, Andreas Bührig-Polaczek  
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Lehrstuhl für das  
gesamte Gießereiwesen  
und Gießerei-Institut

**RWTH**AACHEN  
UNIVERSITY



- Founded in 1870 as “Königlich Rheinisch-Westphälische Polytechnische Hochschule”
- Main research areas: Engineering and Natural Sciences
- Approximately 45.000 students in 173 courses of studies (Bachelor und Master)
- 260 different institutes in 10 faculties
- 572 professors and 6354 scientific researchers (including third-party funded staff)
- Budget 1.234 million € (506 million € third-party funds)
- **3 Clusters of Excellence**



## Research Center for Digital Photonic Production (RCDPP)

Start of Construction: **2015**  
Move-in: May 2019

### Financial Budget:

Total budget: 57,1 Mio. €  
Building costs: 43,4 Mio. €  
Budget for large equipment: 8,8 Mio. €  
Budget for first furnishing  
of labs and offices: 4,8 Mio. €

### Floor Space:

Total floor space app. 2500 m<sup>2</sup>  
Floor space for Material Science and  
Materials Technology: ca. 300 m<sup>2</sup>  
Office space for app. 70 delegated  
scientists of 10 different institutes of  
RWTH Aachen University



# Alloy Design from Powder to Printing

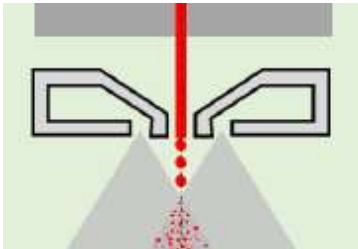
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- Excellence Cluster: Internet of Production (IOP)
- 1 Powder Production
- 2 Analytics
- 3 Printing
- 4 Microstructure Examples
- 5 Sustainable Production  
Excellence Cluster: Internet of Sustainable Production (IoSP)

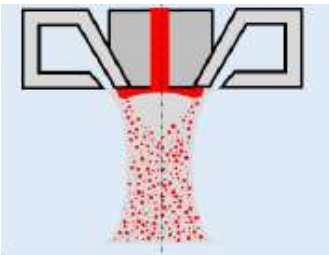


Common method of powder production: gas atomisation

## Free-fall atomisation



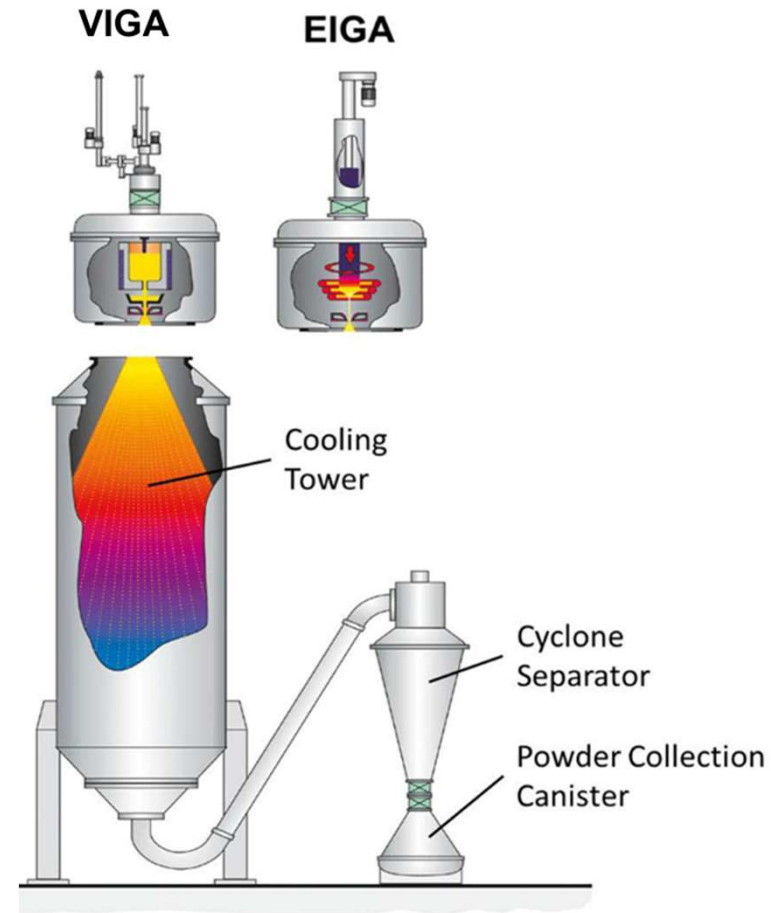
## Close-coupled atomisation



## General units of atomizers

- Melting section
- Atomizer unit with nozzle system (e.g. gases, water)
- Cooling section
- Powder separation

VIGA (Vacuum induction) -, EIGA (Electrode induction) -, Melting Inert Gas Atomization





**VIGA-2B** (Vacuum induction melting inert gas atomization  
2 liter bottom pouring) → **Research batch size**

**EIGA-50-500** (Electrode induction melting inert gas atomization)

## EIGA

- Power HF: 120 kW, 400 A, 200 kHz, 1200 V
- Heating coil voltage 500 V
- Electrode: 50 and 70 mm Ø, length 500 mm EIGA free fall nozzle
- Particle sizes:  $60 \mu\text{m} < d_{50} < 120 \mu\text{m}$
- Melting volume per electrode: ~1 liter

## VIGA

- Power MF: 40 kW, 10 kHz, 260 V Heating coil voltage
- VIGA 2 liter effective melting volume
- VIGA close-coupled gas nozzle
- Particle sizes:  $30 \mu\text{m} < d_{50} < 80 \mu\text{m}$

- **Atomization gas: nitrogen and argon**



# VIGA and EIGA Tops

# 1 Powder Production

Customized cast alloy for Atomizing



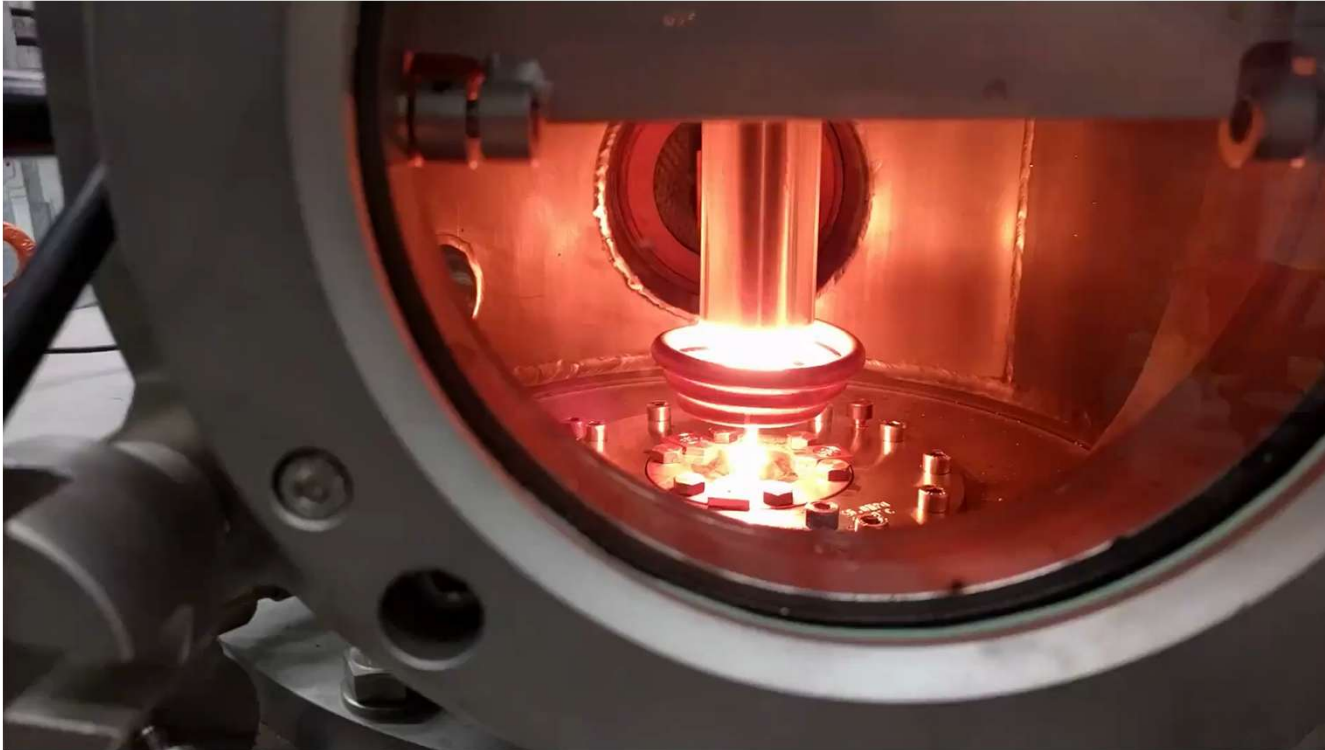
Vacuum induction melting



VIGA



EIGA



Technical or bulk properties: Flowability, apparent and tap density, PSD, sphericity, aspect ratio, humidity



Hall Flow Meter

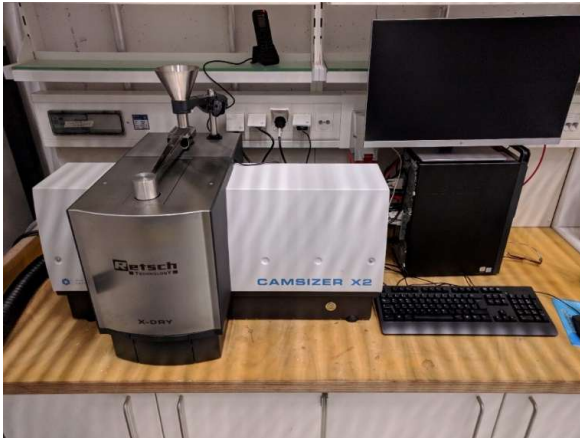


Revolution Powder Analyzer

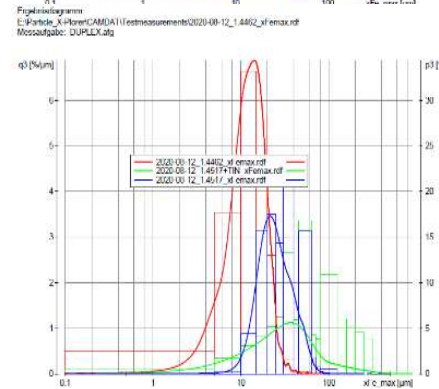
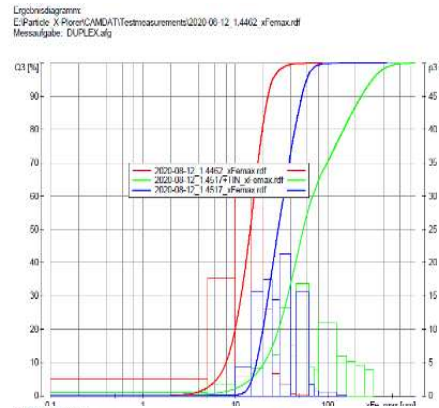


Jolting Meter

Powder size classification via sieve tower, Retsch

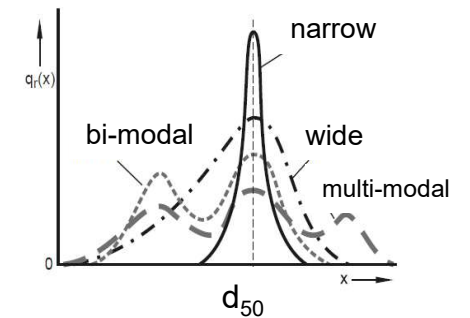
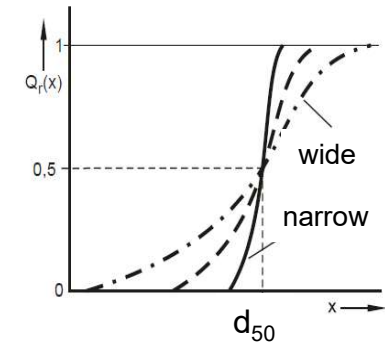


- Camsizer X2 Manufacturer: Retsch GmbH
- Particle size and shape analysis from 0,8  $\mu\text{m}$  to 8 mm
- 2 camera systems with up to 500 pictures per second
- Modular expendable
- With X-Jet compressed air module



PSD (particle size distribution)  
 Sphericity, aspect ratio, circularity, etc.

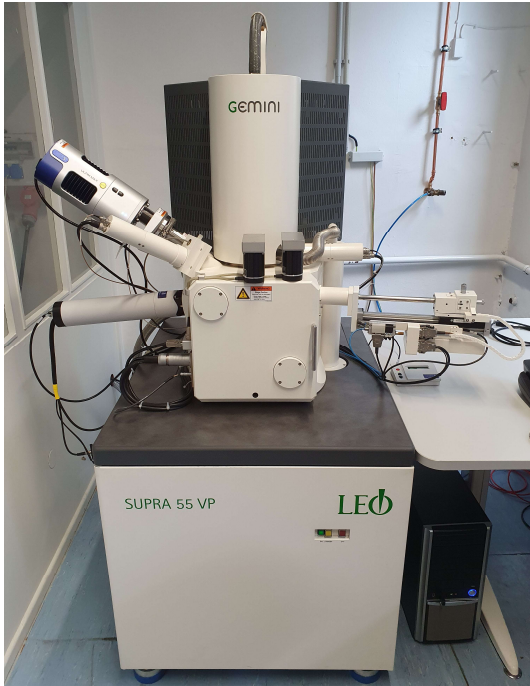
Distribution sum



Different forms of PSD

DOI 10.1007/978-3-642-53885-8

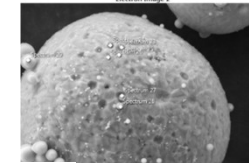
Microscope: Zeiss, Detectors Oxford Instruments, Aztec



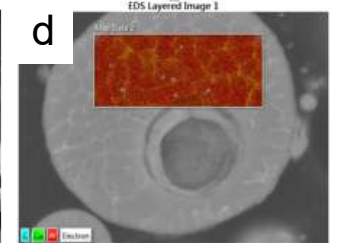
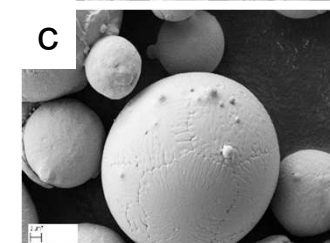
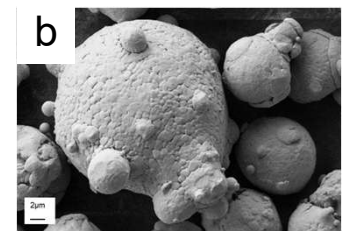
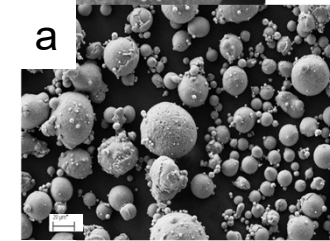
Resolution of detectors (Oxford):  
SE: 5-10 nm, RE: ca. 10 nm, EBSD: ca. 50 nm,  
EDS: spotsize 500nm-1µm, STEM < 10nm

Right of column: In-Lense and STEM  
Left of column: EDS, EBSD, SE2,  
VP-SE2, backscatter detector (not showed)

Field emission cathode,  
Schottky-Emitter



Particle morphologies



Powder particles:  
a, d) AA2099, b) Monel, c) 1.4408

Raffeis, Adjei, DOI: [10.1007/s11837-019-03367-1](https://doi.org/10.1007/s11837-019-03367-1)

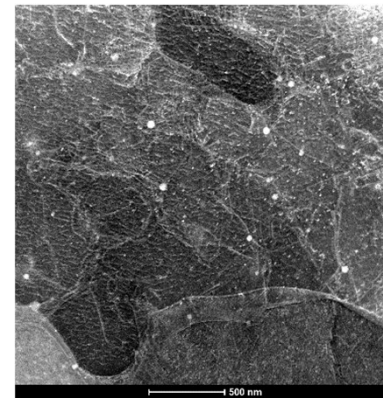
### Transmission Electron Microscope: JEOL JEM-F200, Oxford-EDS and Gatan Camera



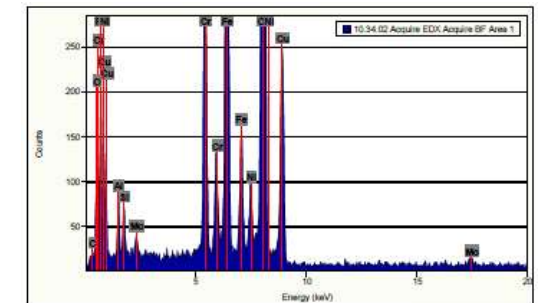
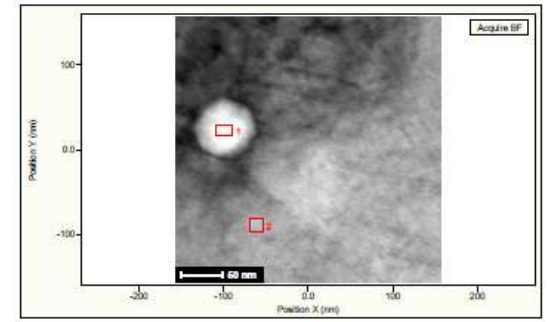
Acceleration voltage 20 - 200 kV

Field emission cathode,  
Schottky-Emitter

Resolution  
TEM, spot: 0.19 nm  
TEM, lattice: 0.10 nm (1 Å)  
STEM-HAADF: 0.14 nm



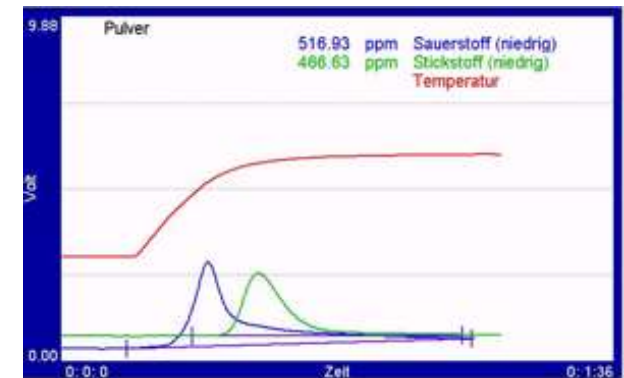
Mixed oxides with  
Si and Al in ferrite  
powder particle



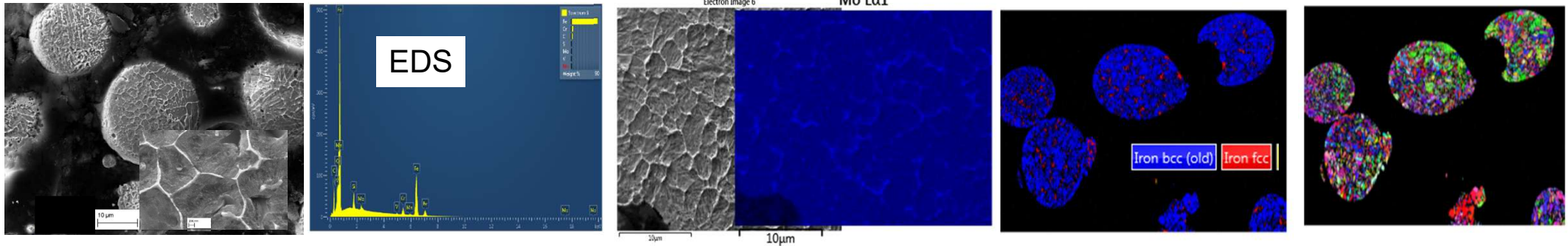
Raffeis, DOI: [10.18154/RWTH-2018-228575](https://doi.org/10.18154/RWTH-2018-228575)

**ON/H Analysis Galileo G8 (Inert Gas Fusion Analyzer)**

- Manufacturer: Bruker
- ON/H measurement from 0,1 ppm to 0,1 %
- Oxygen detection via infrared
- Nitrogen and hydrogen detection via thermal cell



- Measurement of diffusible hydrogen via warm aging till  $T < 900\text{ °C}$
- Carrier gases: He, Ar,  $\text{N}_2$
- Melt extraction furnace till  $3000\text{ °C}$
- Extendable with mass spectrometry



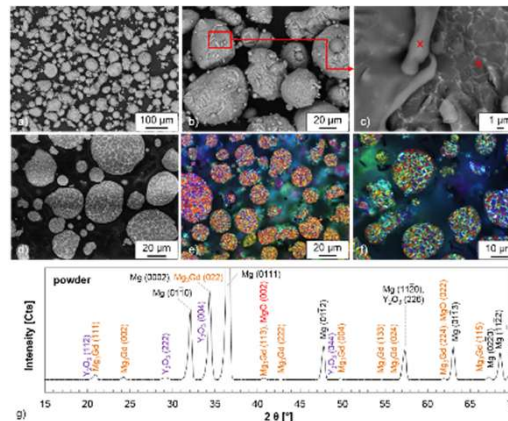
SEM: morphology, EDS, EDS mapping

Segregations on cell boundaries, e.g. Mo, but also Cr, V....

EBSD: Crystallographic structure and orientation

## Analytics (bulk properties):

- Spark spectrometer
- Inert gas fusion analyzer (IGF)
- Combustion analyzer (S and C)
- ICP-OES (inductively coupled plasma optical emission spectroscopy)
- XRD



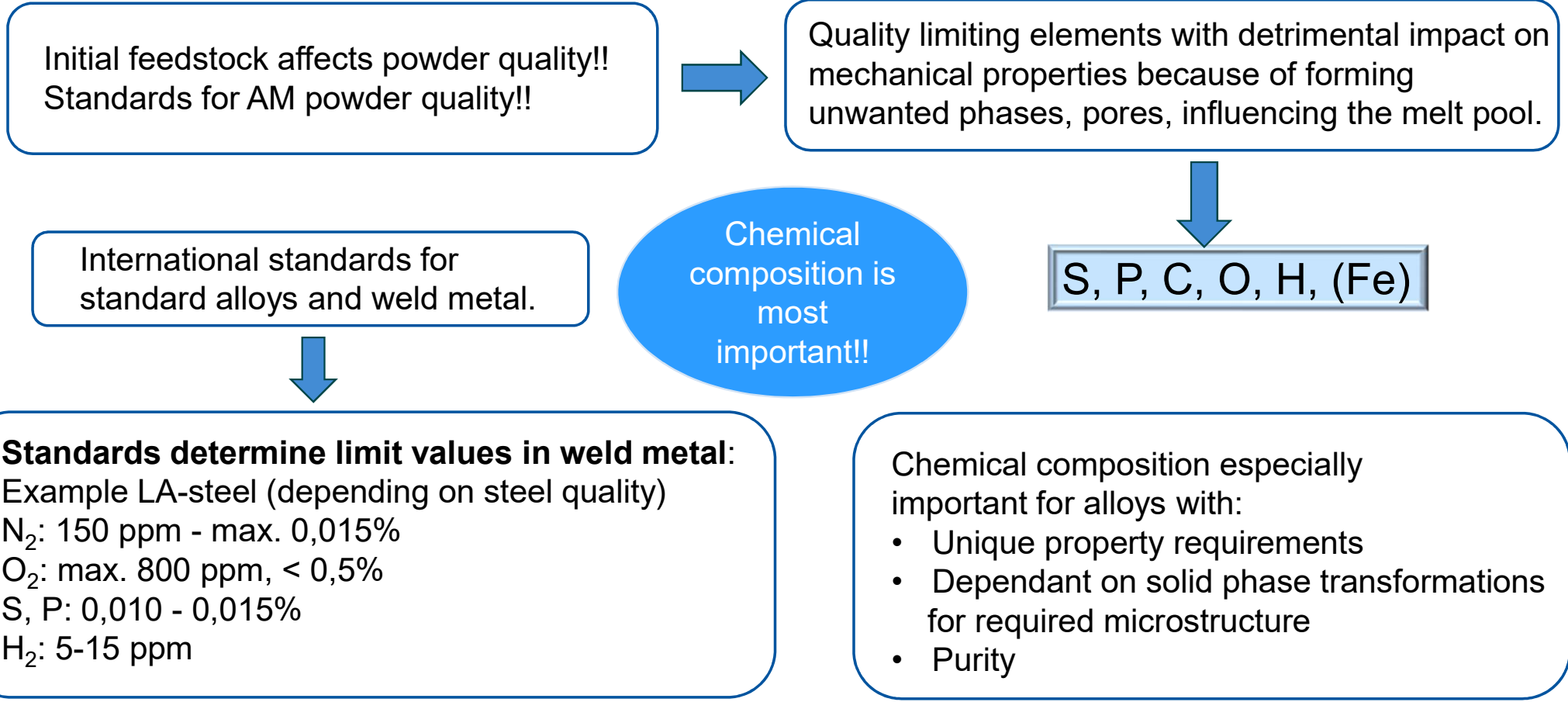
## Electron Microscopy

(Local properties):

- EDS
- EBSD
- EPMA
- TEM

Bold, Raffeis, Adjei-Kyeremeh, et al. DOI: 10.3390/met14010079

Raffeis, Adjei-Kyeremeh, et al. DOI: 10.3390/jmmp6030063



## Critical Elements for Melt Pool Structure

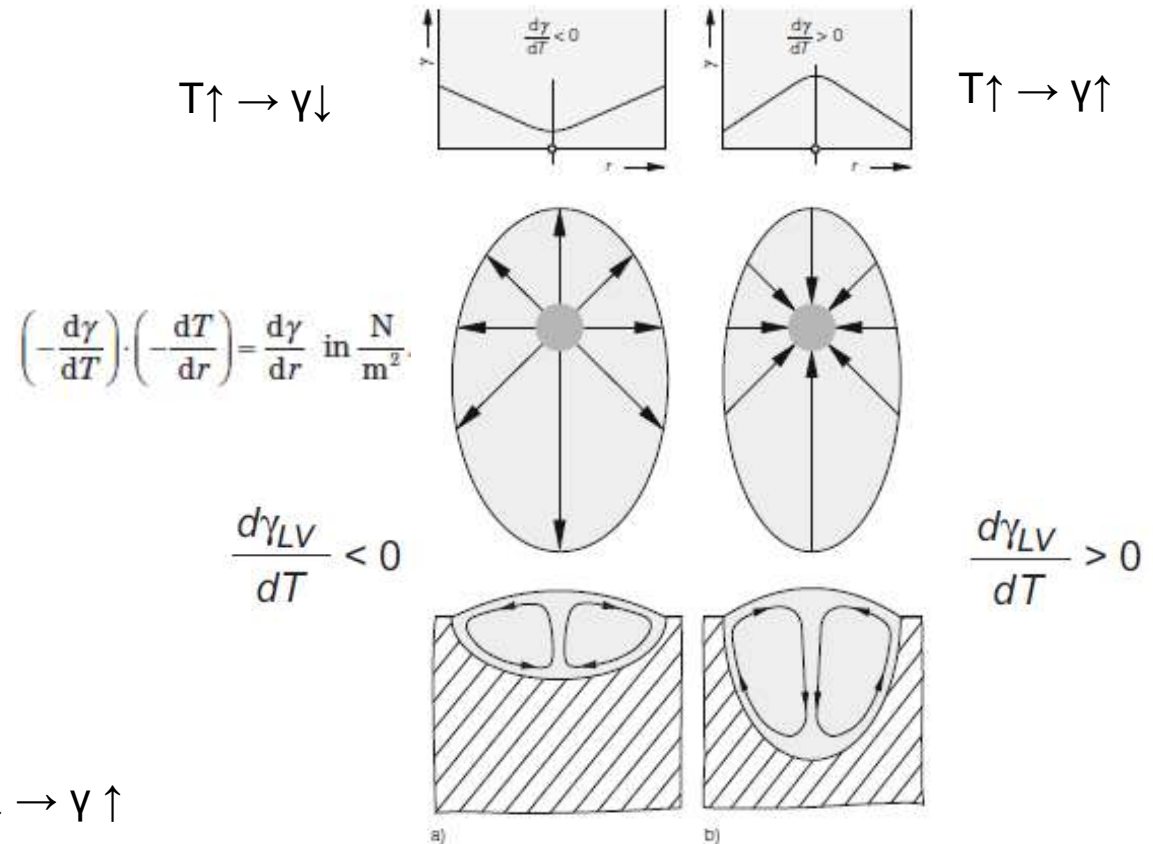
2/4 Analytics

Mass transport in the melt pool due to Marangoni convection due to differences in surface tension (Heiple's model)

Influence of surface tension gradients of a melt pool on Marangoni convection

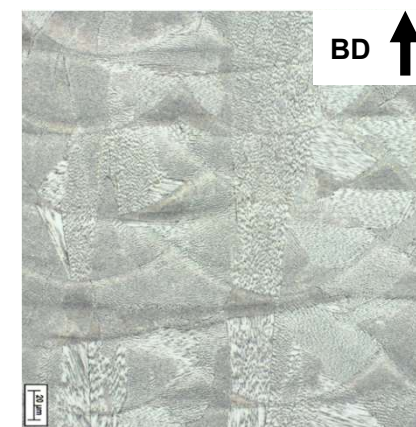
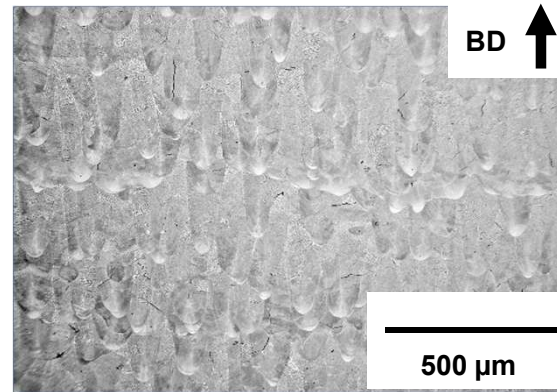
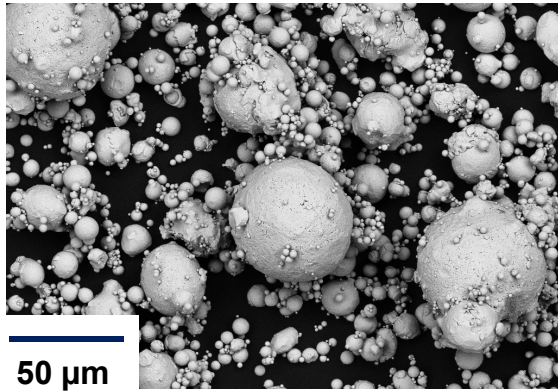
(a) Flat, wide melt pool:  $dy/dT < 0$   
Divergent mass flow, typ. for pure metals and many alloys. Pos. force directed outwards,  $r$  distance from center.

(b) Narrow, deep melt pool:  $dy/dT > 0$   
Convergent mass flow due to surface active elements (B, S, O, As, Te, Se),  $T \uparrow \rightarrow$  solubility  $\downarrow \rightarrow \gamma \uparrow$

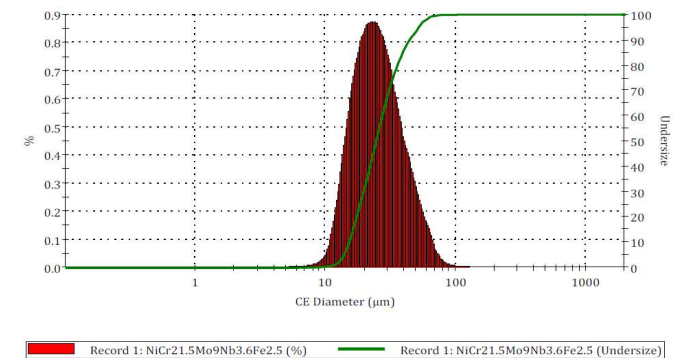
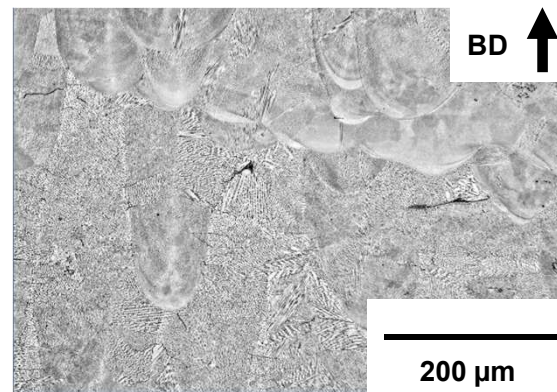
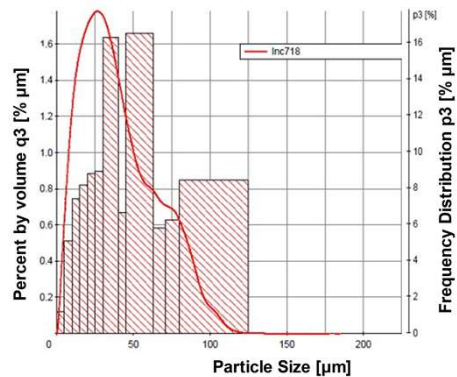


Schulze, G., Die Metallurgie des Schweißens, Springer-Verlag 2010, ISBN 978-3-642-03183-0, DOI 10.1007/978-3-642-03183-0

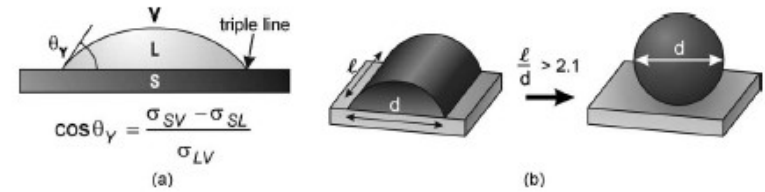
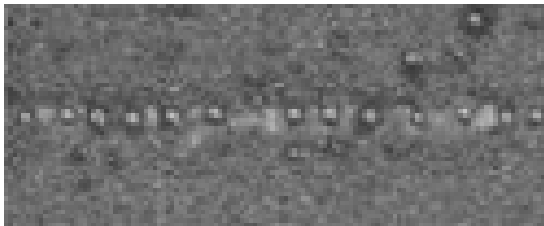
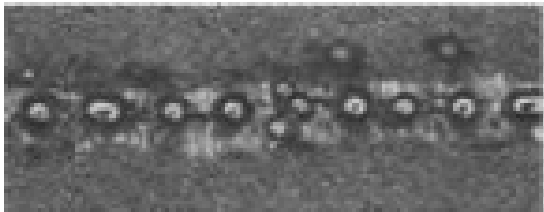
## Argon atomized IN718 with VIGA production



Gaussian PSD Graph

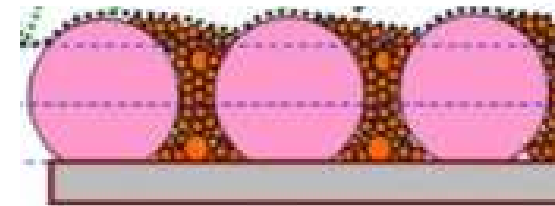


- Increased dissolved oxygen content in the melt
- Change in Marangoni flow due to increase in surface tension gradient
- Capillary Instability → Balling due to poor wettability



J.P. Kruth, L. Froyen, J. Van Vaerenbergh, P. Merckels, M. Rombouts, B. Lauwers, Selective laser melting of iron-based powder, Journal of Materials Processing Technology 149 (2004) 616–622.

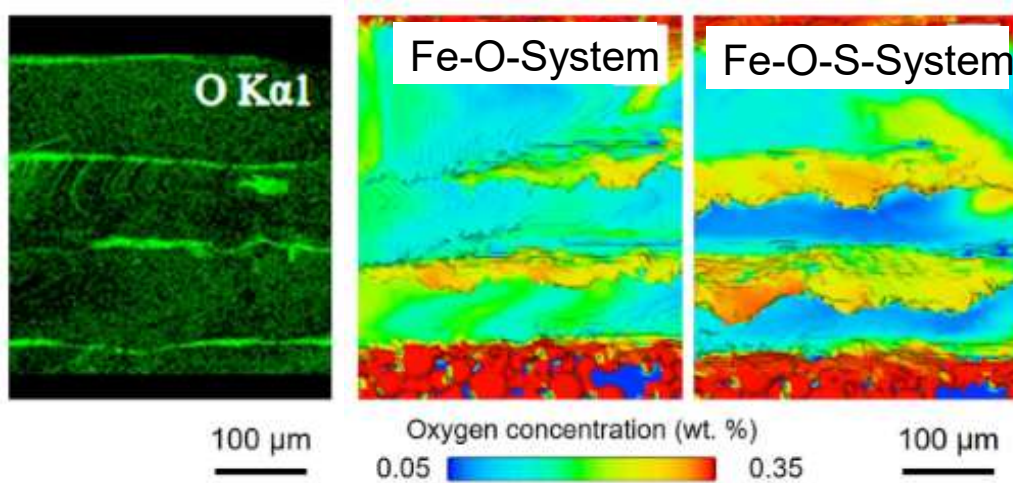
Transition from half cylinder to sphere in Dependence of the melt pool geometry



from R. Li et al., Int. J. Adv. Manuf. Technol. 59, 1025–1035 (2011). Copyright 2011 Springer.

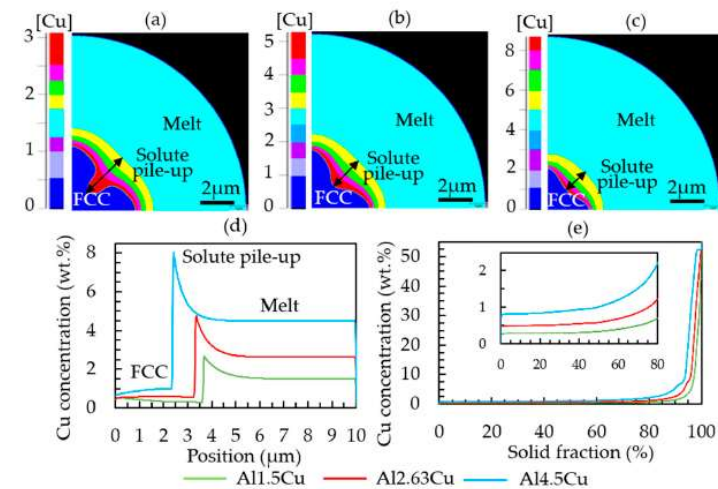
Yap et al. Appl. Phys. Rev. 2, 041101 (2015)

**Multi physics thermal fluid flow simulations**  
show locally oxygen rich streaks



**Kinetic Simulation: Phasefield (MICRESS)**

- Estimation of cooling rate
- Describing microsegregation (solute trapping)



Phyu, Raffeis, Adjei-Kyeremh et.al. DOI: [10.3390/ma16041677](https://doi.org/10.3390/ma16041677)

Exemplary References:

1. Influence of oxygen content on melt pool dynamics in metal additive: High-fidelity modeling with experimental validation, <https://doi.org/10.1016/j.actamat.2023.118824>
2. Micro pool characteristics of 316L and the influence of sulfur during SLM, <https://doi.org/10.1016/j.optlastec.2021.107136>; (finite volume method)
3. Surface-active element transport and its effect on liquid metal flow in laserassisted additive manufacturing, <https://doi.org/10.1016/j.icheatmasstransfer.2017.06.007>; (fluid flow numerical model)
4. New Design of Composites from Fresh Scraps of Niobium for Tribological Applications, [https://doi.org/10.1007/978-3-030-59868-6\\_6](https://doi.org/10.1007/978-3-030-59868-6_6)



**Aconity Mini**

(Powder Bed Fusion-Laser Beam/Metal)

- Manufacturer: Aconity3D GmbH
- Build plate diameter 140 mm, 100 mm, 55 mm
- 400 W fiber laser
- Heating plate up to 800 °C
- High speed camera
- 2 high speed pyrometers
- Spot size ~50 – 500 µm

- Manufacturer: LUNOVU GmbH
- Wire apply system 0,8 – 1,6 mm
- Powder apply system with mixing unit
- Inert gas glovebox
- Heating plate up to 400 °C
- 3-axis system (retrofitable to 5-axis)
- 2000 W diode laser



**Lunovu/Sato**

© www.das-design-plus.de

(DED, Direct Energy Deposition)

Parameters
Laser power $P_L$
Process speed $v_P$
Powder mass flow $\dot{m}_P$
Conveying gas flow $\dot{V}_{FG}$
Shielding gas flow $\dot{V}_{SG}$
Track displacement $\Delta y$
Laser spot diameter $d_L$

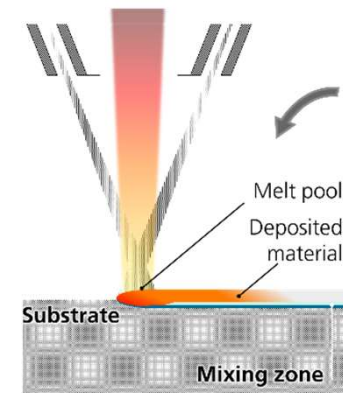


HS DED-LB/M with 8 powder pots

Combination of recycling powder with virgin powder:  
z.B. Ponticon, Lunovu



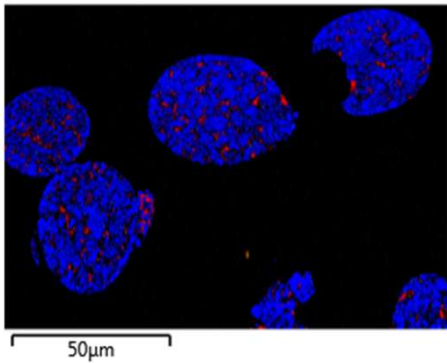
Coaxial powder nozzle



Single layers (10 mm)

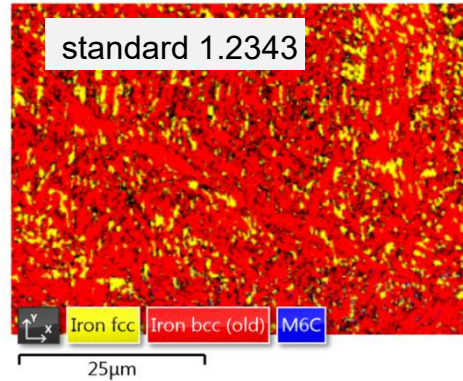
High-speed direct energy deposition with laser beam source

Powder



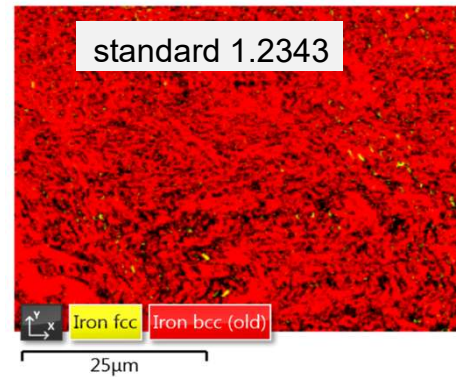
Retained austenite: 1,1%  
martensite, carbides

As built PBF test cube

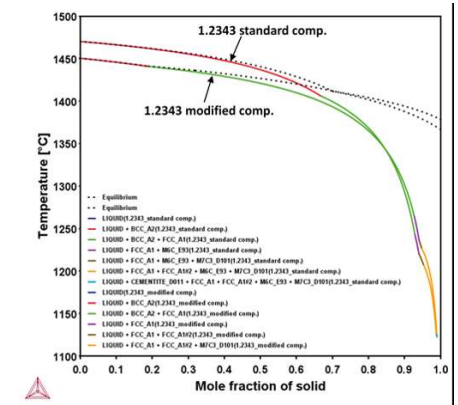


No preheating, RA: 13,8%  
martensite, carbides

Preheated PBF test cube



Preheating at 500°C  
martensite, carbides



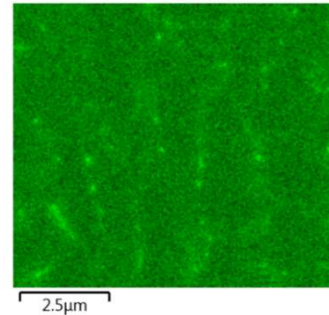
**Goal:** Eliminate RA and cracks

**Solution:**

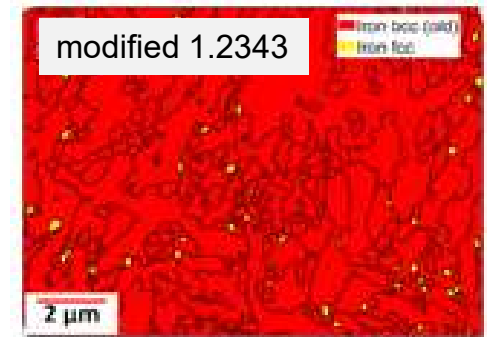
Use heating plate to decrease  
cooling rate and microsegregation

Modify composition

Mo Lα1



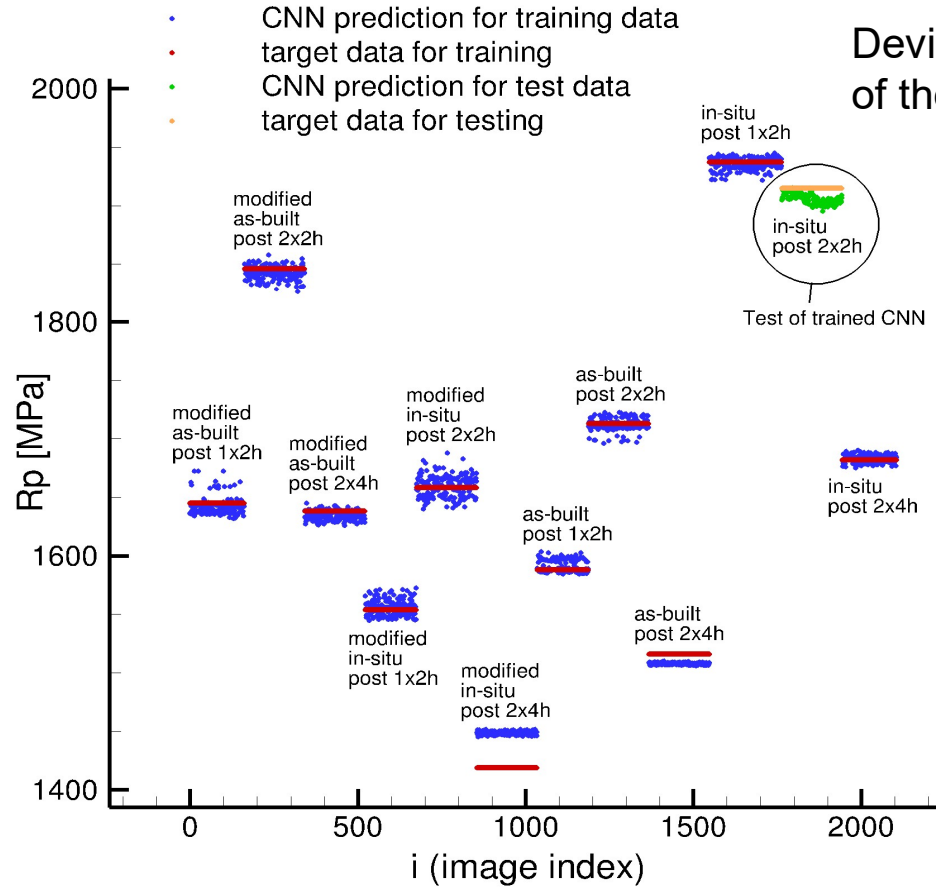
Modified As built  
PBF test cube



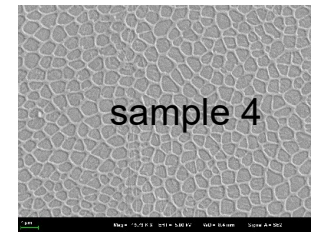
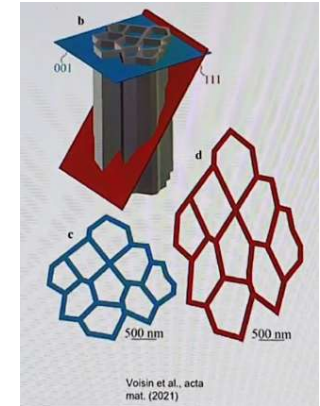
Raffeis, Adjei-kyeremeh, et. al. DOI: [10.3390/jmmp6030063](https://doi.org/10.3390/jmmp6030063)

# Prediction of Strength Properties

# 4 Microstructure Examples



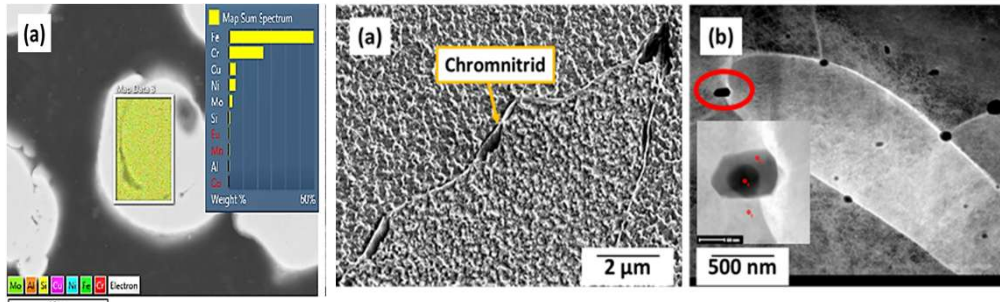
Typical AM cell structures



- Development of two convolutional neural networks:
1. Classifying samples
  2. Prediction of mechanical values

# Phase Transformation in Solid State: Duplex Steel

# 4 Microstructure Examples



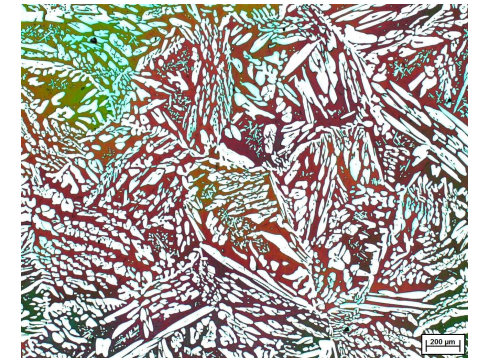
SEM-EDS-image:  
nearly pure ferritic  
powder particle

PBF-LB/M-precipitation  
(a) SEM image of  $\alpha$  with elongated nitrides  
(b) TEM image of a duplex matrix Chromium nitrides,  
oxides (black), nitride, silicon core

Orientation relationships  
between the phases:  
 $(110)_\alpha \parallel (0001)_{Cr_2N} \parallel (111)_\gamma$



LOM: heat treated PBF  
(1h, 1120°C) PBF,  $\alpha$ : 53%,  $\gamma$ : 47%



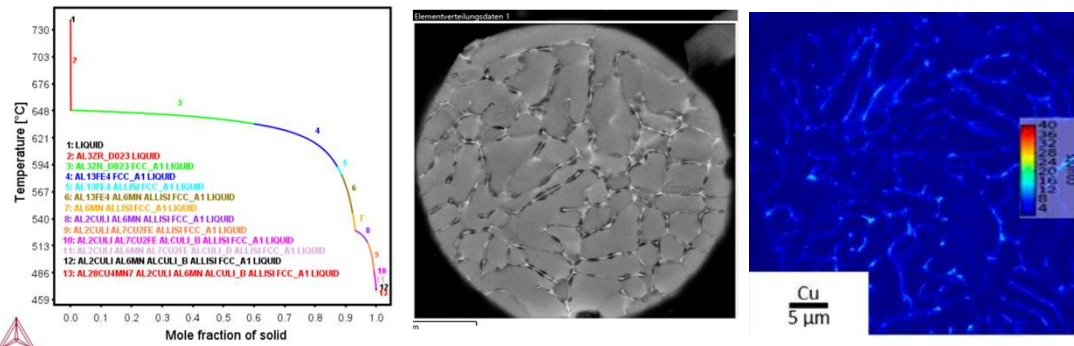
LOM: heat treated cast material

C	Si	Mn	Cr	Ni	Mo	Nb	Cu	P	S	W	V	Ti	Al	Co	N	Fe
0,028	0,69	0,60	25,05	5,09	1,99	0,02	2,87	0,001	0,002	0,01	0,021	0,001	0,023	0,003	0,203	63,39
0,021	0,67	0,57	24,48	4,96	2,03	0,023	2,63	0,012	0,008	0,025	0,050	0,008	0,023	0,003	0,148	64,25

Cast material

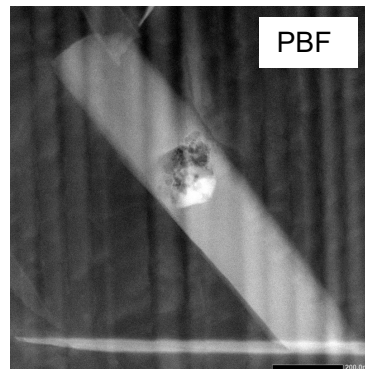
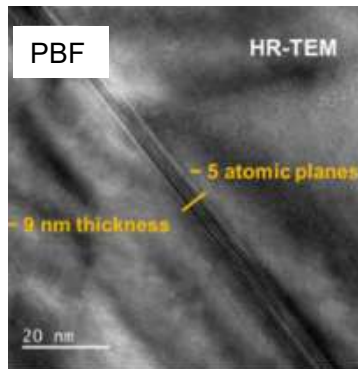
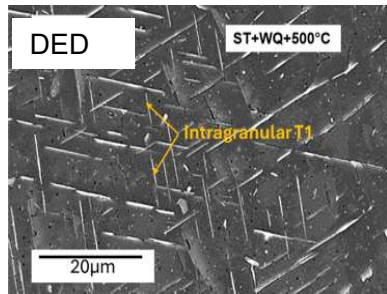
PBF-test cube

Raffeis, Adjei-Kyeremeh et. al. DOI: [10.1002/mawe.201900136](https://doi.org/10.1002/mawe.201900136)



Classical Scheil plot (TCAI20)

EPMA-images of powder particle with segregations



Images of T1 phase: DED, SEM; PBF: 450°C preheating, TEM

## Goals:

1. Formation of T1- precipitates **during** printing without post heat treatment required
2. Crack elimination through grain refinement
3. Elimination of columnar growth

## Results from first tests:

1. No T1-formation in powder particle
2. No T1-formation in PBF
3. Crack formation in PBF

## Solution:

1. PBF In-situ preheating → T1 precipitation
2. DED → T1 precipitation
3. Grain refinement ( $TiB_2$ ) → no cracks

## Next steps:

Investigating nucleation mechanisms

Raffais, Adjei-Kyeremeh et al. DOI: [10.3390/ma13225188](https://doi.org/10.3390/ma13225188)

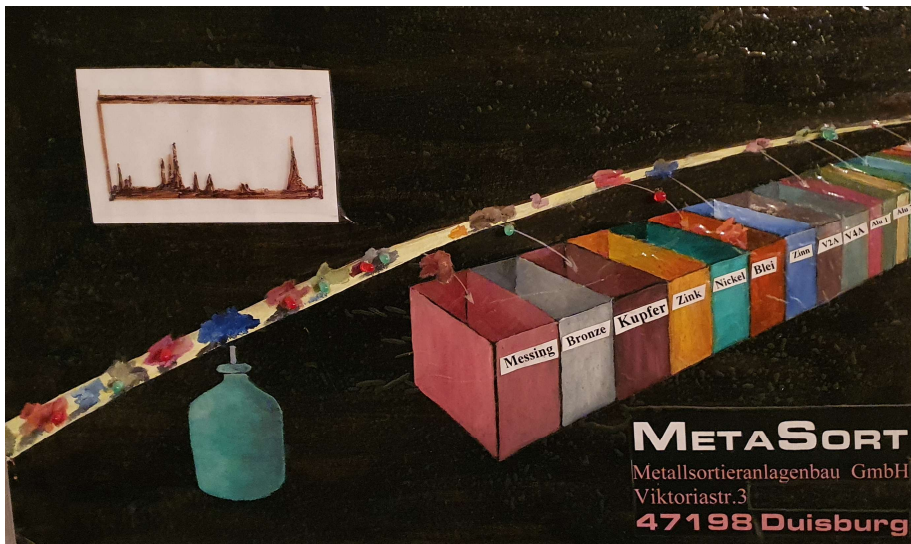
Adjei-Kyeremeh, Raffais et al. DOI: [10.1088/1757-899X/1310/1/012003](https://doi.org/10.1088/1757-899X/1310/1/012003)

### Metal Alloy Sorting for Preconditioning for single grade alloy, Intermediate products, End of Life Scrap, not new

<https://medialib.cmcdn.dk/medialibrary/DEF42602-16D9-4368-83C4-F77E83D08491/3E7AA7E8-D056-EE11-84C7-00155D0B0940.pdf>

Development of spectral analyses

1980s: In-line X-ray fluorescence (**XRF**) analysis



Hand held **XRF**-devices



Laser based sorting:  
**LIPS / LIBS**

**XRT**

X-ray transmission spectrometry

Sorting heavy (high density)  
from light metals (low density).

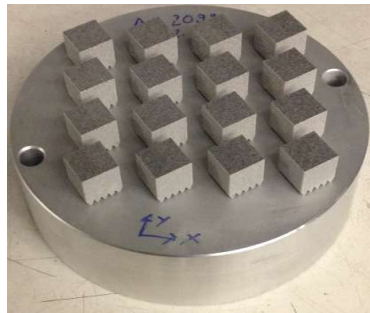
Separation of heavy metals  
Cu, Zn, brass, lead, stainless steel.

## Research Scale Recyclables

## 5 Sustainable Production (xCluster IoSP)



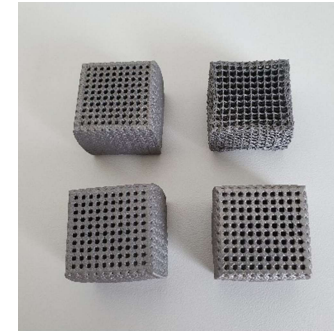
Casting block



PBF test cubes



Tensile test specimens



Pressure test specimens



Lattice structures



Melt retrieved from atomizer

Preparation for atomizing feedstock:

- Sandblasting
- Clean with alcohol
- Store in alcohol if prone to corrosion
- Use if chemical composition is ok as pre-alloys

<https://doi.org/10.18154/RWTH-2024-11018>

<https://medialib.cmcdn.dk/medialibrary/DEF42602-16D9-4368-83C4-F77E83D08491/3E7AA7E8-D056-EE11-84C7-00155D0B0940.pdf>

- Used electrodes, develop as pre-alloys
- Broken tools
- Use if chemical composition is ok



Broken tensile test adapters 1.2709



Electrode remnants (TiAl6V4)



CMSX\_4 alloy

<https://doi.org/10.18154/RWTH-2024-11018>

## Concept of using powder with wider PSD



## EBuild® 850: Turbine Blade

Material	TiAl6V4
Total melt time	12h
Layer height	400 µm
<b>Powder distribution</b>	<b>0-150 µm</b>
Process temperature	850 °C

Electron Beam  
Powder Bed Fusion,  
GIFA 2023 in Düsseldorf

In-situ backscattered electron image of full build area

## RE-Use of powders with or without blending with virgin powders

1. Metal powders in additive manufacturing: A review on reusability and recyclability of common titanium, nickel and aluminum alloys, <https://doi.org/10.1016/j.addma.2021.102017>
2. Effects of powder recycling on stainless steel powder and built material properties in metal powder bed fusion processes, <https://doi.org/10.6028/NIST.AMS.100-6>
3. Revealing the Effects of Powder Reuse for Selective Laser Melting by Powder Characterization, <https://doi.org/10.1007/s11837-018-3305-2>
4. Powders for powder bed fusion: a review, <https://doi.org/10.1007/s40964-019-00078-6>

## Concepts for RE-engineering powder:

Plasma spheroidisation process  
(PIGA 6KUniMelt, GE, Rovalma, Tekna, etc.)  
US process (e.g. Amazemet)

**Conversion of oversized water atomized powders by Plasma spheroidization** into high quality particles and lower oxygen content through reducing agents in the gas nozzle\*; <https://doi.org/10.1016/j.jclepro.2020.122077>  
\*Kelkar, R.M., 2019. US Patent US 2019/0061005A1: High Quality Spherical Powders for Additive Manufacturing Processes along with Methods of their Formation. US Patent Office, USA. wering the oxygen content through reducing agents in the gas inlet.

# Methodology for Sustainable Alloy Design

# 5 Sustainable Production

Use recycled material  
 Reducing powder waste  
 Reducing experiments

Find (new) sources of cleaned, de-oiled scrap as pre-alloys with defined analysis to atomize in research batch size, EOF

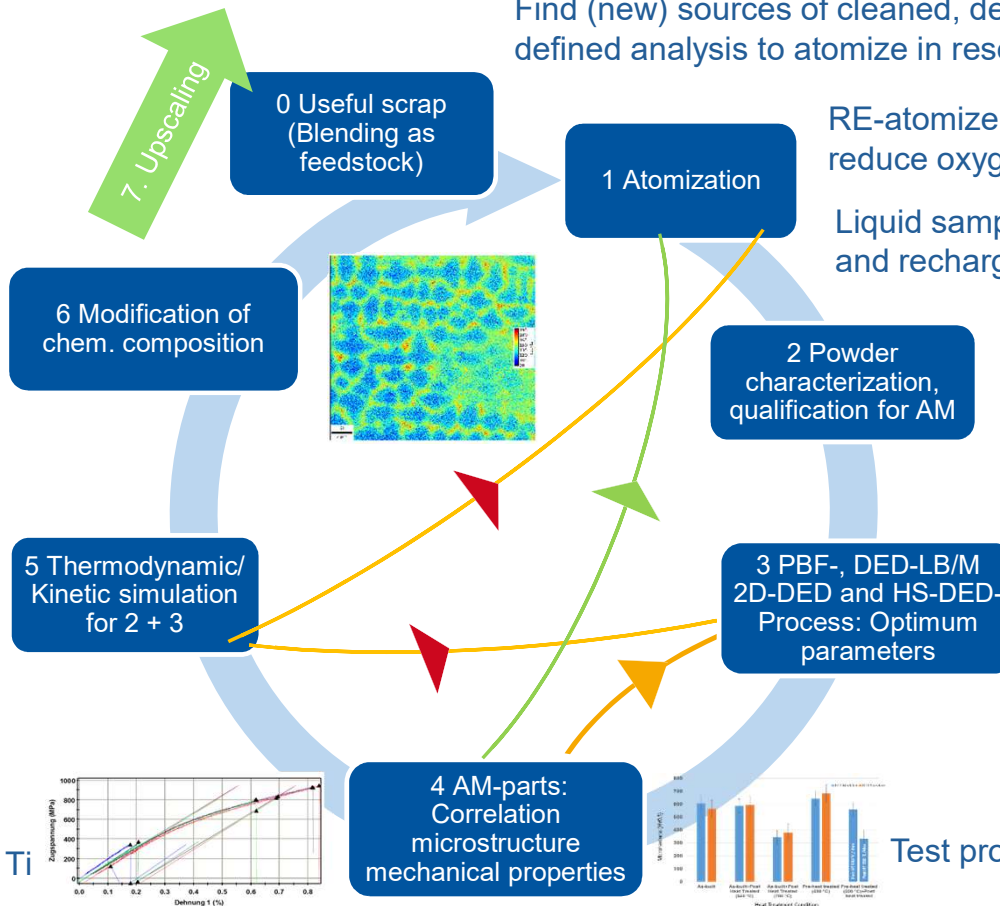
Multi phase systems  
 Control chemistry, chemical reactions, oxide and nitride formation, microstructure...  
 Consider material dependent properties, e.g. aging

Phase Field, Calphad, Solute trapping, Scheil, FEM-modeling

Simulative alloy design, melt pool simulation, microstructure  
 Validation with 4a) and 4b)

Alloy development

Find limits for critical elements: O, N, P, C  
 Find solutions for microalloying elements: B, Ti



RE-atomize powder: spheroidise, reduce oxygen, PIGA, US.....

Liquid sample taking device for monitoring and recharging elements in melt desirable

Analyze powder: Validation with feedstock, AM-part, microstructure

- Find optimum process and parameters for application
1. Dense and crack free
  2. Controlled microstructure
  3. Surface quality
  4. Use wider PSD
  5. Use blended, recycled powder
  6. Print in research scale

Test properties

## Standard and Modified Alloys, Atomized and Printed

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### Steel Alloys

1.4408, 316L, 1.4404 austenitic steels  
1.4517 Duplex steel  
1.4462 Duplex steel  
1.5264 Duplex steel  
1.2343, H11, Hot work steel  
1.2343 modifications  
1.2709, Hot work steel  
AISI 420 Martensitic steels

### High Temperature MMCs

### Nickel Alloys

2.4856 (Inconel 625)  
2.4668 (Inconel 718)  
2.4365 (Monel)  
CMSX\_4, Superalloy

### Titanium Alloys

TiAl6V4

### Aluminum Alloys

(Printed only)

#### Cast alloys

AlSi12  
AlSi10Mg  
Customized alloys  
Wrought alloys  
AA2099  
Modified AA2099  
Scalmalloy  
AA2050  
ZnAlCu-alloys

# Atomizing as a Contribution to the UN Sustainable Development Goals (SDGs)



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# Thank you for your attention

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