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7th International Symposium on Gas-Phase Synthesis of Functional Nanomaterials

From Solar Panel to 3D Print: Scalable Microwave Plasma Processing of End-of-Use PV Silicon for Laser Powder Bed Fusion

S. Kuns[1], C. Kunz[2], F. Kunze[1], N.B. Nezafat[3], G. Schierning[3,4], J.K. Hufenbach[2] and S.M. Schnurre[1,4]

[1] Institut für Umwelt & Energie, Technik & Analytik e.V. (IUTA), Duisburg, Germany

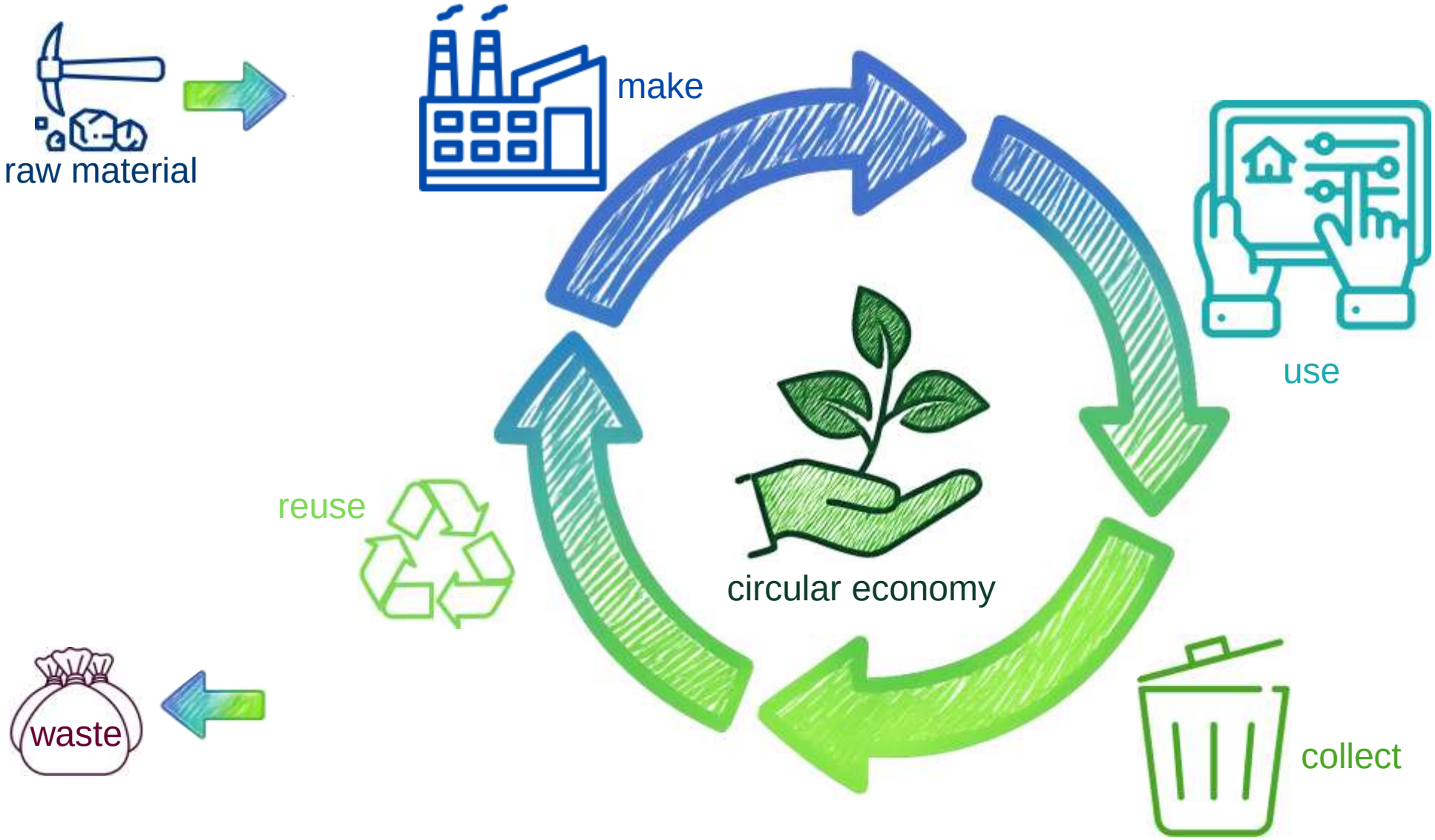
[2] Leibniz Institute for Solid State and Materials Research (IFW) Dresden e.V., Germany

[3] University of Duisburg-Essen, Institute for Energy and Materials Processes Applied Quantum Materials (EMPI), University of Duisburg-Essen, Duisburg, Germany

[4] Center for Nanointegration Duisburg-Essen (CENIDE), Duisburg, Germany

Die Institute der Johannes-Rau-Forschungsgemeinschaft werden vom Land NRW institutionell gefördert.

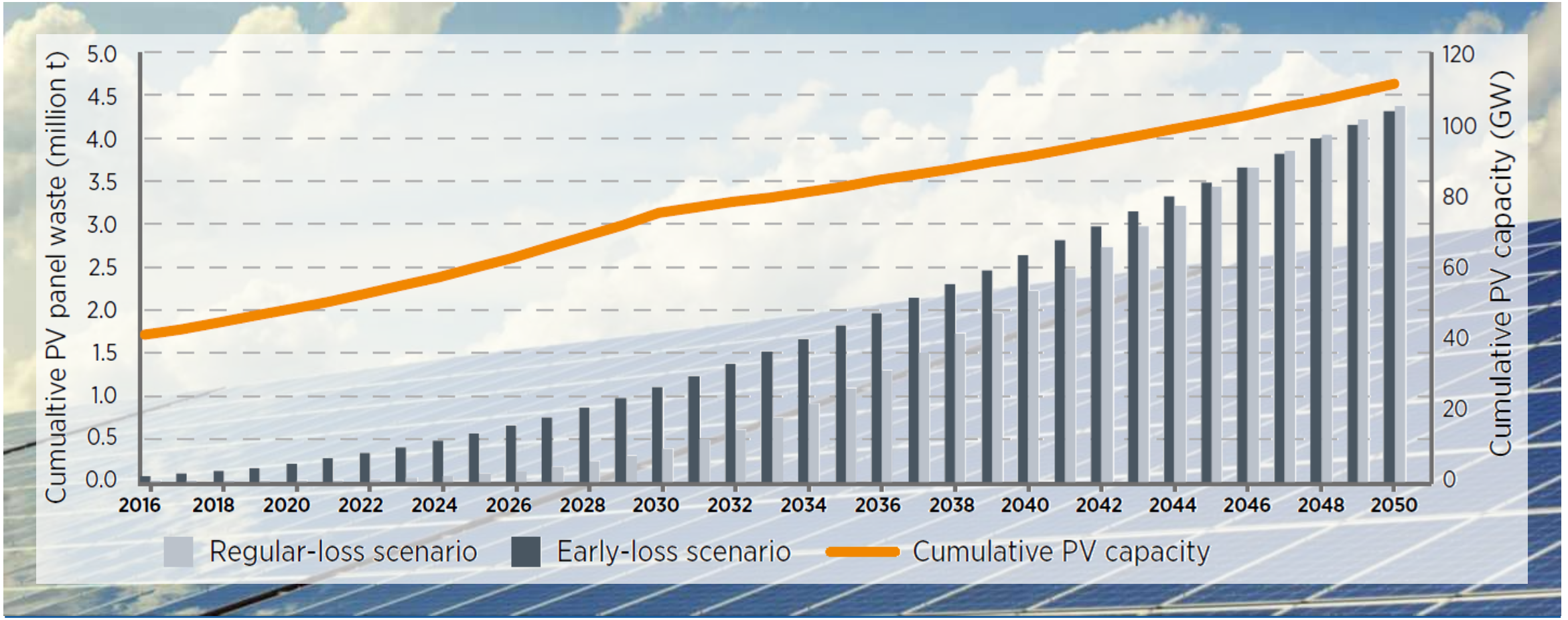
Motivation


raw material
make
use
reuse
circular economy
waste
collect

Motivation

Current situation

- End-of-Use PV modules: 1.7 million tons expected worldwide by 2030, 400 kt in Germany with regular use
- Silicon content is currently hardly used to add value; targeted recovery is just beginning



Starting Material

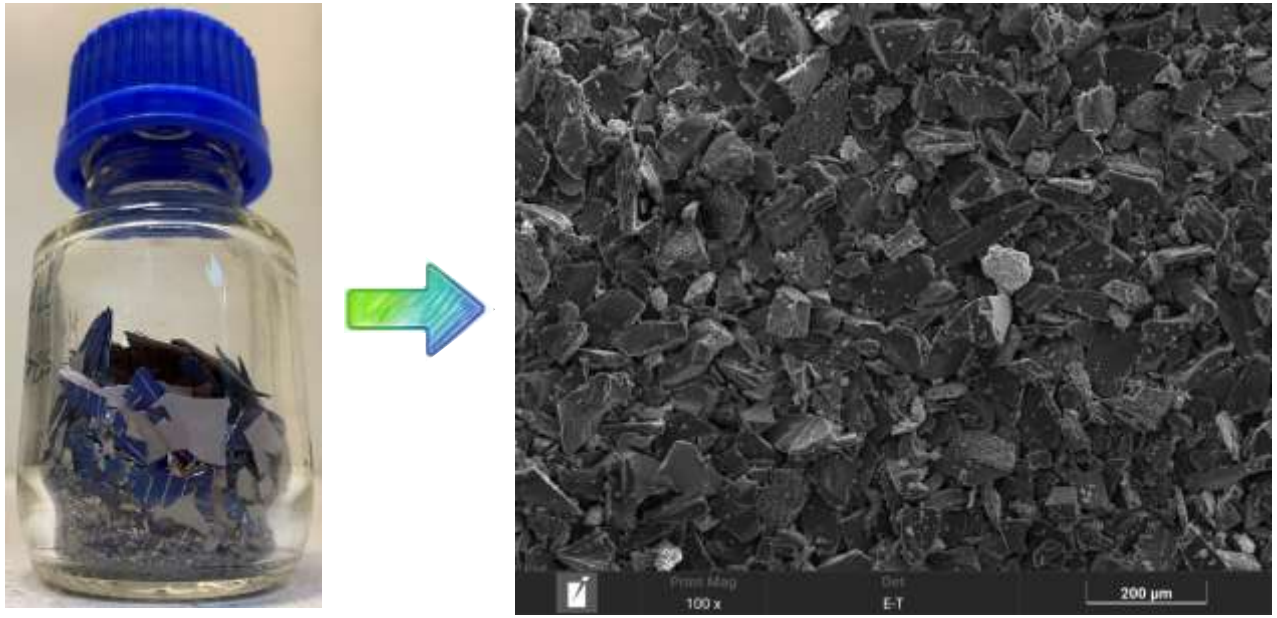
PV-Silicon from different sources

- Several companies developed different recycling methods
- Development ongoing
- Re-use of material must be possible independent of starting specification

Starting Material

Morphology

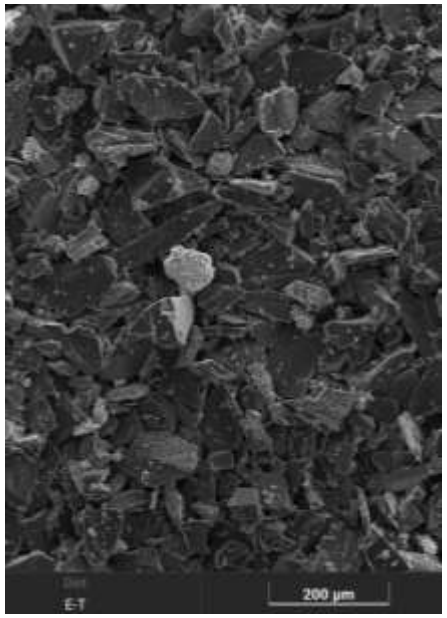
- Coarse and brittle material
- Additive Manufacturing requires size reduction and shape homogenization
  - Targeted particle size for PBF-LB/M: 25 to 80 µm
- Fragmentation and sieving process
  - Ultra centrifugal mill, sieving tower

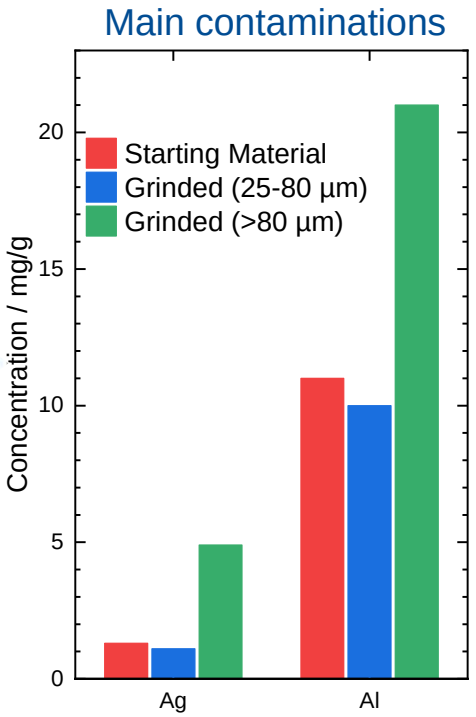


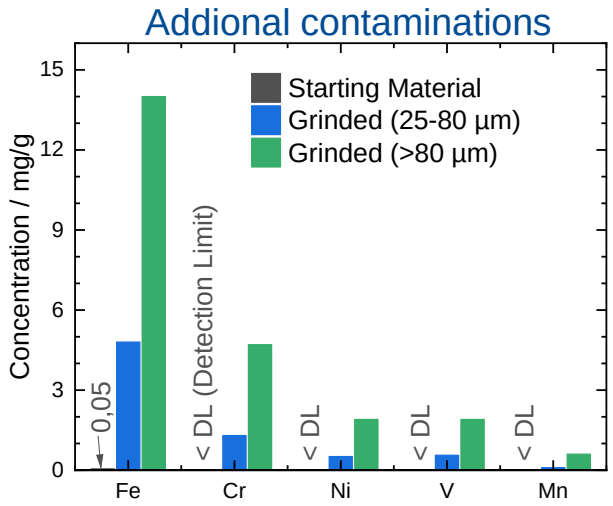
Starting Material

Impurities

- PV-Si contains Silver and Aluminum due to grid lines and backside contacts
  - Amounts differ in the size fractions
- After grinding and sieving additional contaminations from stainless steel
  - Harmful effect for further application possible





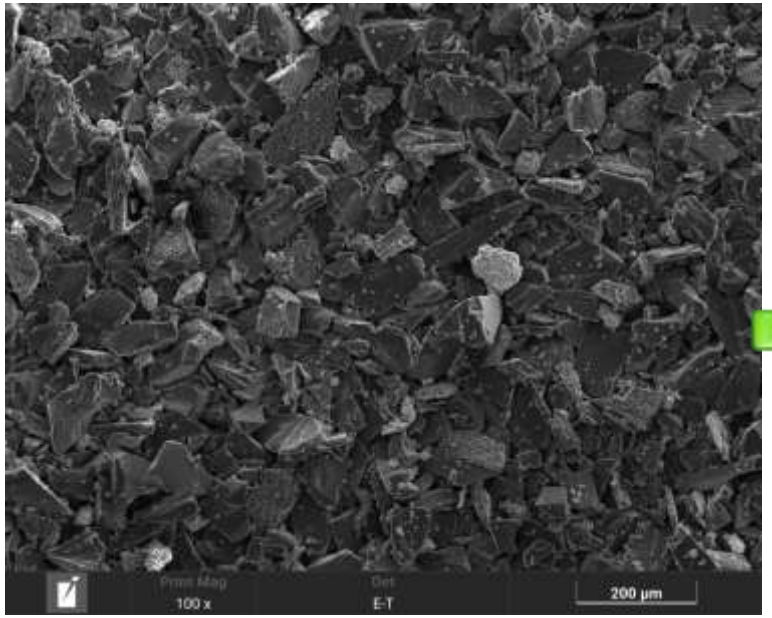


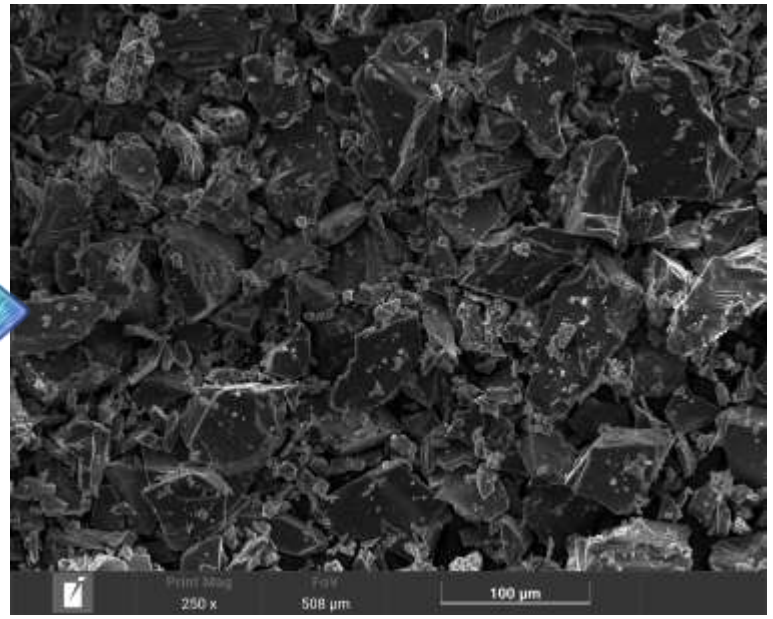
Treatment

Feeding system + Hot wall reactor

- Target: shape homogenization
  - Increase flowability

Setup		VW1	VW2
Residence Time	[s]	4	20
Carrier Gas (Ar)	[slm]	10	3
Sheath Gas (H_2)		40	8
Ring Gap (Ar)		5	-
Temperature	[°C]	1100	
PV-Si Feeding Rate	[g/min]	2	



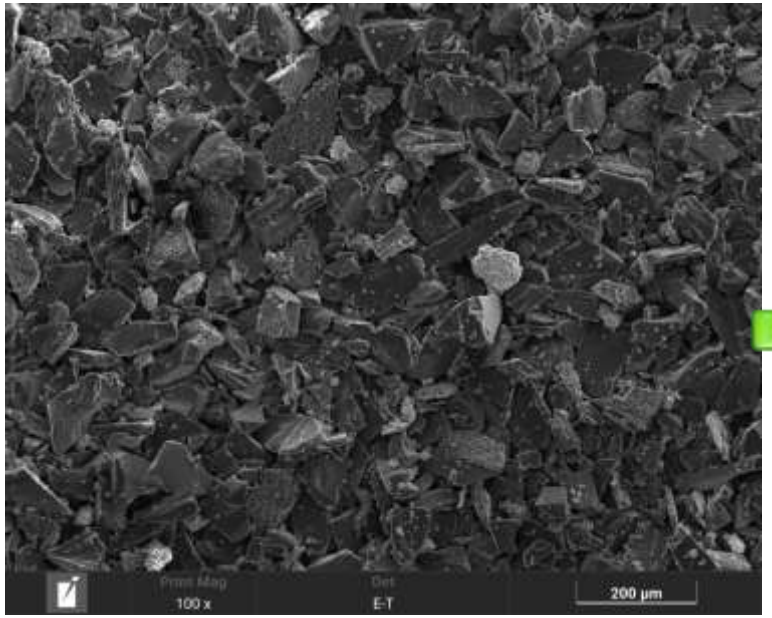


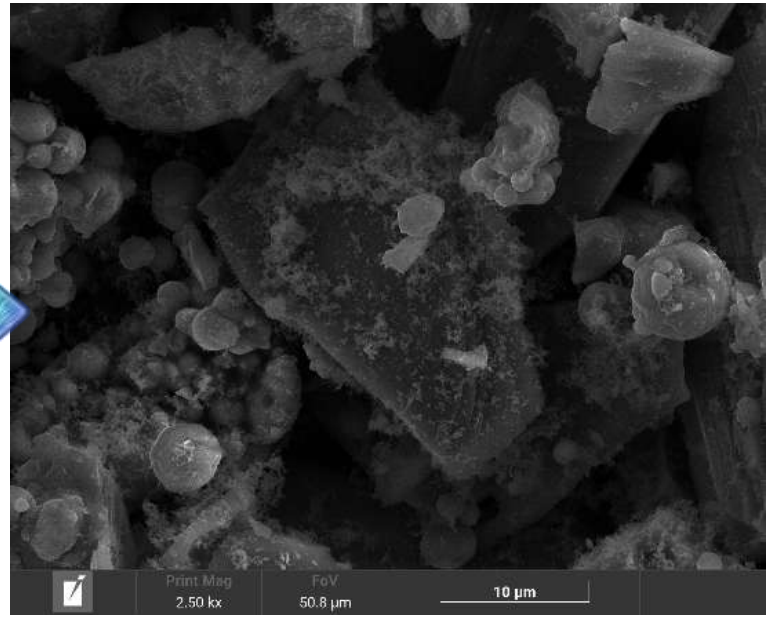
Treatment

Feeding system + Hot wall reactor + SiH_4

- Target: shape homogenization by filling the cracks

Setup		VW1	VW2	SZ1
Residence Time	[s]	4	20	6,5
Carrier Gas (Ar)	[slm]	10	3	3
Sheath Gas (H_2)		40	8	29
Ring Gap (Ar)		5	-	-
Silane (SiH_4)		-	-	1,6
Temperature	[°C]	1100		
PV-Si Feeding Rate	[g/min]	2		

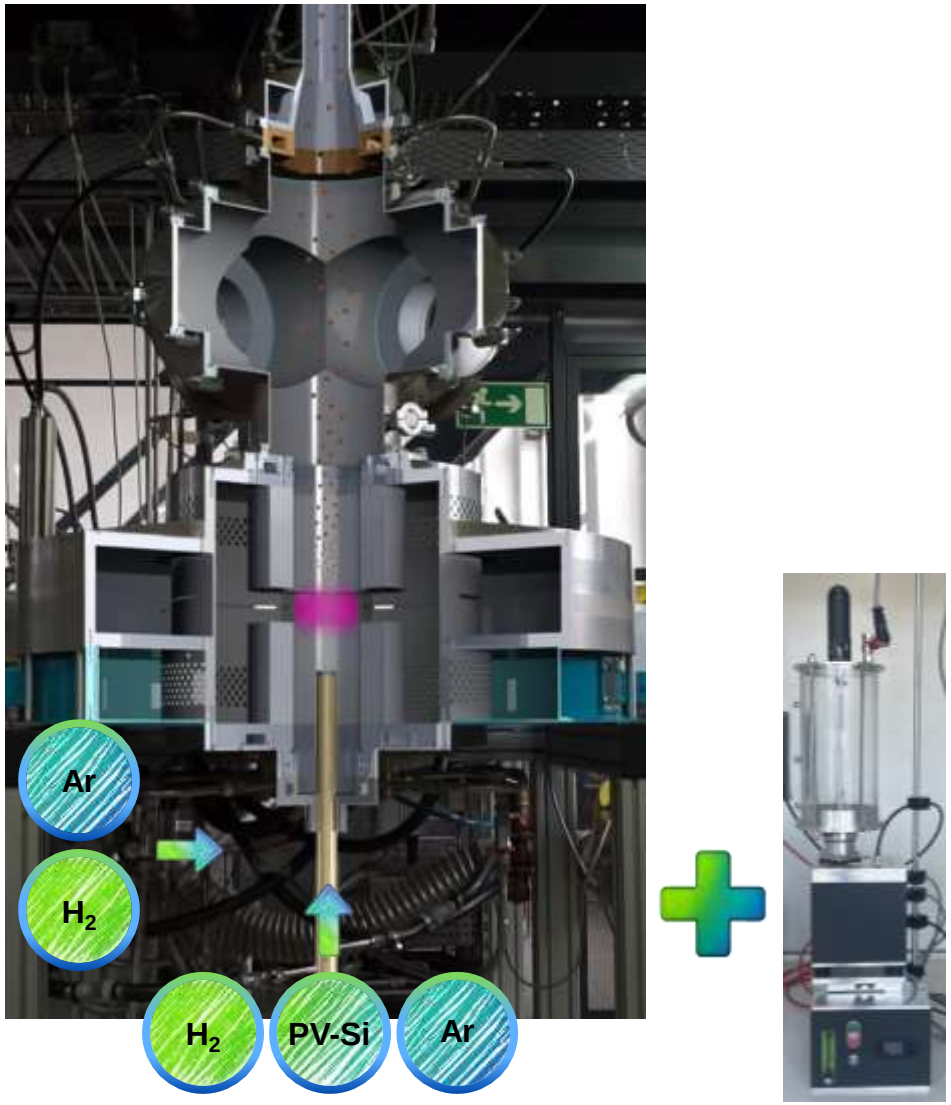




Treatment

Change to plasma reactor

- Microwaves
  - 915 MHz with up to 50 kW
  - Focused inside quartz glass tube
- Two nozzles
  - Inner nozzle carries precursor
  - Coaxial nozzle stabilizes the flow
- Atmospheric Ar/H_2 plasma
- Combination with feeding system

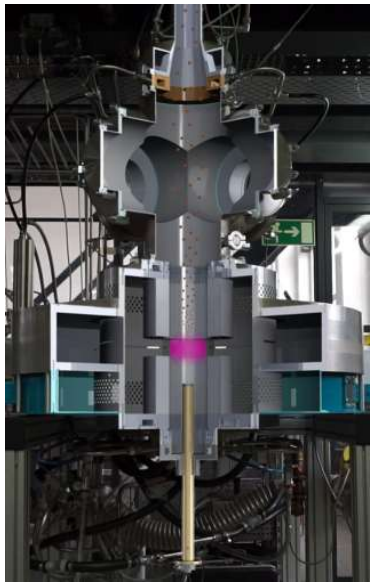


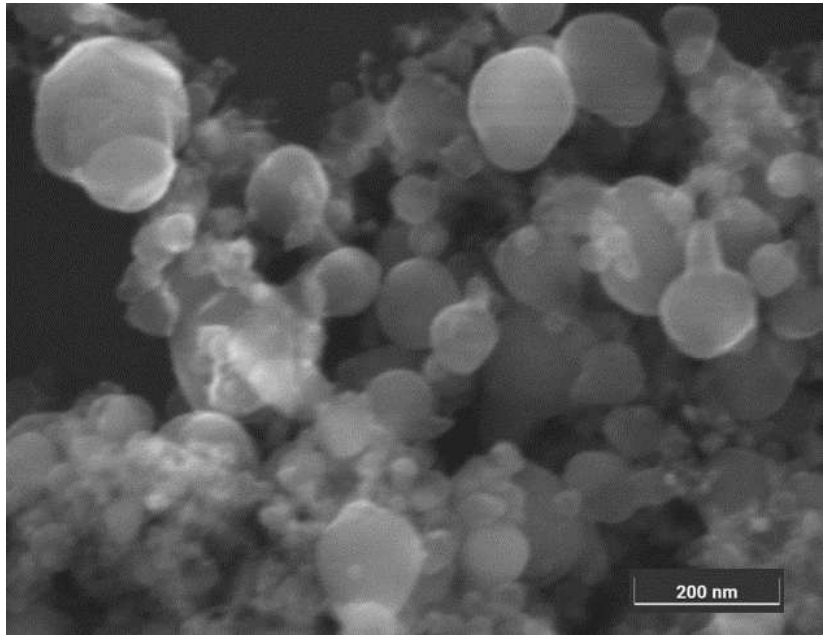
Treatment

Microwave plasma reactor

- Study of residence time by varying volume flow through inner nozzle
  - 5 slm – longer residence time
  - 22 slm – shorter residence time

Parameters	5 slm	22 slm
Carrier gas	2 slm	10 slm
Ar	1 slm	10 slm
H_2	2 slm	2 slm





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- Ideal particle shape and size for additive manufacturing

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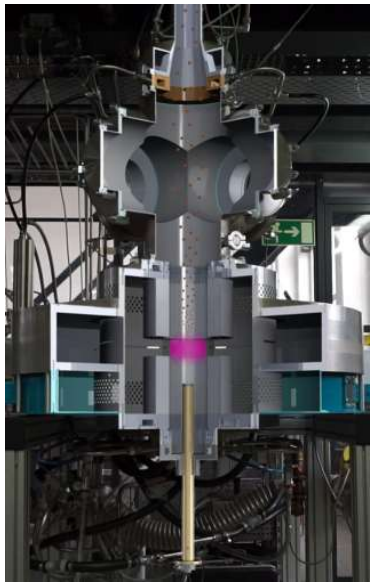

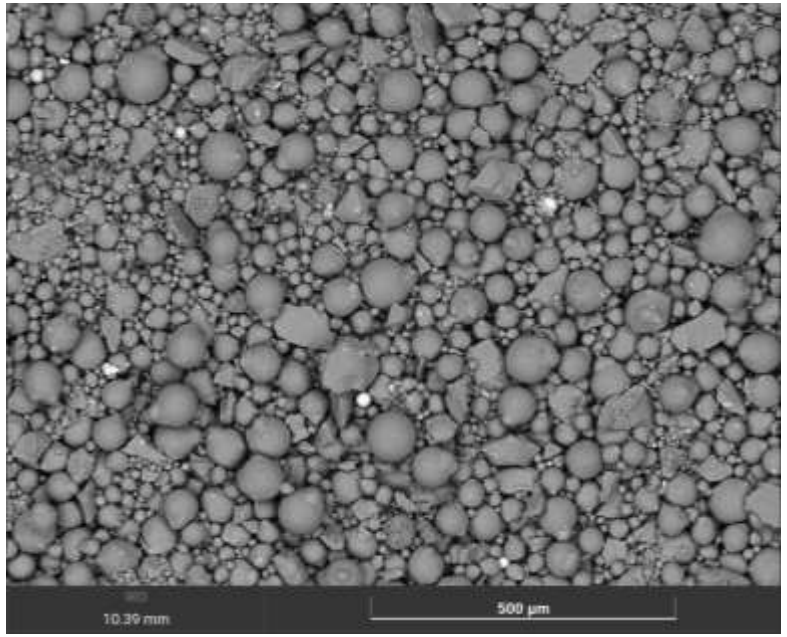

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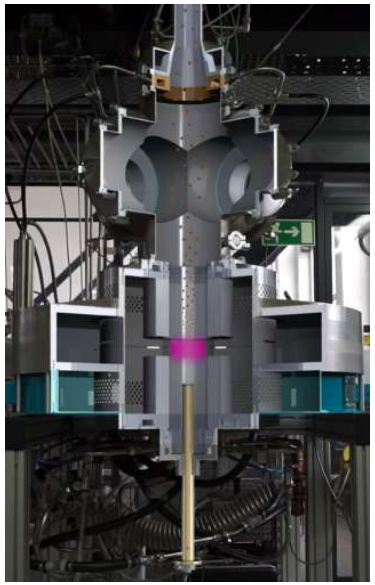
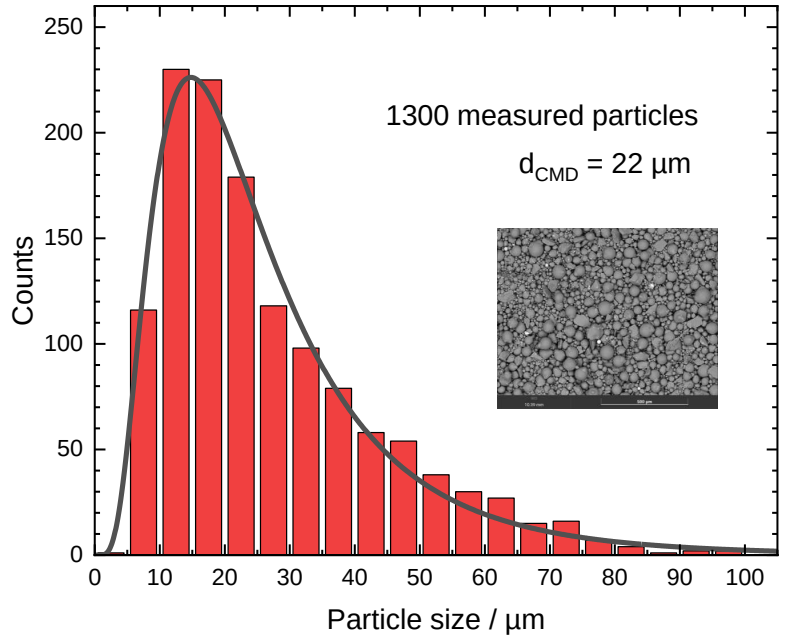
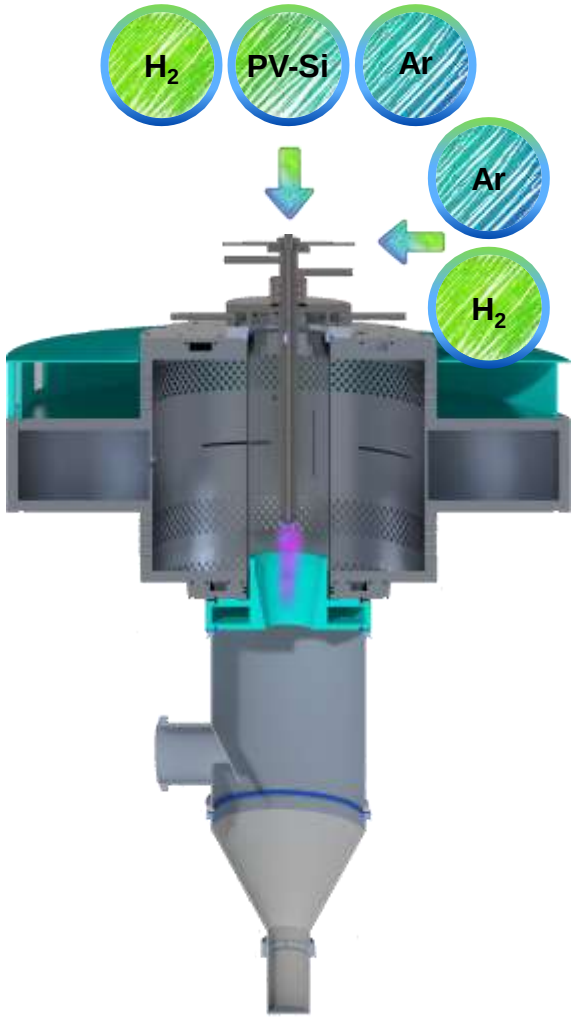




Treatment

Turning of plasma reactor

- Micron sized particles follow gravity
  - Material doesn‘t reach filter
  - Hard to collect
  - Only very small amount produceable
- Improvement of powder feeding and particle collection
- Plasma ignites with standard conditions



Plasma Standard Conditions		
Inner Nozzle	Ar	15 slm
	H_2	2 slm
Coaxial Nozzle	Ar	80 slm
	H_2	5 slm

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  - Pulsation indicates lower stability
  - Arcs move upwards & disappear
  - Increased Argon flows stabilize the plasma

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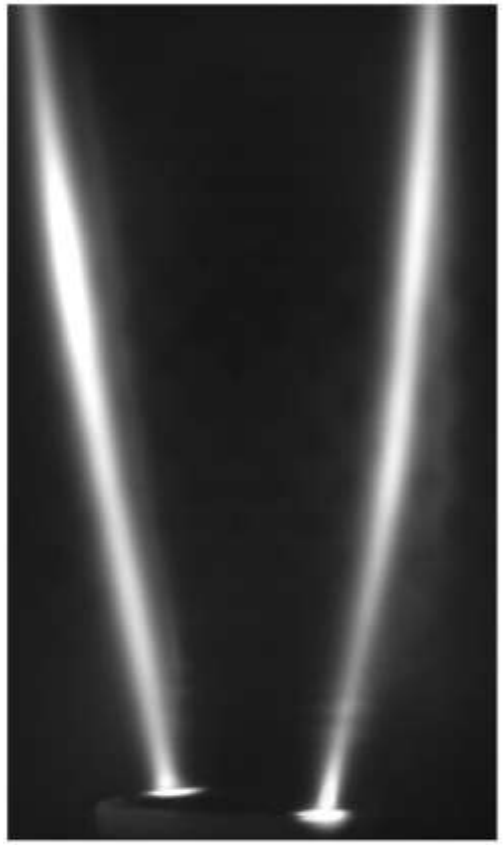

Plasma before reactor turning

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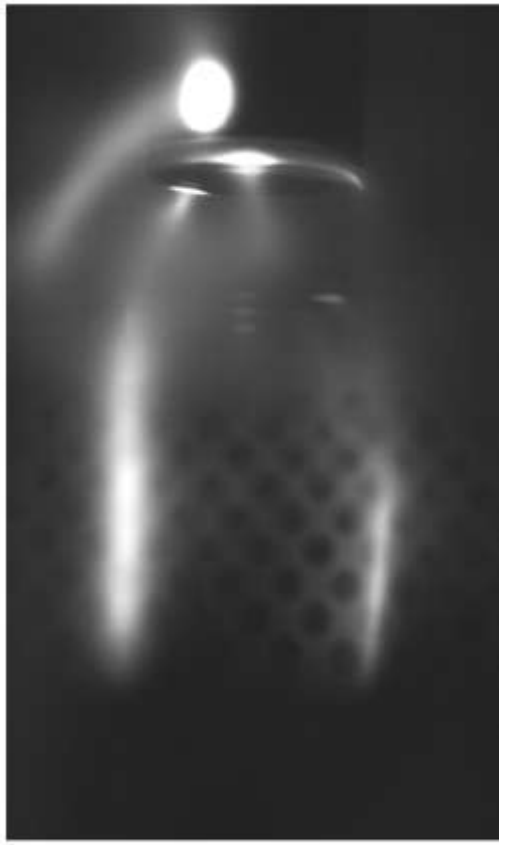

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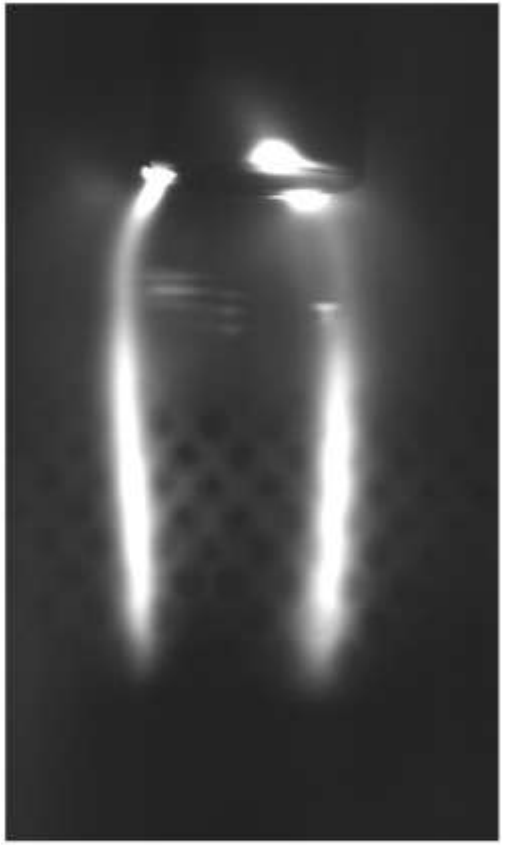
Plasma Standard Conditions			Optimization
Inner Nozzle	Ar	15 slm	30 slm
	H_2	2 slm	2 slm
Coaxial Nozzle	Ar	80 slm	120 slm
	H_2	5 slm	5 slm

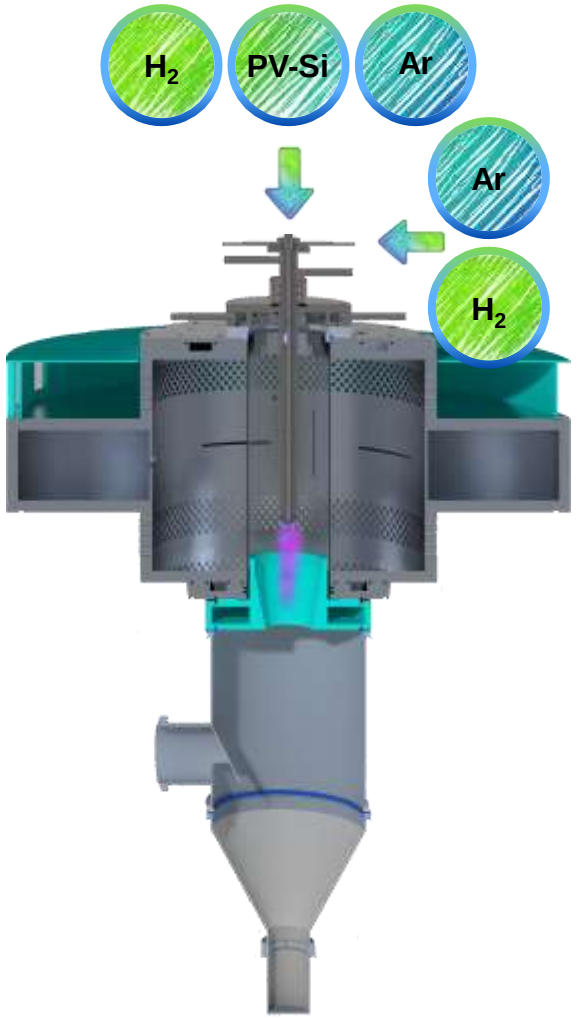


Plasma with optimized gas flows

Treatment

Optimized plasma conditions

New optimized plasma conditions			
Inner nozzle	Ar	30 slm	5 kW
	H_2	2 slm	
Coaxial nozzle	Ar	120 slm	
	H_2	5 slm	



Treatment

Optimized plasma conditions

- Changing the residence time by varying the gas stream of the inner nozzle
- Changing the power
- Combining both beneficial effects

New optimized plasma conditions				d_{CMD}	Rounding
Inner nozzle	Ar	30 slm	5 kW	7.7 µm	74.4 %
	H_2	2 slm			
	Ar	14 slm	5 kW	7 µm	79.4 %
	H_2	2 slm			
	Ar	30 slm	7.5 kW	9.4 µm	89.8 %
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	Ar	14 slm	7.5 kW	26.6 µm	85.9 %
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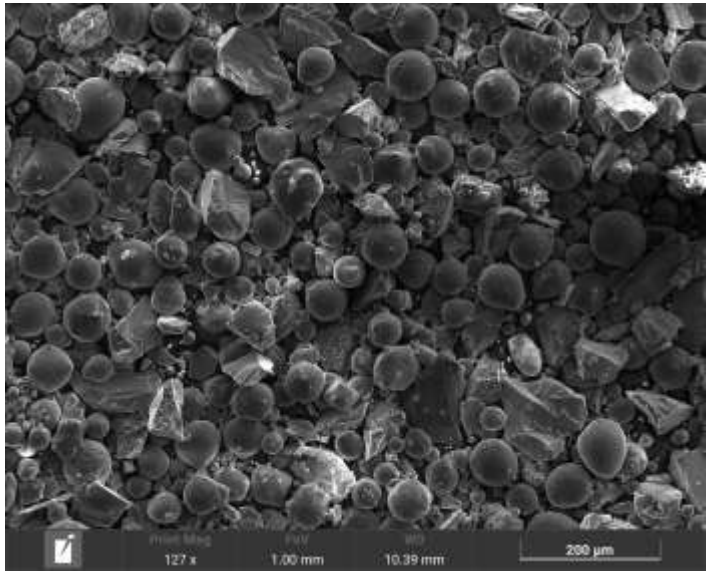

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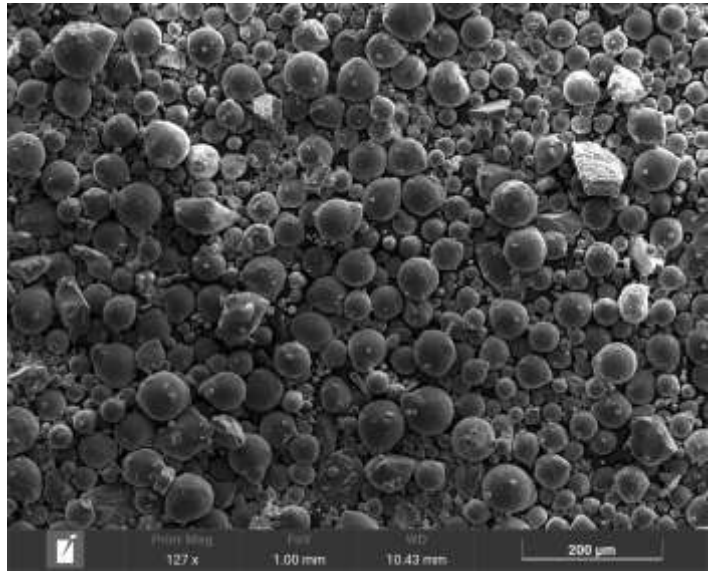

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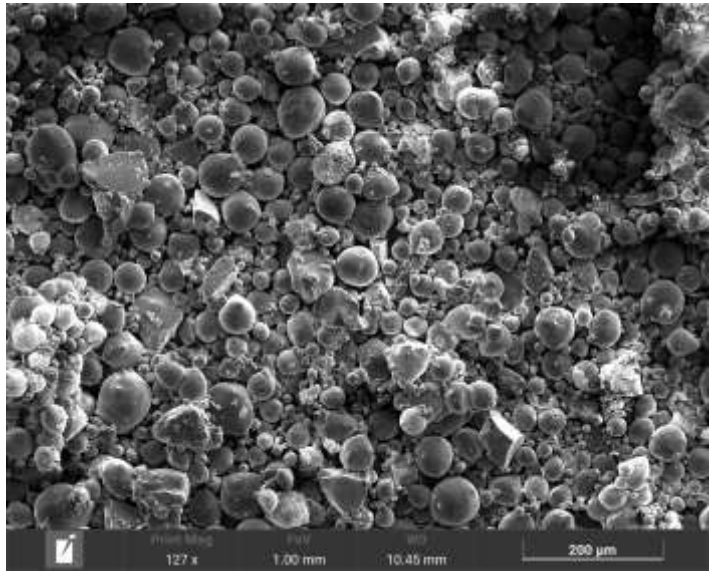

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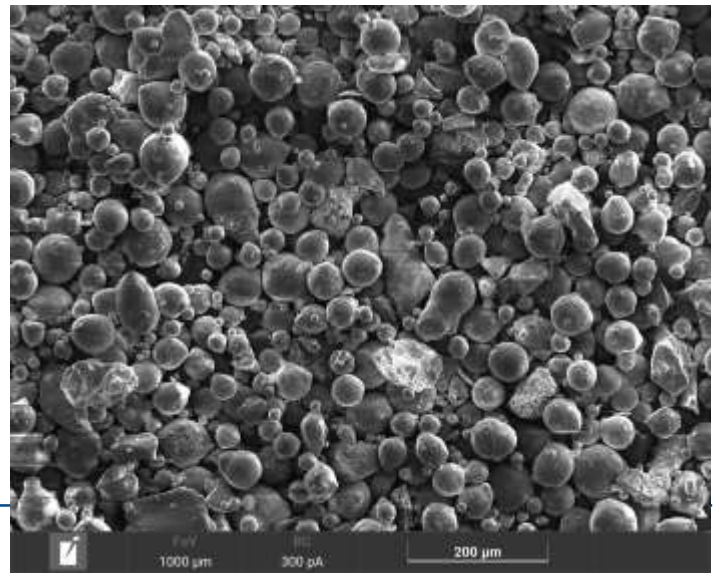
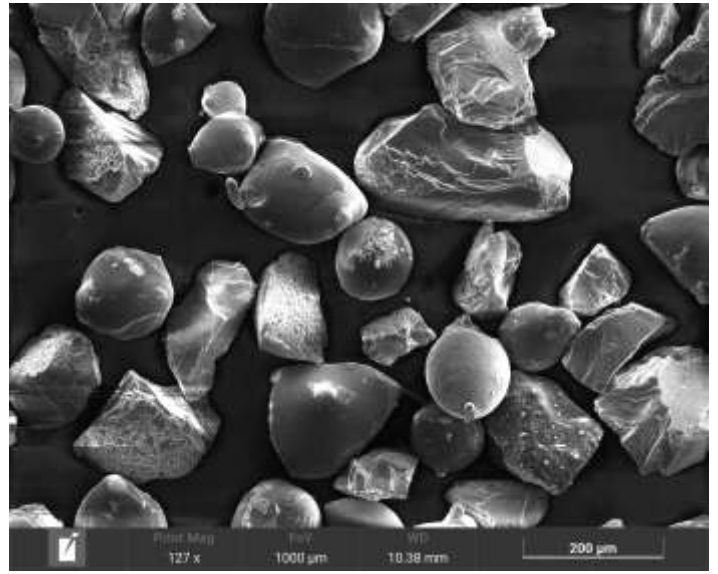


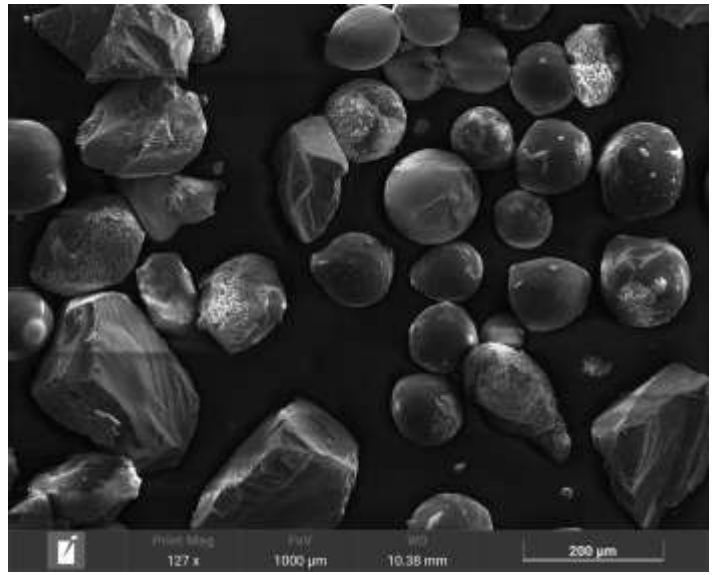
Treatment

Size effect

- Particle size of starting material > 80 µm

New optimized plasma conditions				d_{CMD}	Rounding
Inner nozzle	Ar	14 slm	5 kW	107 µm	47.7 %
	H_2	2 slm			
	Ar	14 slm	7.5 kW	98 µm	73.3 %
	H_2	2 slm			

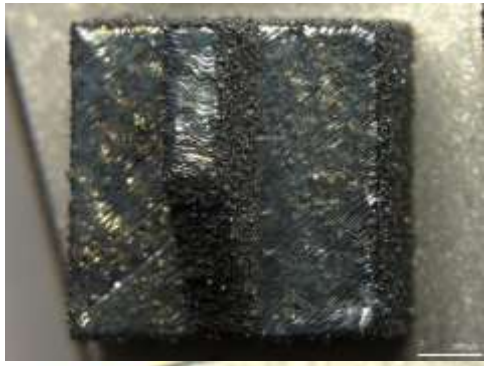


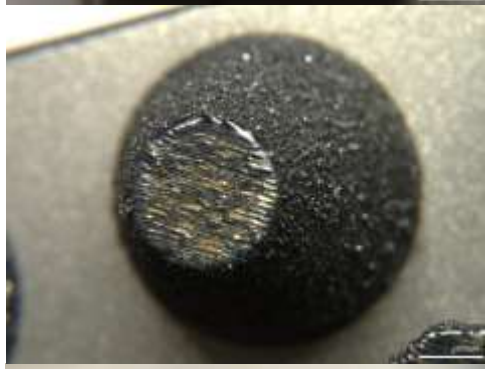


Additive Manufacturing by IFW

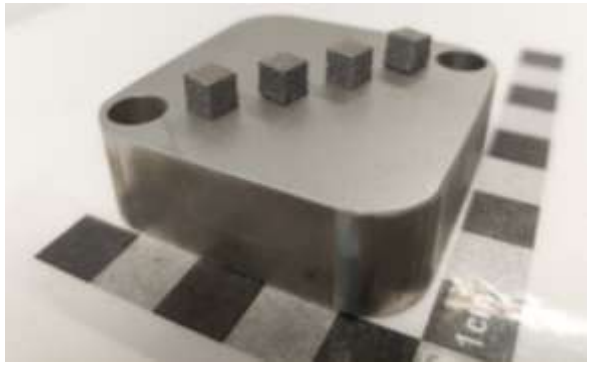
PBF-LB/M

- First parameter studies with conventional Si-powder
- Successful building of cm^3-cubes and larger parts with varying geometries
- Analysis
  - Phase components
  - Structure
  - Relative density
- First successful experiments with PV-Si-powder from hot-wall reactor

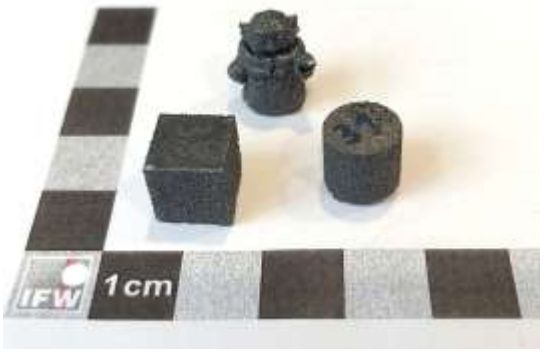




PV-Si-powder (HWR)



Conventional Si-powder

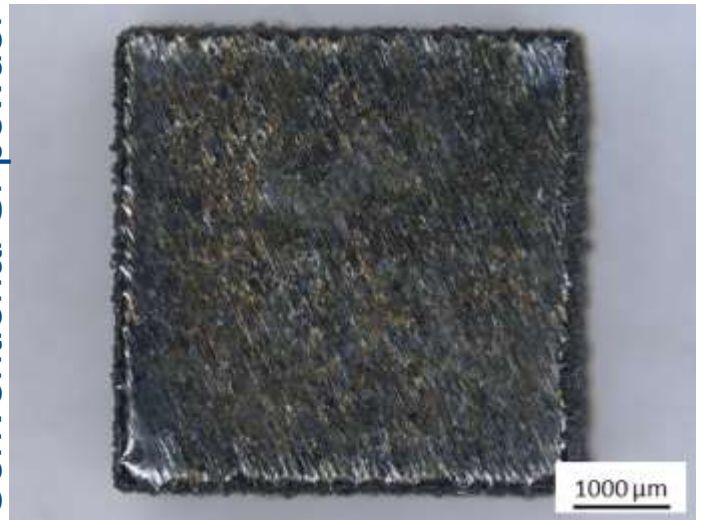


Additive Manufacturing by IFW

Leibniz-Institut für Festkörper- und Werkstoffforschung Dresden

Optical microscope

Conventional Si-powder



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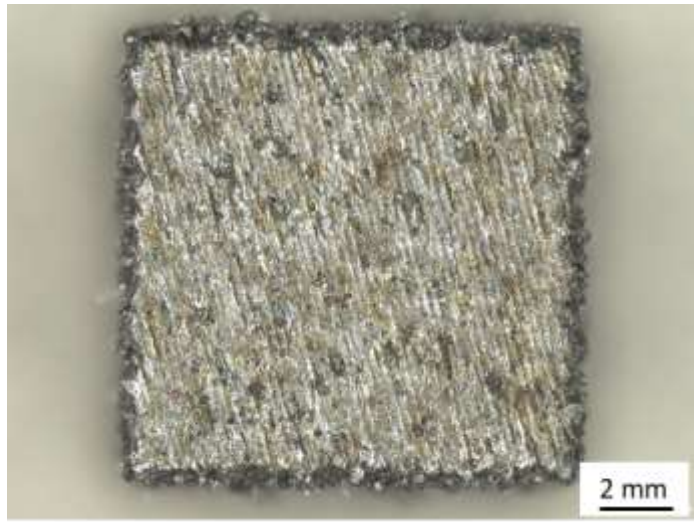
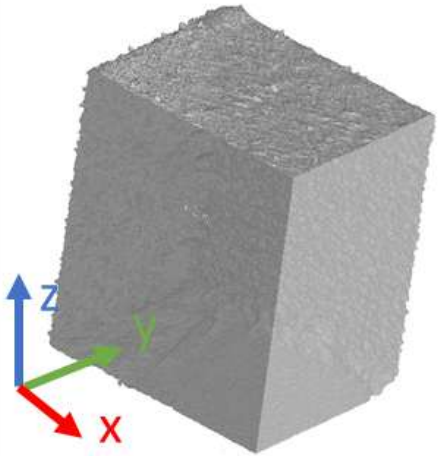
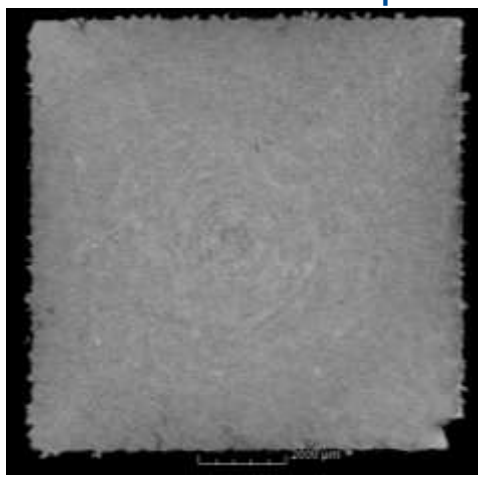


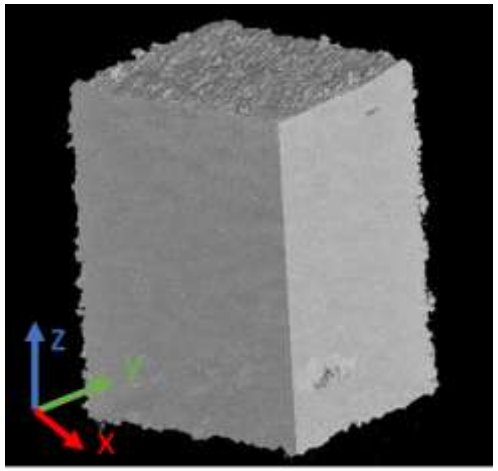
µ-Computed tomography



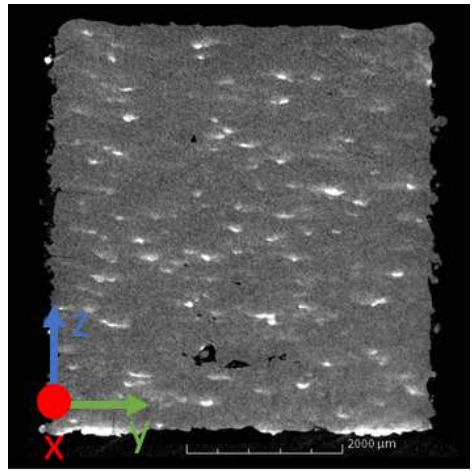
3D-model



Side view



3D-model



Side-view

Characterization by AQM

Key findings

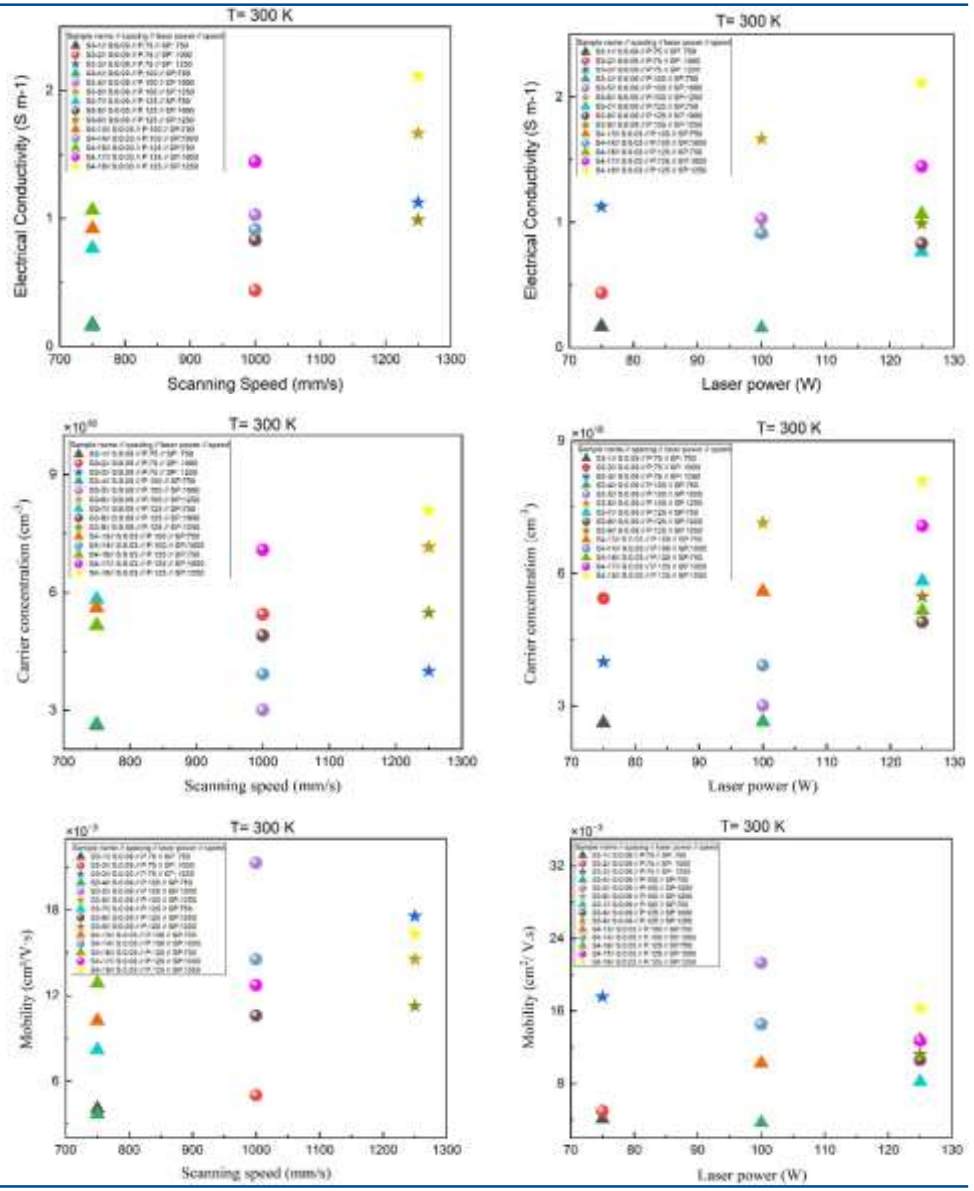
- Influence of laser energy and scan speed
  - Electrical conductivity ↑
  - Charge carrier concentration ↑
  - Low mobility (~10^{-3} cm^2/Vs)

Ongoing Work

- Post-Processing strategy
  - H_2 Autoclave treatment
  - Annealing up to 1000 °C

Characterization methods

- Electrical resistance
- Hall effect
- Seebeck coefficient
- Fermi energy



Outlook

Impurities

- Characterization of material in all phases of the process

Process optimization

- Improvement of grinding and sieving
- Gas flow in PLR
- Powder feeding and particle collection (reduction of sedimentation)

Validation of powder grades and processes

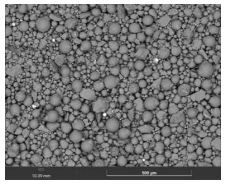
- Identification of impurities
- Characterization on impact of impurities on applications
- Identification of further applications

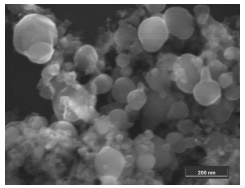
Project MAMBA (IUTA, IFW, AQM)

https://de.wikipedia.org/wiki/Gr%C3%BCne_Mamba#/media/Datei:Dendroaspis_viridisPCCA20051227-1885B.jpg

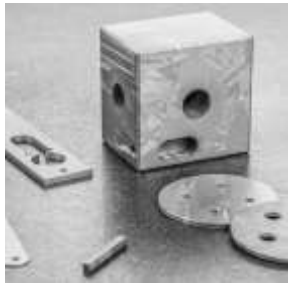
Vision

raw material



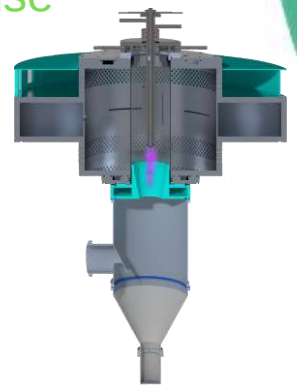


make



use

reuse



collect

waste

Thank you for your attention

Gefördert durch:

Bundesministerium für Wirtschaft und Energie

aufgrund eines Beschlusses des Deutschen Bundestages

INDUSTRIELLE GEMEINSCHAFTSFORSCHUNG

Ergebnisse aus dem Forschungsvorhaben 01IF22324N der Forschungsvereinigung VEU:

Verfahren für die kreislaufwirtschaftliche Nutzung von Silizium mittels Gasphasensynthese (WISENT) – 01.04.2022 bis 31.03.2025

und Anschlussvorhaben 01IF24505N:

Mikrowellenplasmaprozess und laserbasierte additive Fertigung zur kreislaufwirtschaftlichen Komponentenherstellung aus End-of-Use-PV-Silizium (MAMBA) – 01.11.2025 bis 31.10.2027