Summary - FUORCLAM
29 January 2020 – SFA Annual review meeting

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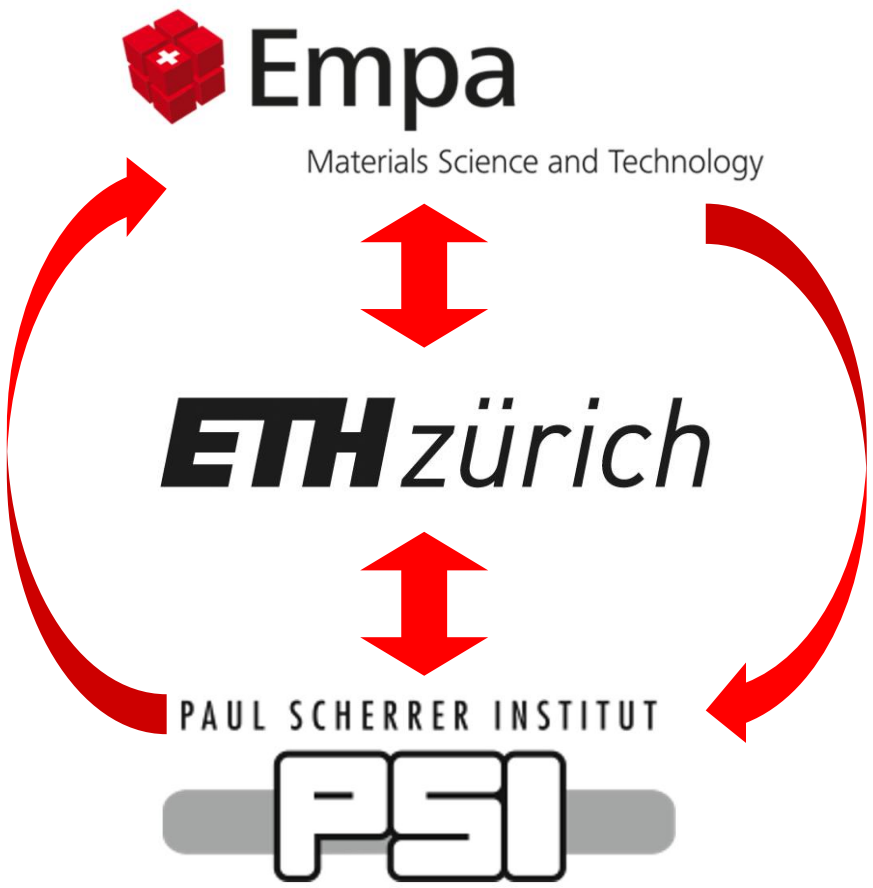
Institute of Machine Tools and Manufacturing

PAUL SCHERRER INSTITUT PSI

Empa
Materials Science and Technology

Direct Selective Laser Melting (SLM) of alumina (one step approach)
Co-development of process and material



Raw material: tailored powder

- Granule design
- Dopants and additives

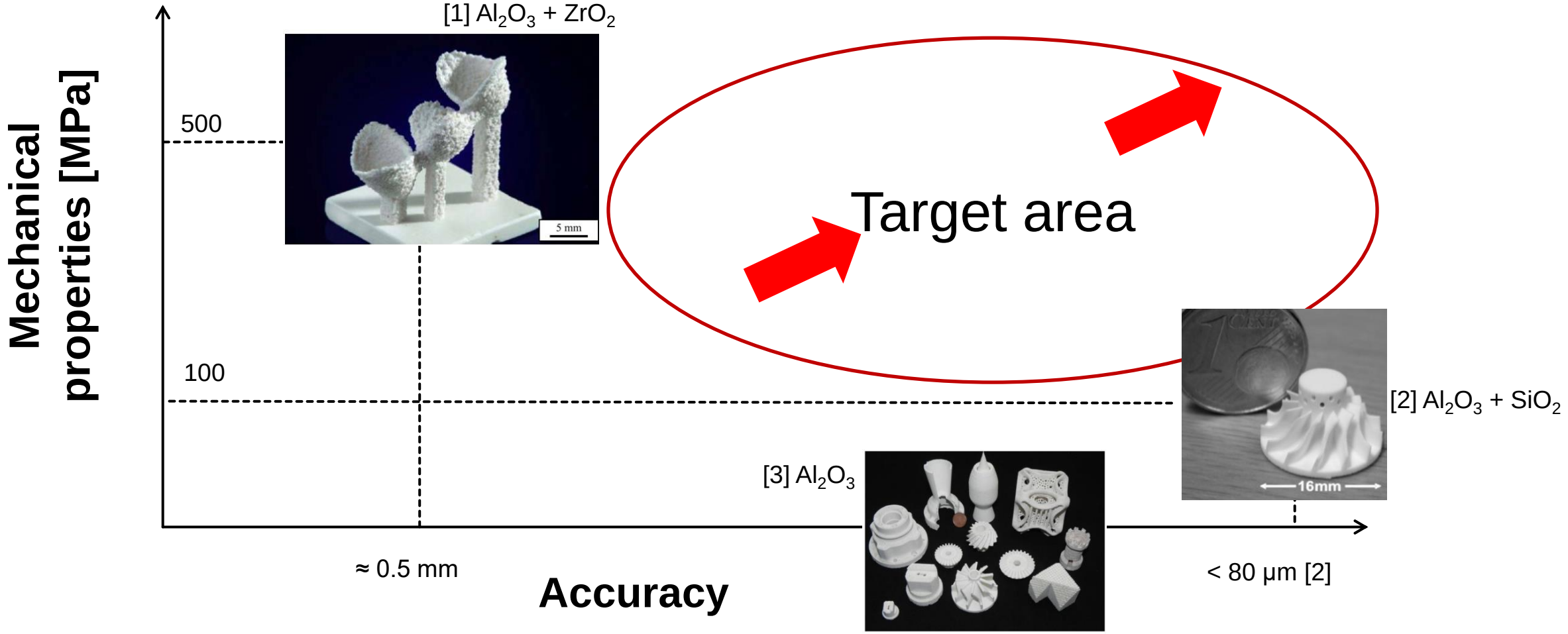
Direct consolidation through laser melting

- Absorption experiments
- Building of experimental setup
- Process windows for pulsed lasers

Material and produced parts characterization

- Raw powder analysis
- Process material analysis

State of the art – direct SLM for oxide ceramics (one-step approach)



[1] Wilkes, J., Hagedorn, Y., Meiners, W., & Wissenbach, K. (2013). Additive manufacturing of ZrO_2-Al_2O_3 ceramic components by SLM. *Rapid Prototyping Journal*, *19*(1), 51–57
[2] Regenfuss, P., Streek, A., Hartwig, … Exner, H. (2007). Principles of laser micro sintering. *Rapid Prototyping Journal*, *13*(4), 204–212.
[3] Juste, E., Petit, F., Lardot, V., & Cambier, F. (2014). Shaping of ceramic parts by SLM […]. *Journal of Materials Research*, *29*(17), 2086–2094.

FUORCLAM – Fundamental Understanding of Oxide Refractory Ceramics and Laser Additive Manufacturing

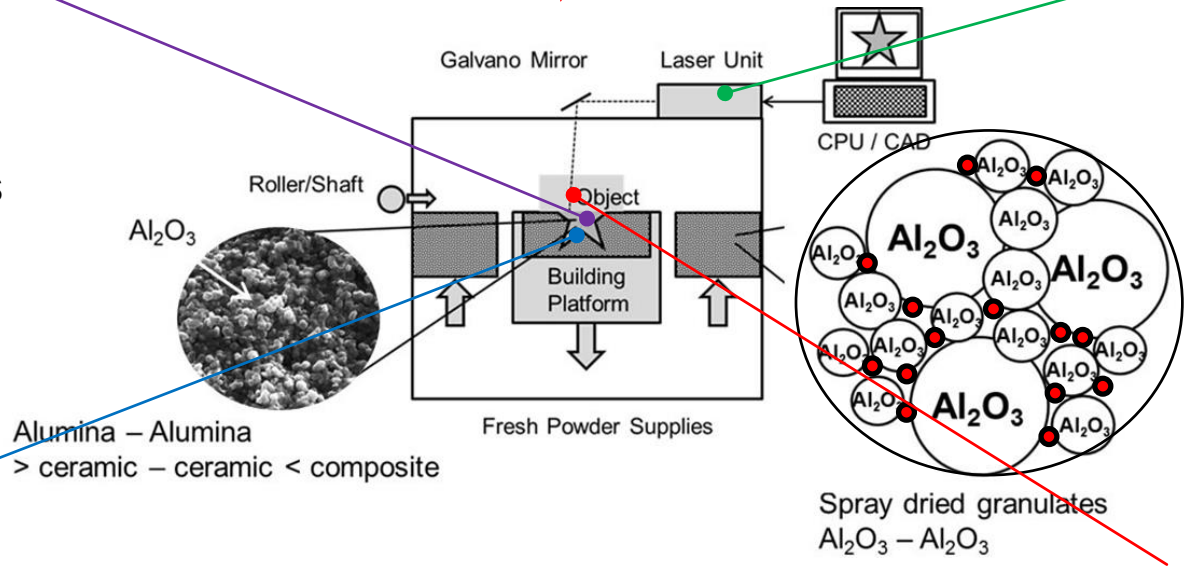
Cracks

Challenge:
Low **thermal shock resistance**

Approach:
Reducing of thermal stresses by additives

Challenge:
Low **thermal shock resistance** and **weak densification**

Approach:
Use of **different lasers**:
1. Pulsed lasers
2. Continuous wave laser



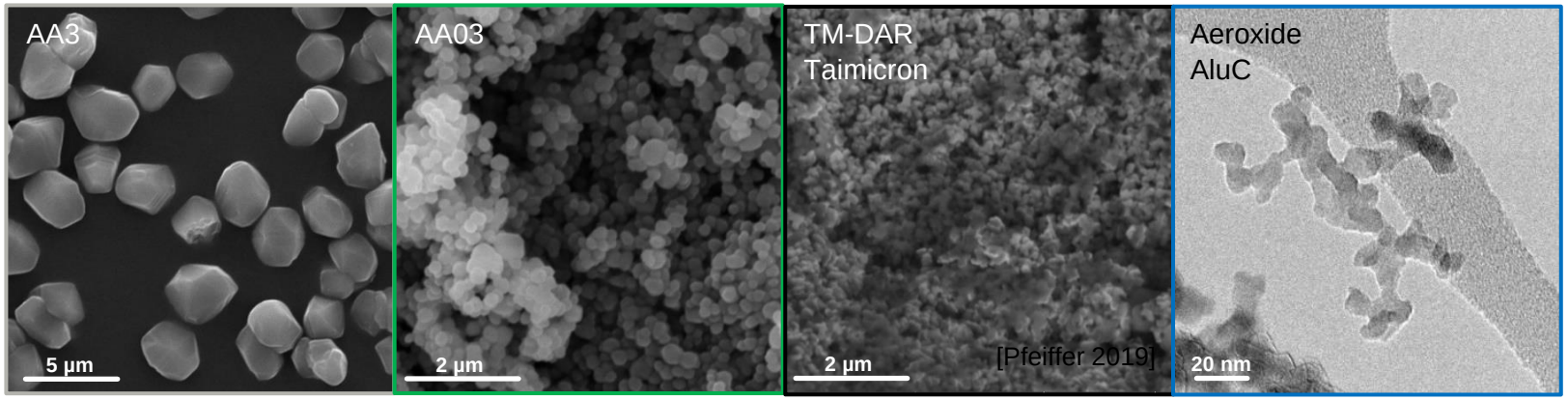
Challenge:
Weak densification

Approach:
Spray granulation of multimodal alumina combinations to increase powder flowability and packing density

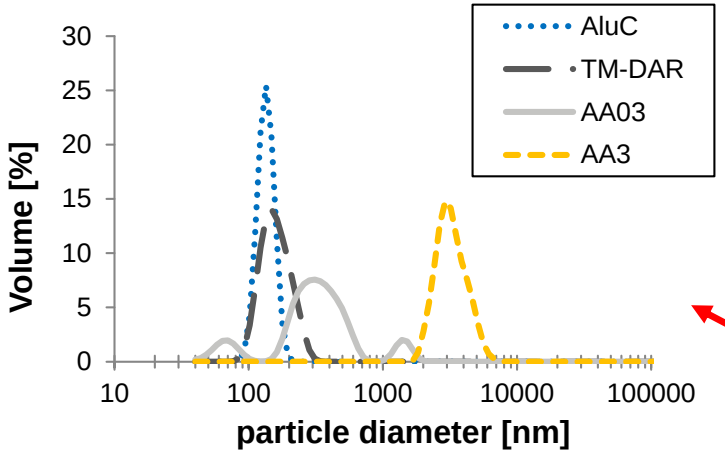
Challenge:
Low laser light absorption of alumina

Approach:
Addition of **coloured nano oxides**

Spray granulation of multimodal alumina compositions

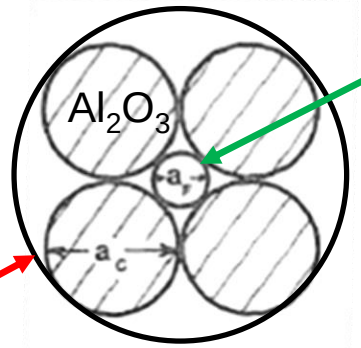


Particle size distributions of dispersed alumina



Ammonium citrate works well as dispersant

Bimodal and trimodal powder combinations



Filling of voids by smaller Al_2O_3 particles

$d_c/d_f > 7/1$

[McGeary 1961]
[Reed 1988]

Homogeneous dispersion required

Why?

Higher packing densities of powder bed by bimodal or trimodal combinations within spray dried granules

Different dopants to improve laser light absorption

Proper dispersing necessary to achieve homogeneous distribution within the alumina granules

Nano-Fe_2O_3 (L2715D/BASF)

Reason for choosing:

- Match of absorption spectrum with green laser light

Nano-"MnO_2" (US3319/US Research Nanomaterials, Inc.)

Reason for choosing:

- Match of absorption spectrum with red laser light

Nano-TiO_2 (P25/Evonik)

Reason for choosing:

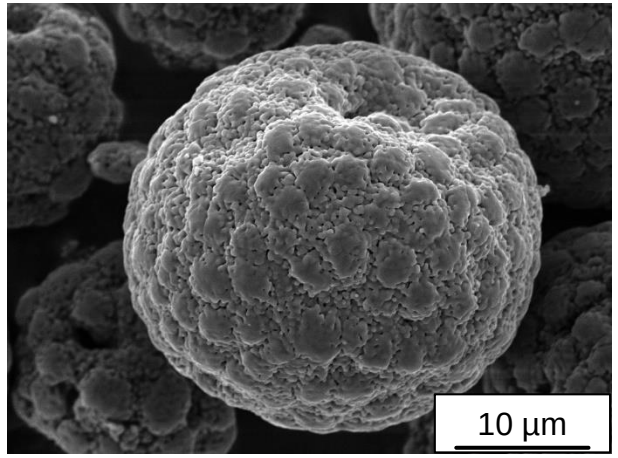
- Known to improve the fracture toughness of alumina
- Good absorption with red laser light when reduced

→ No evidence of agglomerates in the micron range

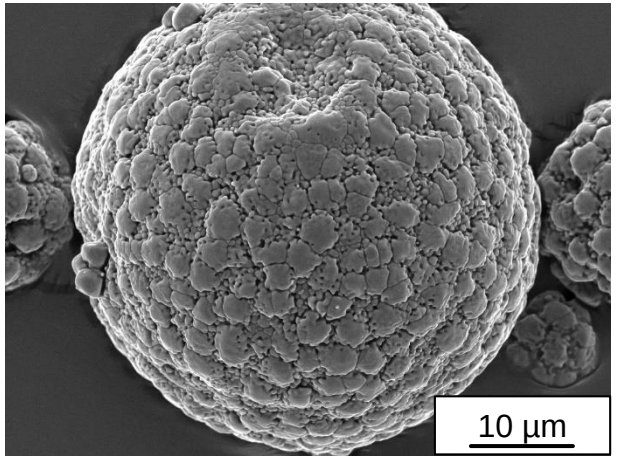
Thermal treatment of bimodal granules

Increase of powder density

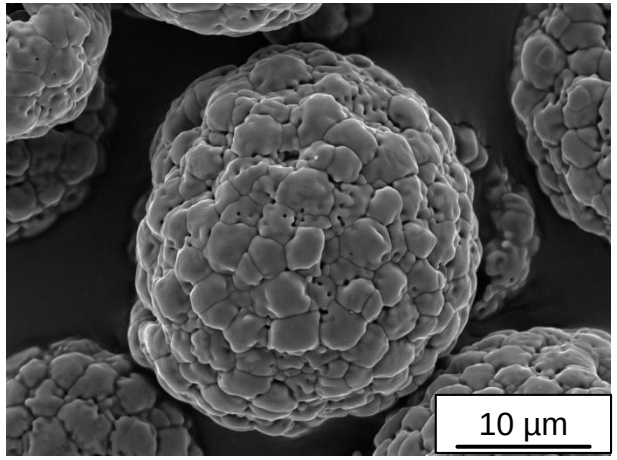
1400 °C/ 2h



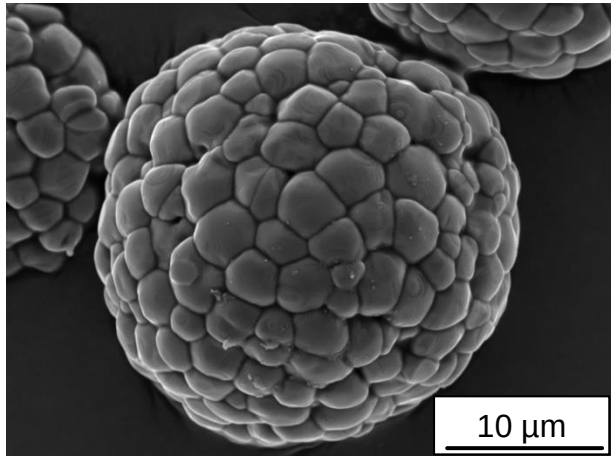
1400 °C/ 4h



1500 °C/ 2h

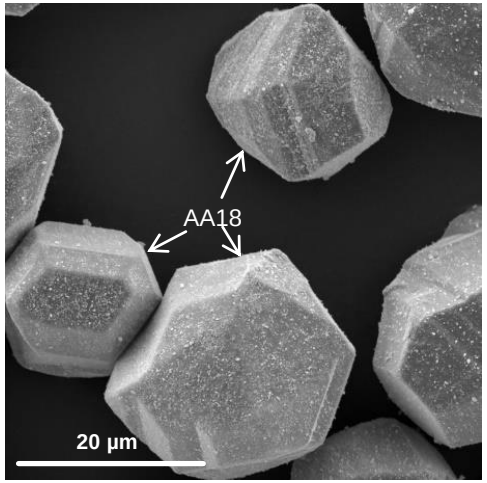


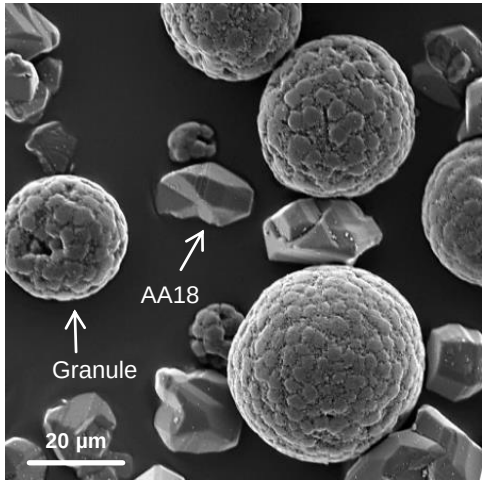
1600 °C/ 2h



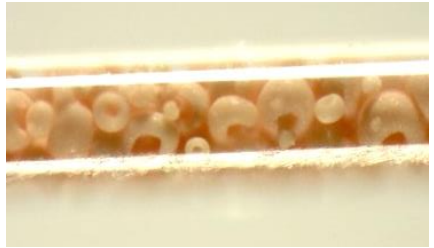
- Breaking of sintering necks in between granules by addition of coarse alumina necessary
- Tapped powder bed densities of up to 56.4% of theoretical density reached

→ **Laser processed part density constant after reaching tapped powder bed densities of 48% of theoretical density**





Chemical 3D imaging at micro-XAS (PSI) - effect of calcination of iron oxide - doped alumina granules on the dopant distribution



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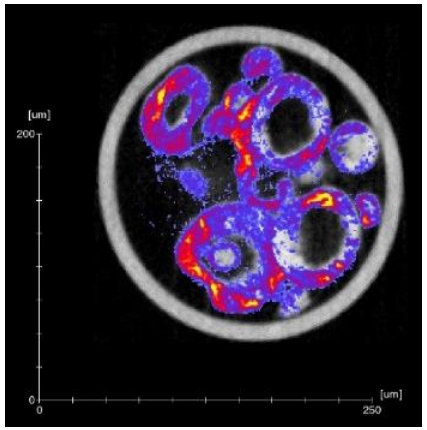
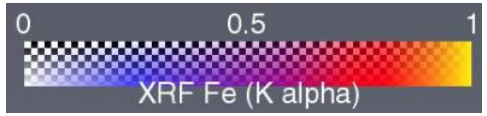
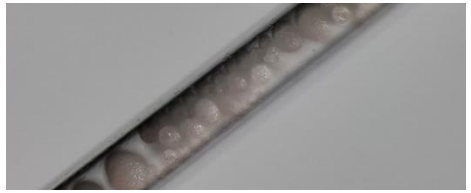
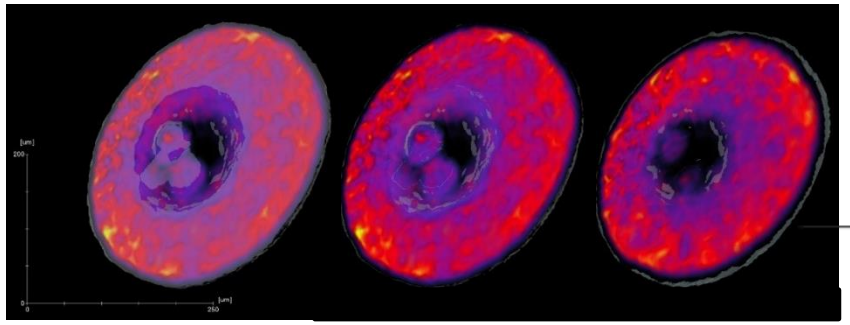


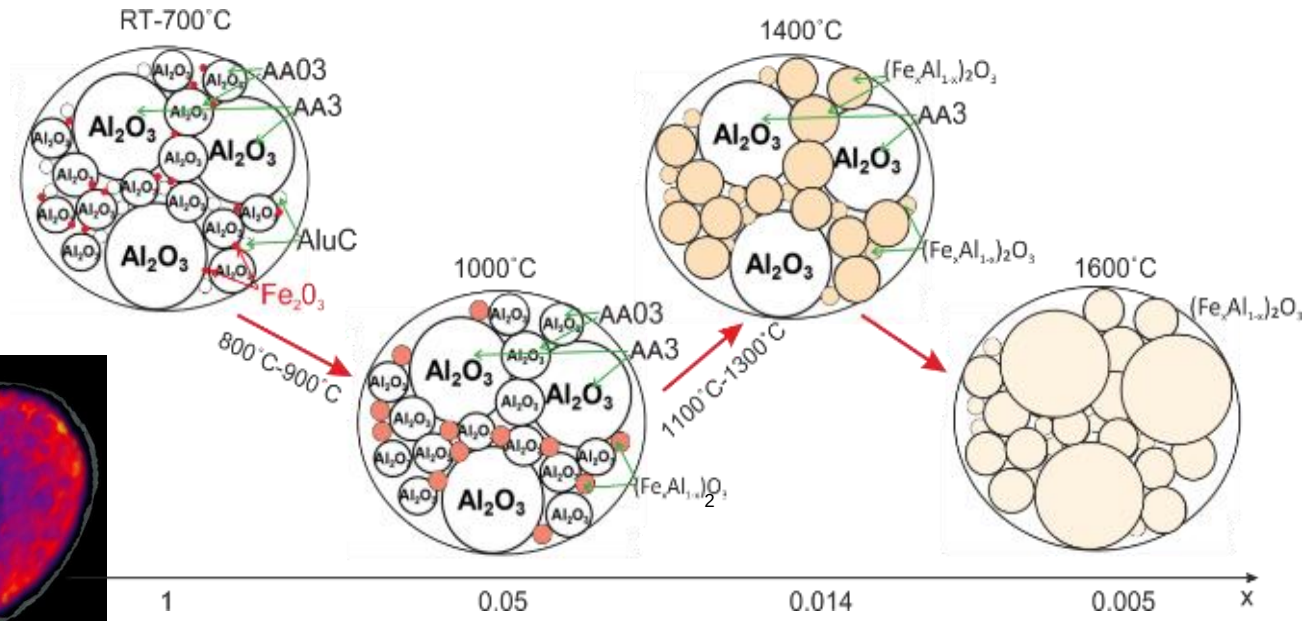


1600 °C





- Good distribution of dopants before and after calcination of the powder
- Dopant is gradually incorporated in alumina lattice, depending on the calcination temperature

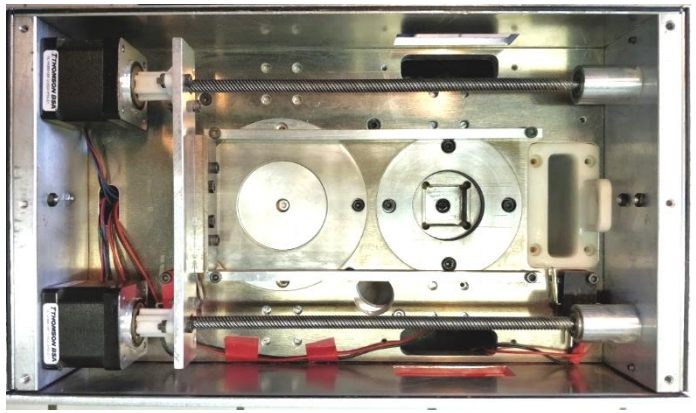


x = amount of iron in doped corundum phase

Ref: Makowska et al. 2019

Short and ultrashort pulsed laser were tested

- Pulse laser because:
  - Higher absorption of powder with these lasers
  - Higher recoil pressure (theory to be proven)



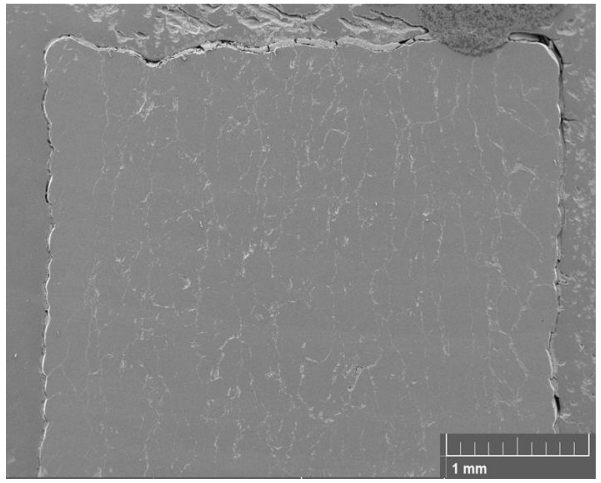
SLM box

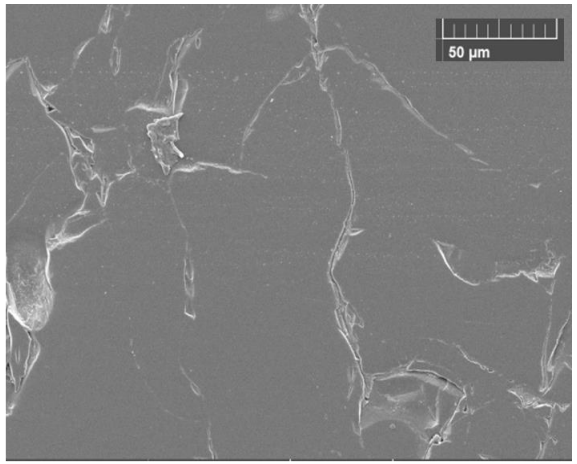
	IPG GLPM-5	TBWP Duetto	SPI 200
Maximum Power (W)	6	10	100
Wavelength (nm)	532 (green)	1064 (IR)	1064 (IR)
Pulse duration	1.5 ns	10 ps	Cw
Rep. Rate kHz	300	8400	-
Scanning speed	2-10 mm/s	2-20 mm/s	10-120 mm/s
Results	**Mechanical test performed**	**Only small cubes built**	**Only small cubes built**
Problems	**Too slow**	**Too much spatter during process**	**Not enough absorption**

Properties of produced parts with green ns laser

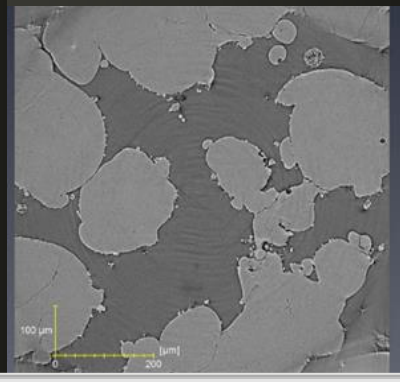
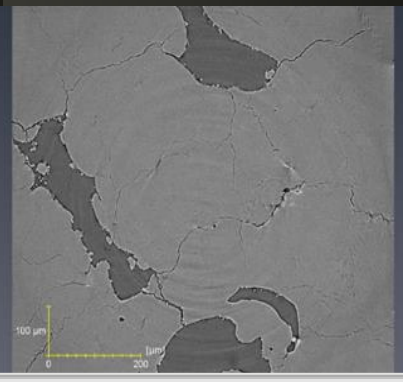
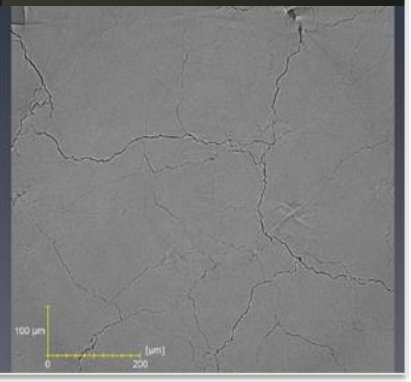
- Density: up to 98% (synchrotron tomography)
- Bending strength: up to 50 MPa (B3B test)
- Compression strength: 250 MPa (uniaxial test)

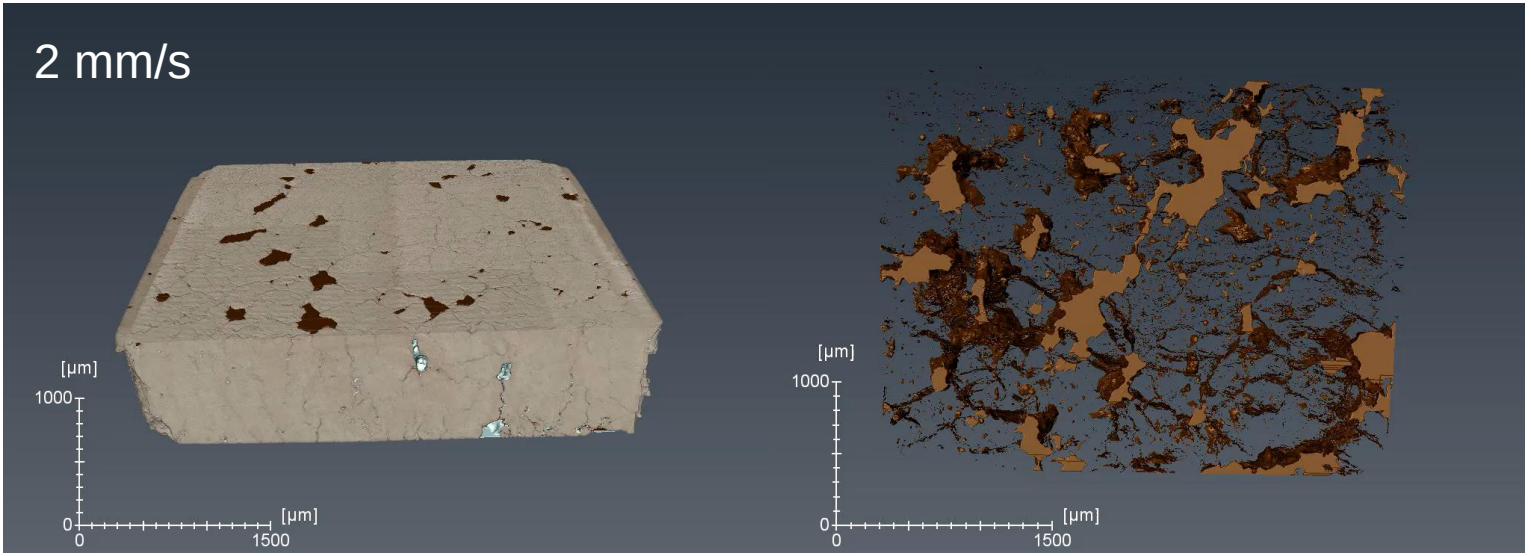
- Limiting factor for strength: cracks and porosity



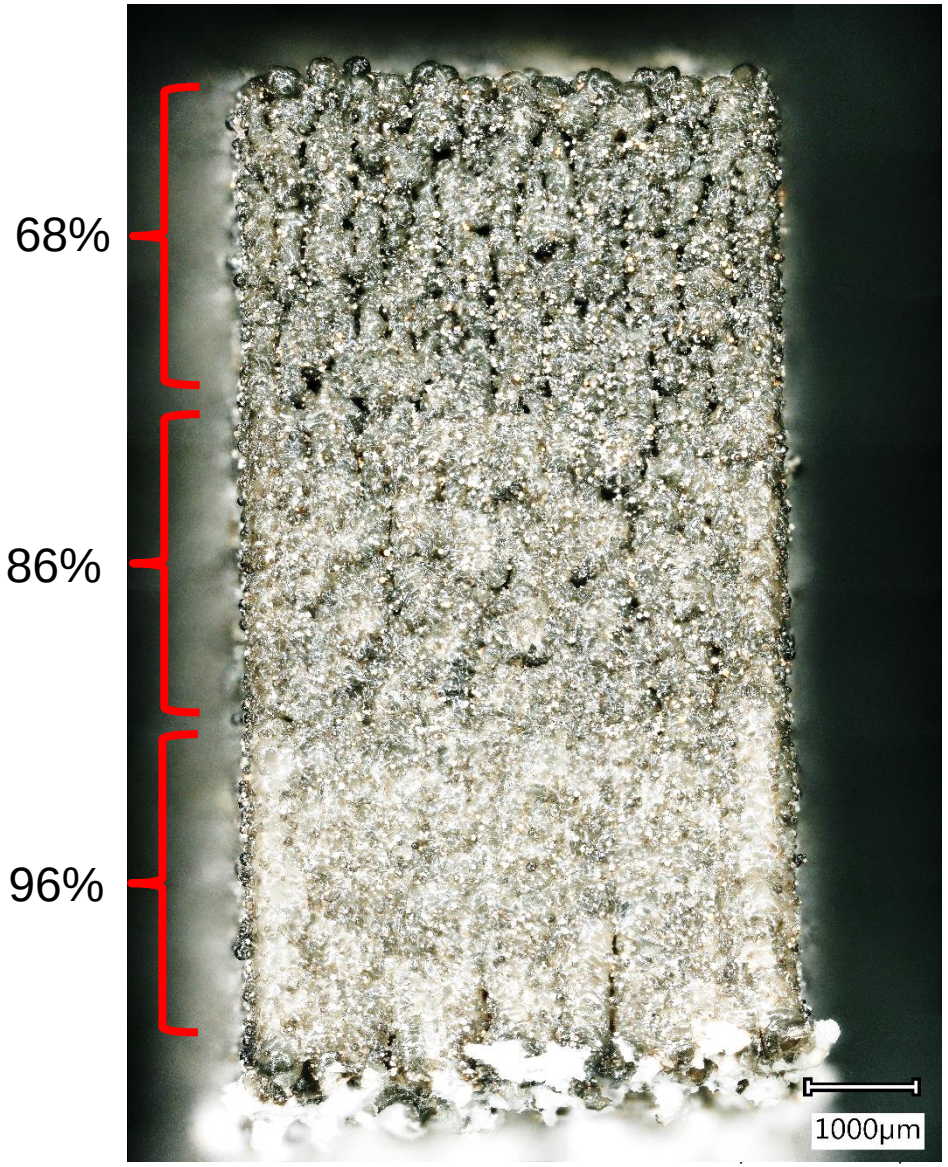


Porosity controlled by laser parameters

Laser scan speed	10 mm/s	5 mm/s	2 mm/s
Density	68 %	86 %	96 %
Micrograph			

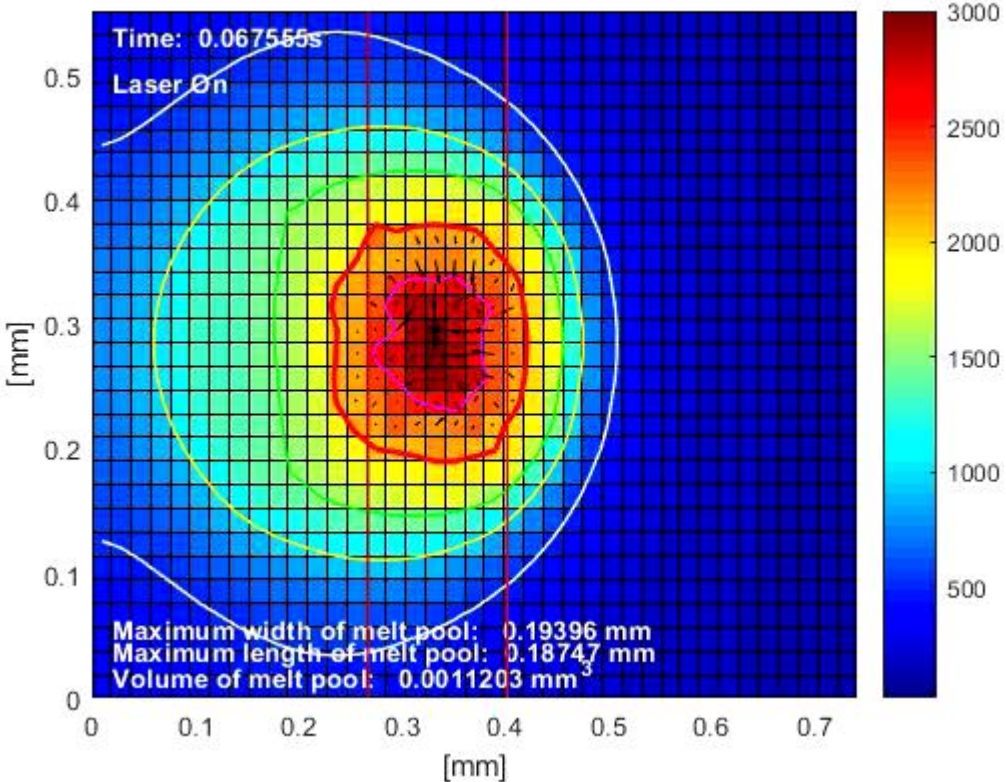


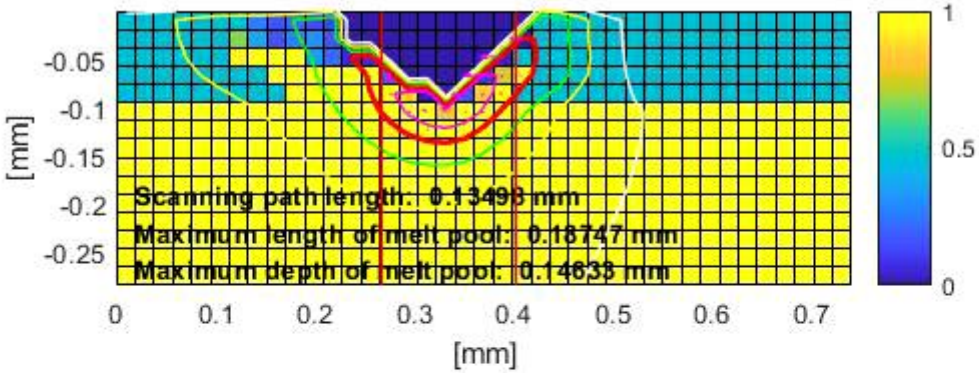
Example of gradient structure

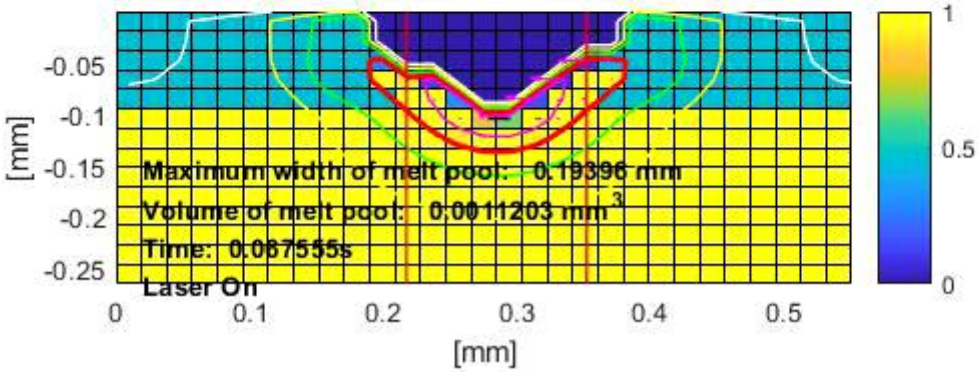


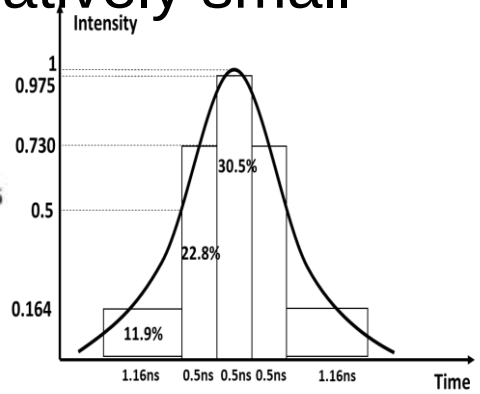
Improving understanding – Recoil pressure calculation with FEM simulation

- FEM model internally developed at IWF
- Changes of melt-pool size **irrelevant between laser pulses**
- **Recoil pressure**: 0.15-0.2bar → heat input in a single pulse is relatively small



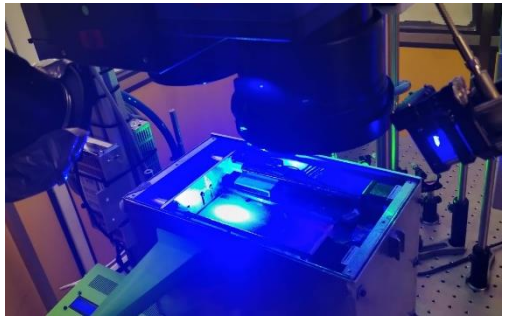




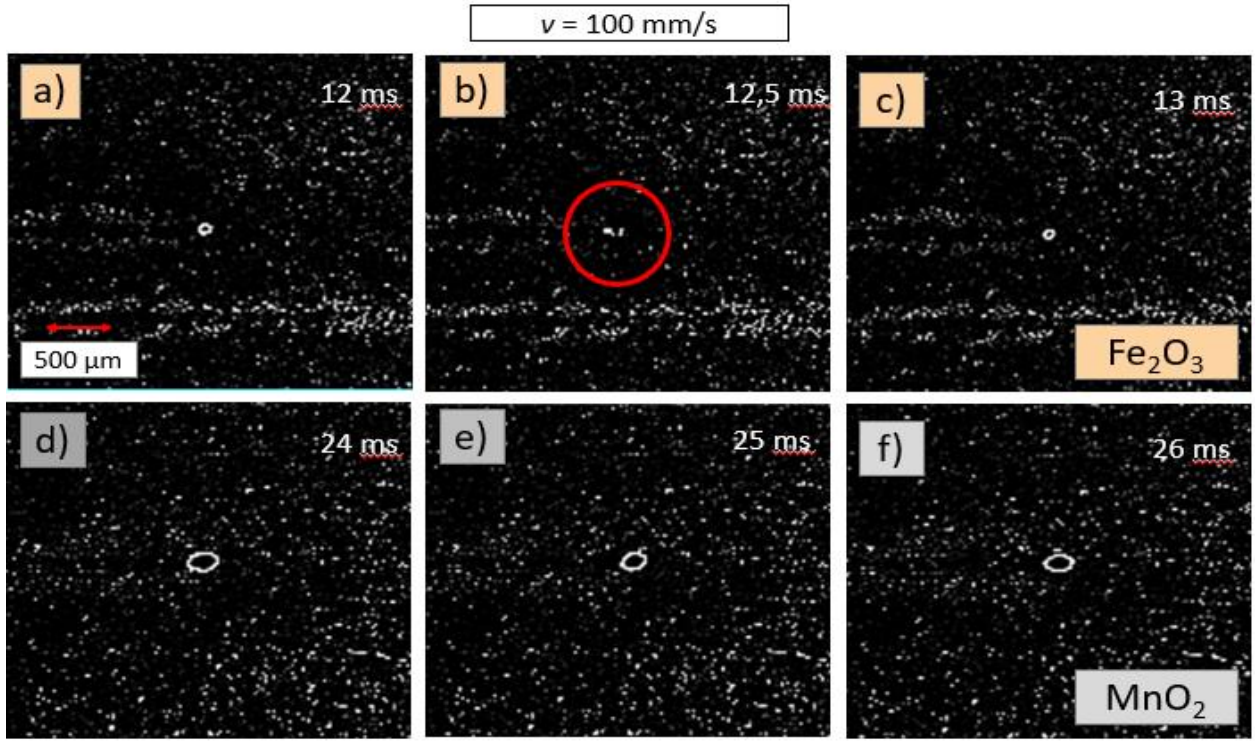


Comparison of different powders with in-situ measurements

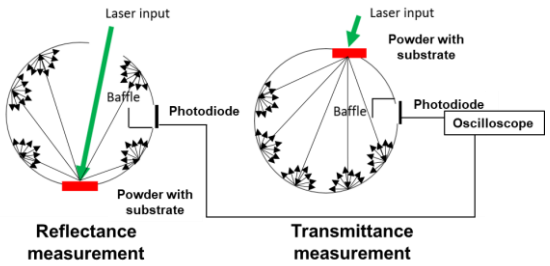
- **Manganese** oxide and **iron** oxide dopants for alumina are compared
- **High speed camera** and in-situ **reflection measurements** shows that there are some **instabilities** with iron oxide powder

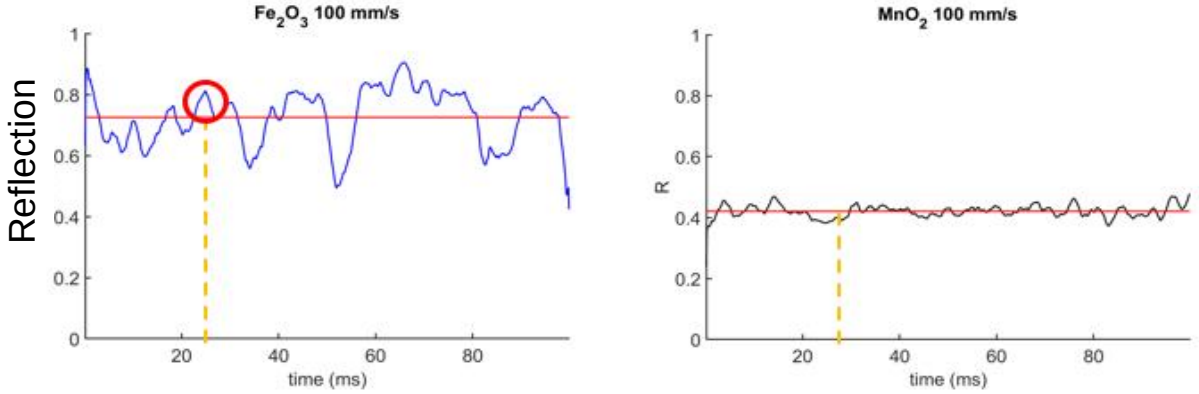


High speed camera with external blue illumination

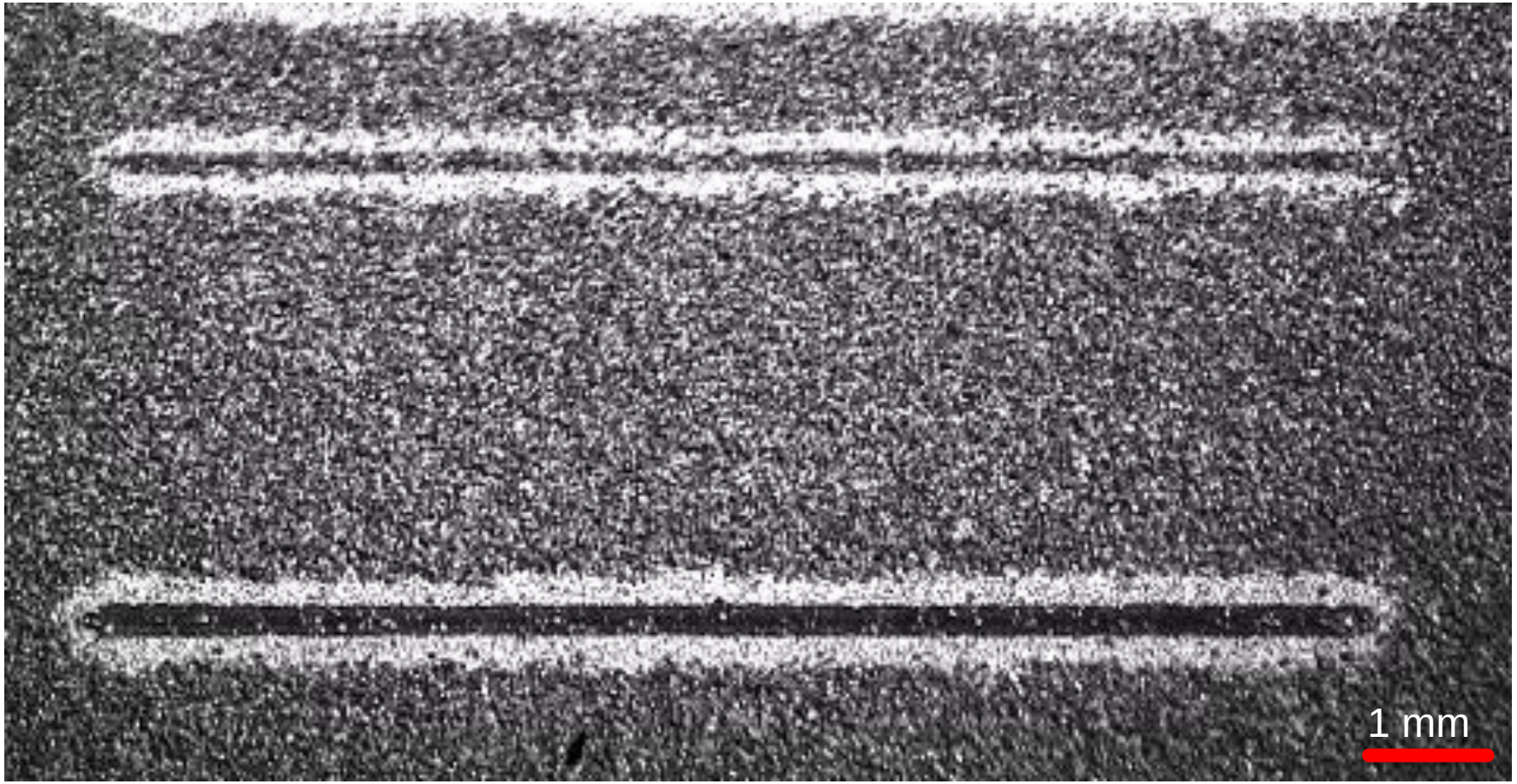


In-situ absorption with integrating sphere



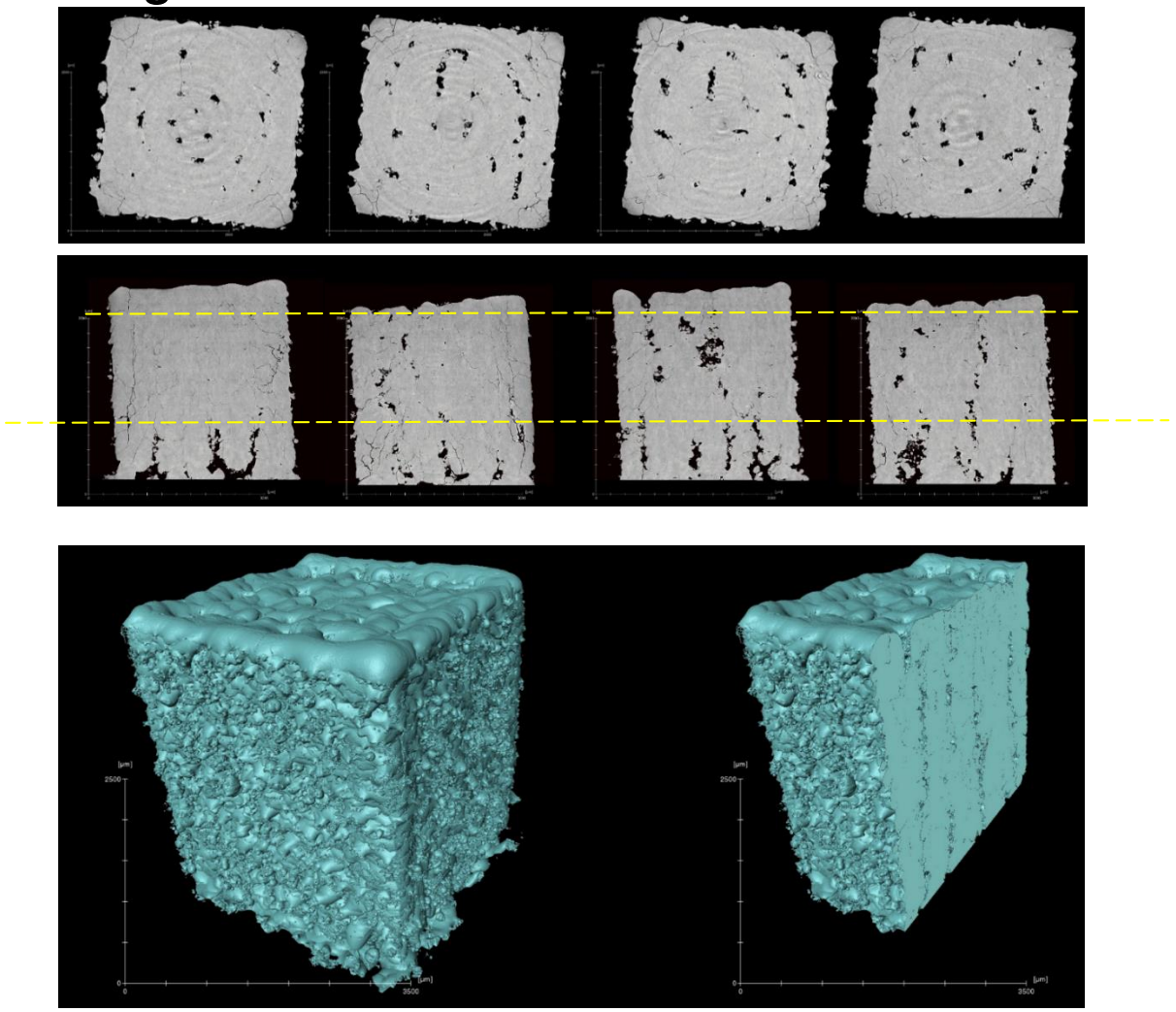


Understanding the process
Denudation of the powder bed during laser melting



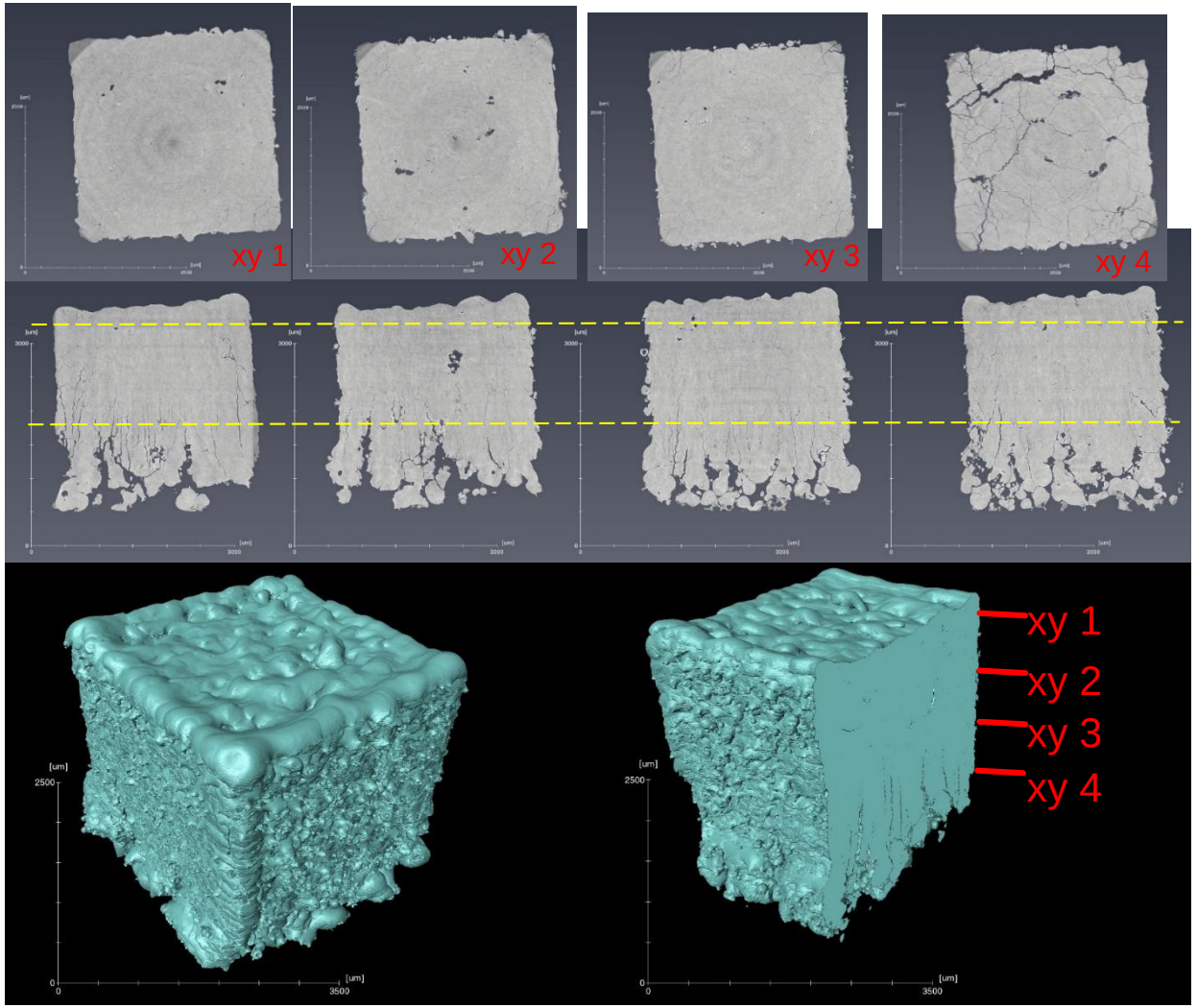
Cw IR 30 W
40 mm/s
100 um powder layer
175 um spot size

Parts produced with Mn doped alumina – granules before calcination



Density: 94.63%
inner part: 96.19%

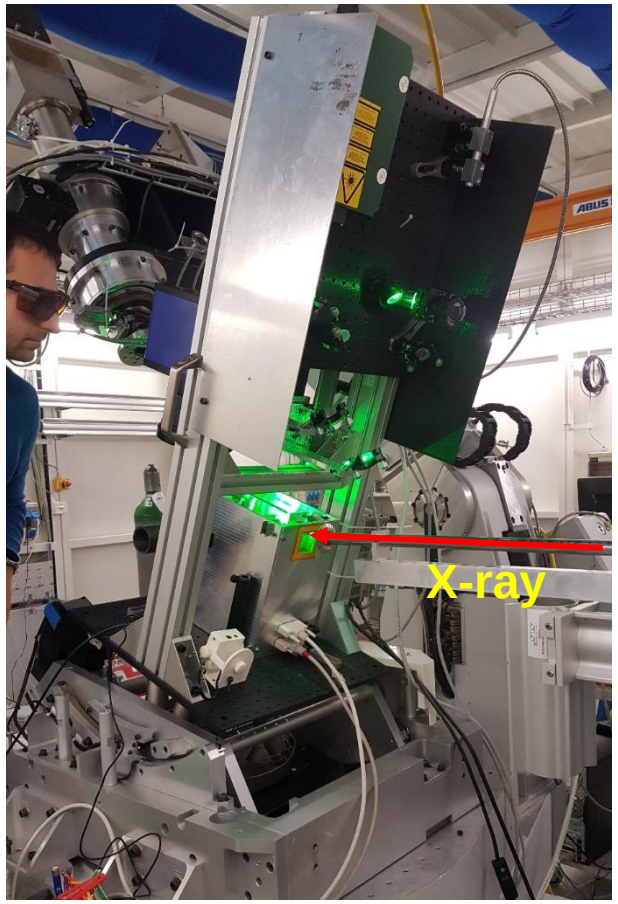
Parts produced with Mn doped alumina – granules after calcination

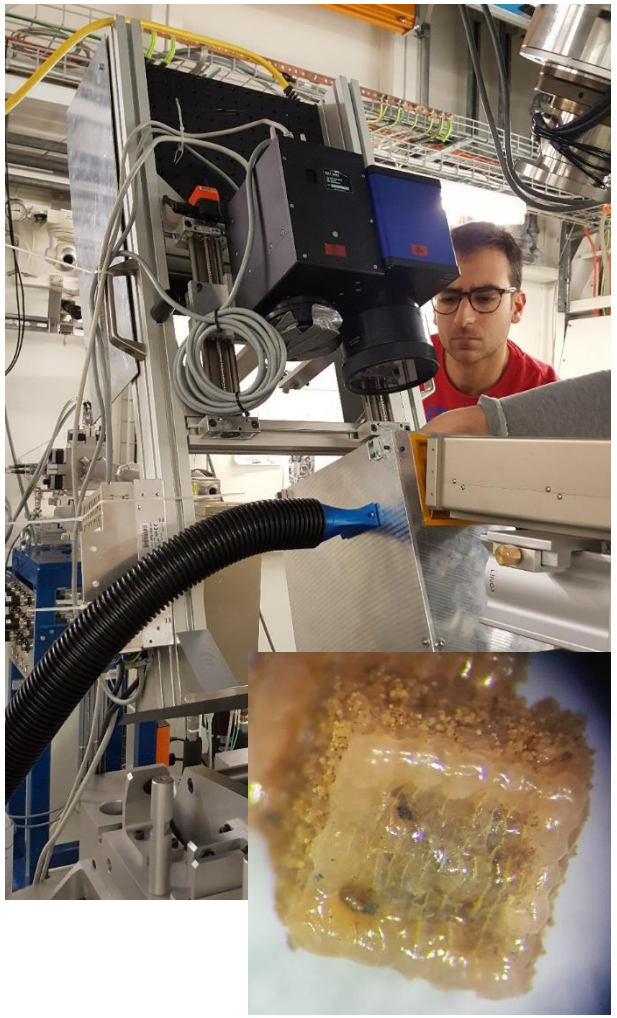


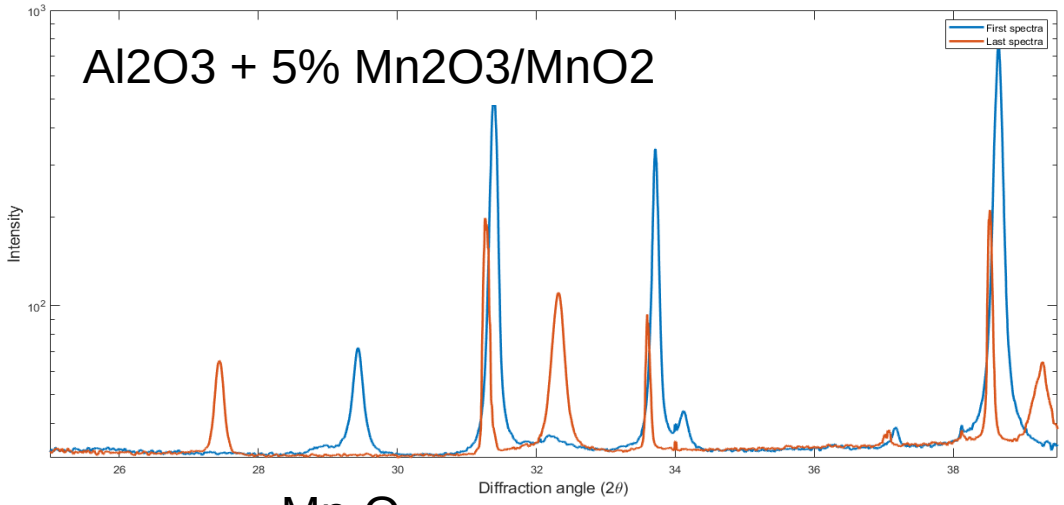
3 mm cube

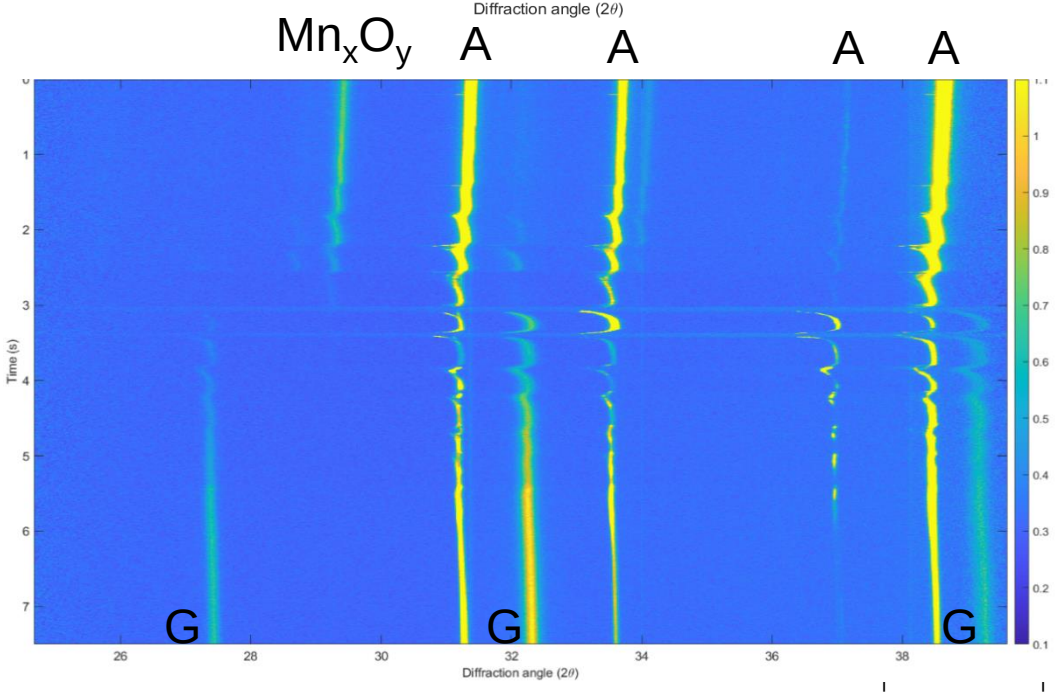
Density: 96.18%
Inner part: 98.64%

In-situ powder diffraction studies of SLM process at the Swiss Light Source (PSI)









Milestone

i. Decision for most promising, doped alumina made (12 months)
Iron oxide for green laser. Manganese oxide for IR laser

ii. Powder bed packing density above 65% (36 months)
56.4% packing density achieved. No indication that higher packing densities are useful

iii. Cube with graded density in normal direction (24 months)
A cube with variable density between 67 and 95 % is shown and available at the project desk

iv. Dense Cube with 5 mm edge length (36 months)
Examples of such cubes is at the project desk. Density is up to 98%

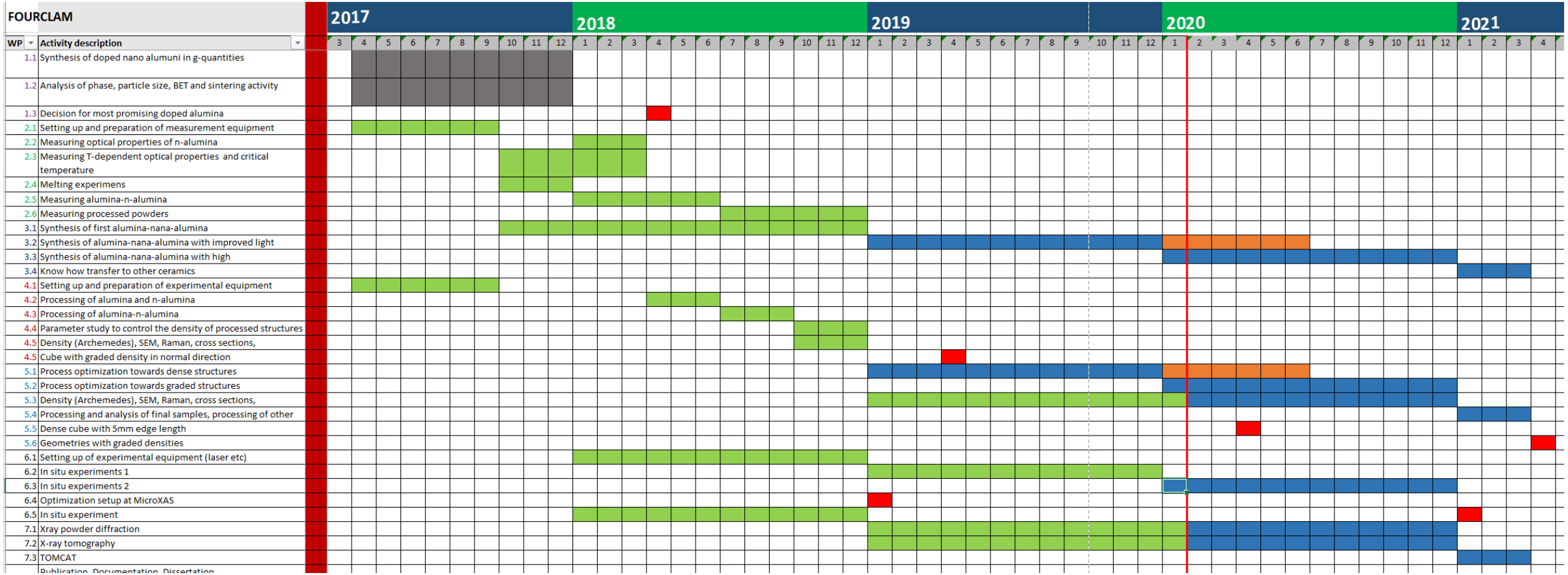
Next steps

- Test more powerful green laser (up to 20 W available since January 2020), to increase production speed and densities
- Test in-situ tomography and powder diffraction at Swiss Light Source PSI
- Test of new materials with higher resistance to crack formation

Thank you for your kind attention
Questions welcome

Timeline

cancelled
concluded on time
extended deadline



Lower roughness achieved with cw laser