Summary - FUORCLAM
29 January 2020 – SFA Annual review meeting

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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)

Target area

Mechanical properties [MPa]

[1] $\text{Al}_2\text{O}_3 + \text{ZrO}_2$

[2] $\text{Al}_2\text{O}_3 + \text{SiO}_2$

[3] $\text{Al}_2\text{O}_3$

Approximately 0.5 mm

Accuracy

< 80 μm [2]

Target area


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

**Challenge:** Low thermal shock resistance

**Approach:** Reducing of thermal stresses by additives

**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

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

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

**Cracks**
Spray granulation of multimodal alumina compositions

Particle size distributions of dispersed alumina

Bimodal and trimodal powder combinations

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

Ammonium citrate works well as dispersant

Homogeneous dispersion required

Filling of voids by smaller Al₂O₃ particles

McGeary 1961
[Reed 1988]
Different dopants to improve laser light absorption

Proper dispersing necessary to achieve homogeneous distribution within the alumina granules

**Nano-Fe$_2$O$_3$ (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

- 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**

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

Ref: Makowska et al. 2019

$x = \text{amount of iron in doped corundum phase}$
Short and ultrashort pulsed laser were tested

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

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

Problems:
- Too slow
- Too much spatter during process
- Not enough absorption

SLM box
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

<table>
<thead>
<tr>
<th>Laser scan speed</th>
<th>10 mm/s</th>
<th>5 mm/s</th>
<th>2 mm/s</th>
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<tbody>
<tr>
<td>Density</td>
<td>68 %</td>
<td>86 %</td>
<td>96 %</td>
</tr>
</tbody>
</table>

**Micrograph**

- 2 mm/s

**Example of gradient structure**

- 68%
- 86%
- 96%
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.

In-situ absorption with integrating sphere

High speed camera with external blue illumination

\( v = 100 \text{ mm/s} \)
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

Density: 96.18%
inner part: 98.64%

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

Al₂O₃ + 5% Mn₂O₃/MnO₂

X-ray
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