INSTITUTO DE PESQUISAS TECNOLÓGICAS

## COMUNICAÇÃO TÉCNICA

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# Comparing spherical and irregular powders in the selective laser melting of Ti-53%Nb alloy

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# COMPARING SHPERICAL AND IRREGULAR POWDERS IN THE SELECTIVE LASER MELTING OF Ti-53%Nb ALLOY

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2 Institute for Technological Research (IPT)

3 National Service for Industrial Training (SENAI)

# Objetive of the project

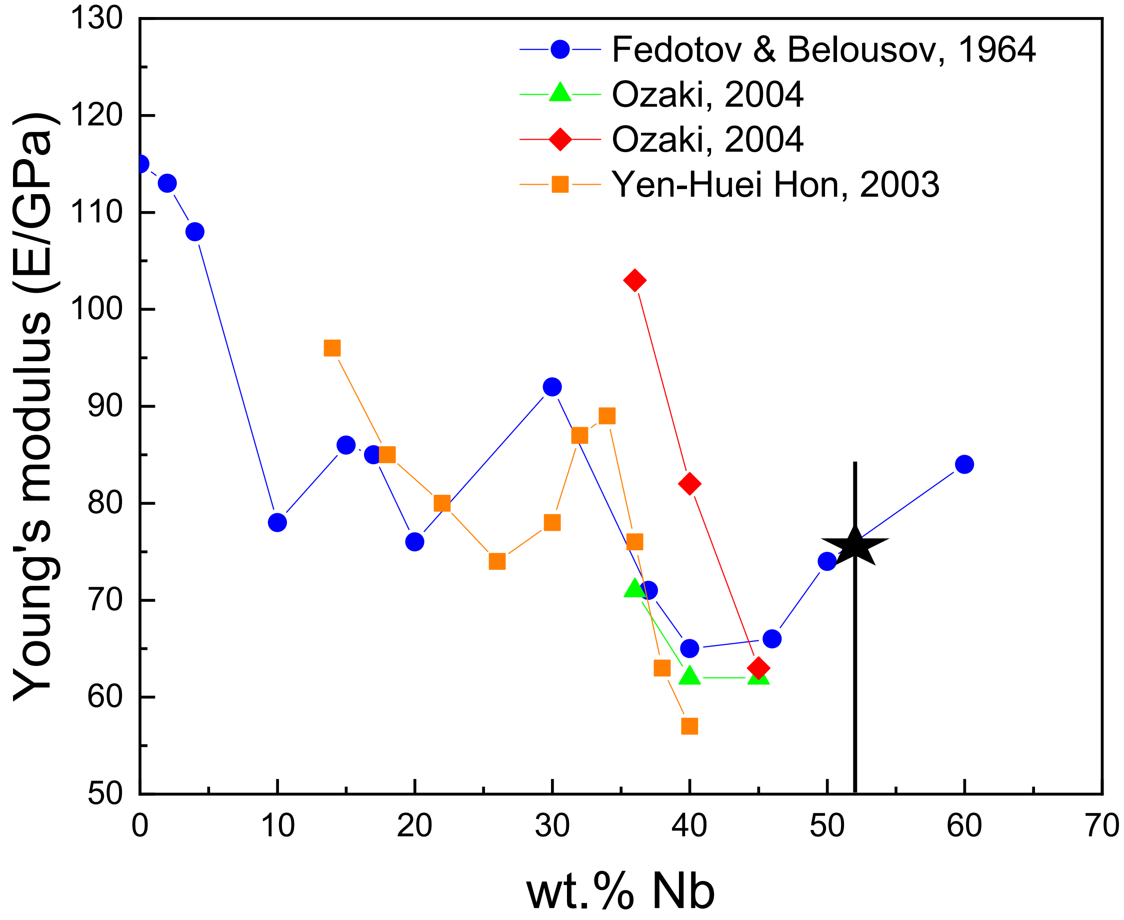
- To evaluate the possibility of producing orthopaedic Implants of Ti-53%Nb alloy by Selective Laser Melting
  - The Niobium Company (CBMM) wants to promote the use of Niobium in new applications
  - Niobium, like titanium, promotes osteointegration
  - Orthopedic implants work better when their elastic modulus is similar to that of the bones
  - Niobium addition to titanium lowers the elastic modulus

# Young's modulus Ti–Nb

Niobium decreases the elastic module of titanium

There is some scatter about the effect of niobium on the elastic constant

The Ti-53%NB was chosen because of alloy commercial availability: it is a superconductor composition.

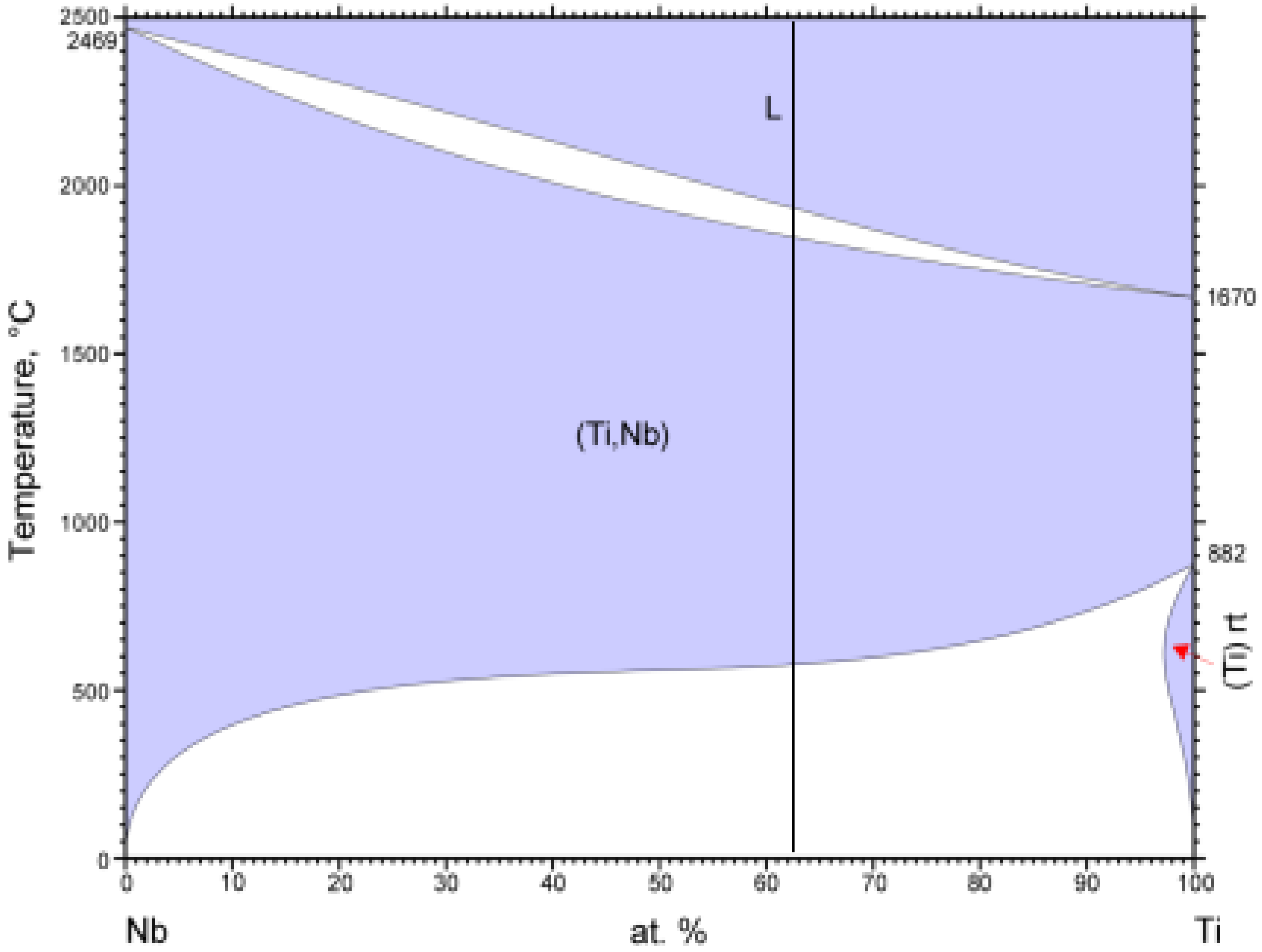



(7) C. Schulze, M. Weinmann, C. Schweigel, O. Keßler, R. Bader, Mechanical Properties of a Newly Additive Manufactured Implant Material Based on Ti-42Nb, Materials (Basel). 11 (2018) 124. doi:10.3390/ma11010124.

# Niobium – Titanium system

| Phases of the system Ti – Nb | | |
|---|---|---|
| Phase | Crystal structure | Composition at%. Nb |
| α | HCP | 0-2.5 |
| β | BCC | 0-100 |

Although the phase diagram predicts a α+ β structure, beta is strongly metastable.

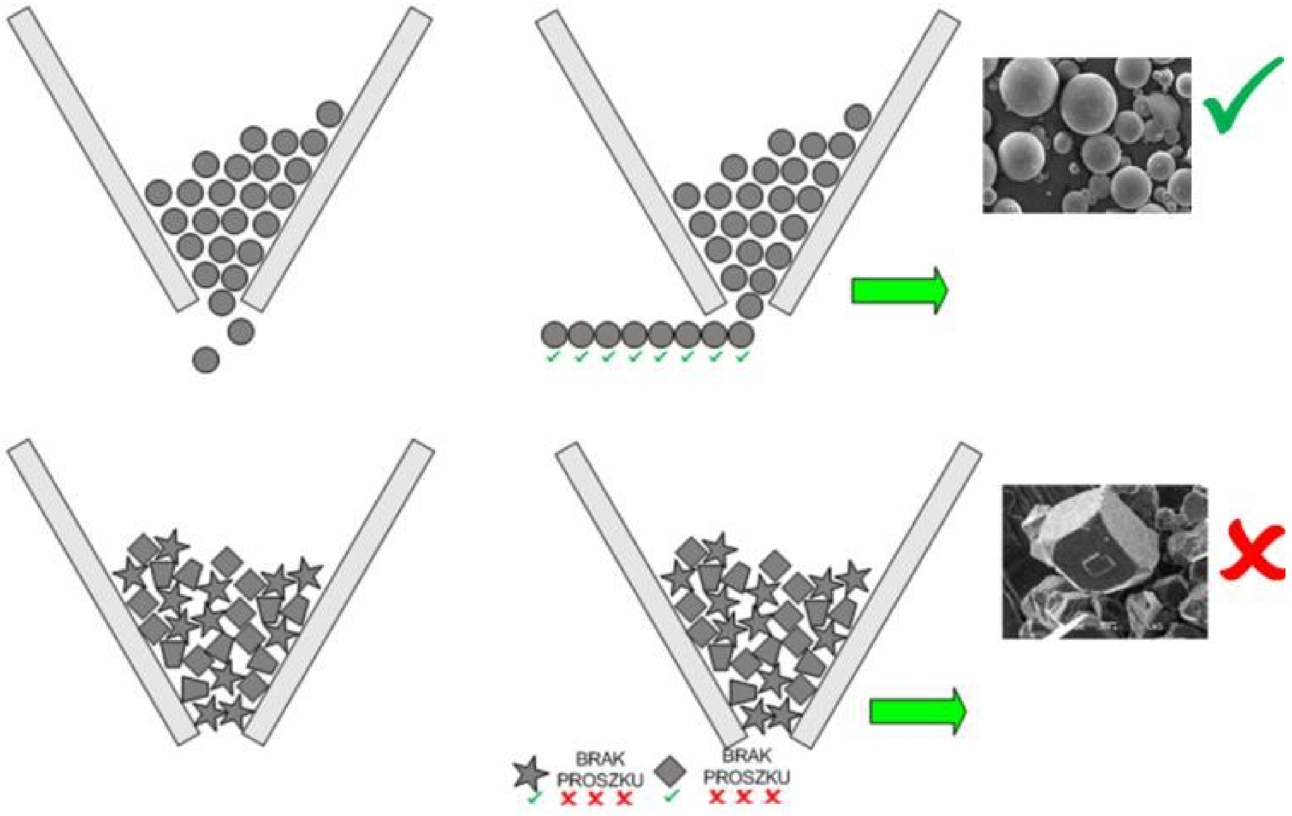



(7) M. Hansen, E.L. Kamen, H.D. Kessler, D.J. McPherson, Systems Titanium-Molybdenum and Titanium-Columbium, JOM. 3 (1951) 881–888. doi:10.1007/BF03397396.
(8) J.L. Murray, The Nb−Ti (Niobium-Titanium) system, Bull. Alloy Phase Diagrams. 2 (1981) 55–61. doi:10.1007/BF02873704.
(9) C.M. Lee, C.P. Ju, J.H. Chern Lin, Structure-property relationship of cast Ti-Nb alloys., J. Oral Rehabil. 29 (2002) 314–22. http://www.ncbi.nlm.nih.gov/pubmed/11966963.

# Powders

## Cost and flowability

- Spherical shaped powder is the common solution for Ti (U$ 333/kg)
- Ti irregular shaped powder may be a better solution (U$ 50/kg ?)
- Apparent density and flowability may be impaired.




Layer deposition: a) spherical powder, b) irregular powder (6).

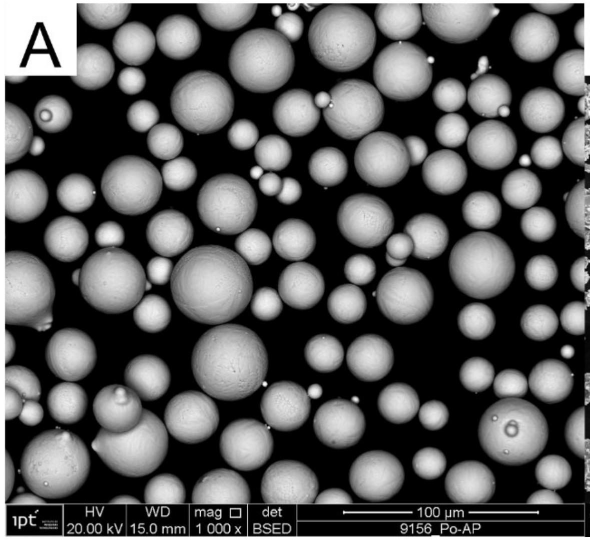
(5) W. Xu, S. Xiao, X. Lu, G. Chen, C. Liu, X. Qu, Fabrication of commercial pure Ti by selective laser melting using hydride-dehydride titanium powders treated by ball milling, J. Mater. Sci. Technol. 35 (2019) 322–327. doi:10.1016/j.jmst.2018.09.058.
(6) T. Kurzynowski, E. Chlebus, B. Kuźnicka, J. Reiner, Parameters in selective laser melting for processing metallic powders, in: E. Beyer, T. Morris (Eds.), 2012: p. 823914. doi:10.1117/12.907292.

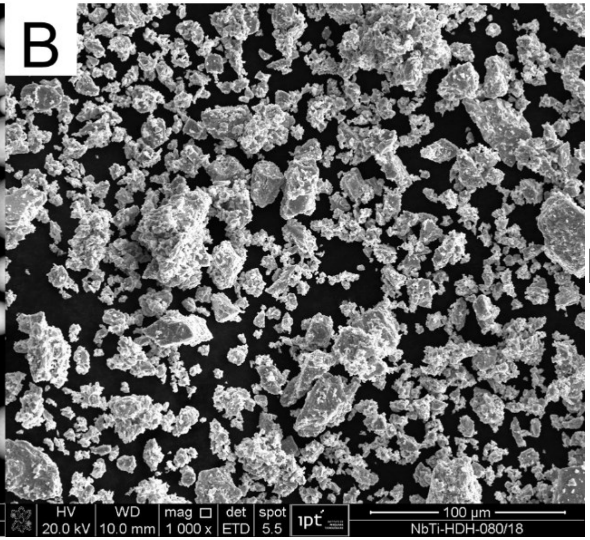
# Objective of this paper

- To evaluate the viability of obtaining high density parts using HDH powder

# Materials

AP spherical shaped powder




HDH irregular shaped powder

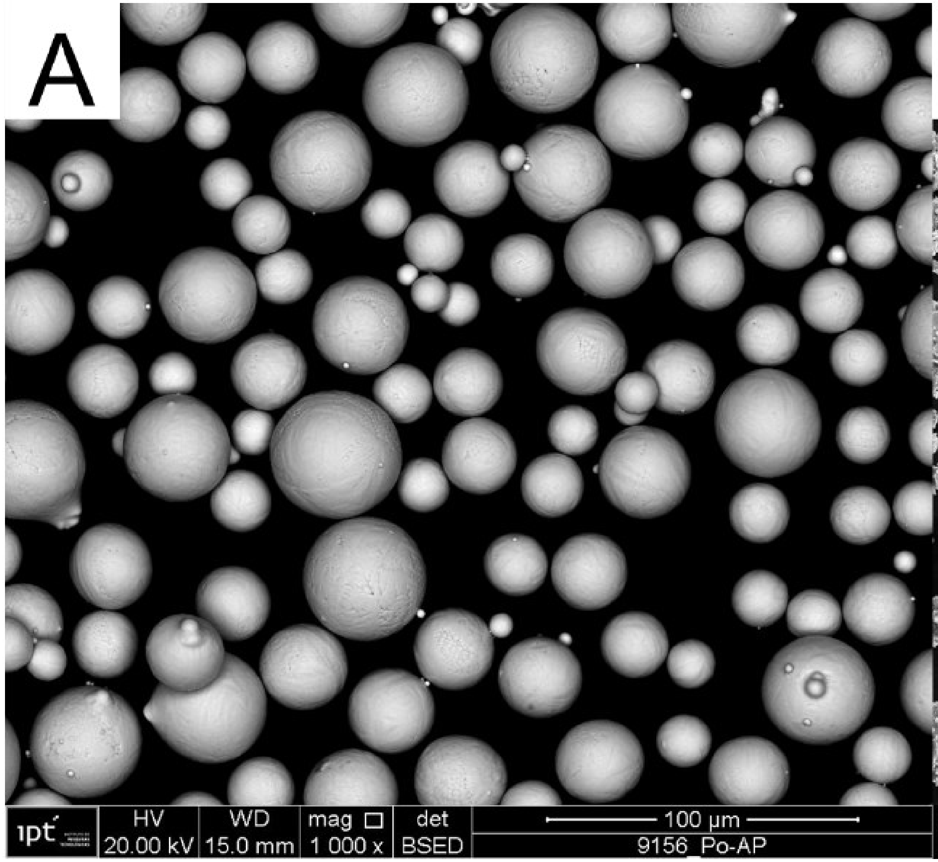
Powder characteristics.

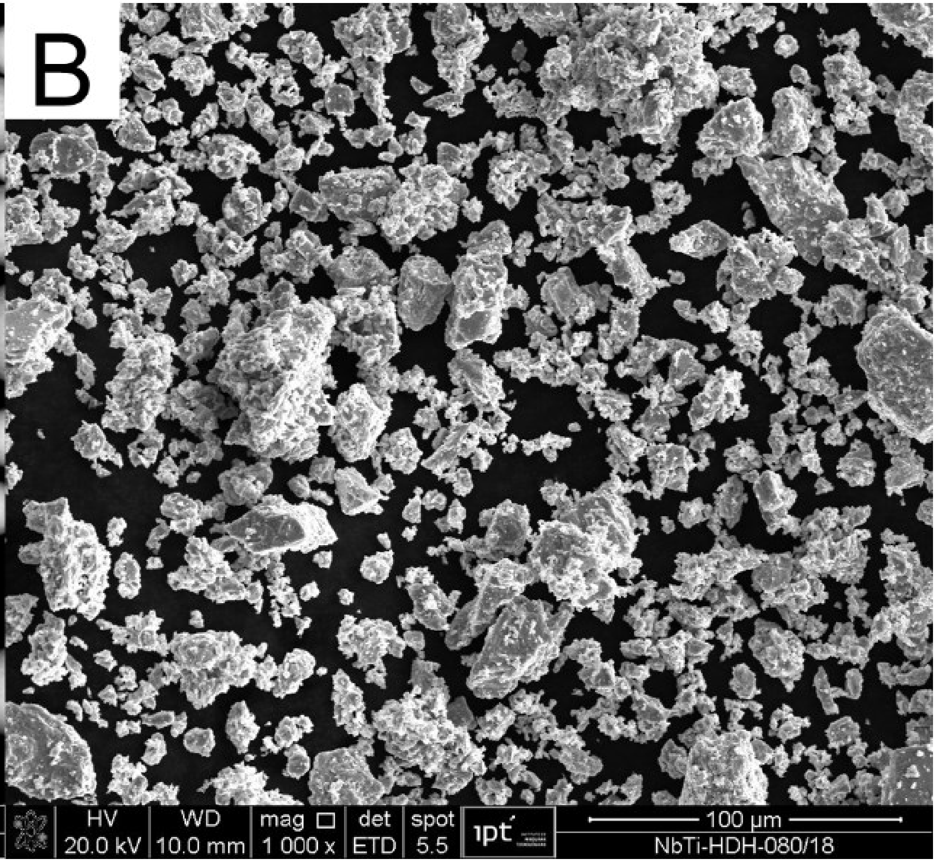
| **Powder process** | **Plasma Atomization** | **Hydrogenation-Dehydrogenation** |
|---|---|---|
| Morphology | Spherical | Irregular |
| $\rho_{DRX}$ (g/cm$^3$) | 6.059 | 6.046 |
| $\rho_a$ (mg/mm$^3$) | 3.49 | 1.85 |
| O initial (ppm) | 1200 | 3400 |
| N initial (ppm) | 209 | 430 |
| D10, D50, D90 | 19 µm, 31 µm, 51 µm | 12 µm, 31 µm, 67 µm |
| Hall flow (s/50g) | 20 | No flowability |

# Materials

AP spherical shaped powder

HDH irregular shaped powder



# SLM parameters

- As the apparent density of the HDH powder was 50% of the atomized powder,  a double layer thickness was chosen, so that the mass energy density was similar.

| | Atomized powder | HDH powder |
|---|---|---|
| Average particle size (µm) | 31 | 31 |
| Apparent density $\rho_a$ (g/cm3) | 3.49 | 1.85 |
| Layer thickness $D_S$ (µm) | 30 | 60 |

$$Ev = \frac{P_L}{V_S * h_S * D_S}$$

$$Em = \frac{Ev}{\rho_a}$$

Ev is Volumetric energy density

Em is mass energy density

# SLM parameters

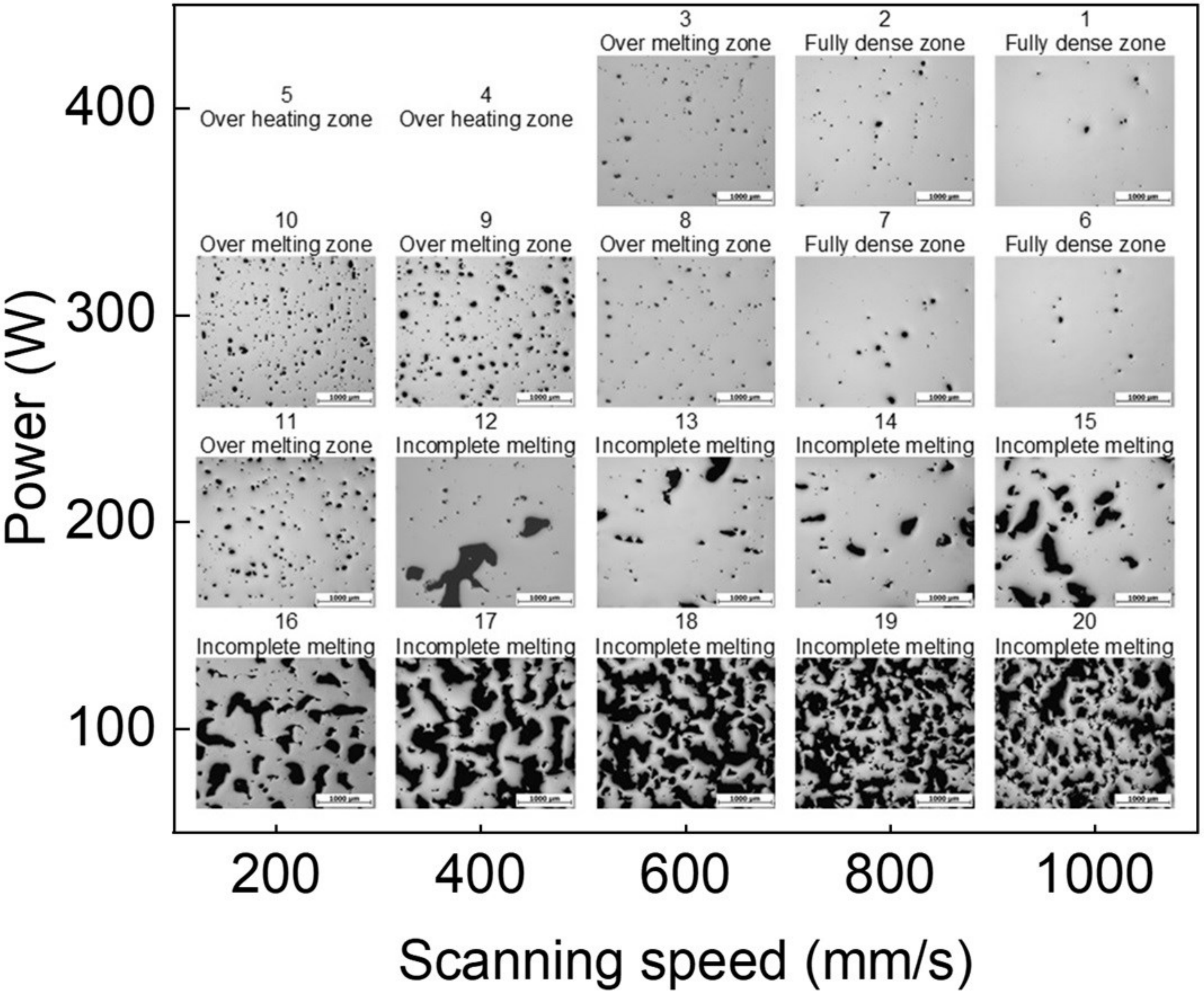
- Samples were made in a Concept Laser M2 machine, at ISI Laser Labs, Joinville, Brazil.
- For the HDH series, 20 conditions were investigated
  - Laser Power from 100 to 400W
  - Scanning speed from 200 to 1000 mm/s
  - Results suggested a better screen for the second trial
- For the atomized poder, 24 conditions were investigated
  - Laser power from 150 to 300W, from 900 to 1400mm/s

# Results

# Methods

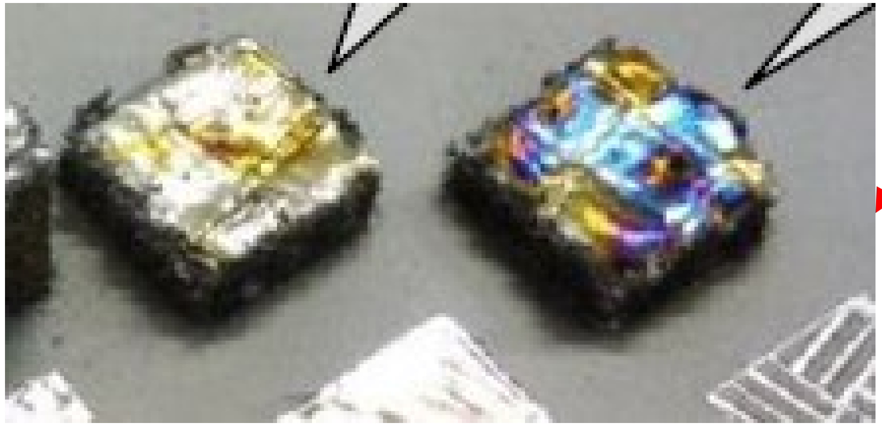
- Relative density was obtained by the Archimedes' principle (hydrostatic density) and pore area fraction with the Fiji Software.
- Microstructural analyses by optical microscopy and scanning electron microscopy.
- Microhardness testing using Vickers indentation with a load of 200 g.
- Measurements of oxygen and nitrogen were performed by the inert gas fusion-infrared absorption.

# Influence of power and scanning speed

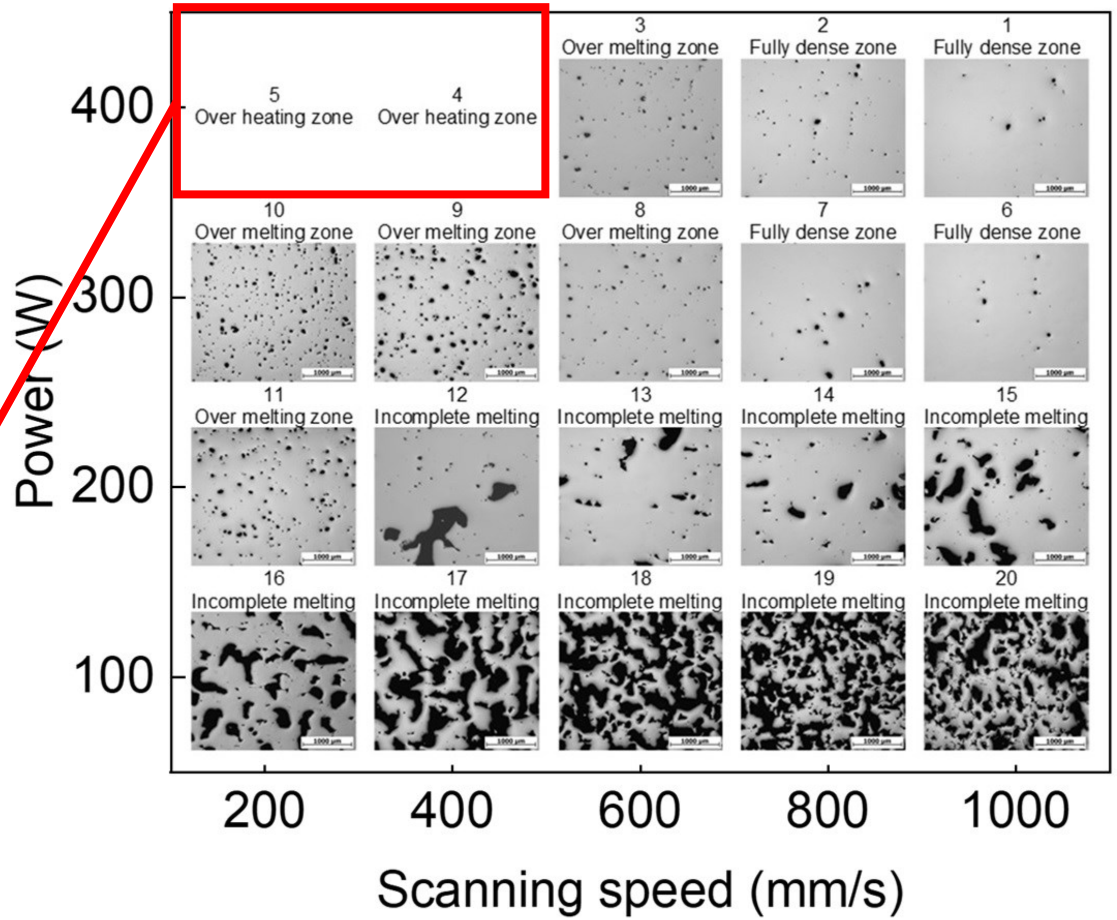


# Over heating zone

High power and low scanning speed. Pieces are deformed due to high deposited energy, so it is impossible to manufacture final parts.

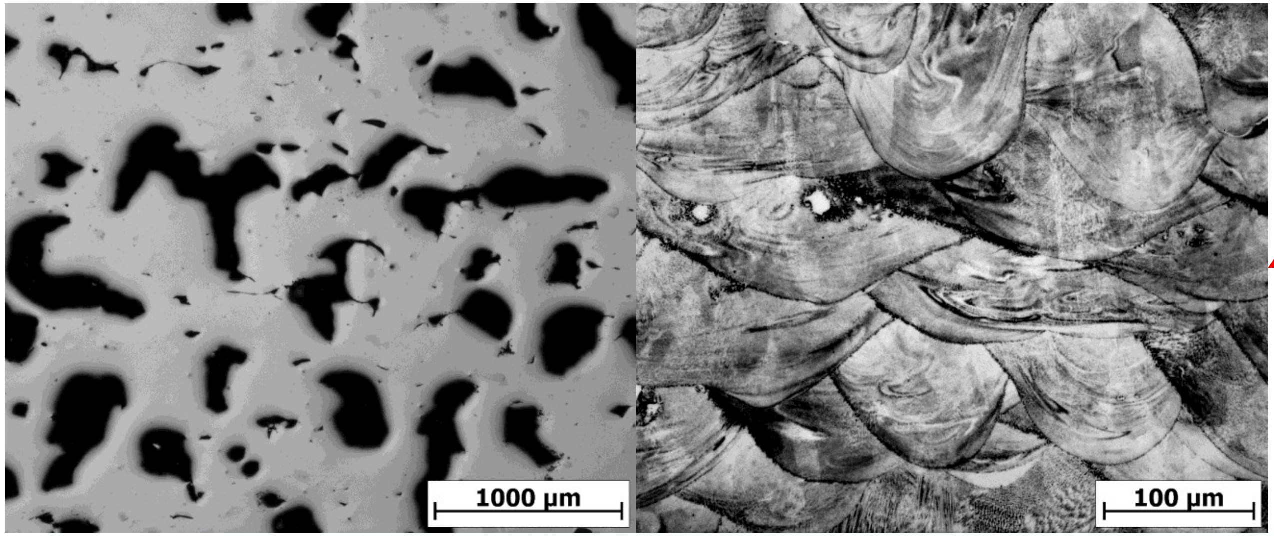


## Samples from HDH powder

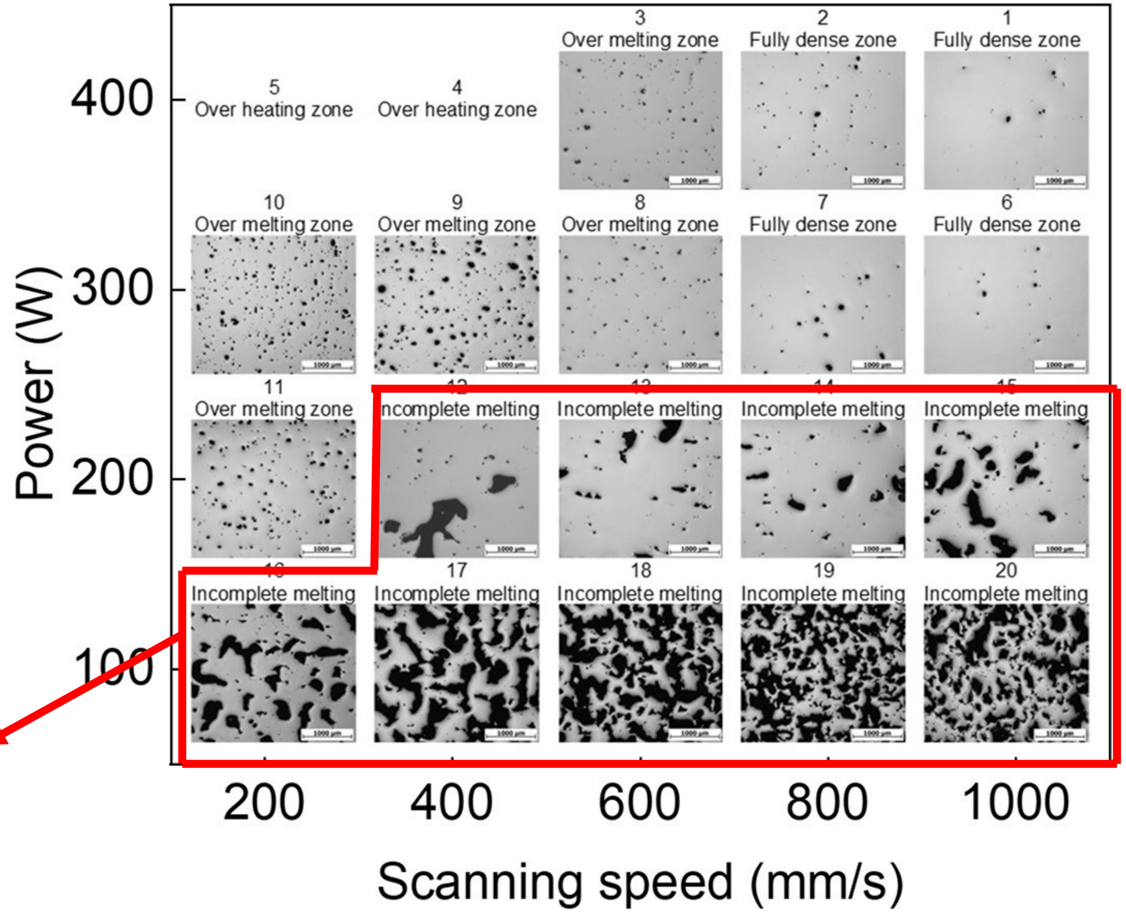


# Incomplete melting zone

Low power and high scanning speed. It is possible to manufacture pieces with present of irregular porosity and high content of segregation. It is also known as lack of fusion.

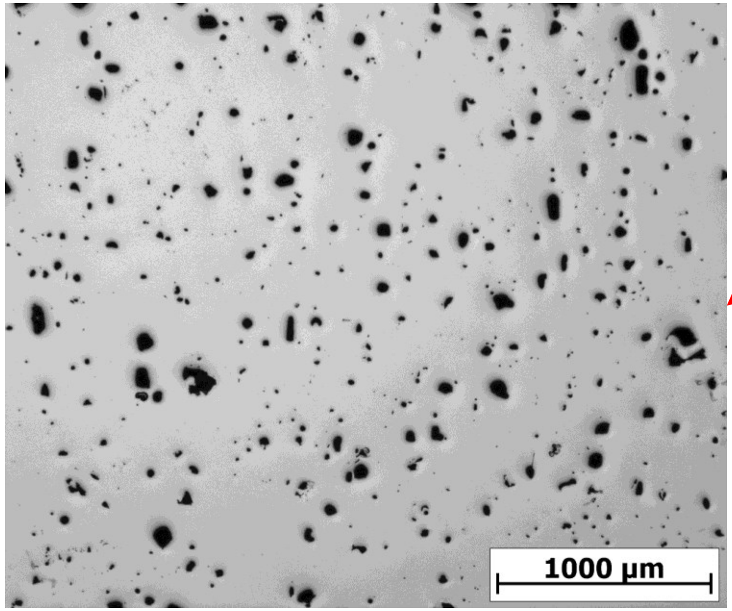



## Samples from HDH powder

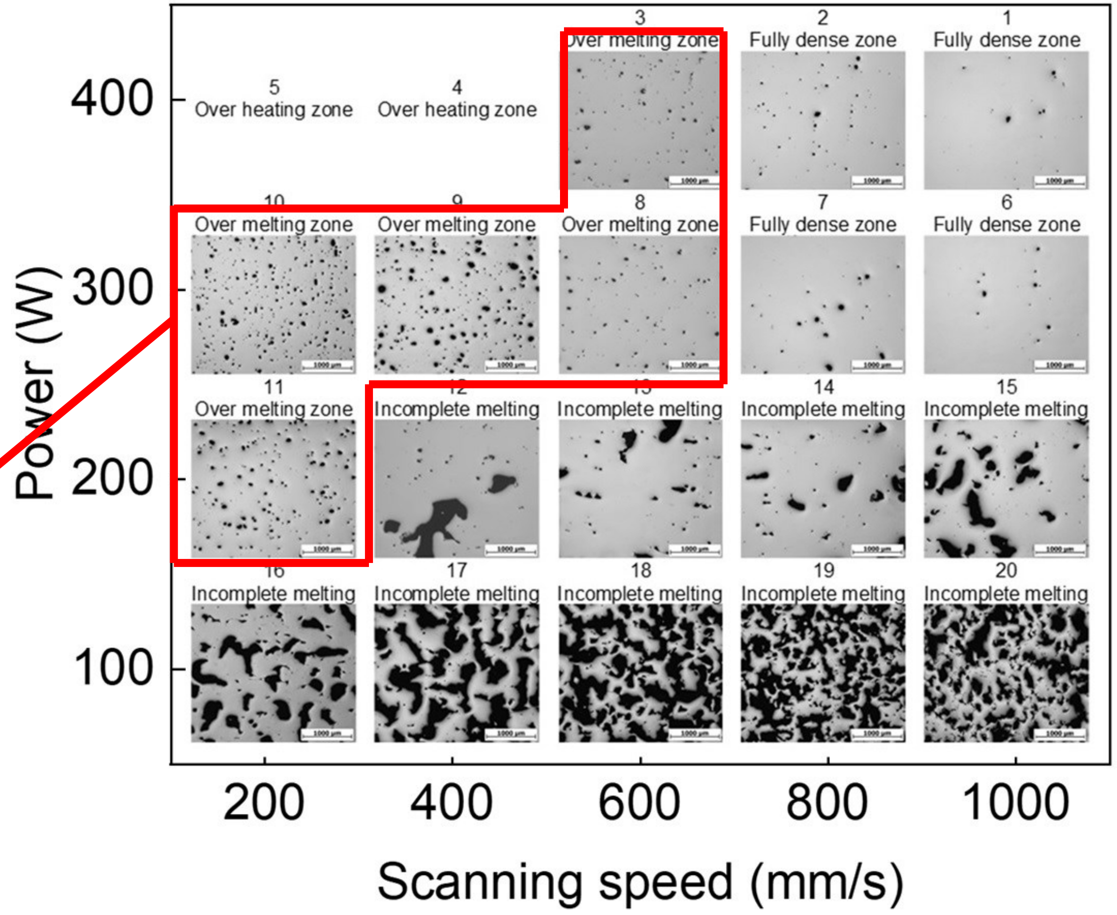


# Over melting zone

High power (slightly less than in the first zone) and low scanning speed. It is possible to manufacture pieces with spherical porosity due to trapped gas.

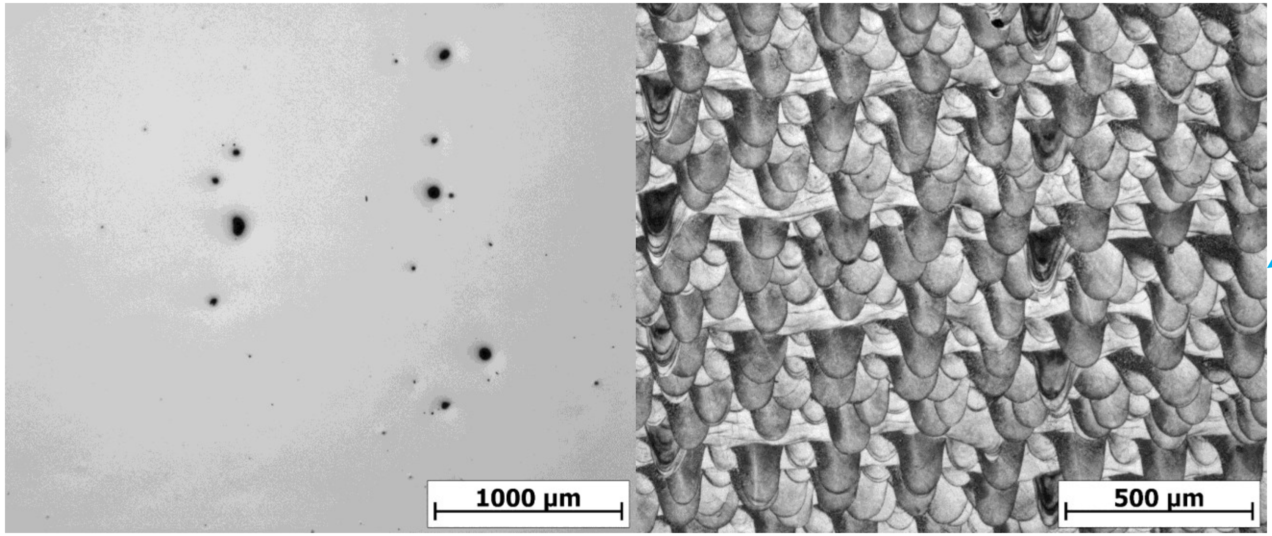



## Samples from HDH powder

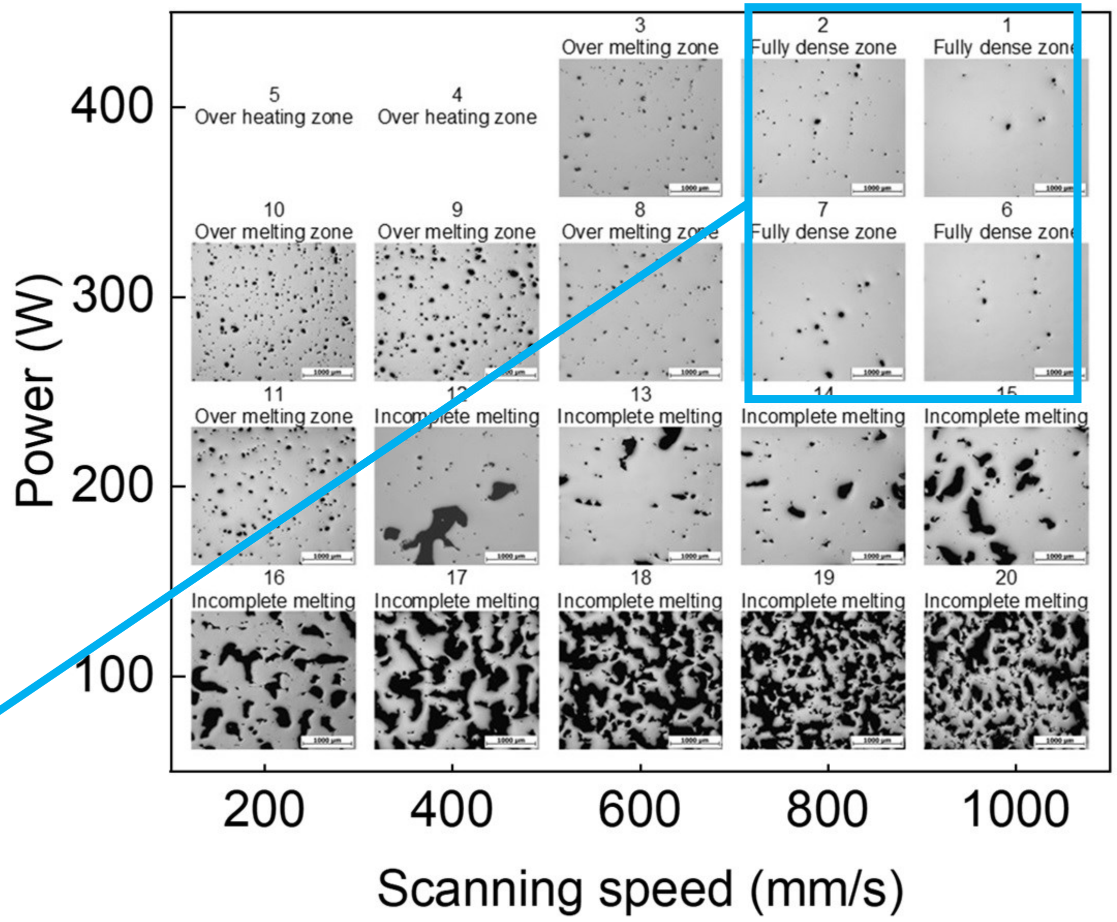


# “Fully dense” zone

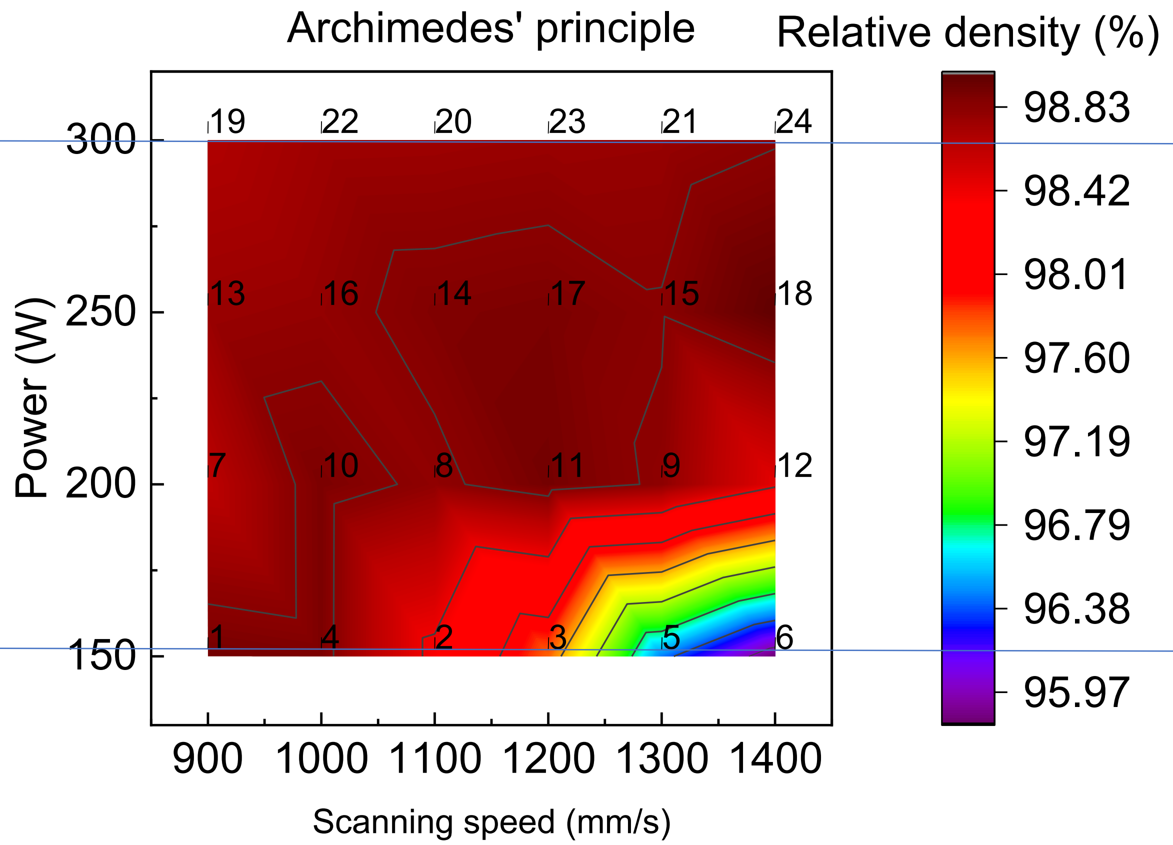
A steady relation between power and scanning speed is achieved and it is possible to obtain pieces with densities higher than 98 %.




## Samples from HDH powder

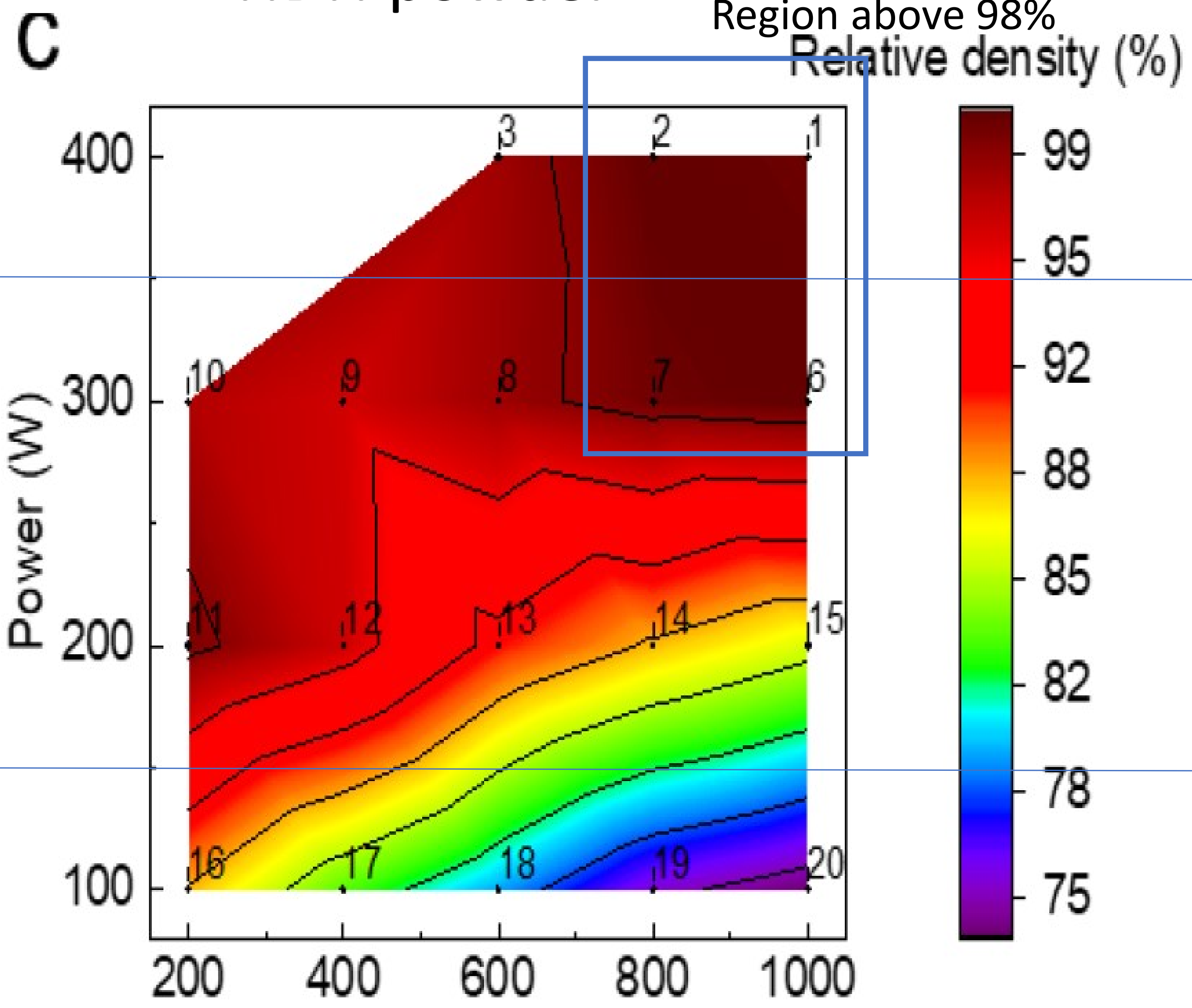


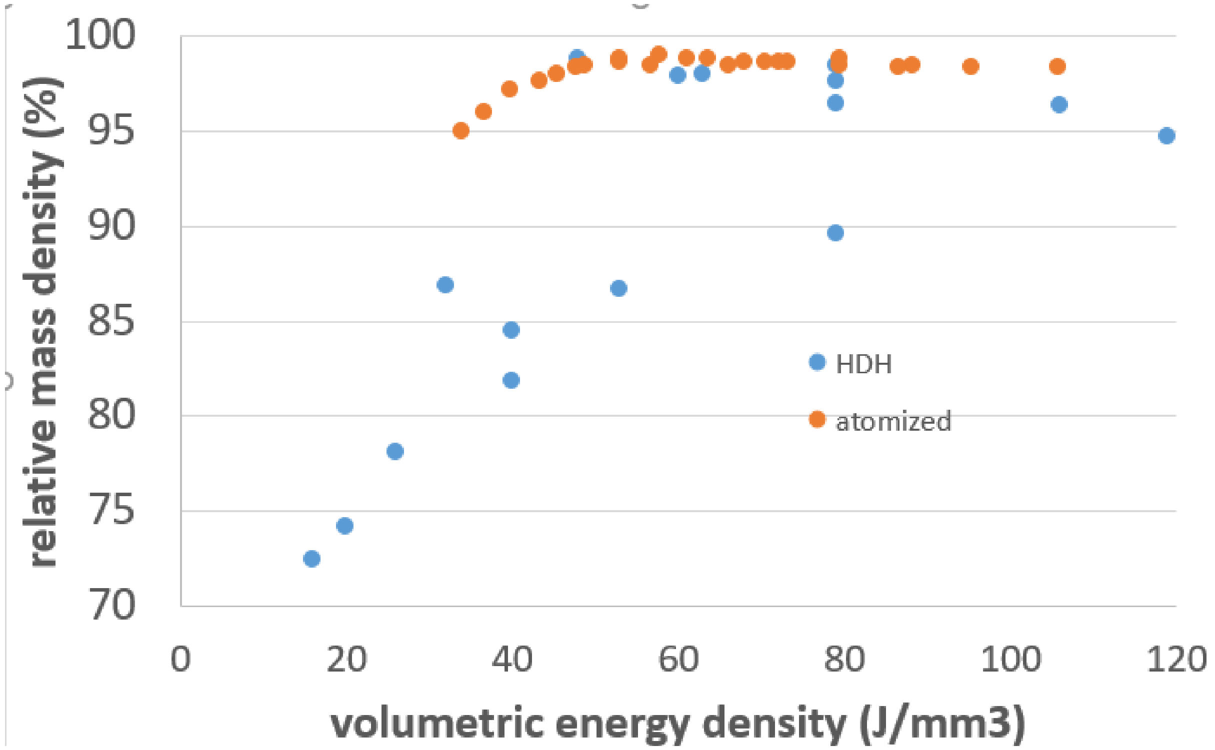
## Atomized powder

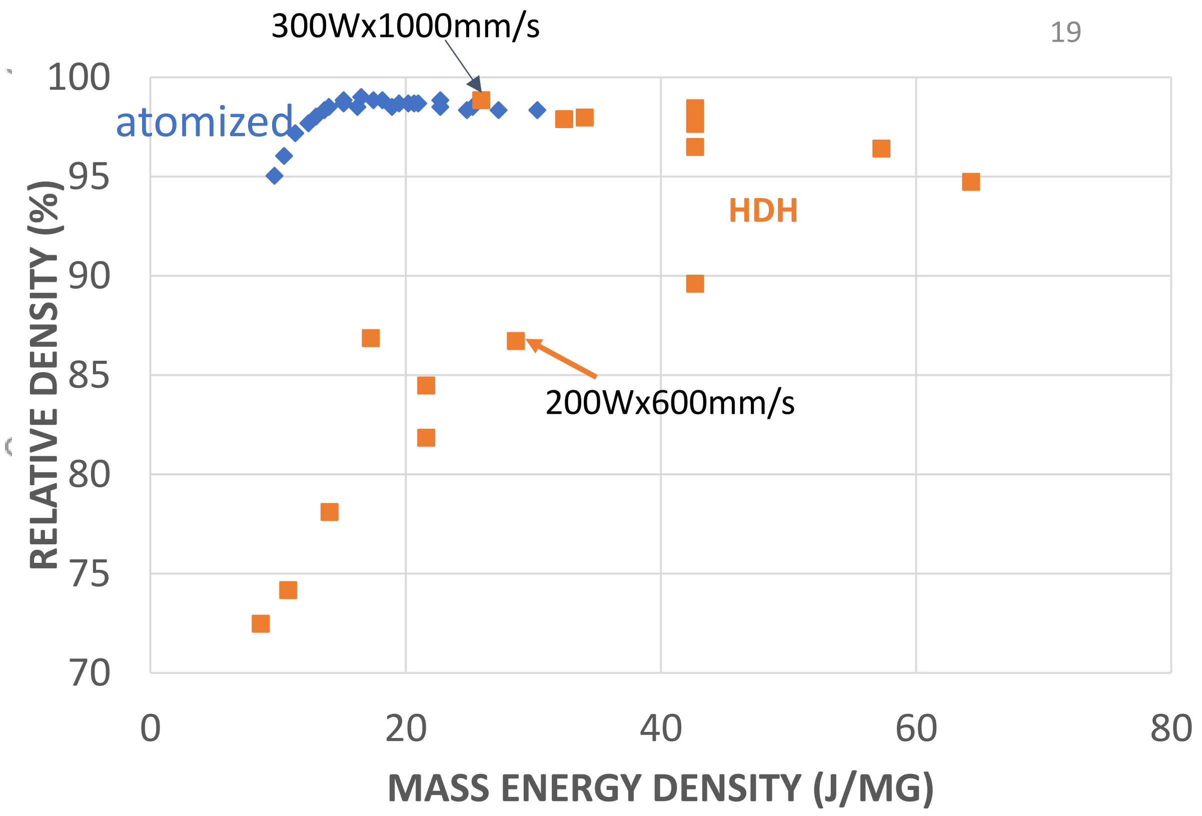



## HDH powder

Region above 98%






Two different approaches to the energy density

Relative density was sensitive to the combination of power and speed.
Low power was unsufficient to promote densification, even when speed was decresed.

# More detailed comparison

SLM parameters of fabrication:
Same laser speed in four samples, comparing two powders at two laser power.

| **Sample** | **1AP** | **2AP** | **3HDH** | **4HDH** |
|---|---|---|---|---|
| $P_L$ (W) | 200 | 300 | 200 | 300 |
| $V_S$ (mm/s) | 1000 | 1000 | 1000 | 1000 |
| $H_S$ (µm) | 105 | 105 | 105 | 105 |
| $D_S$ (µm) | 30 | 30 | 60 | 60 |
| $E_V$ (J/mm$^3$) | 63 | 95 | 32 | 48 |
| $E_M$ (J/mg) | 18 | 27 | 17 | 26 |

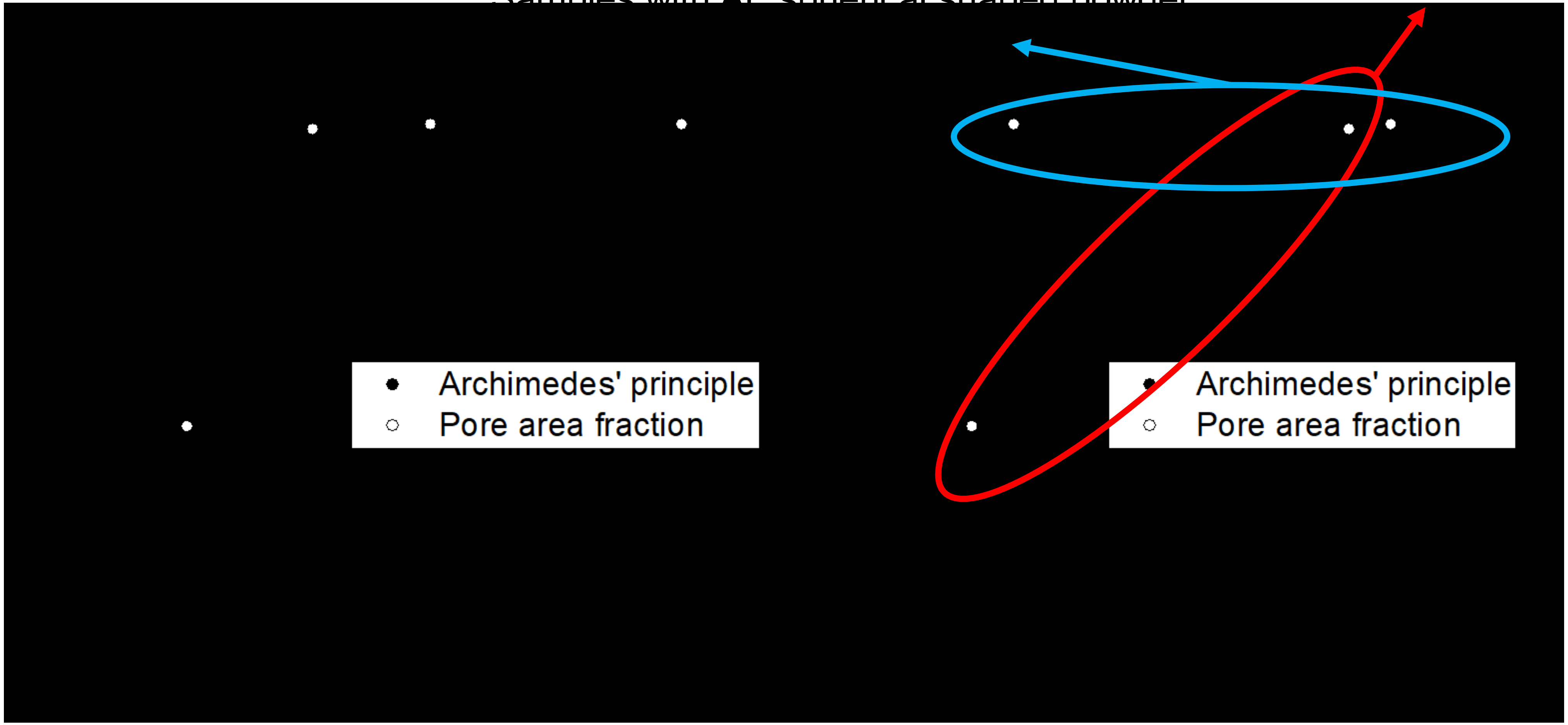
For samples manufactured with AP powder

For samples manufactured with HDH powder.

# Results: Relative density

Samples with HDH irregular shaped powder

Samples with AP spherical shaped powder



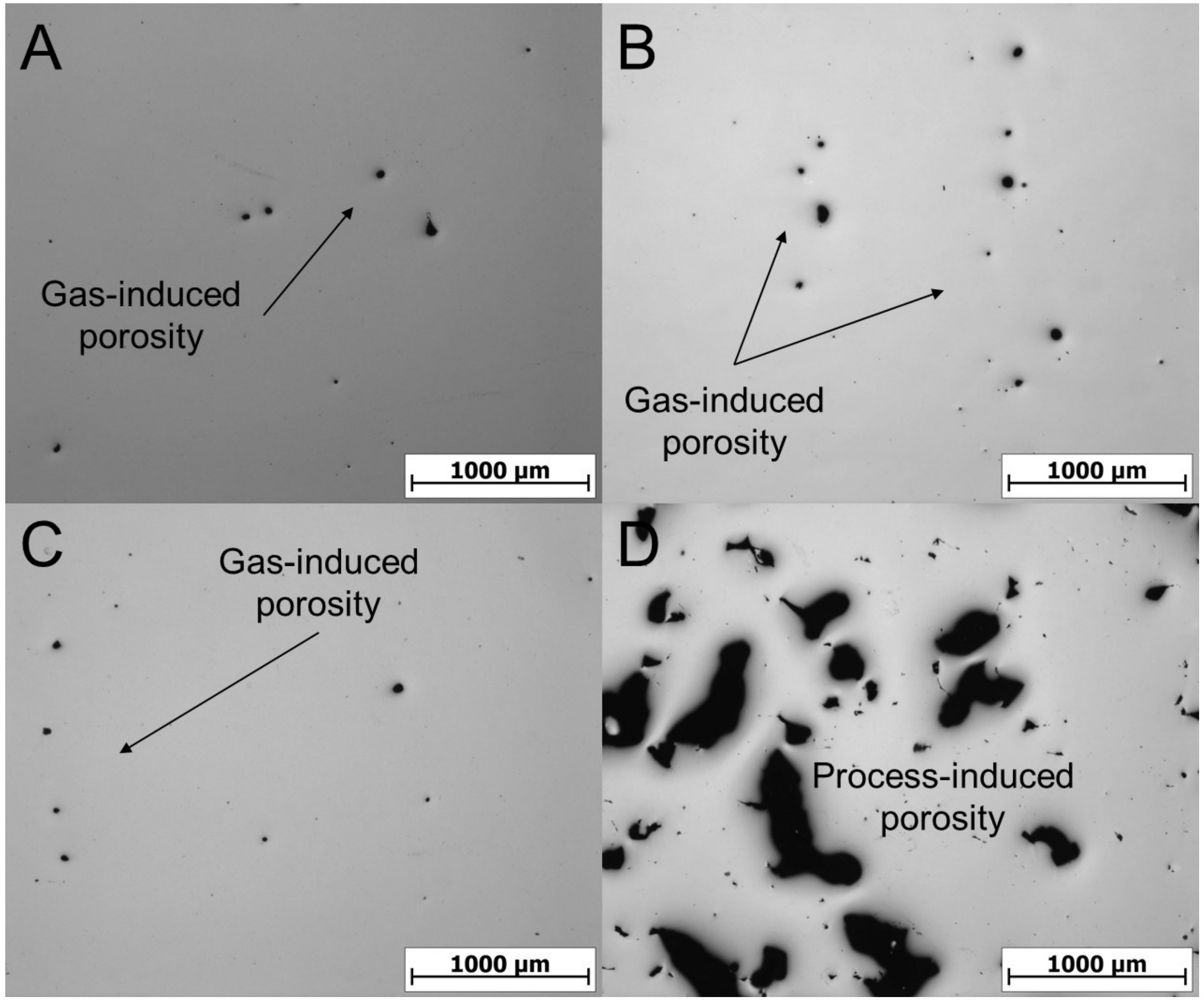

Values of relative density as a function of A) energy density and B) energy mass density.

# Results: Microstrutural analysis

300 W

Samples fabricated with AP spherical shaped powder

200 W

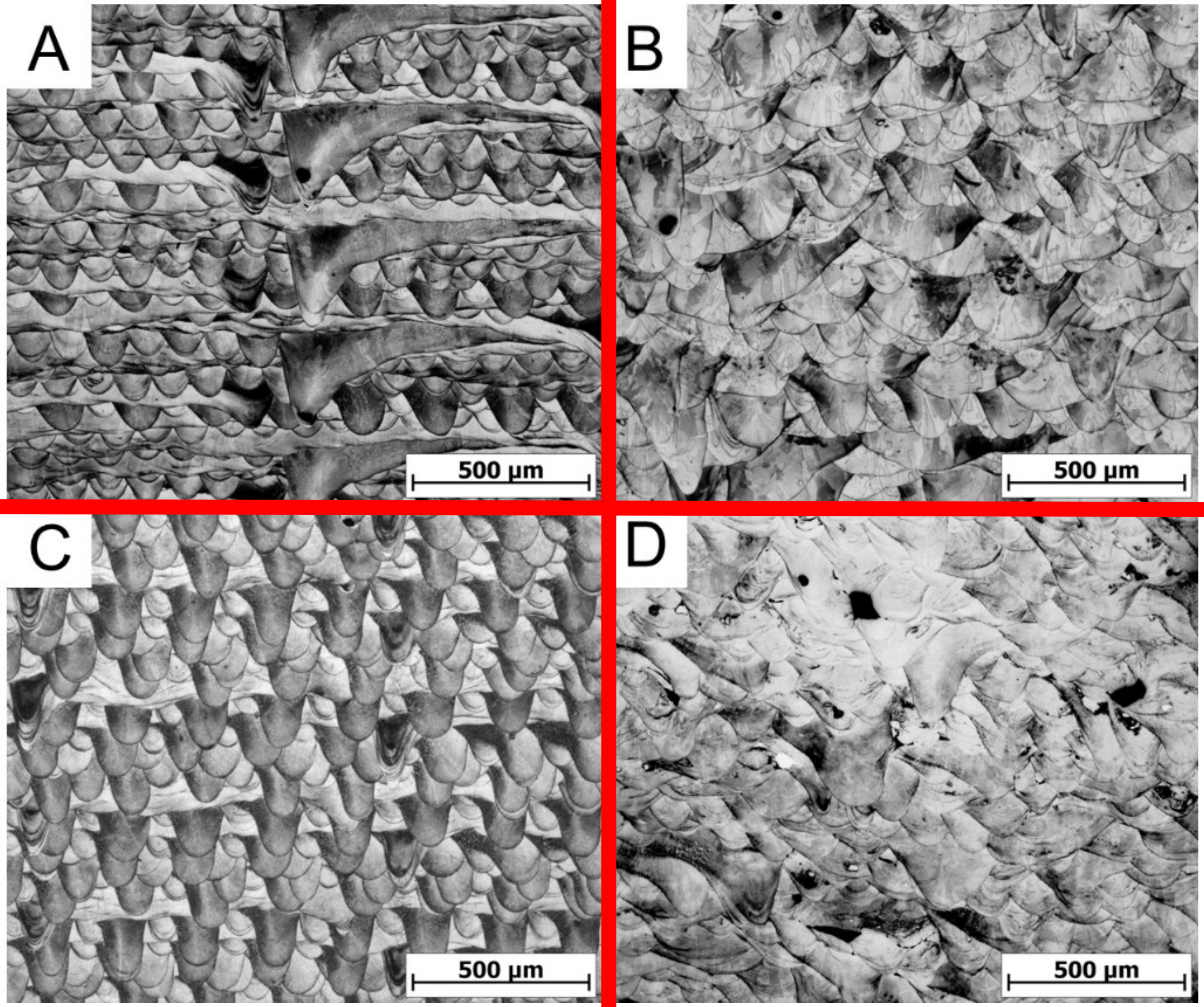



samples fabricated with HDH irregular shaped powder

AP spherical shaped powder | HDH irregular shaped powder

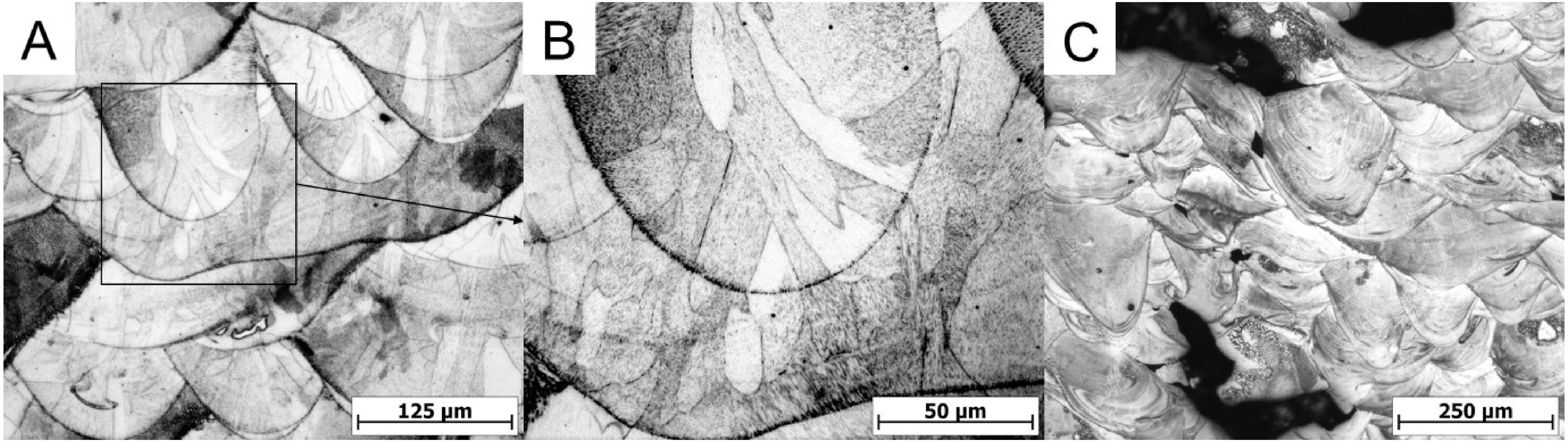
300 W

200 W



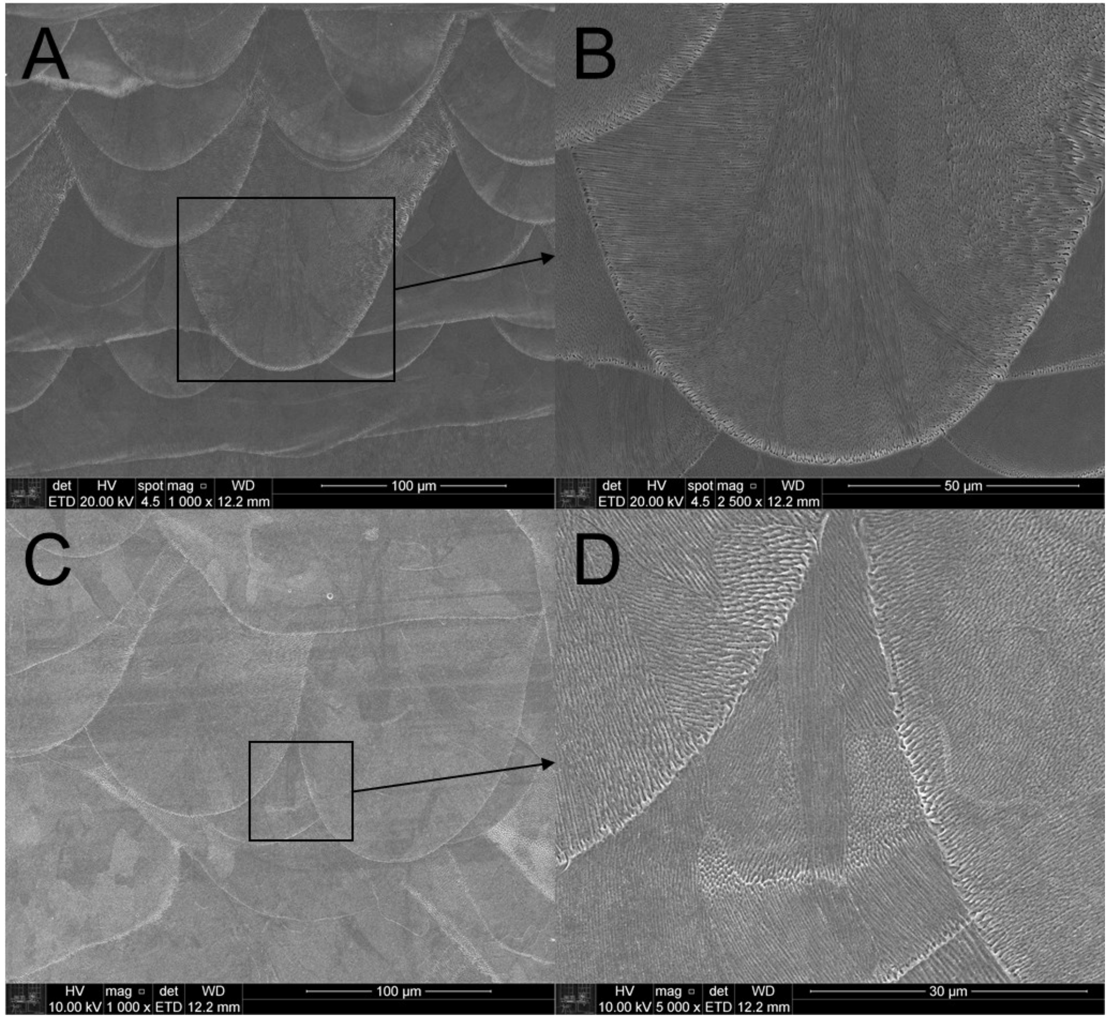
# Results: Samples from HDH powder

300 W　　　　　　　　200 W



# Results: Microstrutural analysis

Sample 2AP fabricated with AP spherical shaped powder




Sample 4HDH fabricated with HDH irregular shaped powder

300 W

# Conclusions

1. Its has been possible to obtain density above 98%, with HDH powder, even though its apparent density is half of the atomized powder.
2. It was possible to obtain density above 98%, with atomized powder, even with laser power of 150W
3. Different processing windows was need for HDH powder: Laser power above 200W was necessary for 98% densification.

# Acknowledgment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AACD

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# Thank you!

Fernando J.G. Landgraf

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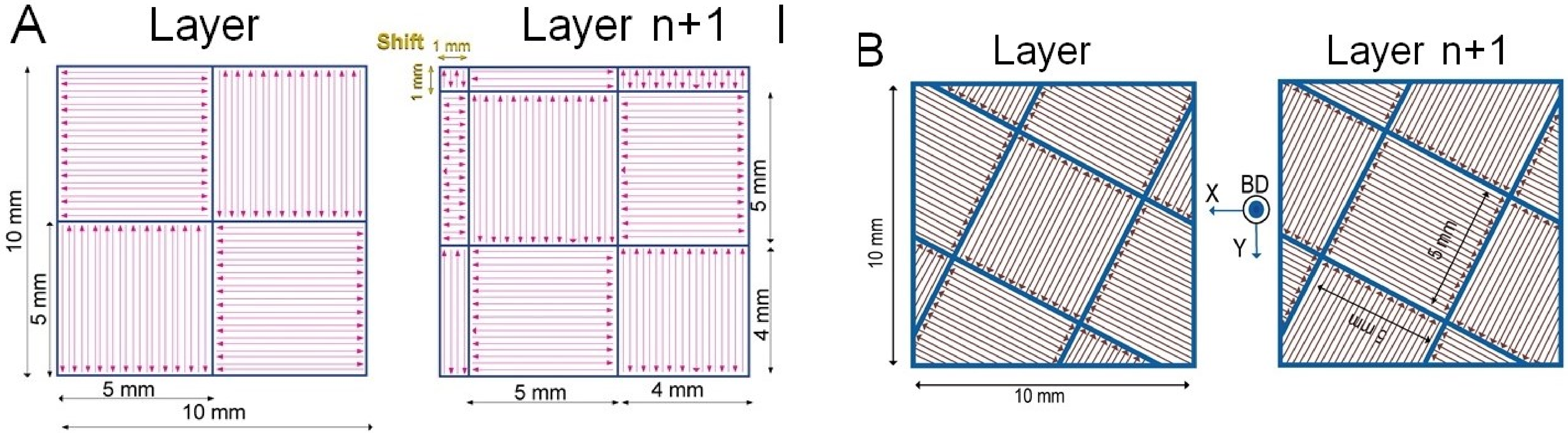
# Some SLM parameters were changed




For samples manufactured with AP powder

For samples manufactured with HDH powder.

Content of oxygen inside the chamber
AP Spherical powder: Less than 0.1 %
HDH Irregular powder: between 0.4-0.6 %