

# **In-depth investigation of the sub-micronic equiaxed grain microstructure of a NdFeB permanent magnet fabricated by Laser Powder Bed Fusion**

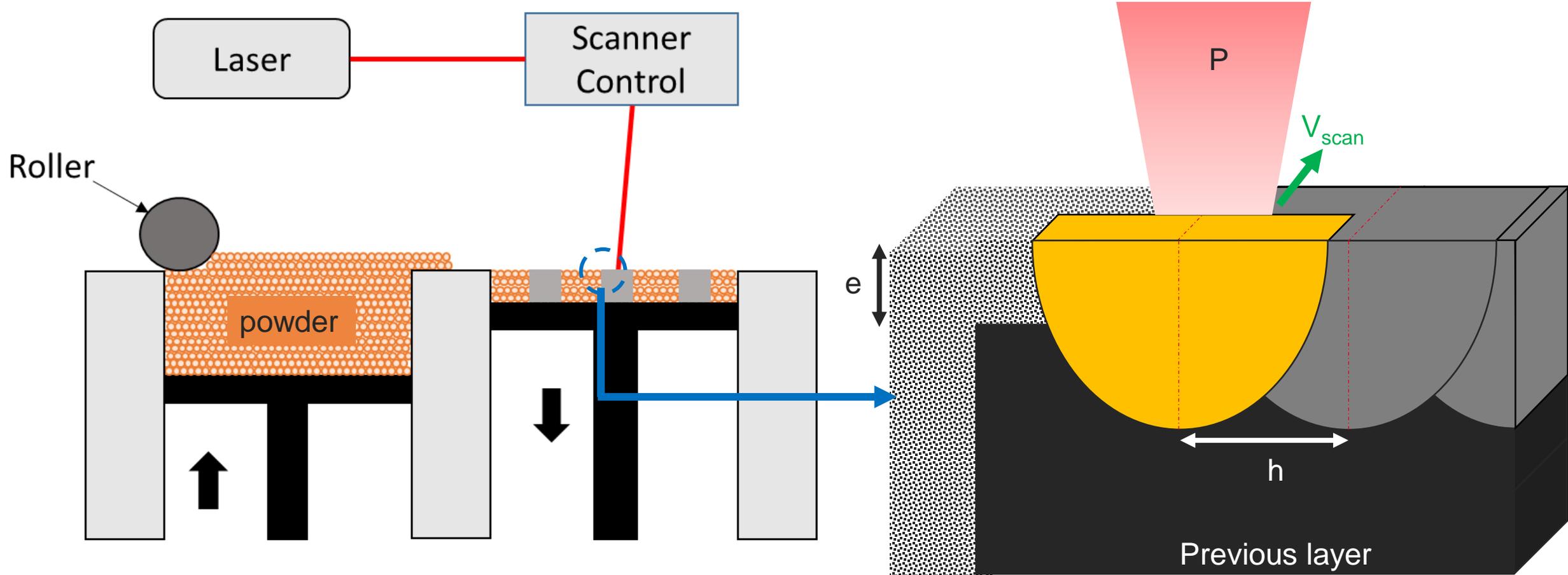
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*Univ. Grenoble Alpes, CEA, LITEN, Grenoble, 38000, France*



# The Laser Powder Bed Fusion Technique

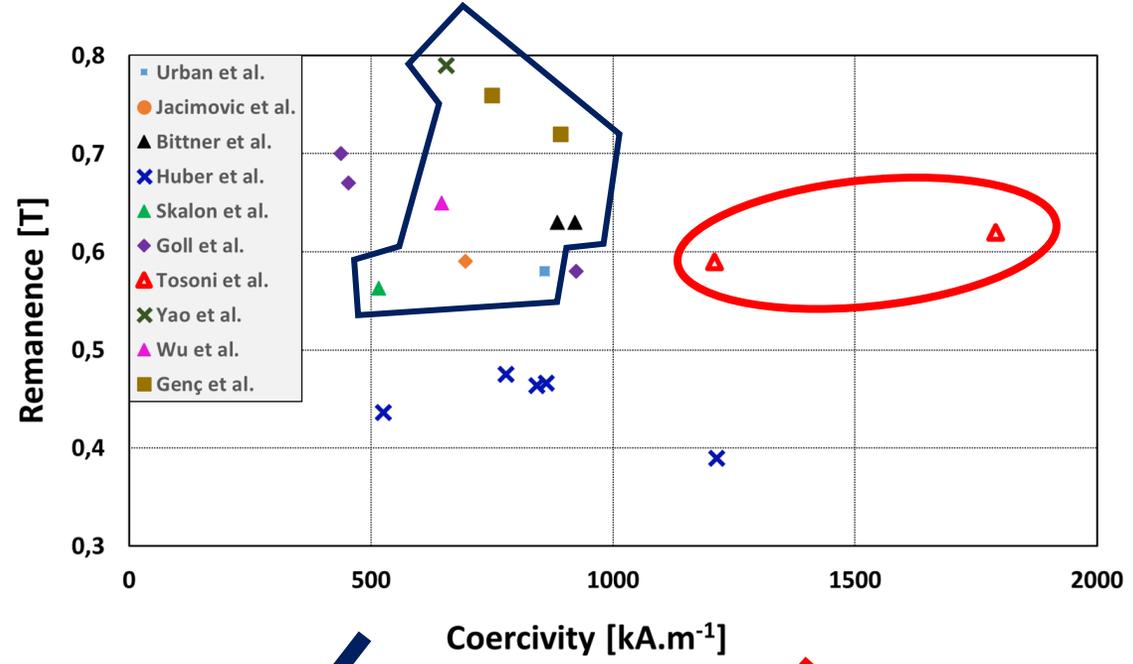
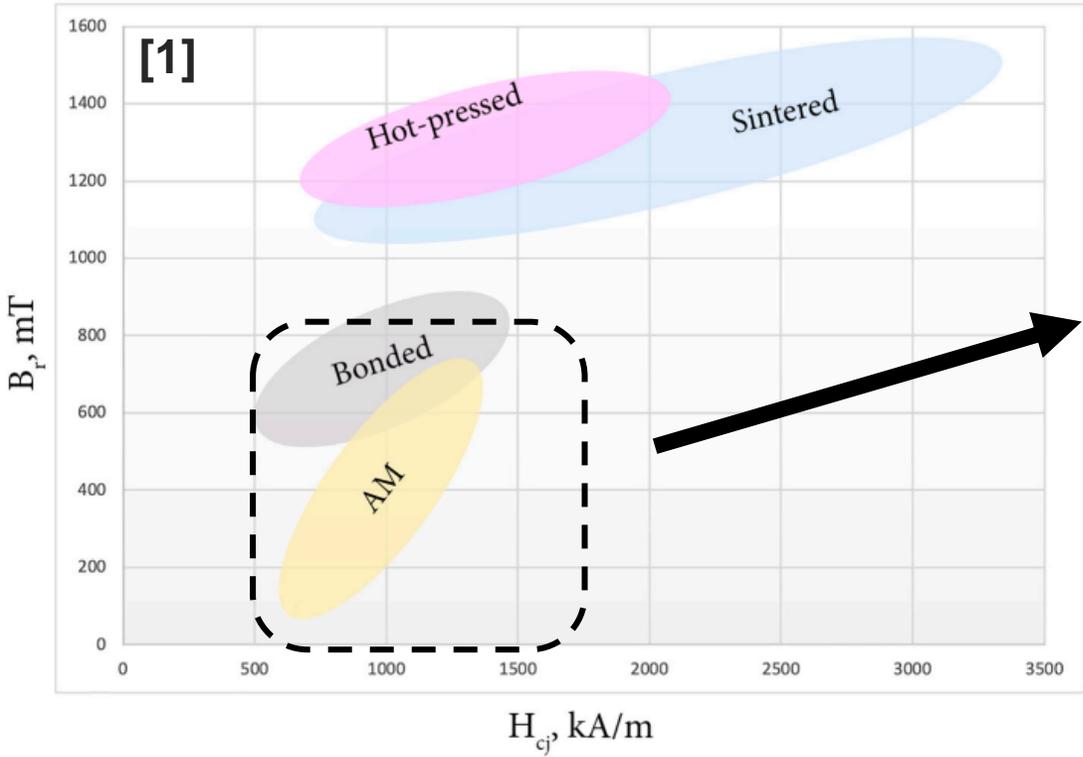


Alimentation piston system

Fabrication zone

$P$  : laser power  
 $V_{scan}$  : Scan speed  
 $e$  : powder bed thickness  
 $h$  : hatch distance

# State-of-the-art



Commercial powder:  
8%at RE  
2.5%at Ti  
2.5%at Zr

Tailored powder:  
14.4%at RE  
2%at Cu

$RE_2Fe_{14}B$   
stoichiometry :  
11.7%at. RE



No possibility for grain alignment  
→  $B_r < 0.8 T$

Commercial Powder (RE content **under** 2:14:1 stoichiometry )

Fe-rich grain boundary phases

Low coercivity

Tailored Powder (RE content **over** 2:14:1 stoichiometry )

Nd-rich grain boundary phases

Higher coercivity



[1] Pelevin I, Lyange M, Fedorenko L, Chernyshikhin S, Tereshina I. The Laser Powder Bed Fusion of Nd<sub>2</sub>Fe<sub>14</sub>B Permanent Magnets: The State of the Art. Condensed Matter. 2025; 10(2):22. <https://doi.org/10.3390/condmat10020022>

# Objectives and Approach



## OBJECTIVES

01

- Fabricate **samples by L-PBF** with a specific high RE content powder composition but free of HRE
- Describe **the microstructure** of a L-PBF sample and propose a formation mechanism

## APPROACH

02

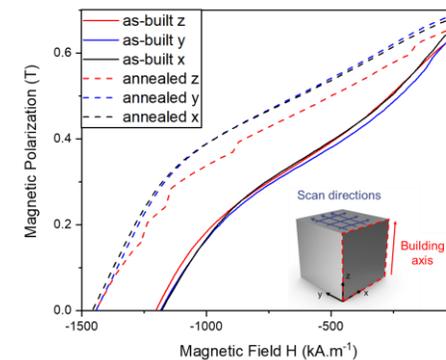
- Fabrication of a **tailored powder**
- Fabrication of **NdFeB samples by L-PBF**
- Magnetic characterization and selection of an optimal sample
- Microstructure investigation down to the nanoscale
- Elaboration of a solidification model to explain the formation of the microstructure



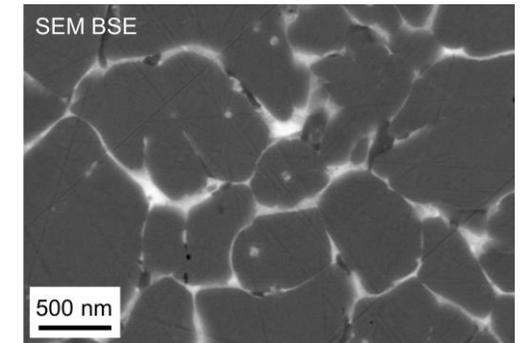
Powder



Samples



Properties

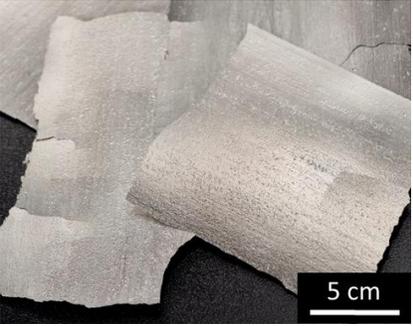
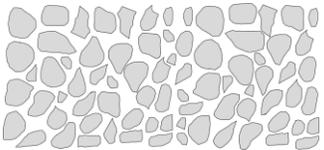
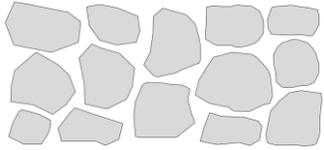
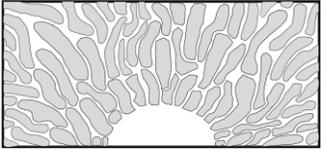
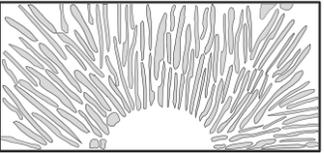
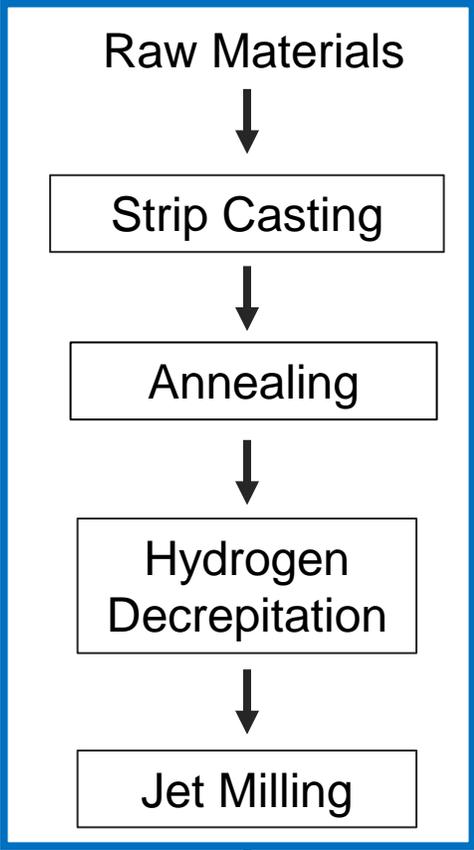


Microstructure

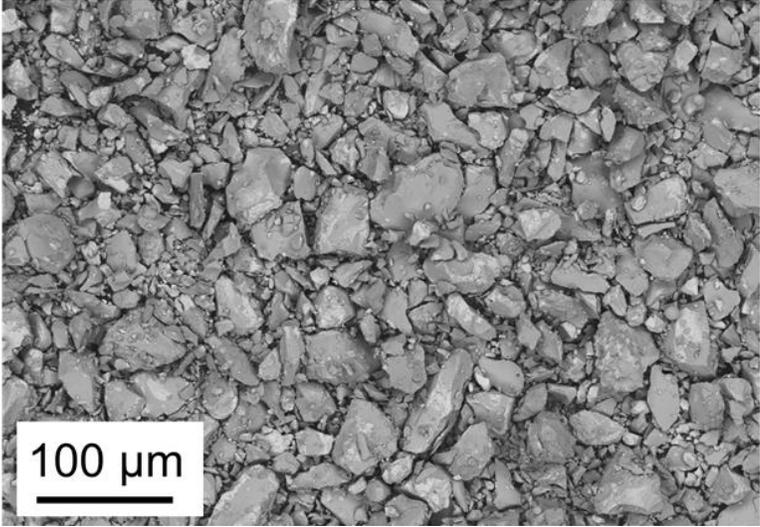
# Sample elaboration



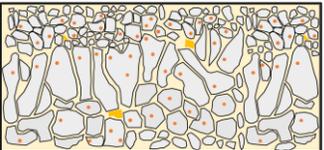
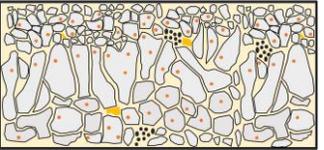
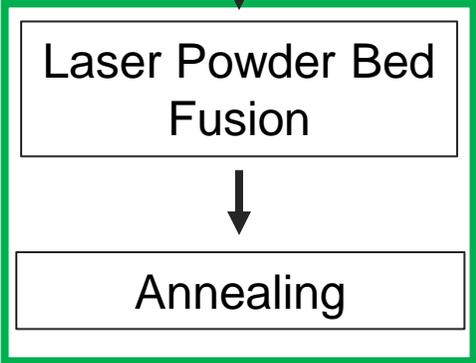
Powder



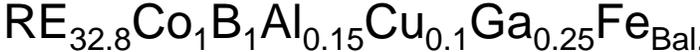
SEM BSE powder micrograph  
40 μm angular powder



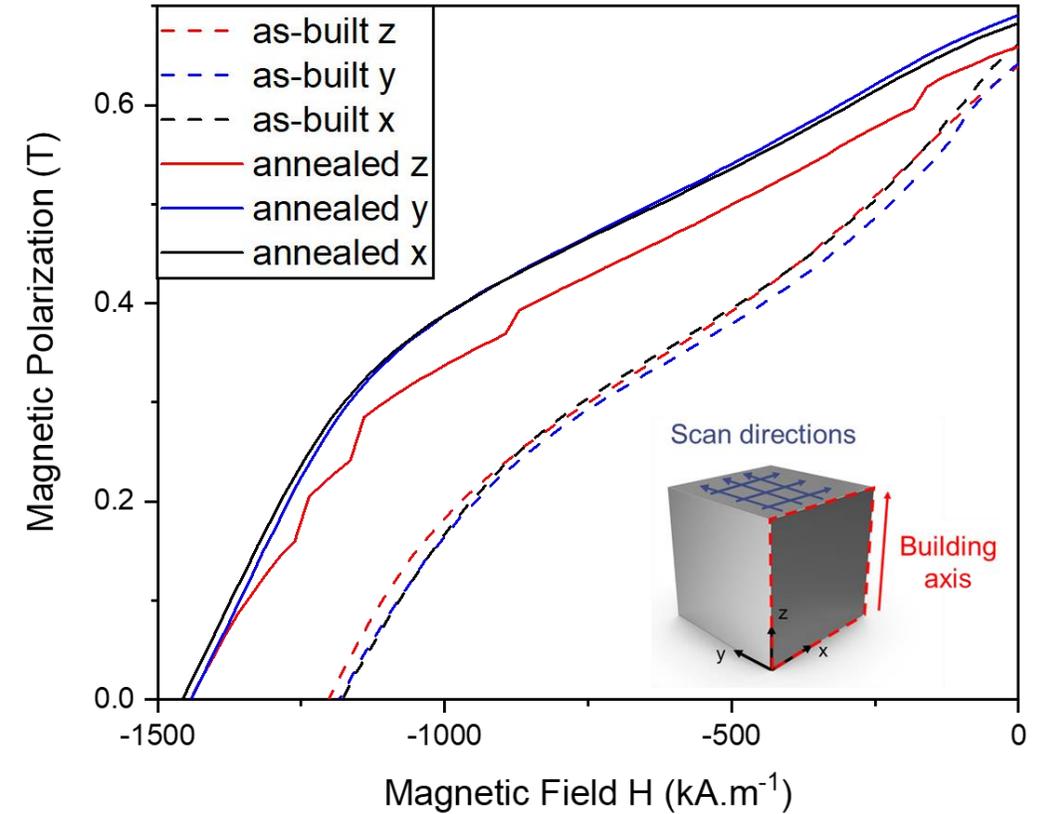
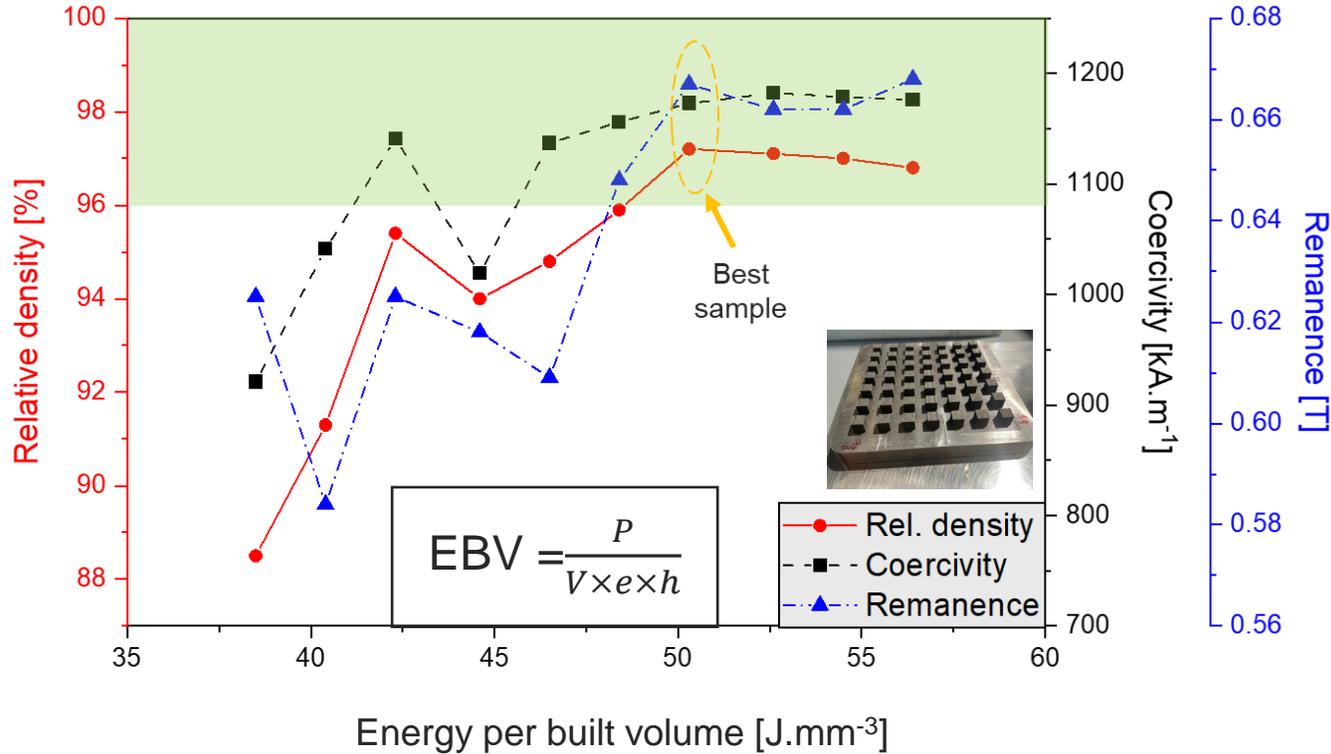
Samples



Powder composition (%mass.)



# Magnetic properties



## Best parameter set

$P=120 \text{ W}$

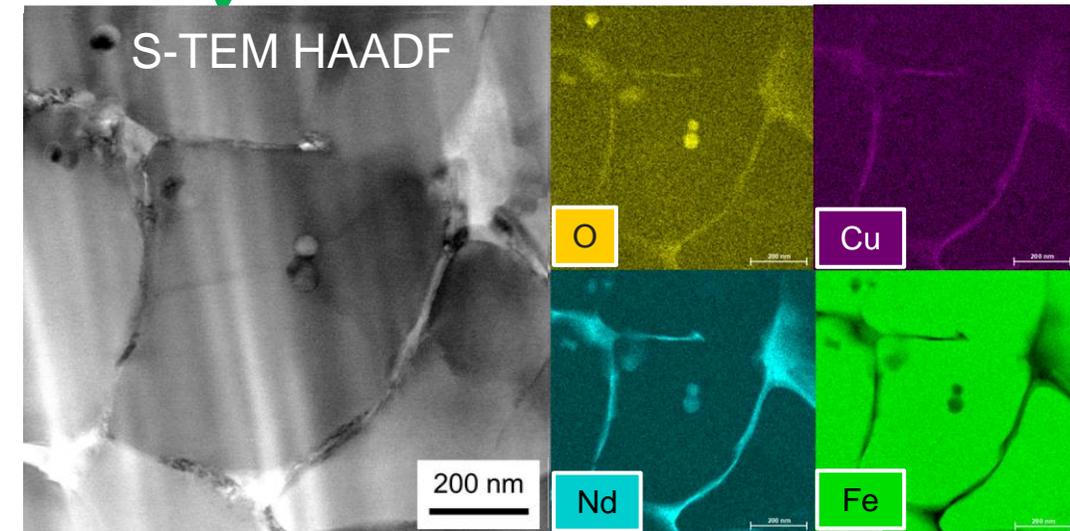
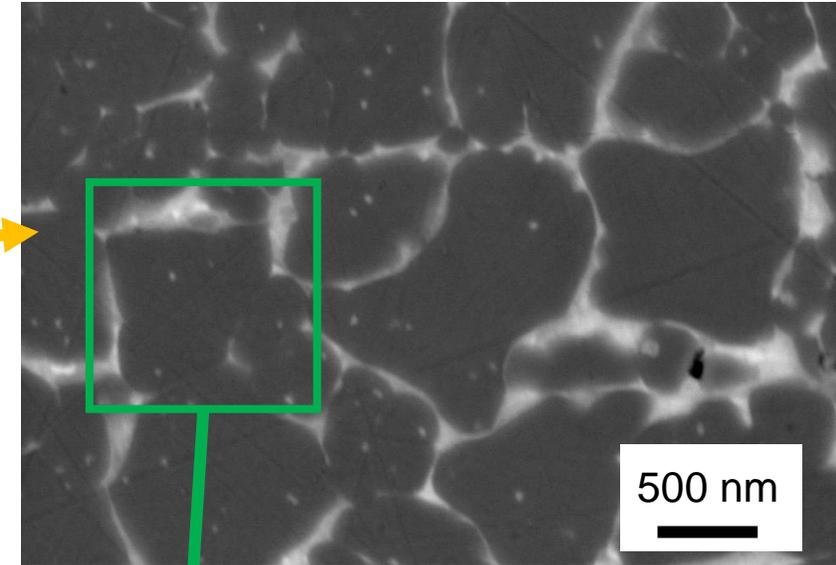
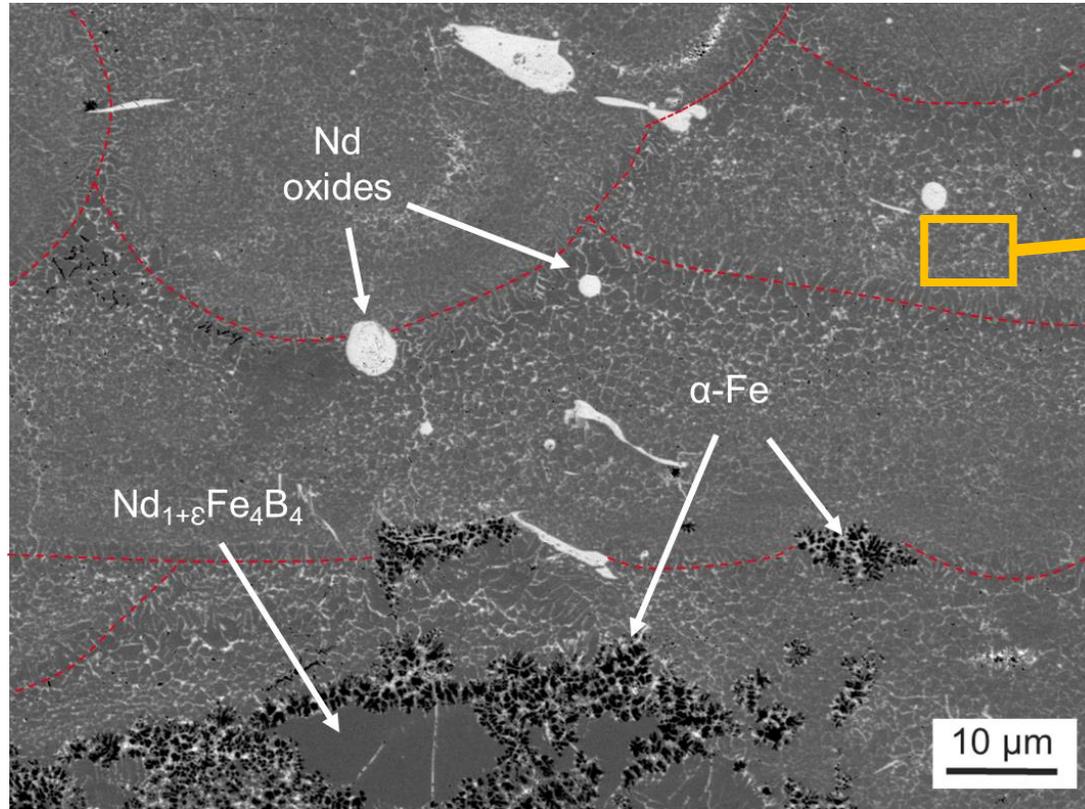
$V=1250 \text{ mm}\cdot\text{s}^{-1}$

$h=70 \text{ }\mu\text{m}$

$e=30 \text{ }\mu\text{m}$

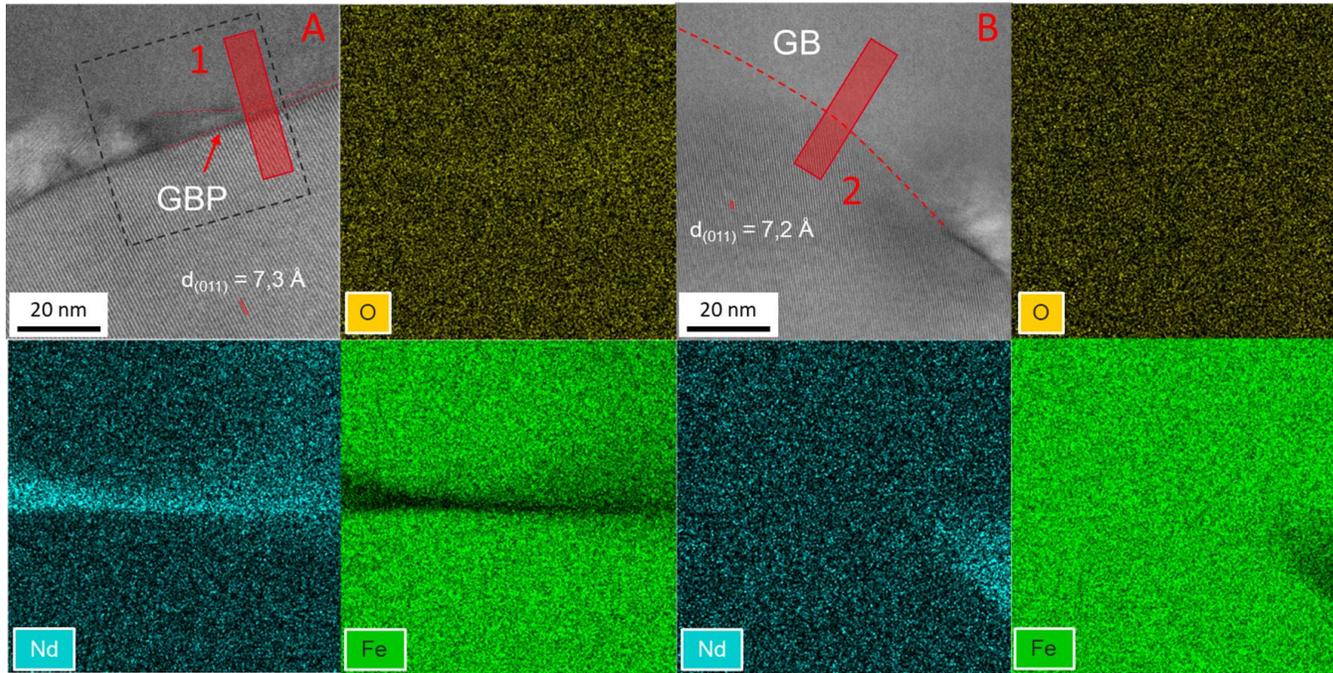
- Annealing realized at  $600^\circ\text{C}$  for 10 min. +  $470^\circ\text{C}$  for 1h
- Maximum as-built properties :  $B_r = 0.67 \text{ T}$  and  $H_{cJ} = 1170 \text{ kA}\cdot\text{m}^{-1}$
- Maximum annealed properties :  $B_r = \mathbf{0.69 \text{ T}}$  and  $H_{cJ} = \mathbf{1440 \text{ kA}\cdot\text{m}^{-1} (1.8 \text{ T})}$

# Microstructure investigation

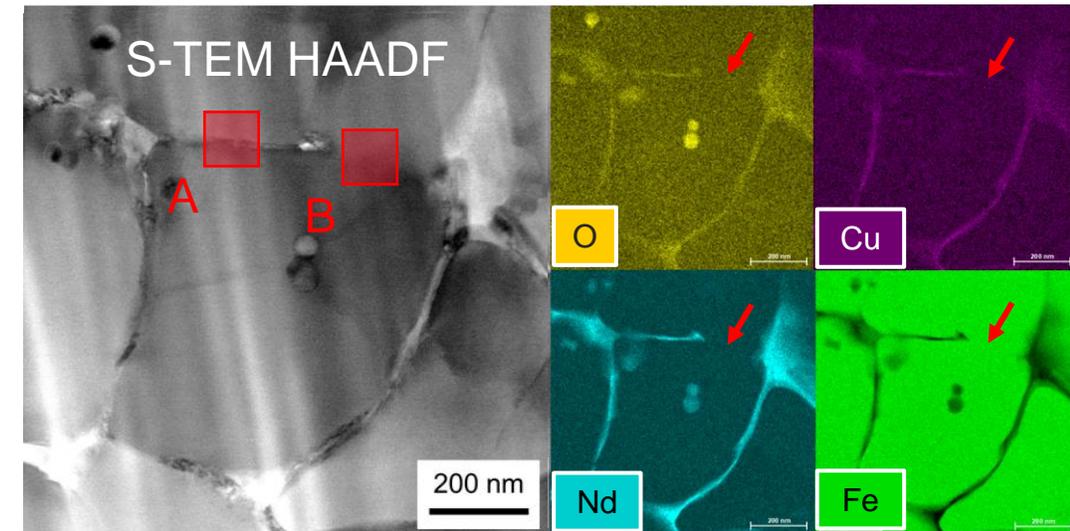
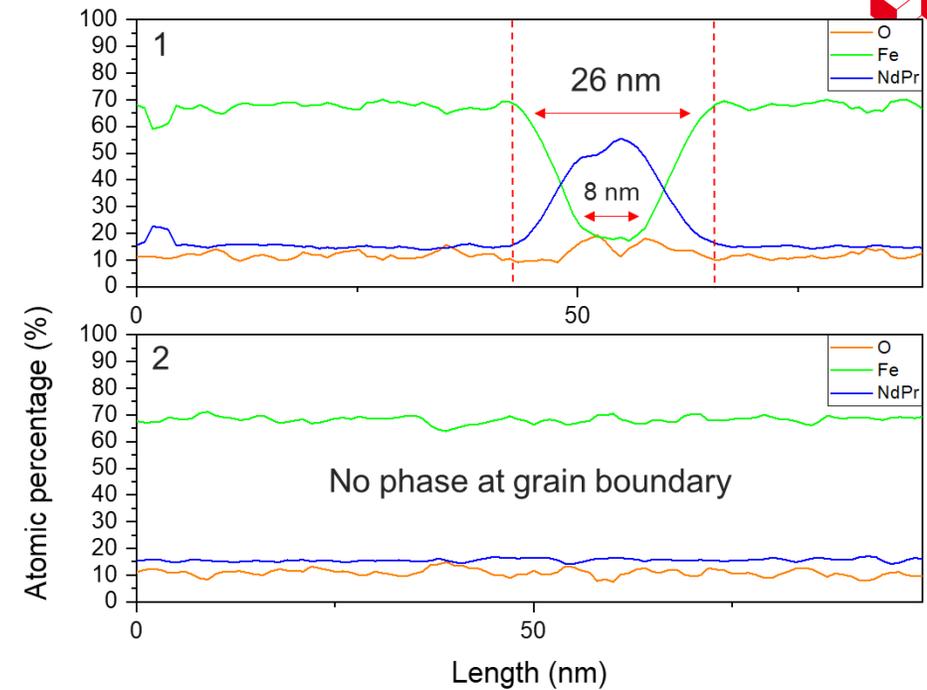


- Most of the microstructure is composed of small magnetic grains
- Non continuous grain boundary phase (GBP)
- Annealing does reorganise the GBP plus recombine  $\alpha$ -Fe clusters with Nd-rich to obtain additional  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grains

# Microstructure investigation



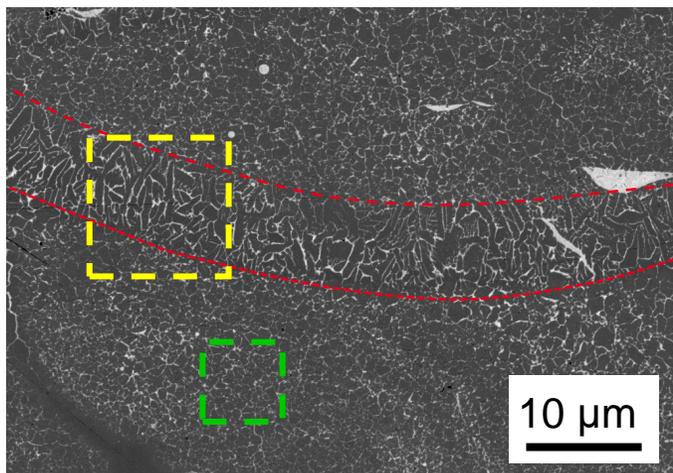
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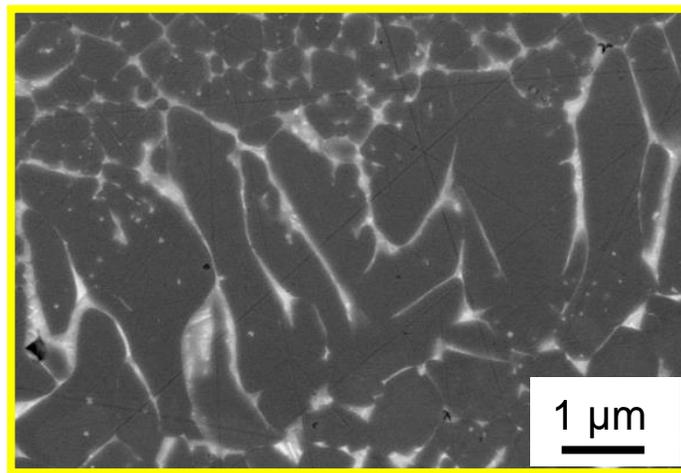
# Morphology of magnetic grains



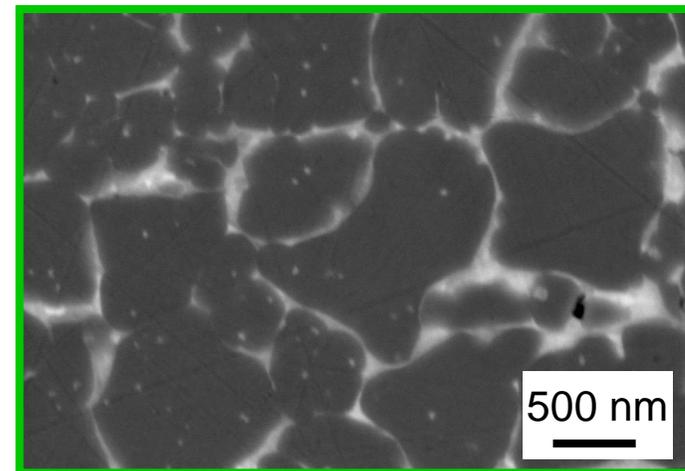
SEM Imaging



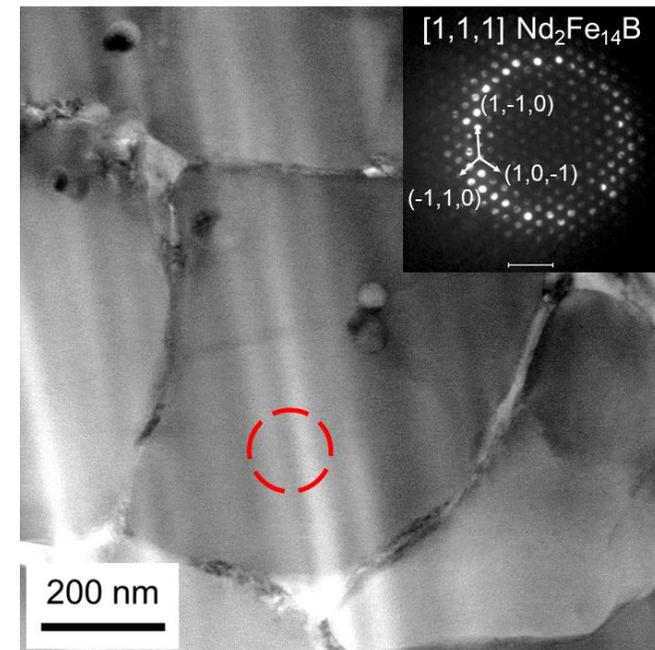
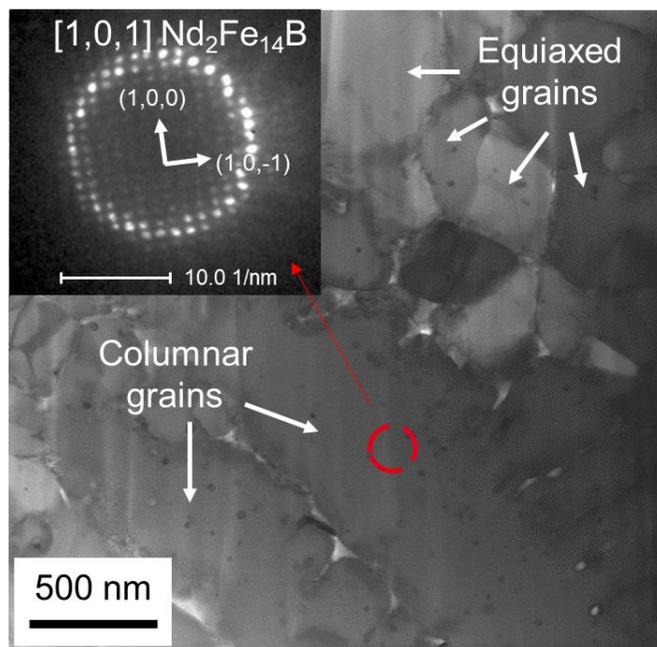
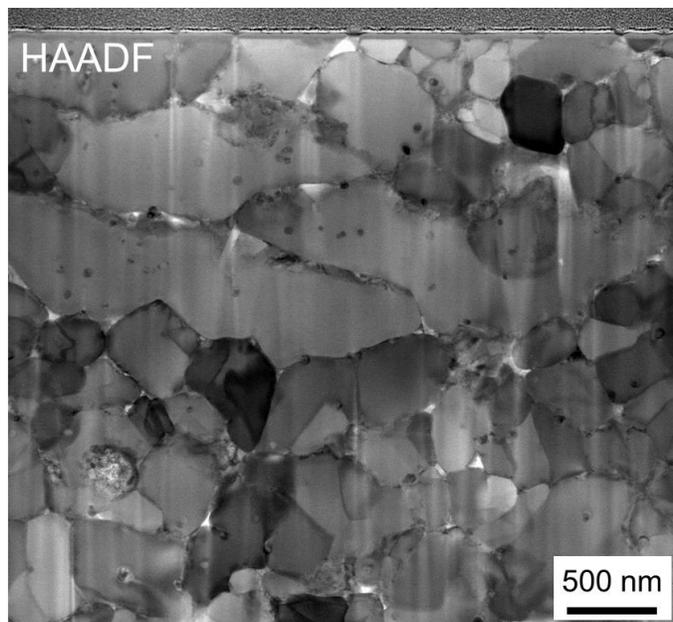
Columnar grains



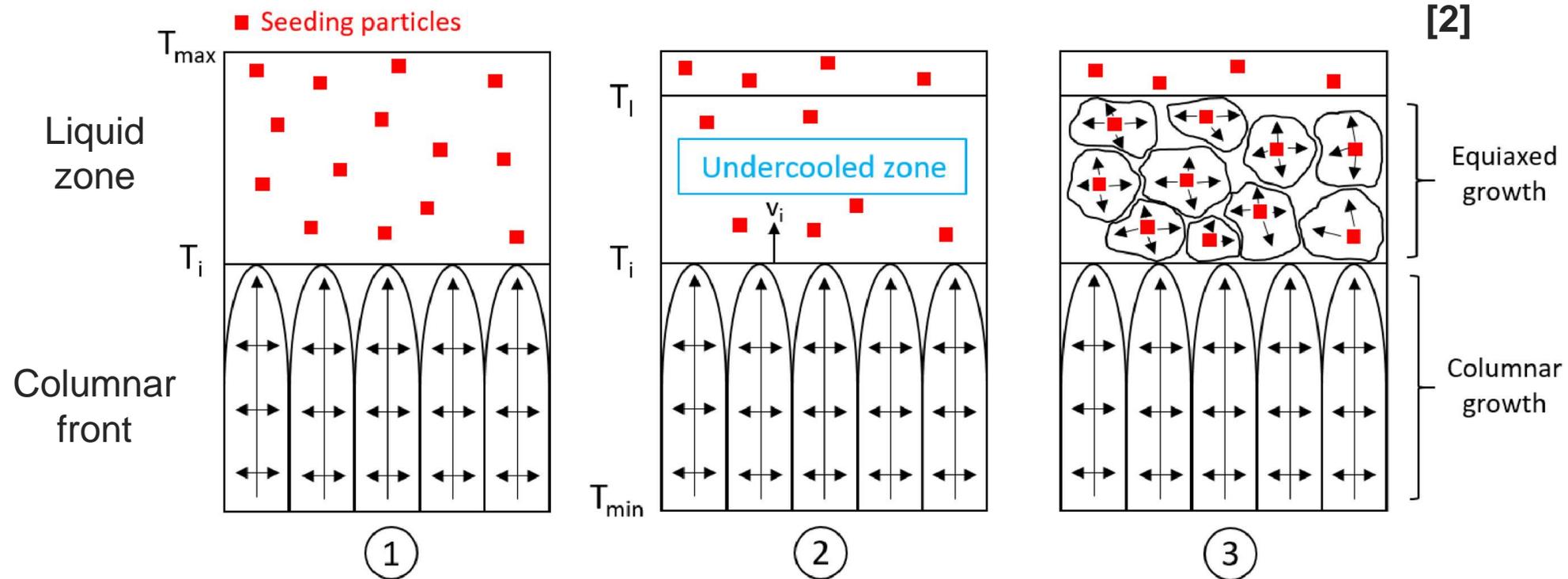
Equiaxed grains



S-TEM Imaging

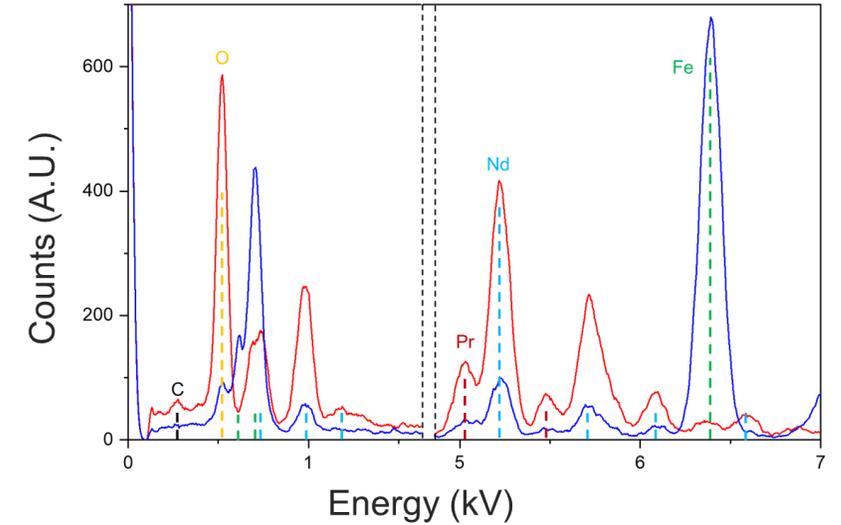
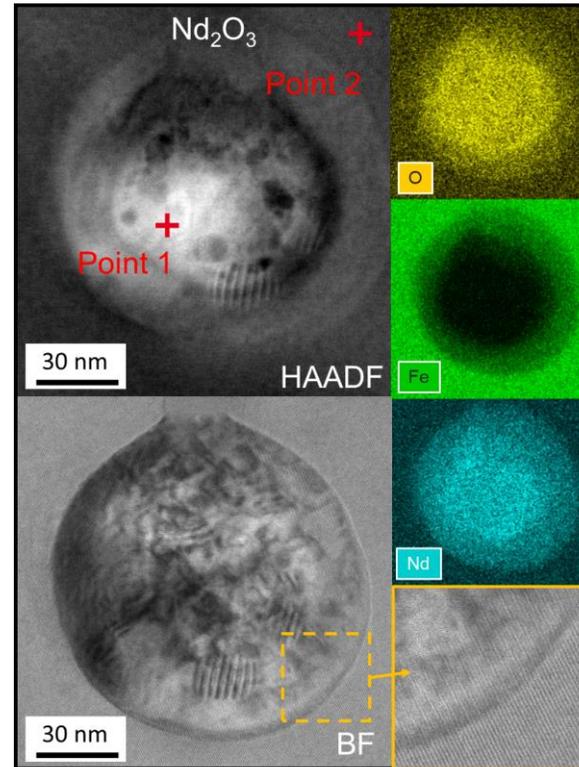
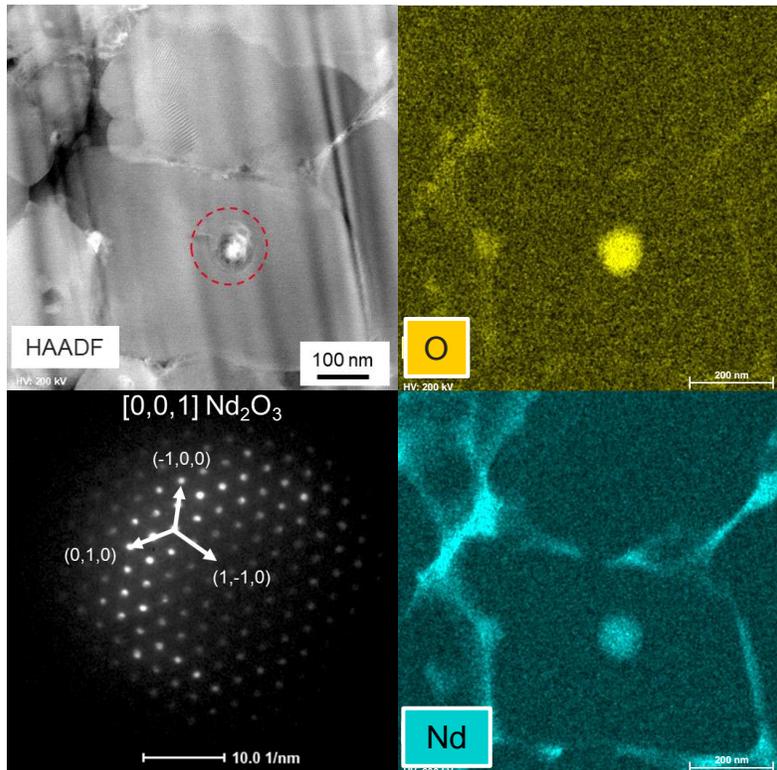
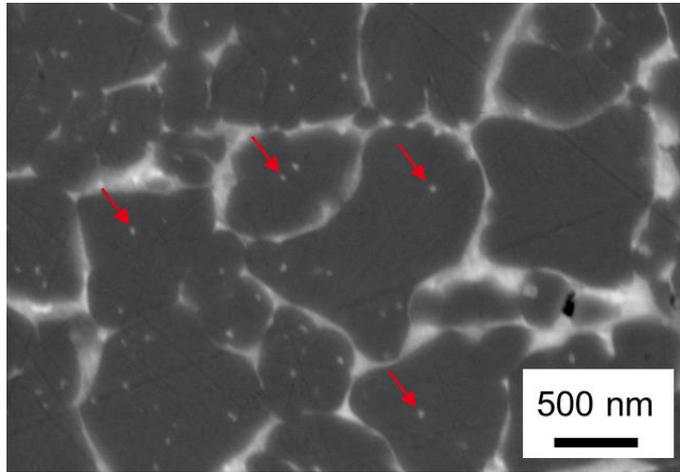


# Nucleation and growth process of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase



- For the formation of **equiaxed grains** by a **heterogeneous nucleation** process, 3 components are needed :
  - Seeding/Nucleating particles** already in solid state in the melt
  - Sufficient **undercooling** to initiate the nucleation
  - Enough time to allow the equiaxed grains to grow and to block the columnar front

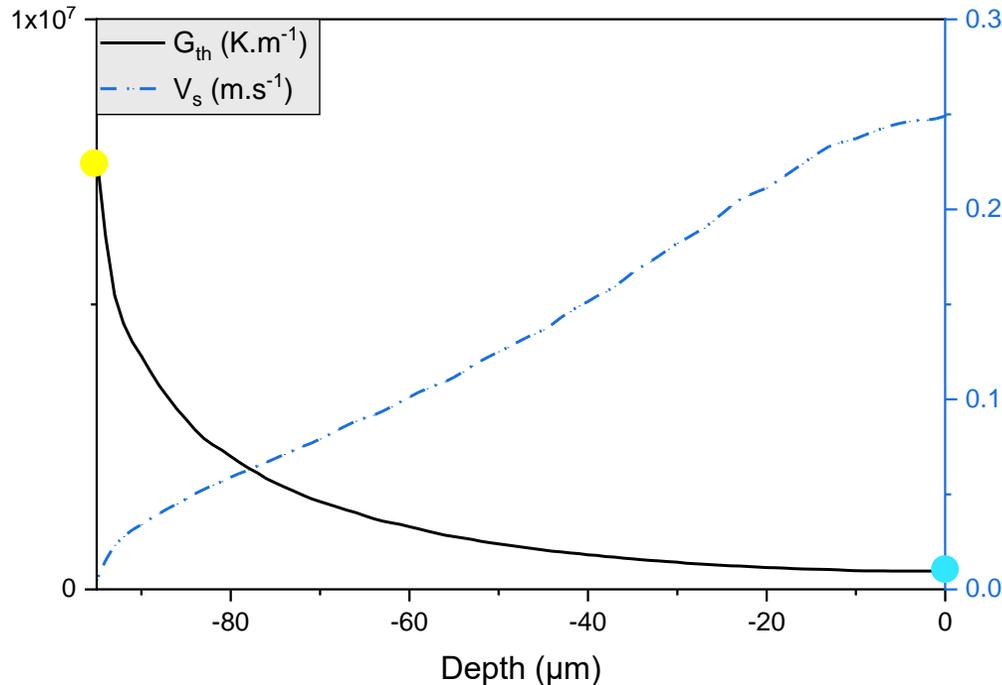
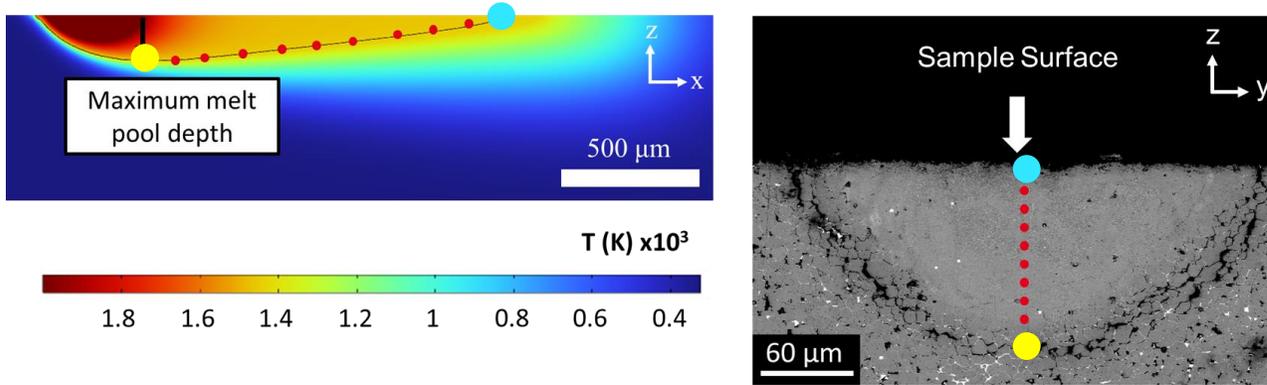
# Intragrain Nd oxide inclusions



% at	O	Fe	RE	Co,Cu,Ga,Al
Point 1	57,6	1,6	40,9	1,4
Point 2	19,2	53,3	24,5	3

- Nd oxide inclusions as the only intragranular phase
- Size of  $\approx 90$  nm for this case (Typically 30 to 100 nm)
- $\text{Nd}_2\text{O}_3$  hexagonal structure

# Kinetic aspect of the growth process



*Determination of cooling conditions by Finite Elements simulation*

- The growth of the equiaxed grains depends on the local cooling conditions. The following relationship can be established in LPBF [2]:

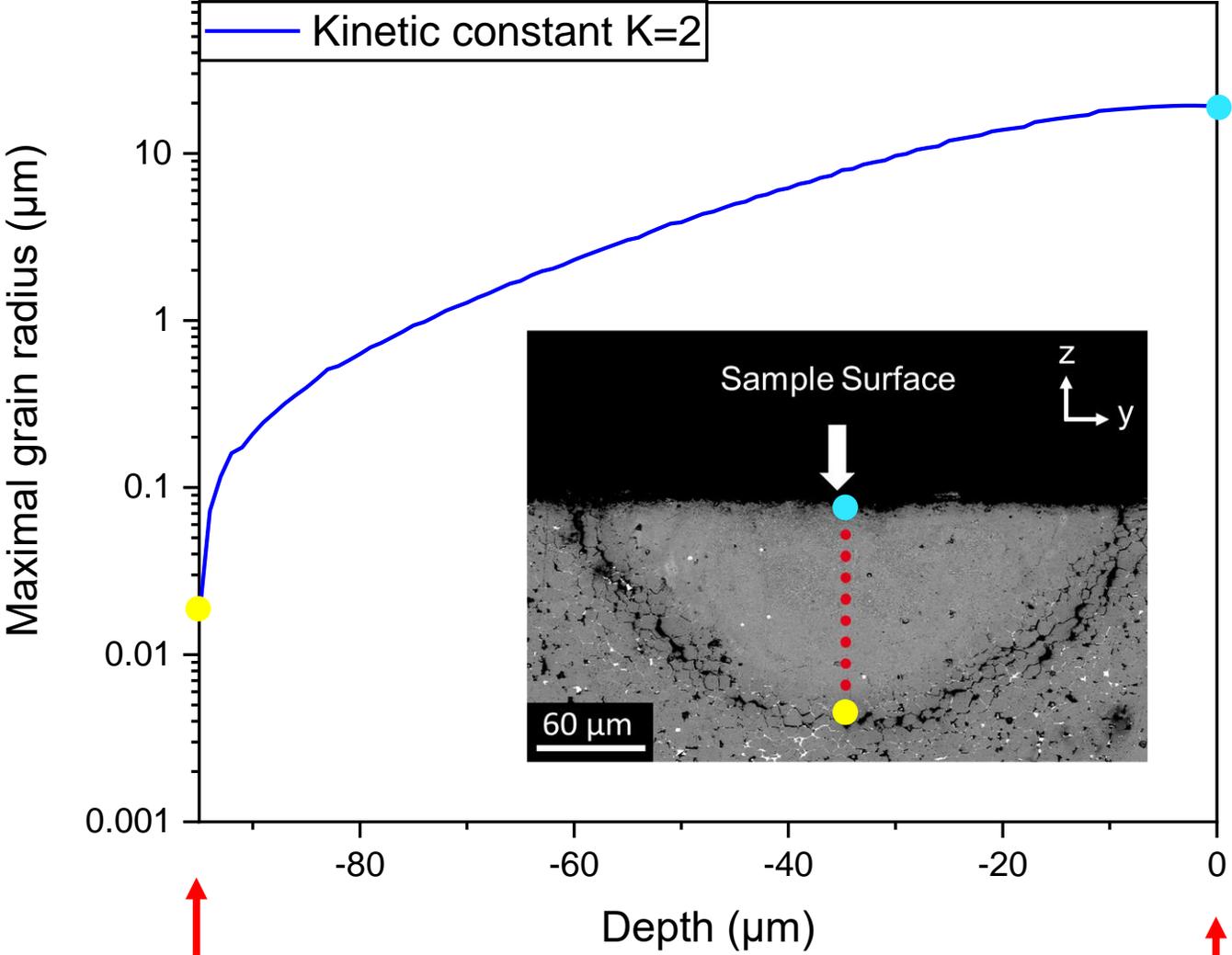
$$R_{max} = f(V_s, G_{th}) = \frac{V_s}{2 \times G_{th} \times K}$$

With  $K$  a kinetic constant ( $\text{cm}\cdot\text{K}^{-1}\cdot\text{s}^{-1}$ ),  $V_s$ ,  $G_{th}$

- $V_s$  (solidification velocity) and  $G_{th}$  (thermal gradient) can be estimated thanks to Finite Elements thermal model developed using Comsol Multiphysics software [3].
- Thermal cooling conditions have been extracted vertically at the center of a melt pool

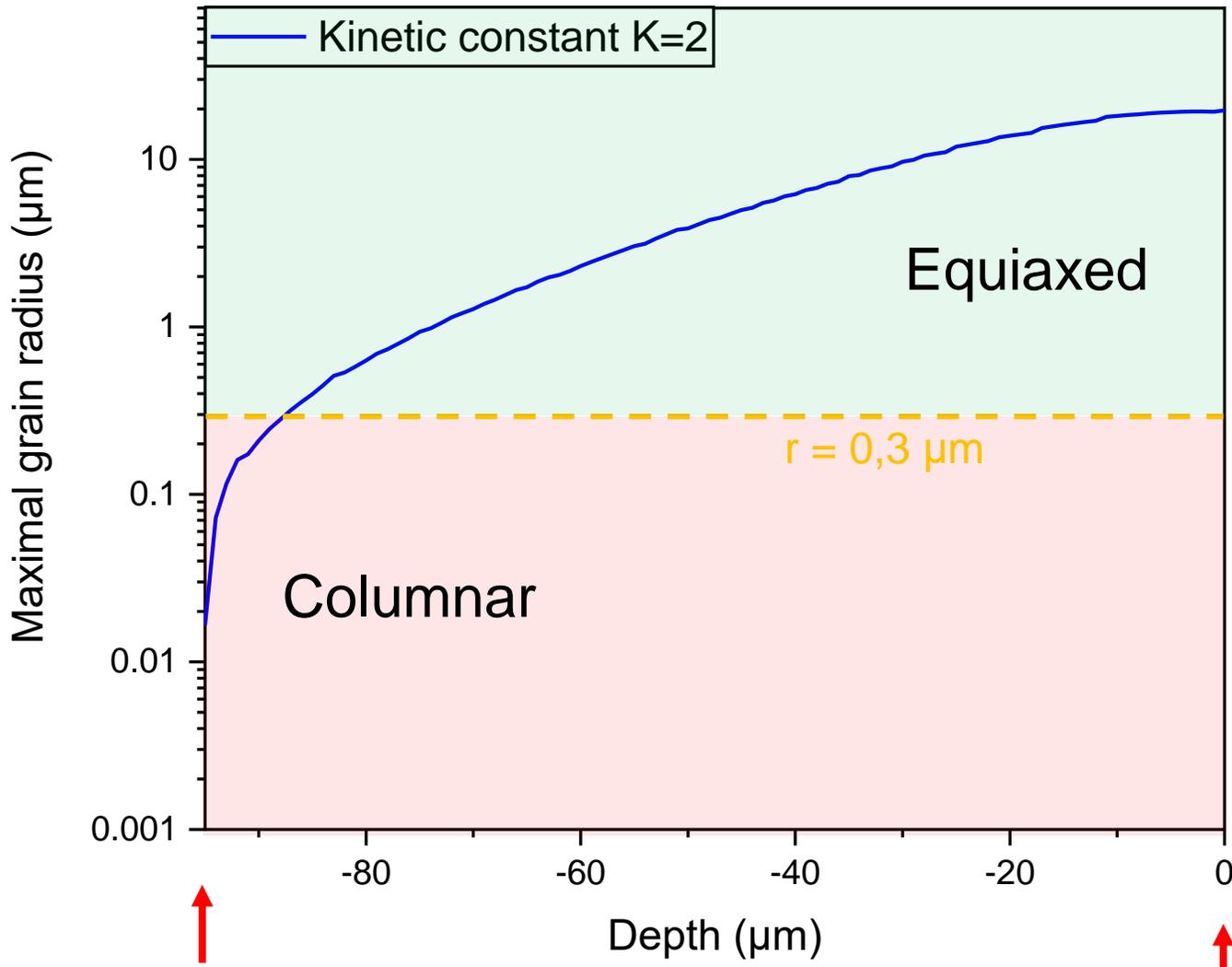
[3] **A. Wolz**, R. Caniou, O. Tosoni, C. Rado, et J.-P. Garandet, « On the Effect of Cooling Parameters on Solidification Structure in NdFeB Alloys », *Advanced Engineering Materials*, vol. n/a, n° n/a, p. 2400978, doi: 10.1002/adem.202400978.

# Evaluation of the grain growth



- We've been able to represent the maximum equiaxed grain size possible depending on the vertical location inside of the melt pool

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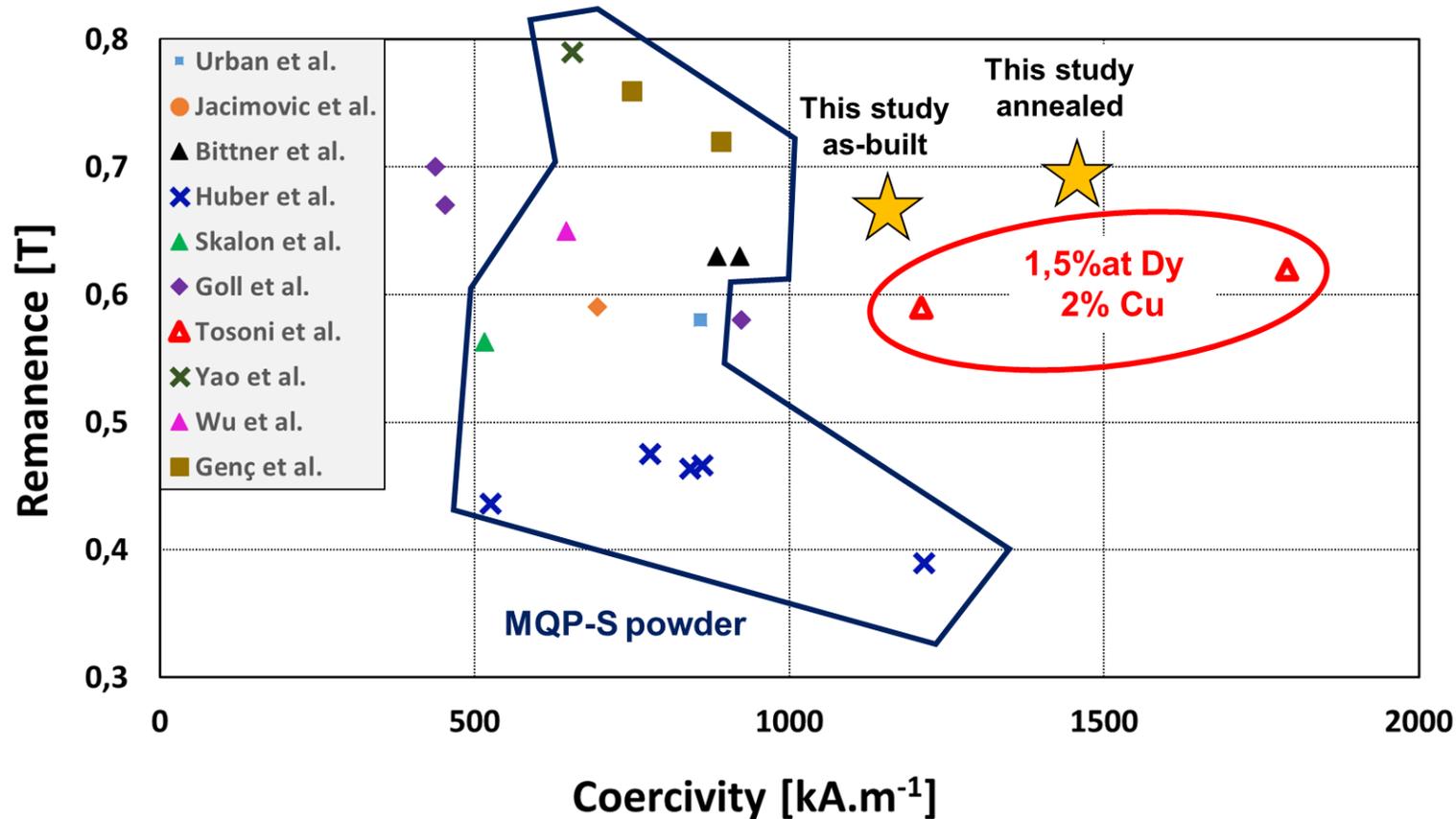
- We've been able to represent the maximum equiaxed grain size possible depending on the vertical location inside of the melt pool
- We placed the critical value to reach to block the columnar front ( $r=300 \text{ nm}$ ) which is the size of the effective equiaxed grains observed in the samples
- This confirm that in the edges of the melt pool the microstructure is columnar, and a transition to an equiaxed structure occurs after a few microns.

# Conclusion

01

## MAGNETIC PROPERTIES

- High coercivity samples compared to the L-PBF state of the art, up to  $1440 \text{ kA}\cdot\text{m}^{-1}$ .
- Nearly isotropic samples and remanence still limited ( $<0,7 \text{ T}$ )
- Annealing heat treatments at  $600^\circ\text{C}+470^\circ\text{C}$  strongly improve the coercivity



# Conclusion

01

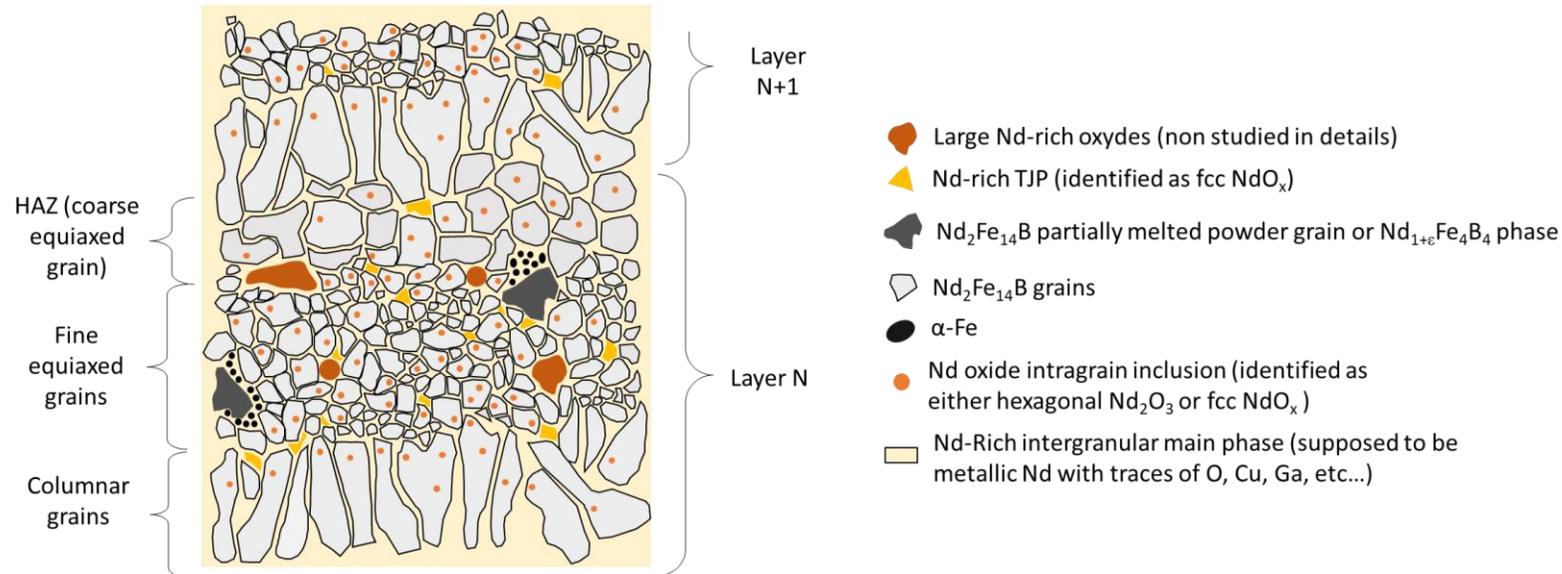
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## MICROSTRUCTURAL ANALYSIS [4]

- A mainly **sub-micronic equiaxed Nd<sub>2</sub>Fe<sub>14</sub>B grains** microstructure was obtained.
- The grain boundaries are mainly composed of Nd-rich phase
- Isolated punctual phases like Nd oxides,  $\alpha$ -Fe, Nd<sub>1+ $\epsilon$</sub> Fe<sub>4</sub>B<sub>4</sub>



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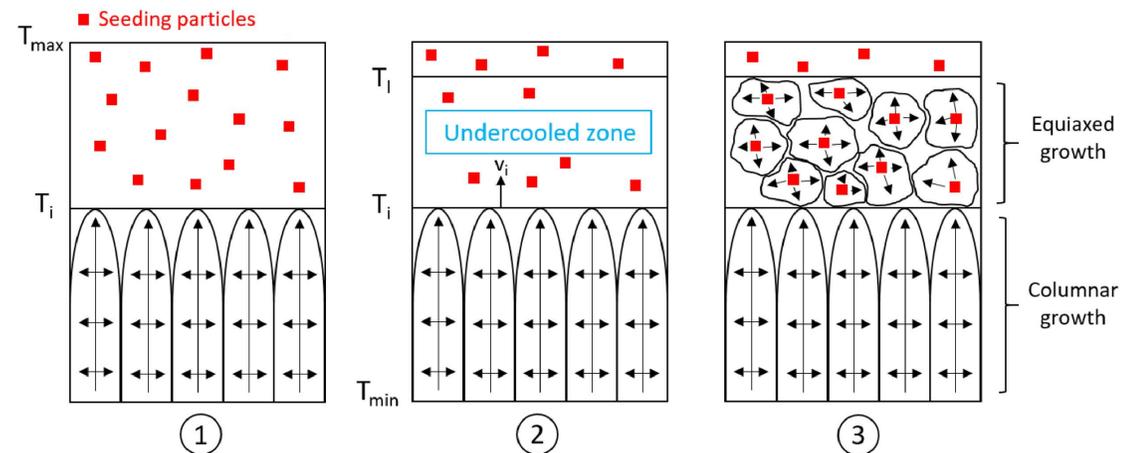
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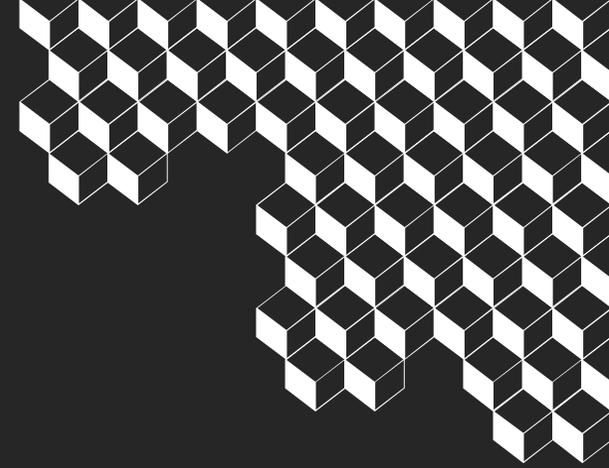
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03

## FORMATION OF THE MICROSTRUCTURE

- Systematic **Nd-oxide** inclusions (30 to 100 nm) appear as **likely candidates** to play the role of **nucleating agent**.
- A **simple kinetic model** can explain the presence of both equiaxed and columnar grain inside of the samples.





**Many thanks for your  
attention**

*Feel free to ask questions !*

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