



liten

Low temperature densification of $\text{Nd}(\text{Fe},\text{Mo})_{12}$ nitrided powders

Gabriel Gomez Eslava¹, Patricia de Rango² and Sorana Luca¹

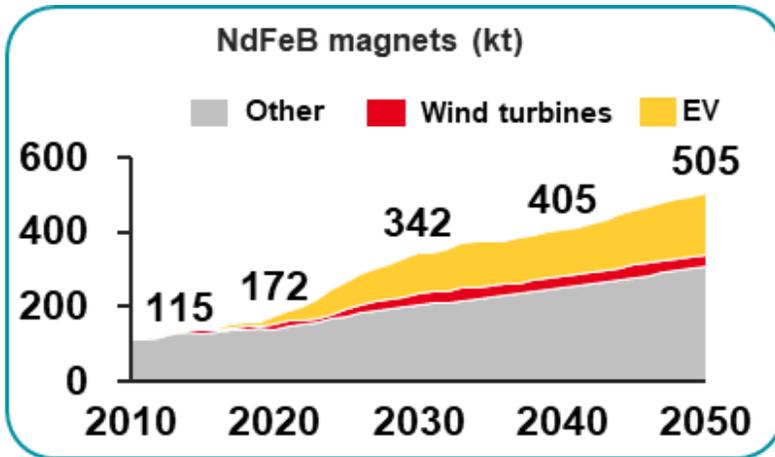
¹Univ. Grenoble Alpes, CEA, Liten, DTNM, 38000 Grenoble, France

²Univ. Grenoble Alpes, CNRS, Institut Neel, 38000 Grenoble, France



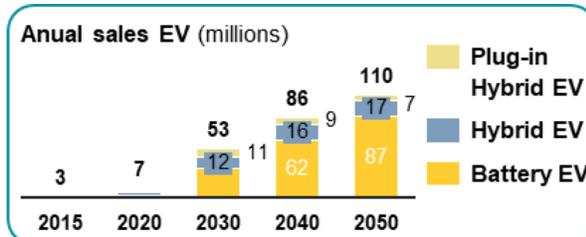
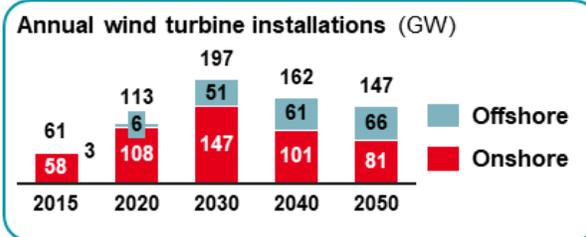
Context

- NdFeB are strategic materials for our future society and especially for the green energy transition
- E-mobility and wind turbines are expected to be the key driver of rare-earth permanent magnets demand growth over the future



To mitigate the risk of shortages and ensure future supplies in permanent magnets (PM) -> big challenges for our society:

- Design of components to reduce the weight of PM
- Reduce the waste of critical material by net shape process
- Efficient recycling
- **Searching RE-free or RE-lean permanent magnets capable to replace the benchmark NdFeB magnets**

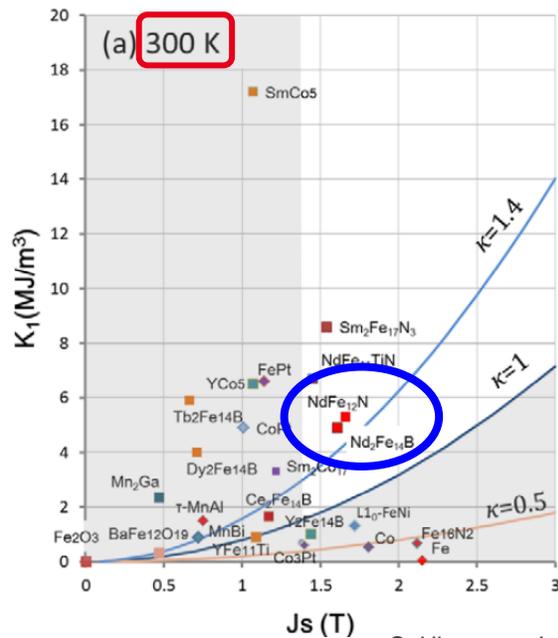


Context

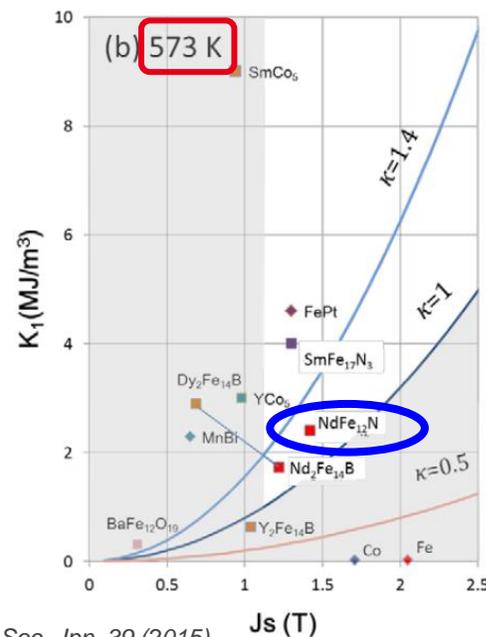
Searching RE-free or RE-lean permanent magnets capable to replace the benchmark NdFeB magnets

New material should fulfil the following conditions:

- Sufficient magnetic properties at room temperature and high temperatures → **Magnetic hardness factor $K = (\mu_0 H_A / 2J_s)^{1/2} > 1$**
- Allow an efficient economy of critical material = energy efficiency (**RE economy vs available magnetic performances**)



S. Hirosawa, J. Magn. Soc. Jpn. 39 (2015)



Nd₂Fe₁₄B
11.8 at% RE

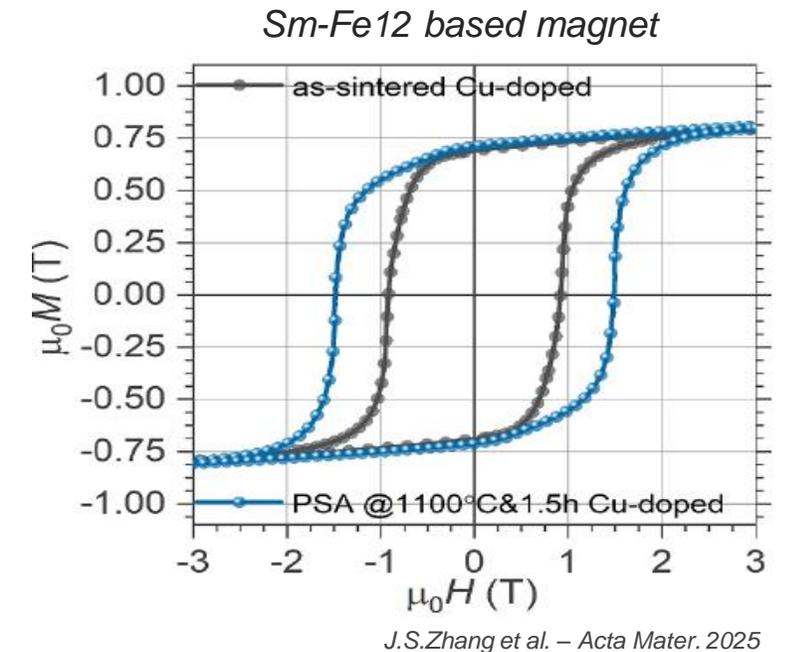
vs

RE-Fe₁₂
7.7 at% RE

30% RE economy
K > 1 from RT up to 200°C

Potential of the RE-Fe₁₂ magnets compared to the NdFeB – Performance Factor

- Dense Sm-Fe₁₂ based magnets have been recently reported in the literature, with coercivity as high as 1.5T and remanence of 0.71T (*J.S. Zhang et al., Acta Mater. 2025*)
- Nd-Fe₁₂ based compounds suffer from low magnetocrystalline anisotropy at room temperature, that can be enhanced by the insertion of light atoms (N) into the 1-12 structure → densification of the nitrogenated powders proves to be difficult and up to now no dense Nd-Fe₁₂ based magnet has been reported



Potential of the RE-Fe12 magnets compared to the NdFeB – Performance Factor

- To evaluate the potential of these magnets in a system, compared to the NdFeB, the energy efficiency should be estimated
- **Performance Factor (PF) = available magnetic performances (BHmax)/ RE content**

$$PF(\text{arb. unit}) = \frac{BH_{max} \text{ (kJ/m}^3\text{)}}{x_R \text{ (wt. \%)}}$$

Here x_R = RE % wt

When values of BH_{max} and M_r are not measured, they are estimated using the formulas below:

$$BH_{max} = \mu_0 M_r^2 / 4$$

$$M_r = 0,9 * M_s$$

Potential of the RE-Fe12 magnets compared to the NdFeB – Performance Factor

	$\mu_0 M_s$ (T)		$\mu_0 M_r = 0.9 \mu_0 M_s$ (T)		PF	
	25	180	25	180	25	180
Temp. (°C)	25	180	25	180	25	180
N50 NdFeB	1.6	1.3	1.42	1.15	13.3	10.8
NdFe_{10.5}Mo_{1.5}N [1]	1.1	0.9	1*	0.81*	14.6	12.0
Sm ₈ Fe _{73.5} Ti ₈ V ₈ Al ₂ Cu _{0.5} [2]	0.74	0,42	0.62	0.45	9.6	6.9
Sm ₈ Fe _{76.5} Ti ₅ V ₈ Al ₂ Cu _{0.5} [3]	0.81	-	0.71		10.9	

[1] S. Luca et al., JALCOM 2025

[2] A. Srinithi et al., Acta Mater. 2023

[3] J.S.Zhang et al., Acta Mater. 2025

$$PF(\text{arb. unit}) = \frac{BH_{max} \text{ (kJ/m}^3\text{)}}{x_R \text{ (wt. \%)}}$$

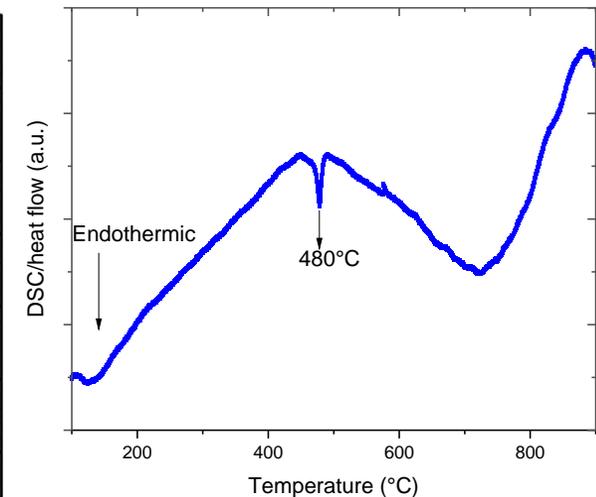
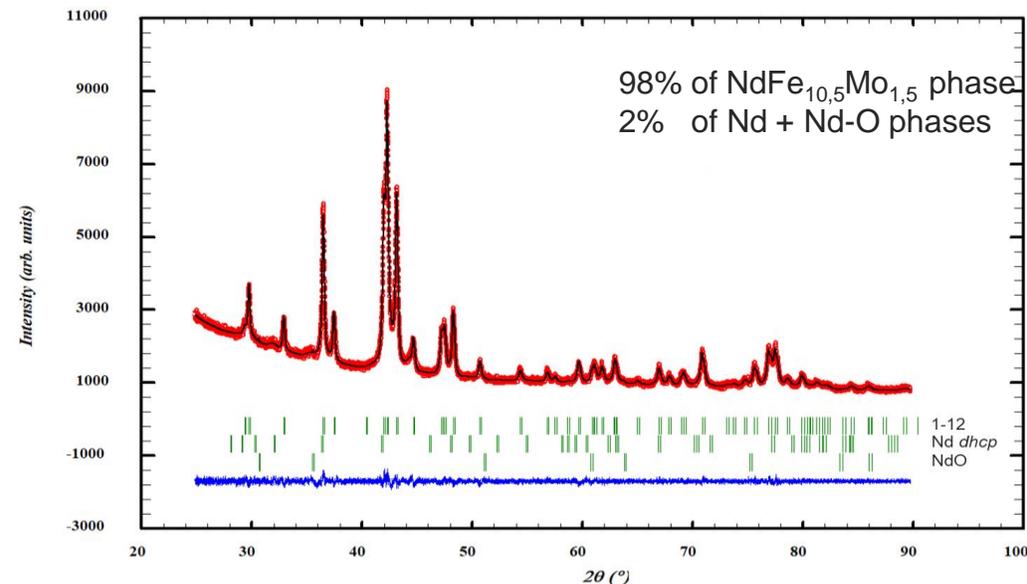
$$BH_{max} = \mu_0 M_r^2 / 4$$

*Represents calculated values using:
 $M_r = 0,9 * M_s$ and $M_s = \text{measured value}$

- ⇒ The potential of the Sm-Fe12 based magnets is hindered by the low remanence values → the challenge is to enhance the Br values
- ⇒ **Great potential of the Nd(Fe,Mo)₁₂N based magnets** for PM applications for temperatures up to 180°C (economy of approximately 10% of RE for the same BH_{max}) **without using HRE elements**
- ⇒ **However, the densification of the nitrogenated powders** is the biggest challenge

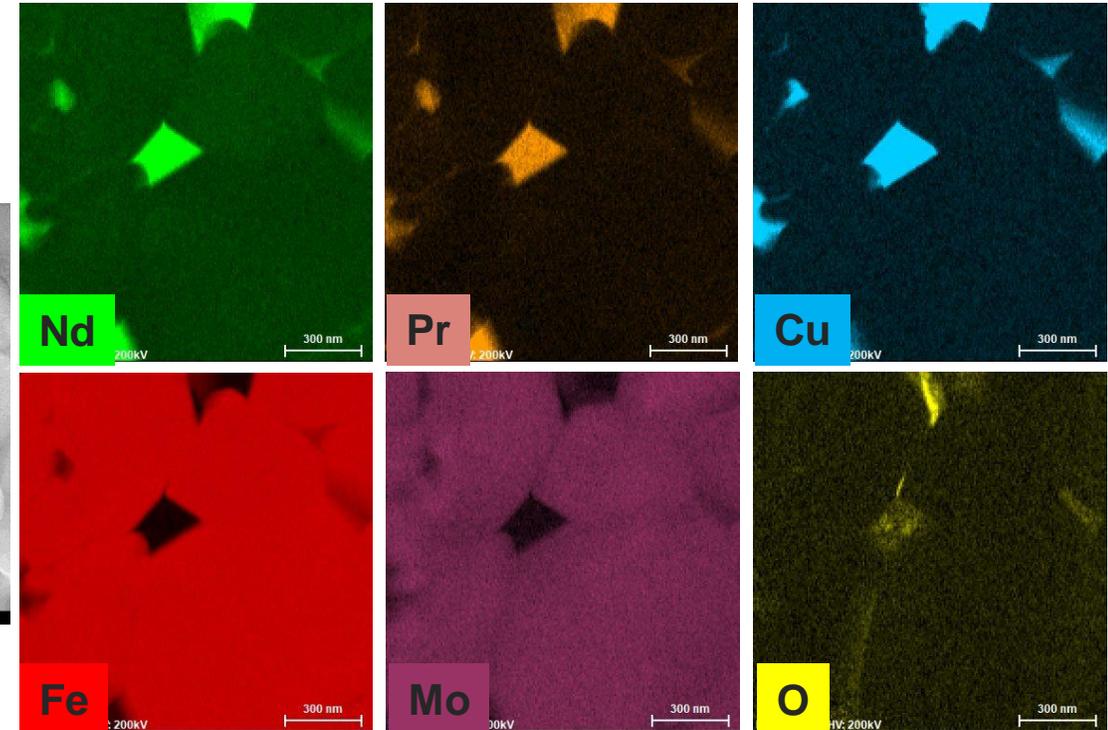
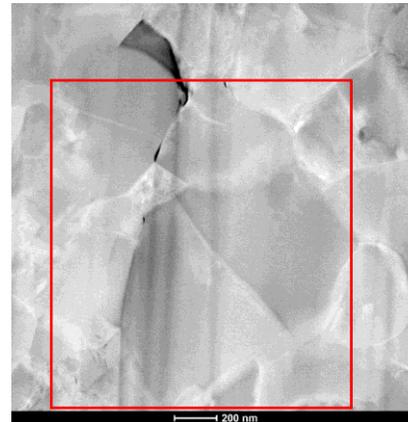
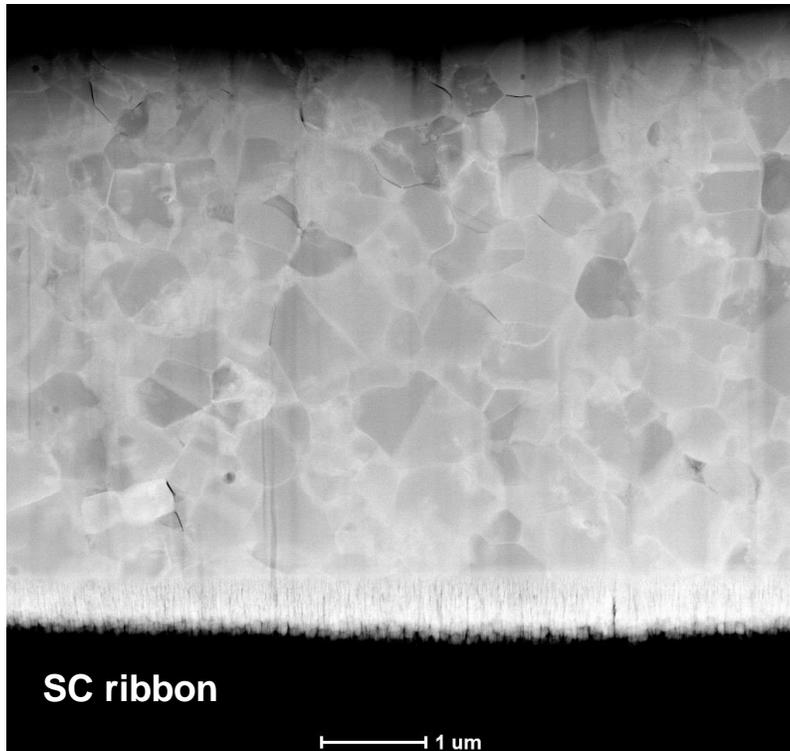
Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders

- $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders are not stable at high temperatures ($> 600^\circ\text{C}$) \rightarrow conventional sintering cannot be applied
- **Low melting temperature phase** at grain boundaries can assist the **densification at low temperatures**
- Elaboration of $(\text{Nd},\text{Pr})_{1.35}\text{Fe}_{10.5}\text{Mo}_{1.5}\text{Cu}_{0.1}$ alloy by SC – almost single 1-12 phase



- Presence of the RE-Cu eutectic
- Nitrogenated powders have: $H_A = 11T$, $M_S = 1.1T$, $T_C = 300^\circ\text{C}$

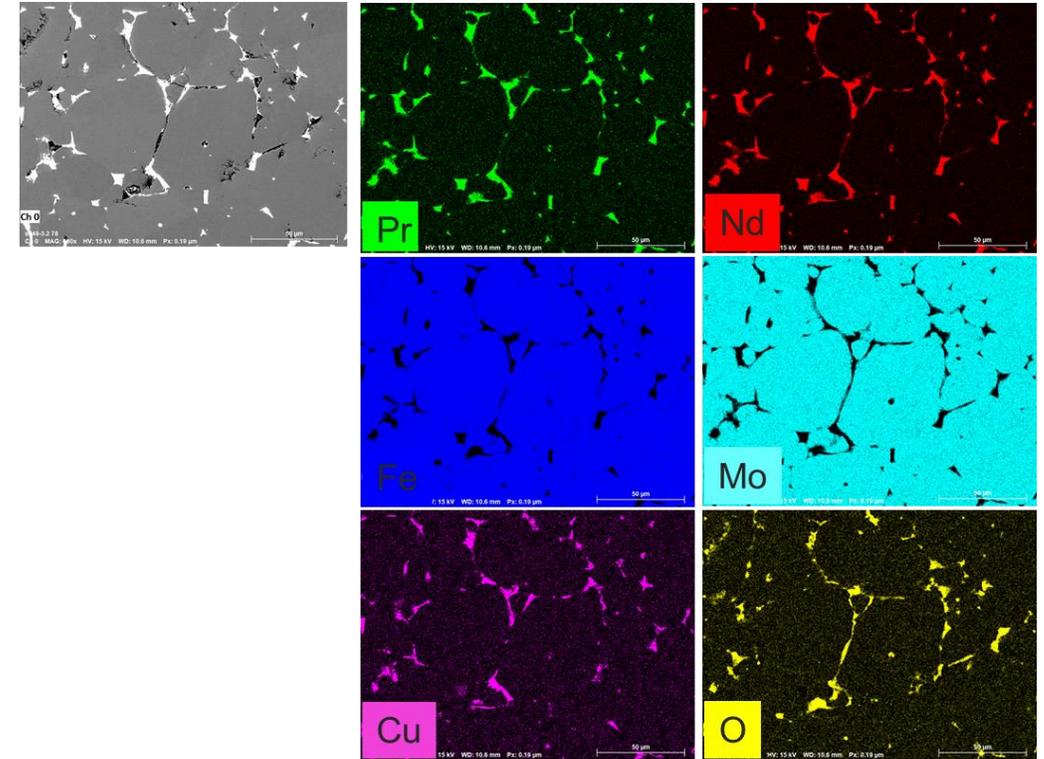
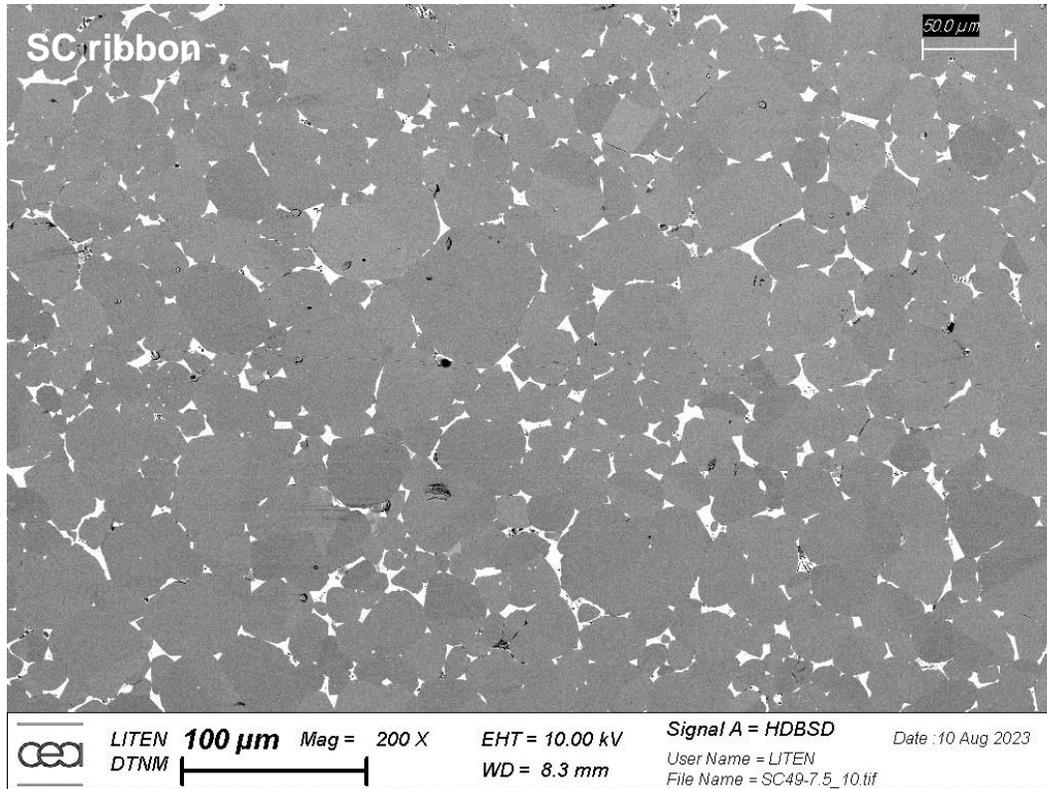
Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders



S. Luca et al., JALCOM (2024)

- Fine microstructure with grains of 100 to 1000 nm
- Cu is present predominantly at triple points together with RE elements
- Well defined RE-Cu phase cannot be observed at GBs → difficult to promote the liquid phase sintering assisted by the low melting eutectic

Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders



- Combination of high temperature + low temperature annealing treatment → better wettability of the RE-Cu phase at grain boundaries
- However, high temperature annealing also induces grain growth → detrimental for coercivity development

Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders

Our approach:

- **Spark Plasma Sintering (SPS)**: allows to densify the powders by simultaneous application of a pressure + temperature, in a closed system under vacuum or N_2 or Ar gas
- **Two powder blending** – assists the densification at low temperatures
- ⇒ Search for **low melting temperature compounds** fulfilling the following conditions:
 - Melting temperature $< 600^\circ\text{C}$
 - The constituents are not soluble into the 1-12 phase
 - Paramagnetic phase -> allow to develop the coercivity

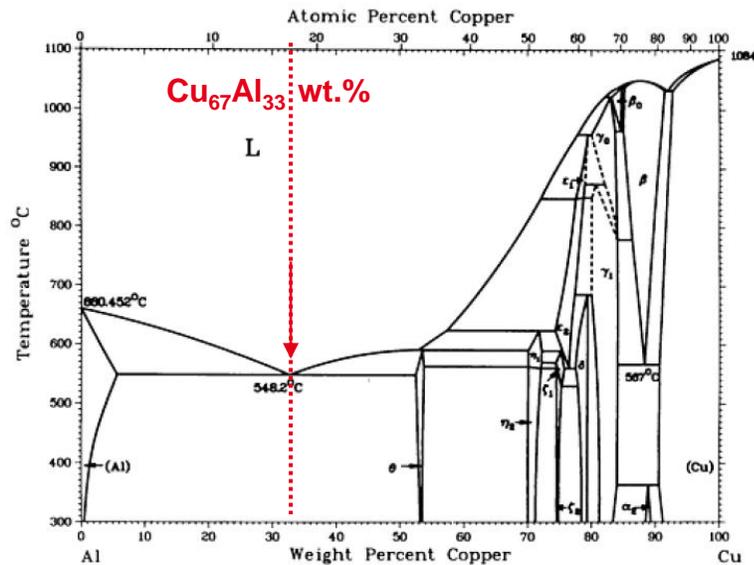


Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders

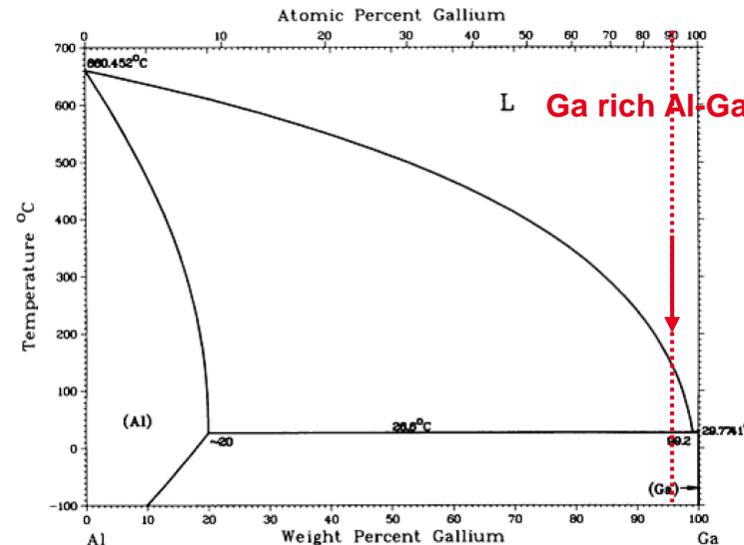
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$T_{\text{melt}} = 548.2^\circ\text{C}$

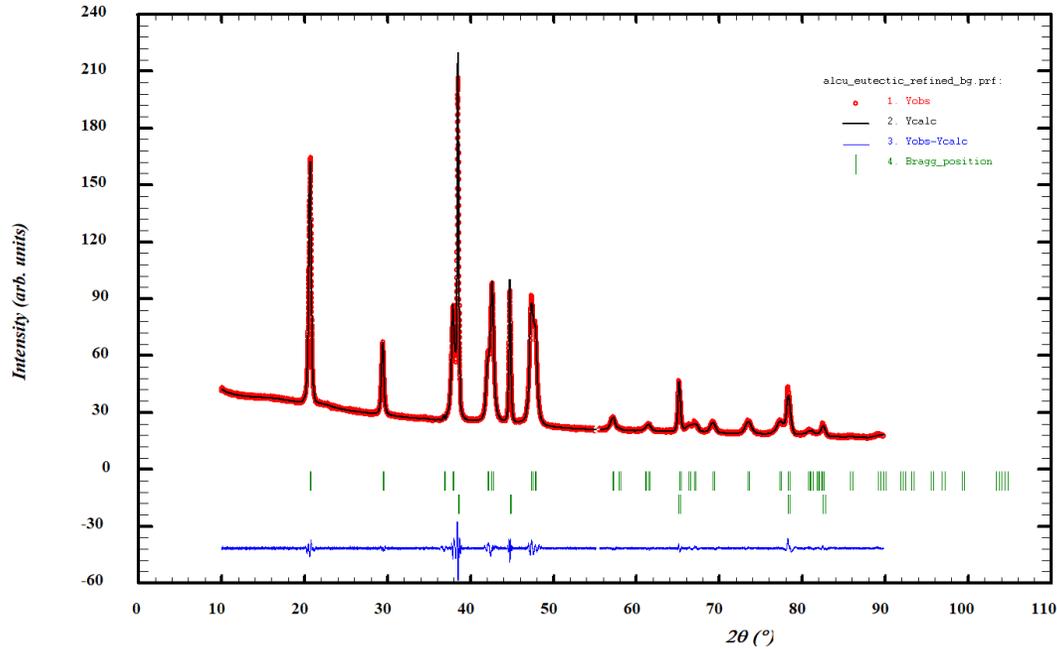


liq > 200°C

- **Al, Ga** can stabilize the 1-12 structure
- **Cu** – do not stabilize the 1-12 structure
- easier to manipulate

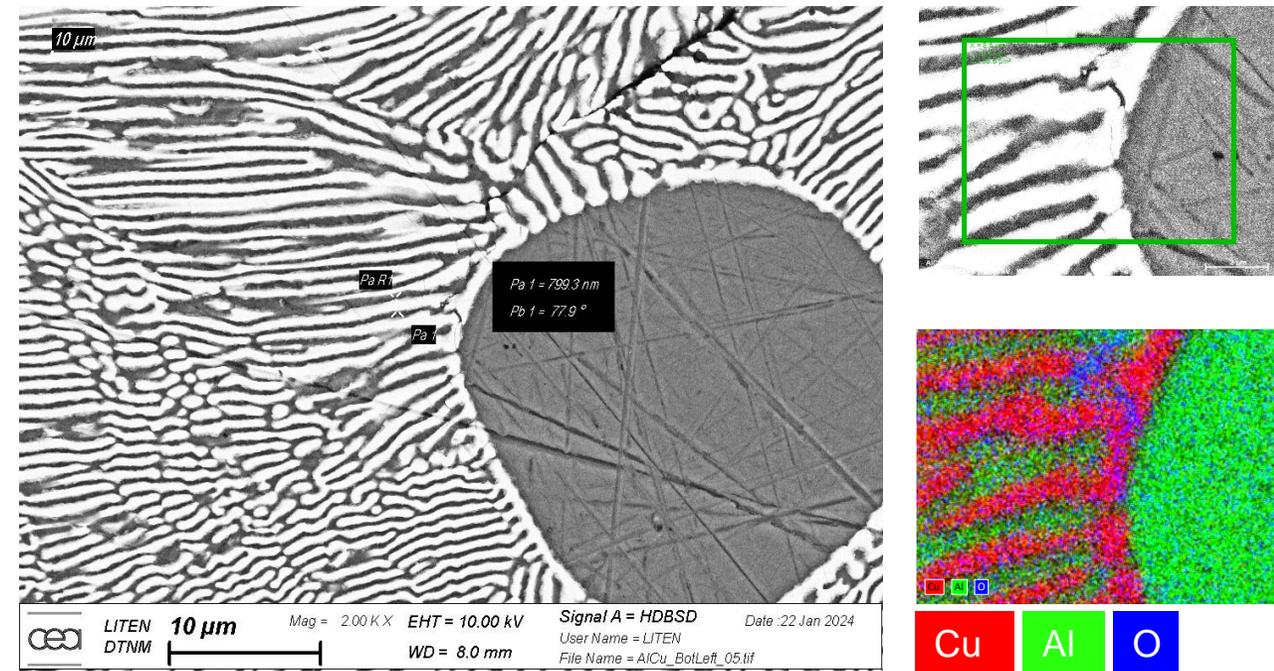
Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders

Al₆₇Cu₃₃ wt% Chi2: 0.0108



Phase	Space Group	Lattice parameters (nm)	%
Al ₂ Cu	I 4/m c m	a=b=6.066 c=4.880	54
Al	F m 3 m	a=4.047	46

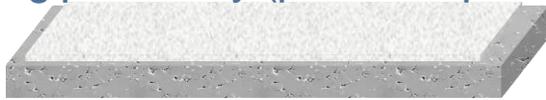
- Al₆₇Cu₃₃ alloy fabricated by melting the pure elements in an induction furnace



Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders

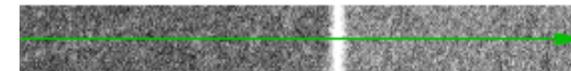
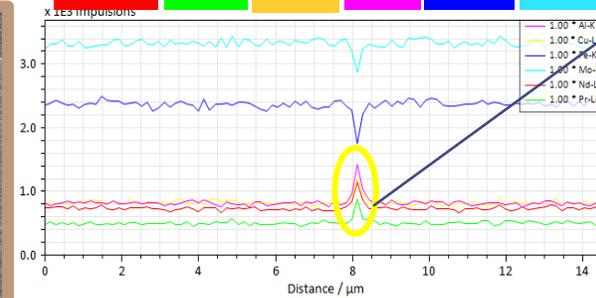
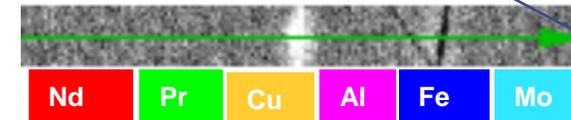
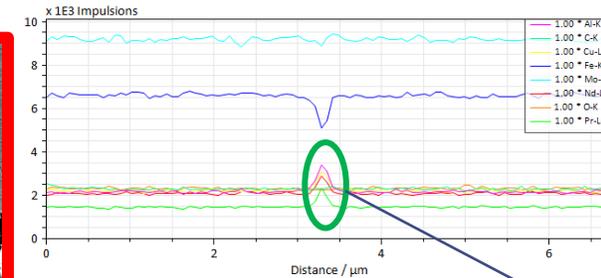
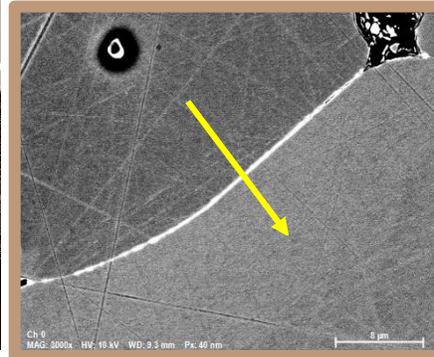
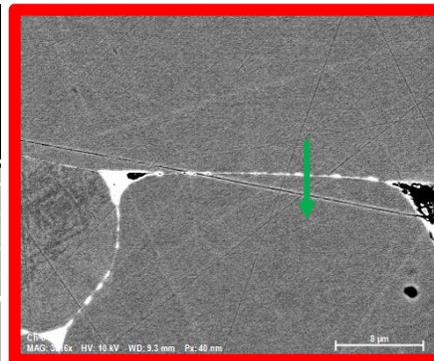
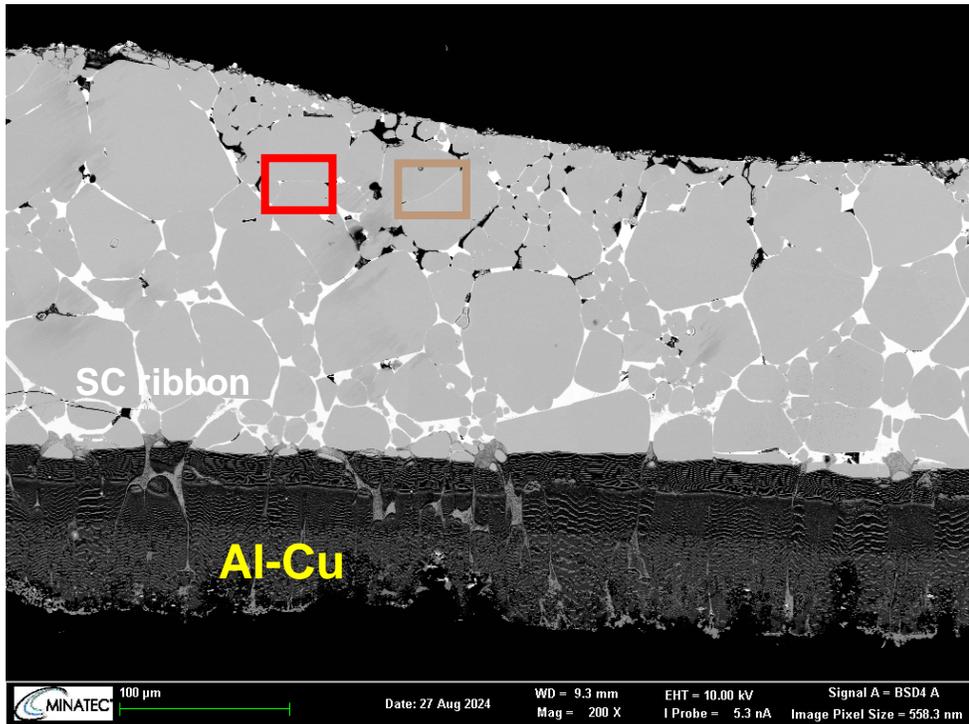
Test of the ability of the eutectic alloy to form GB phases – infiltration experiment on a SC ribbon

Low melting point alloy (pieces or powder).



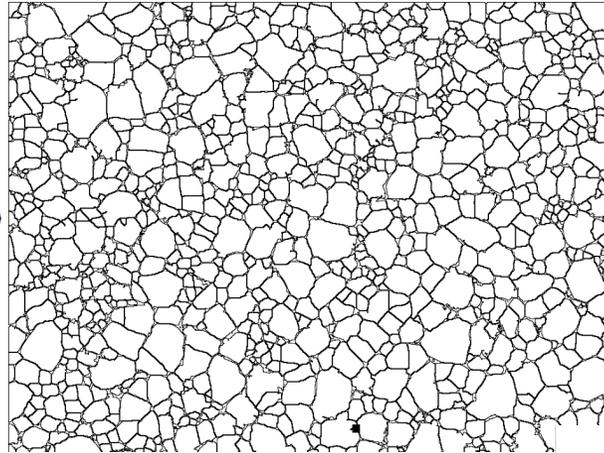
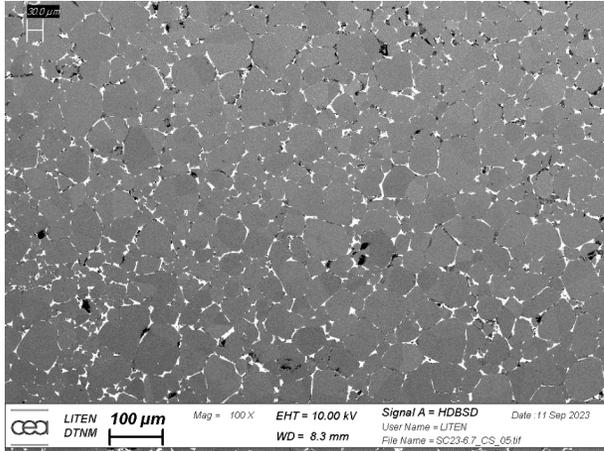
Low temperature heat treatment ($< 600^\circ\text{C}$)

Annealed $\text{Nd}(\text{FeMo})_{12}$ flakes with optimized μ -structure



RE-Al-Cu rich phase with low Fe content

Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders



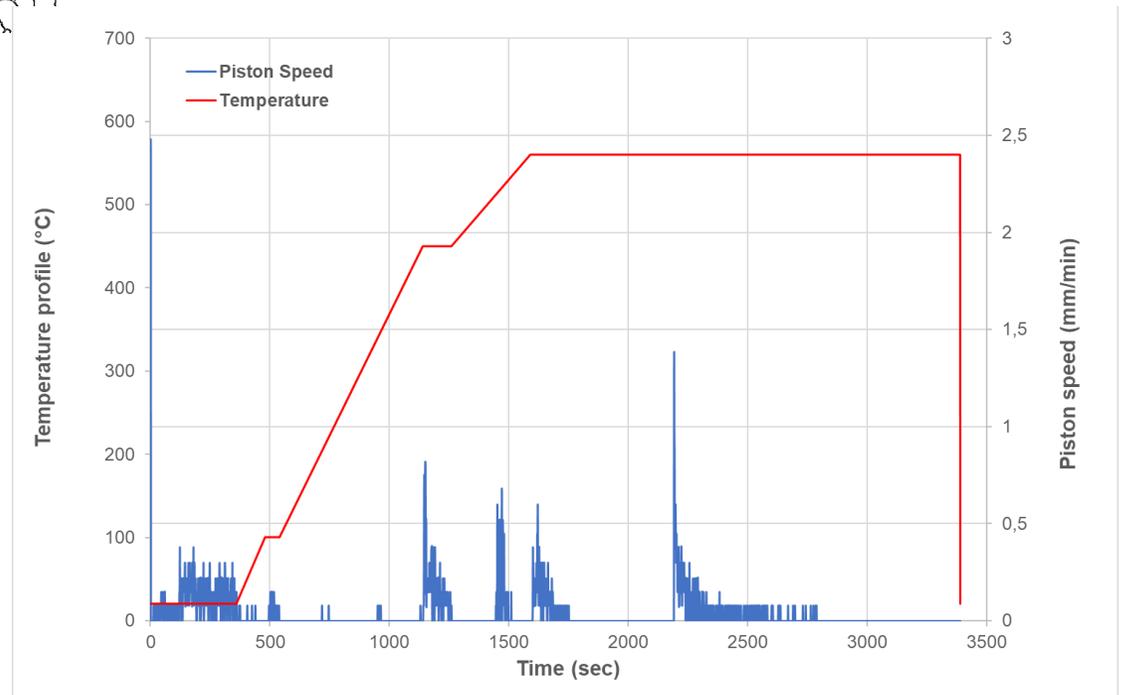
SEM image analysis \rightarrow grains / GB = 80/20 % vol



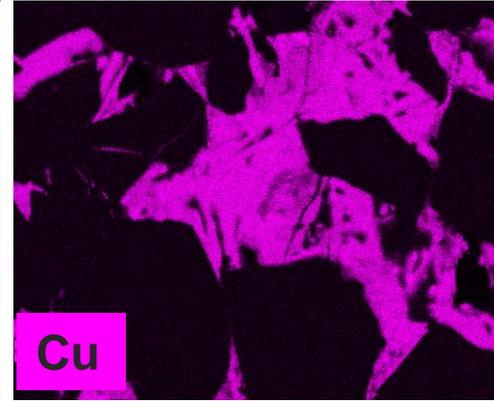
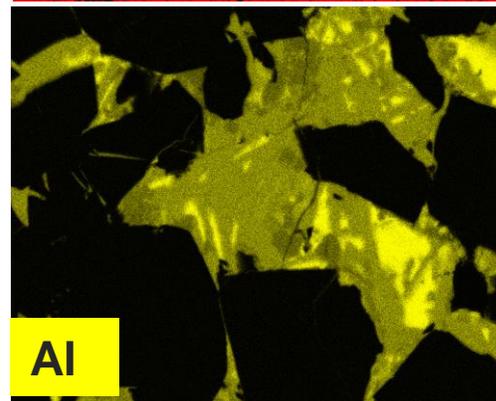
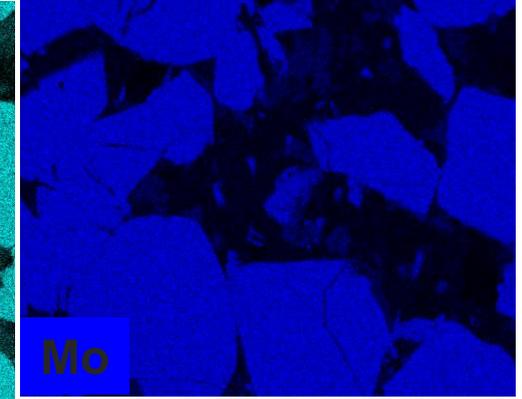
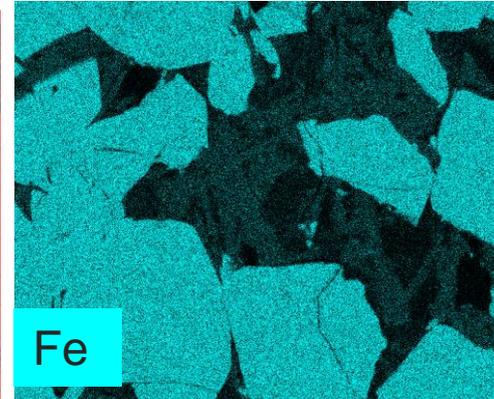
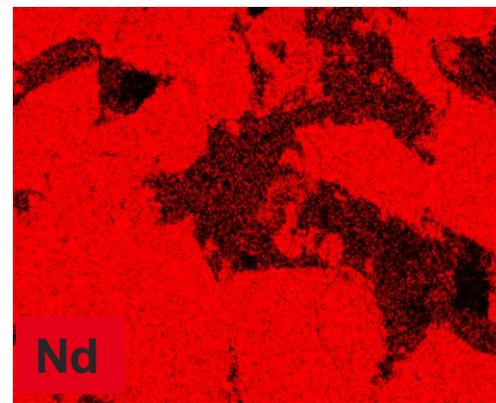
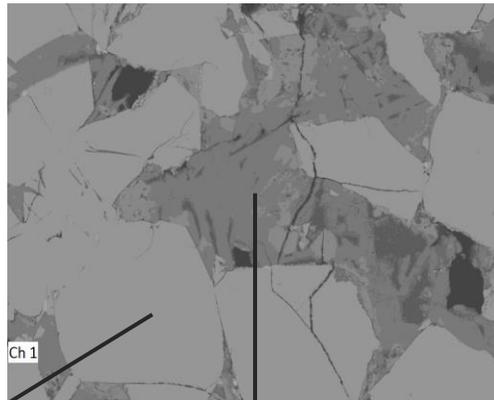
75% vol. of jet mill $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{Cu}_{0.1}\text{N}_x$ + 25% vol. mechanical milled AlCu



- T = **560°C**, under **600 MPa**
- Measured density = $6,69 \text{ g/cm}^3$ representing **94.8%** of the theoretical density



Densification of the $\text{Nd}(\text{Fe},\text{Mo})_{12}\text{N}$ powders



1-12 grain

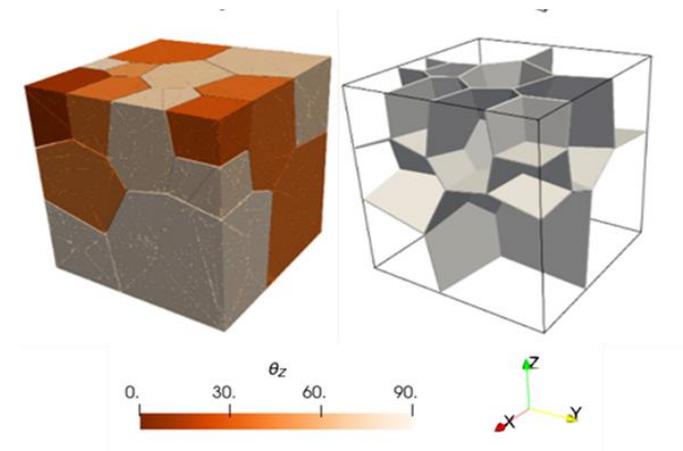
Al-Cu

- Grains of nitrogenated 1-12 phase are separated by Al-Cu phase, which is spread out around the magnetic grains → it seems that the **low melting eutectic assisted the densification of the nitrogenated powders**

Conclusion

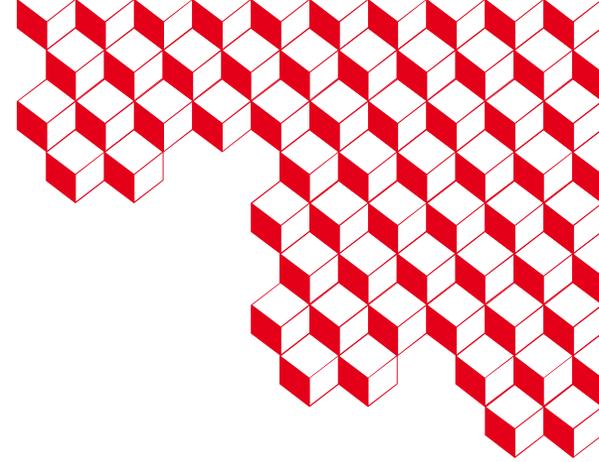
- The RE-Fe₁₂ compounds are very promising material for next generation PM
- **Nd(Fe,Mo)₁₂Cu compound** has a great potential for future permanent magnet
 - High magnetocrystalline anisotropy (11T)
 - High saturation magnetization (1.1T)
 - Moderate Curie temperature (300°C)
- The big challenge is to **densify the nitrogenated powders** under the decomposition temperature of the nitride (< 600°C)
- **Powder blend method + SPS** have been applied in order to densify the powders at 560°C → **density of 95%**, without the decomposition of the nitride
- Fe-lean RE-Al-Cu-rich grain boundaries are formed with an appropriate thermal treatment that can be beneficial for coercivity development

Next challenge: develop the coercive microstructure formed of fine 1-12 grains + non-magnetic grain boundaries





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Thank you

Merci

ありがとう

CEA Liten

38229 Grenoble Cedex

France

sorana.luca@cea.fr

