Influence of Microstructure on the Interphase Exchange Coupling of $Nd_2Fe_{14}B$ + 10 wt%Fe Nanocomposites Obtained by Mechanical Milling

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Abstract: The influence of the microstructure on the magnetic interphase exchange coupling of $Nd_2Fe_{14}B$+10 wt% α-Fe nanocomposites obtained through mechanical milling was studied as a function of the milling energy (shock, friction and the ball impact energy) by varying only the size of the grinding media (steel balls with diameters of 5, 10, 15, and 20 mm). The mean crystallite sizes in both $Nd_2Fe_{14}B$ and α-Fe were studied through X-ray diffraction, by the Scherrer method. The variation in milling energy had little impact on the mean crystallite sizes for α-Fe; approximately 10 nm after annealing at 700 °C for 1.5 min and 20 nm after annealing at 800 °C for 1.5 min. However, after annealing, the $Nd_2Fe_{14}B$ crystallites present average sizes ranging from 50 nm (for the lowest impact energy used) to 20 nm (for high impact energy), with no significant variation due to annealing temperature in the investigate range. Increasing the milling energy does not lower the mean crystallite size beyond 20 nm for the hard magnetic phase. The influence of the microstructure on the magnetic interphase exchange coupling between the hard and soft magnetic phases is reflected in the demagnetization curves and dM/dH vs. H plots for the annealed samples. The samples in which the $Nd_2Fe_{14}B$ phase presents lower mean crystallite sizes show an improvement in both coercivity and energy product over the samples with larger grains. Further improvement in the magnetic interphase coupling can be observed as the average crystallite sizes decrease to 20 nm, for which the highest values for the coercive field H_c = 0.65 T and energy product $(BH)_{max}$ = 110 KJ/m³ were recorded.

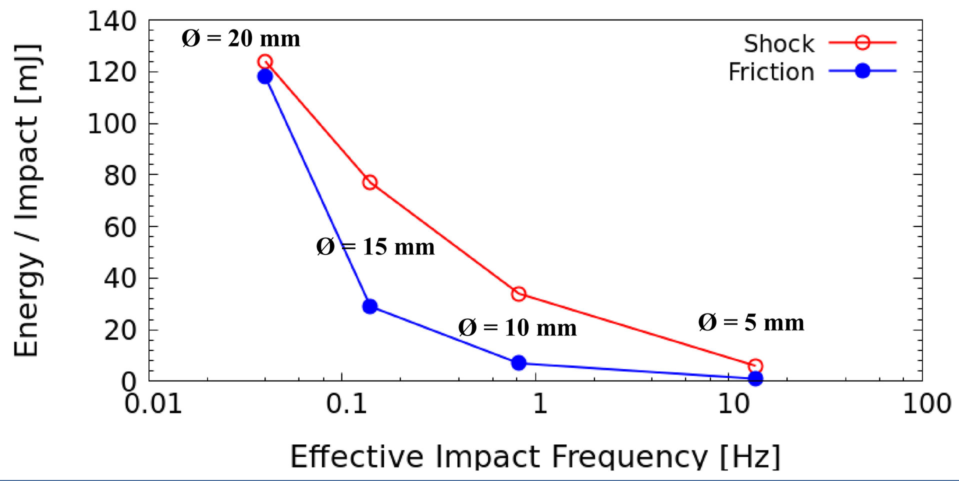
Experimental Details:

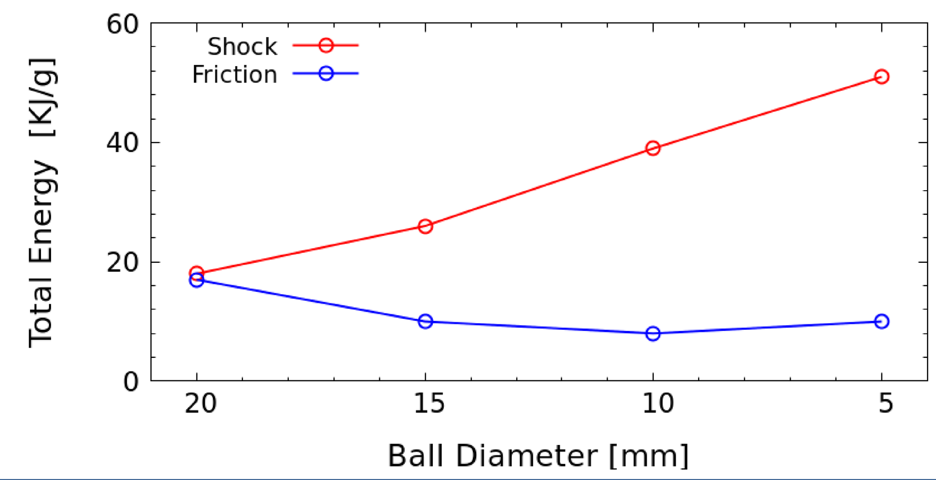
- The $Nd_2Fe_{14}B$ phase was prepared : arc melting $Fe_{14}B$ → induction melting $Fe_{14}B$ + Nd (under purified Ar gas) → annealeling in vacuum for 72 h at 950 °C. → ground to a fine powder under 500 µm.
- The soft magnetic phase consisted of NC 100.24 commercial Fe powder Höganäs product.
- Nanocomposite synthesis: starting powder 90wt% $Nd_2Fe_{14}B$ + 10wt%Fe → 6h MM → Short Time Annealing 1.5 min
- The milling energies and impact frequencies were calculated by simulating the milling process [1, 2]
- X-Ray diffraction measurements were performed on a Brüker D8 Advance diffractometer using Cu - Kα radiation.
- Scanning electron microscopy (SEM) studies were performed on a Hitachi SU8230 device.
- Magnetic measurements were carried out on powder fixed in epoxy resin at 300 K in applied fields up to 10 T.
- Internal Field: demagnetization factor of 1/3

Results and Discussions:

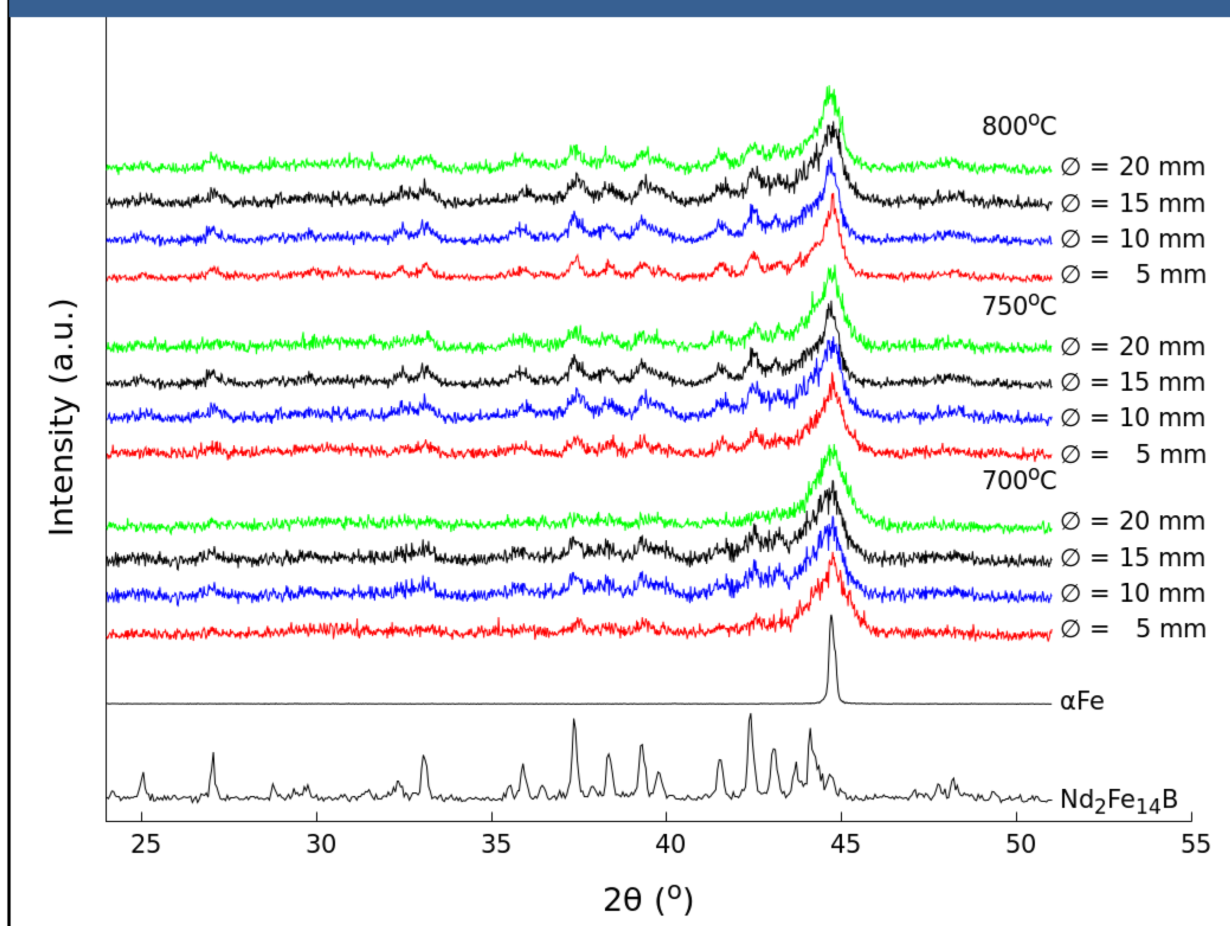
Calculations

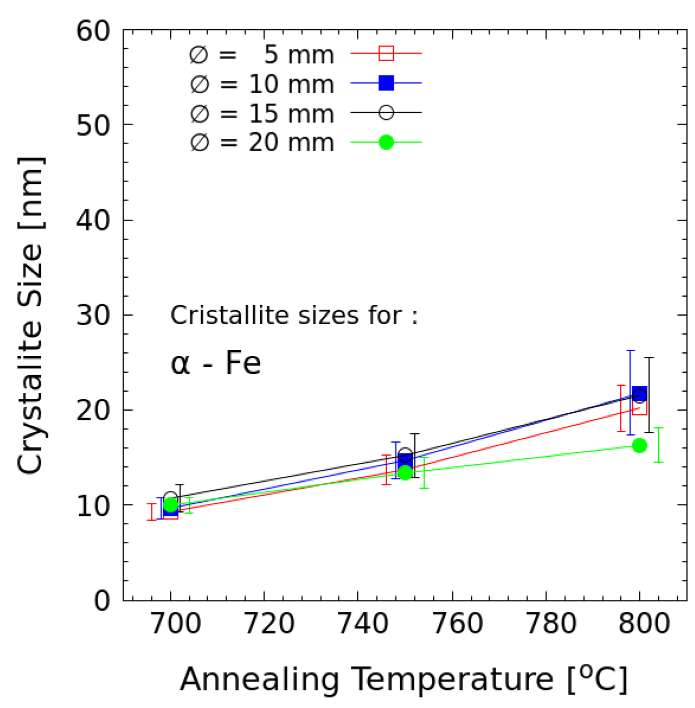
Ball diameter [mm]	Number of Balls
5	209
10	26
15	9
20	4

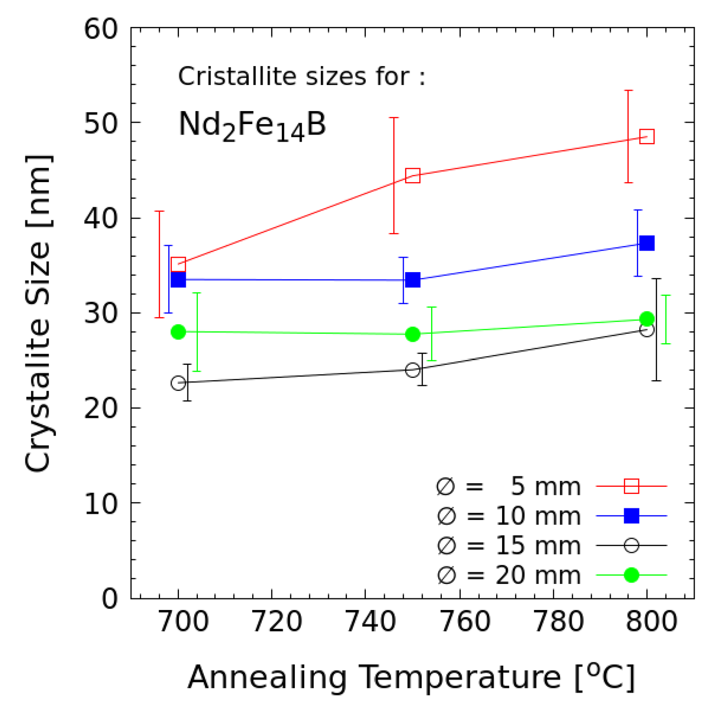


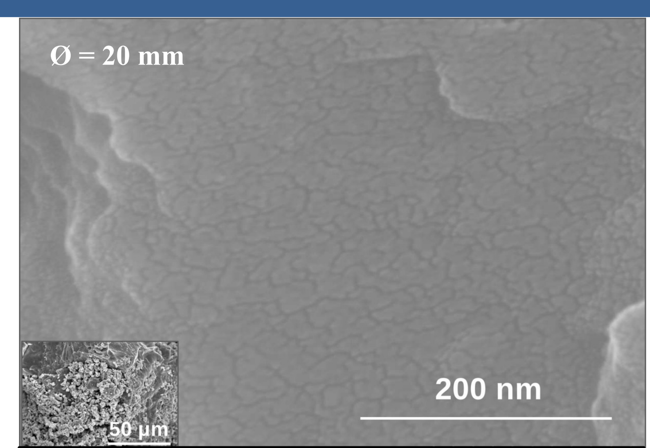


Structure and Microstructure

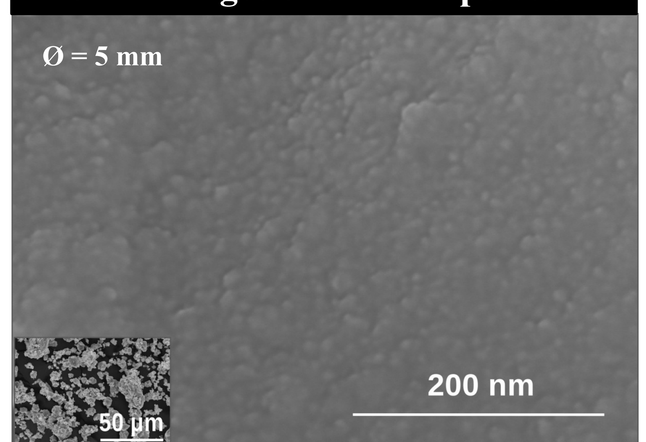






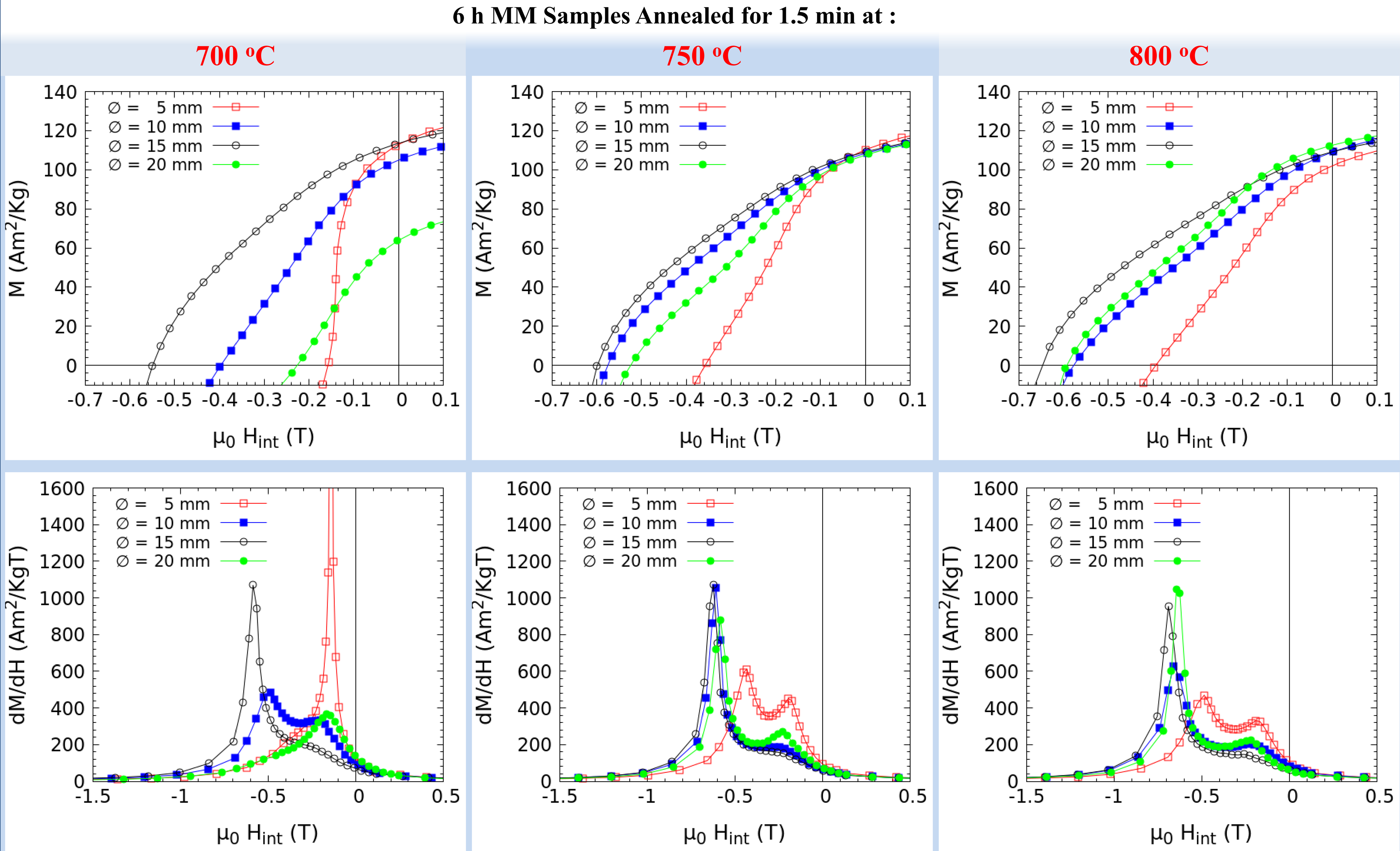


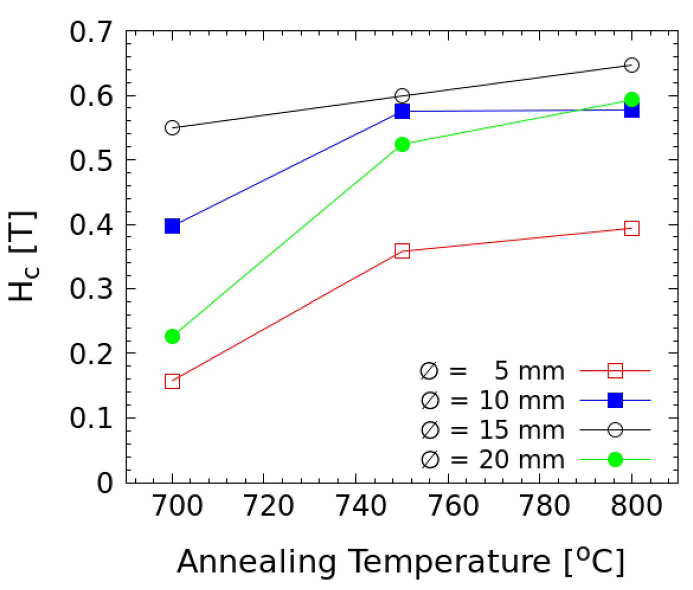
SEM images of 6 h MM powder

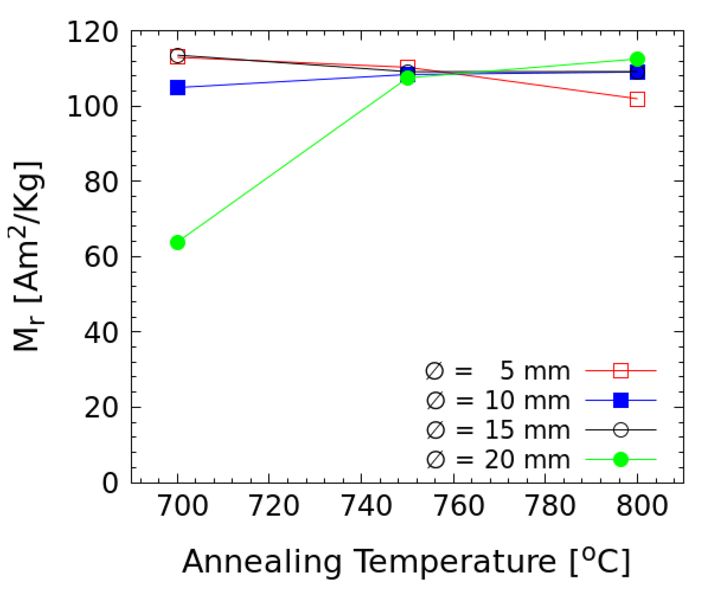


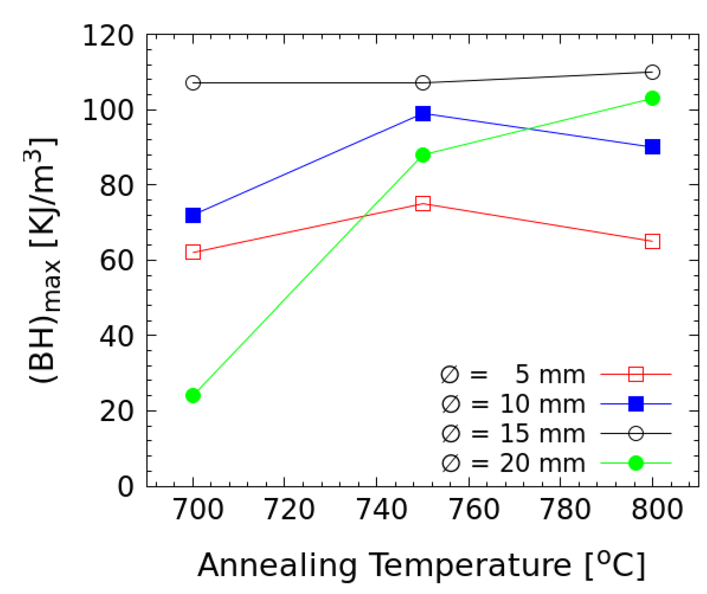
Magnetic Proprieties

6 h MM Samples Annealed for 1.5 min at :









Conclusions:

- **Higher impact energy** → the **lower $Nd_2Fe_{14}B$** mean **crystallite** sizes become (minimum 25 nm).
- **α-Fe crystallite size** are **affected only by annealing** temperature
- **Smaller α-Fe and $Nd_2Fe_{14}B$ crystallite** sizes result in a **better interphase exchange coupling** between the hard and soft phase [3]
- **Best results** in the sample set were obtained for the samples **milled with Ø = 15 mm** diameter steel balls and **annealed at 800 °C: H_c = 0.65 T and $(BH)_{max}$ = 110 KJ/m³**

[1] M. Abdellaoui, Acta. metall. mater, vol. 43, no. 3, pp. 1087-1098, 1995.
[2] D. Maurice, Metal. Mater, Trans. A, vol. 26A, pp. 2431-2435, 1995.
[3] J. Fiddler and T. Schrefl, J. Appl. Phys., vol. 79, no. 8, pp. 5029-5034, 1996.

Acknowledgement
*This work was made possible trough the financial support of Romanian Ministry of Education and Research, grants **PN-II-ID-PCE-2012-4-0470** and **PN-II-RU-TE-2014-4-2360***
We would also like to thank the support staff at Institut Néel for all of their help and also George Crisan and Teodor Biter Lucian for their council.