Effect of Milling Conditions on the Microstructure and Interphase Exchange Coupling of $Nd_2Fe_{14}B/\alpha$-Fe Nanocomposites

Sever Mican[1], Răzvan Hirian[1], Olivier Isnard[2], Ionel Chicinaş[3], Viorel Pop[1]

[1]Faculty of Physics, Babeş-Bolyai University, Cluj-Napoca, RO-400084 Romania
[2]Institut Néel, CNRS, Université Grenoble Alpes, BP 166X, 38042 Grenoble, Cédex 9, France
[3]Materials Science and Engineering Department, Technical University of Cluj-Napoca, Cluj-Napoca, RO-400641 Romania

Abstract: The effects of milling conditions on the microstructure and interphase exchange coupling of $Nd_2Fe_{14}B$+10wt% α-Fe nanocomposites were investigated. The α-Fe crystallite size is critical for obtaining an efficient interphase exchange coupling. The problem of damaging the $Nd_2Fe_{14}B$ crystal structure during milling was addressed by using different milling conditions. $Nd_2Fe_{14}B$+10wt% α-Fe powder samples were prepared through mechanical milling for 6 h with Ø = 10 mm and Ø = 15 mm balls respectively. The restoration of the $Nd_2Fe_{14}B$ crystal structure after annealing was confirmed by XRD, with a limited growth of α-Fe crystallites. The magnetic behavior was investigated from hysteresis curves and dM/dH vs. H plots. The samples milled with Ø = 10 mm diameter balls show good interphase exchange coupling, however, milling with larger diameter balls determined higher coercivities and better interphase exchange coupling due to the reduced damaging of the $Nd_2Fe_{14}B$ crystal structure during milling. The best exchange coupling was obtained for the samples annealed at 800 ºC for 1.5 minutes, with a maximum coercive field of about 0.65 T. The $Nd_2Fe_{14}B/\alpha$-Fe exchange coupling was analyzed as a function of milling and annealing conditions.

Experimental:

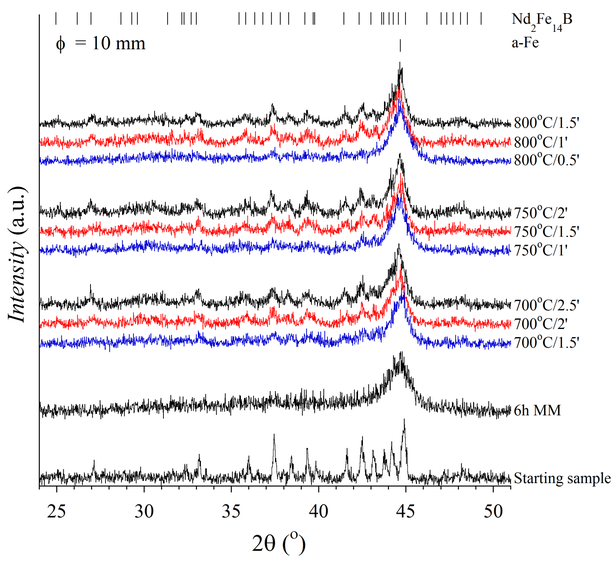
- The $Nd_2Fe_{14}B$ hard phase was prepared by induction melting in an Ar atmosphere, followed by annealing in vacuum at 950 ºC for 72 h. The ingot was ground to a fine powder under 500 µm. The soft magnetic phase is NC 100.24 commercial Fe powder – Höganäs product
- The $Nd_2Fe_{14}B$ powder was mixed with the Fe in a weight ratio of 90% $Nd_2Fe_{14}B$/10% Fe. The mixture was dry-milled (with Ø = 10 mm and Ø = 15 mm diameter balls respectively, in Ar for 2, 4 and 6 h respectively with a ball to powder weight ratio of 10:1. The milled samples were annealed in an Ar atmosphere at 700, 750 and 800 ºC for 0.5-2.5 min and quenched in water.
- X-Ray diffraction measurements were performed on a Brüker D8 Advance diffractometer using Cu K_α radiation.
- Magnetic measurements were carried out on powder samples fixed in epoxy resin using the extraction method at 300 K in applied fields up to ±10 T. Assuming isolated spherical magnetic particles we used a demagnetization factor of 1/3 for magnetic data.

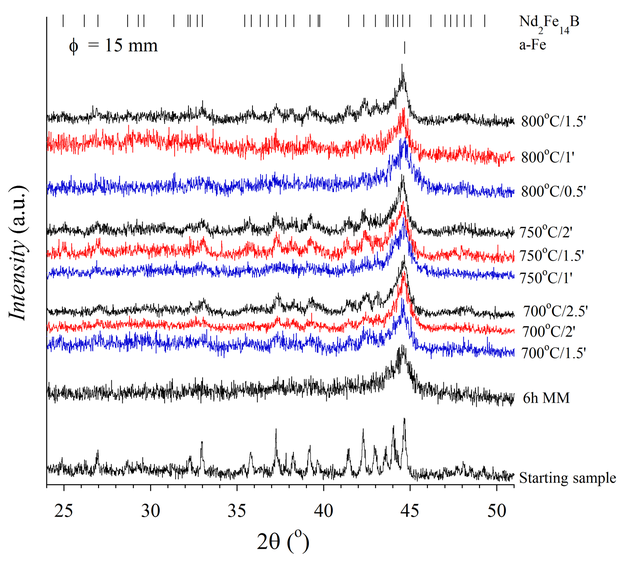
Results and Discussions:

$Nd_2Fe_{14}B$+10wt% α-Fe

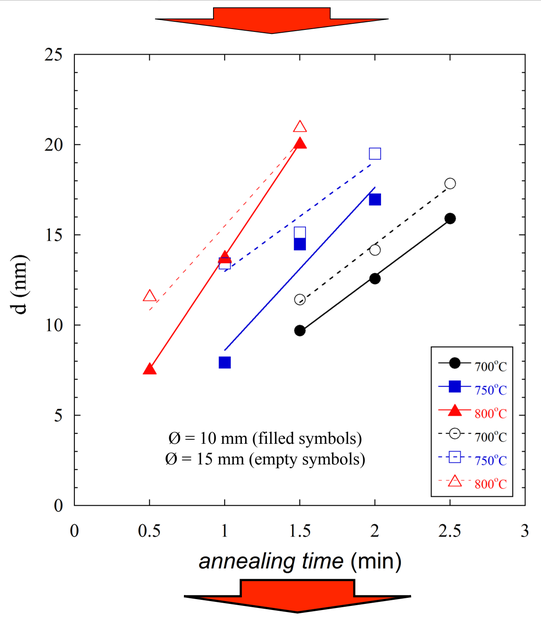
Damaging of the crystal structure and increased internal stress and defect density by MM.

Hard phase XRD peaks disappear after 6 h MM.

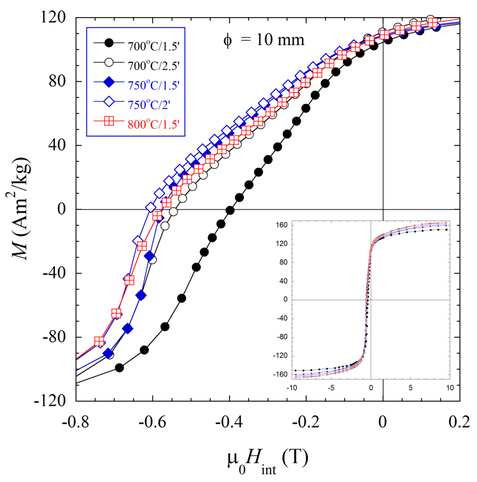


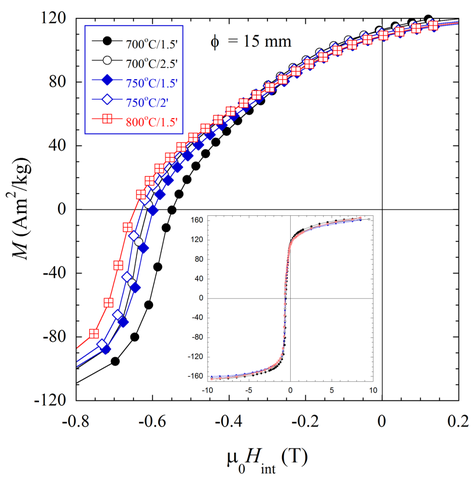


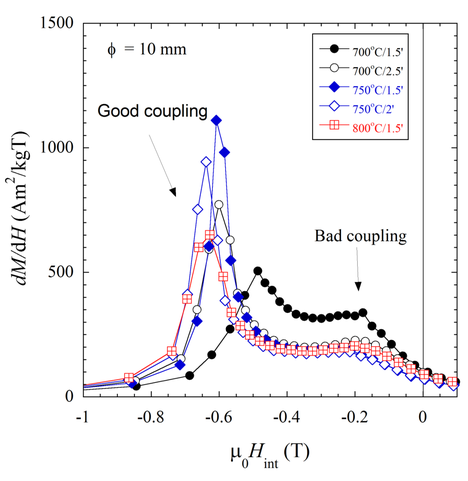
The Scherrer formula $d = \frac{K\lambda}{\beta \cos\theta}$ for the Fe peak at $2\theta = 82.3^o$.

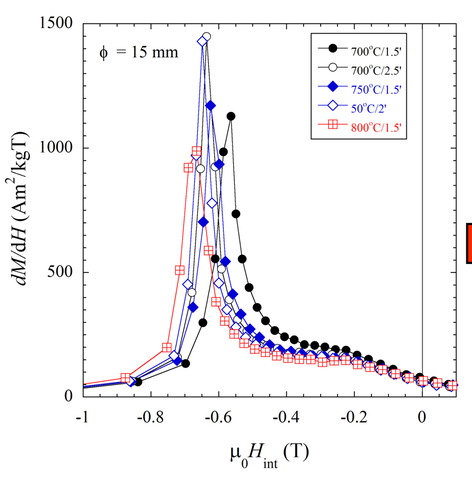


$d \approx$ 5-20 nm
No excessive growth of the soft phase crystallites during the ***short time annealing***.

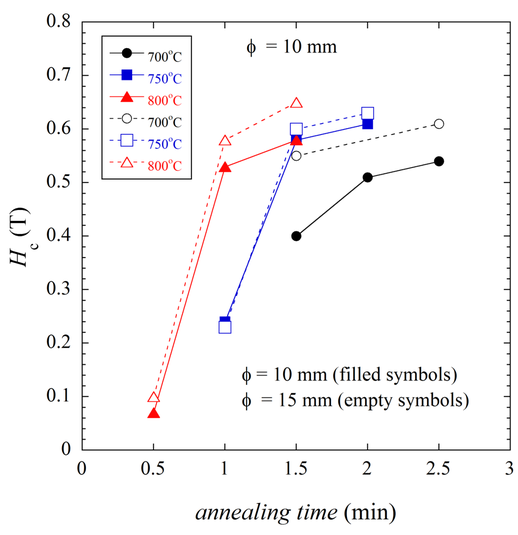


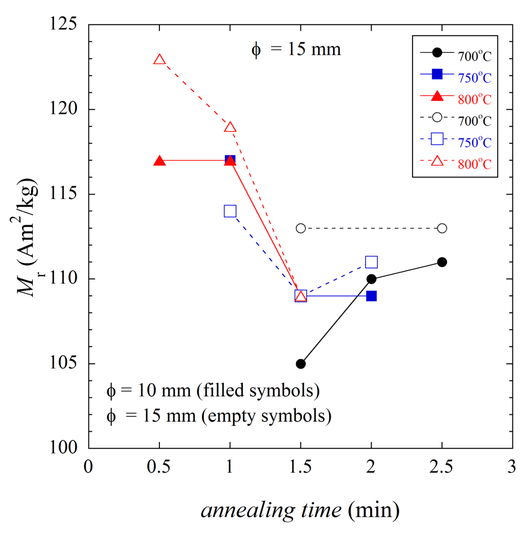


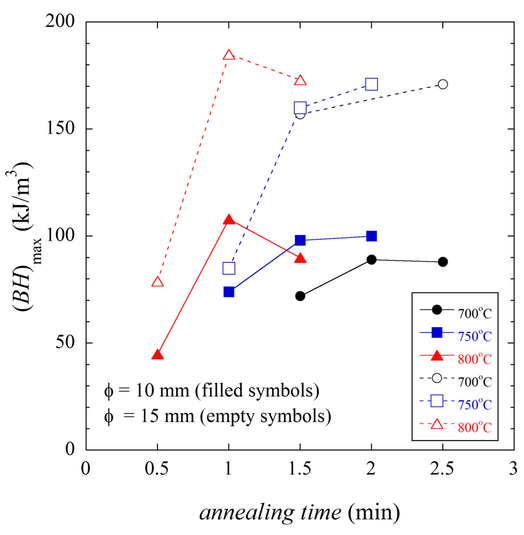




Better coupling for Ø = 15 mm balls. Smaller balls tend to increase the degree of amorphisation during milling [1]. This is most obvious for the annealing at 700 ºC for 1.5 minutes.







The $(BH)_{max}$ values increase with annealing time and slightly increase with annealing temperature.

The largest $(BH)_{max}$ values were obtained for samples milled with Ø = 15 mm diameter balls.

Conclusions:

- ***Short time annealing restores the structure of the hard phase*** destroyed by milling with a ***limited growth of the soft magnetic crystallites.***
- The exchange ***coupling strength*** is higher when milling with Ø= 15 mm diameter balls, ***due to less damage to the crystalline structure of the hard phase.***
- The ***best exchange coupling*** was obtained for the ***6 h MM with Ø= 15mm*** sample annealed at ***800ºC for 1.5 minutes*** with a maximum coercive field value of ***0.65 T***.
- The highest ***energy product*** *185 KJ/m³* is ***comparable*** with the best reported values on rare-earth nanocomposites [2].

[1] Suryanarayana C, *Prog. Mater. Sci.* **46** (2001) 1
[2] Coey J M D, IEEE Trans. Magn. **47** (2011) 4671

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