Magnetic Properties of $Nd_2Fe_{14}B$/α-Fe Nanocomposites Obtained by Mechanical Milling and Rapid Annealing

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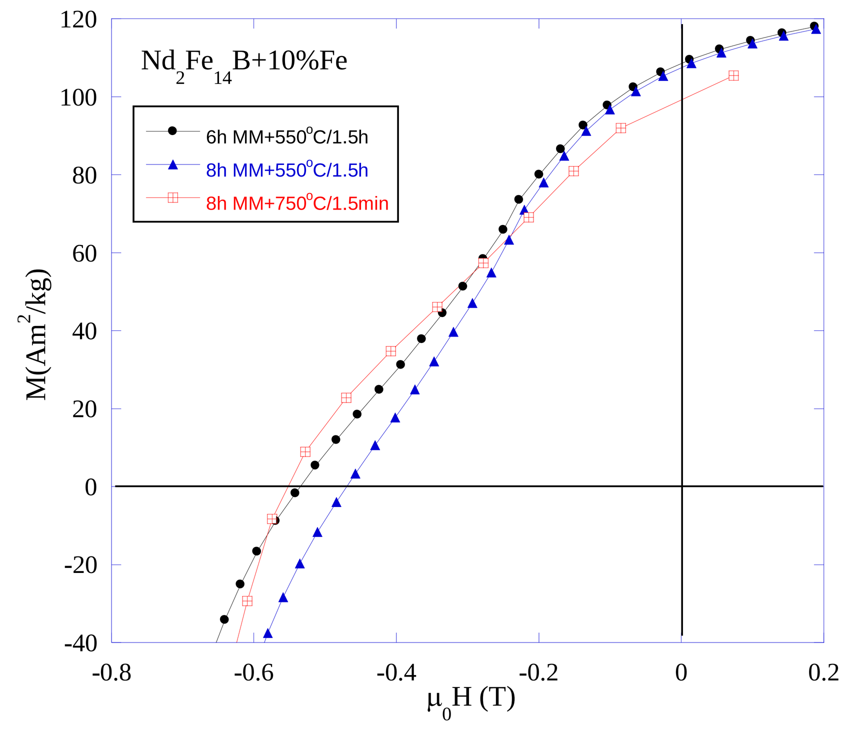
Abstract: The structural and magnetic properties of $Nd_2Fe_{14}B$/α-Fe hard/soft magnetic nanocomposites were investigated. Previous results have suggested a better interphase exchange coupling for samples milled for 6 hours [1]. The width of the X-ray diffraction peaks of the hard magnetic phase increases with milling time, with the peaks disappearing after 6 hours of milling. Following annealing, the characteristic peaks of the hard magnetic phase are restored and are more resolved for the air-quenched samples. Higher annealing times lead to higher coercivity and remanence values and to a better interphase exchange coupling. These results are discussed in comparison with previous results obtained for 8h milled samples.

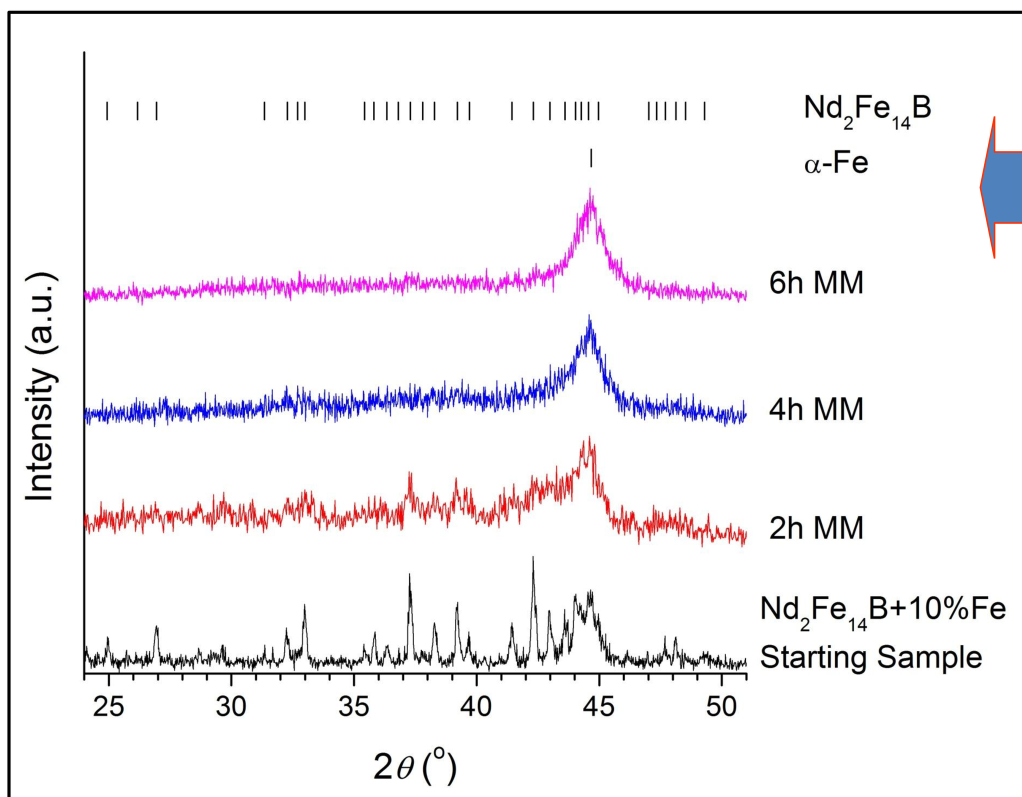


For ***classically annealed samples***, the interphase exchange coupling is better after 6 h of milling in comparison with 8 h of milling [2].
What will happen for short time annealing?

Experimental

- The $Nd_2Fe_{14}B$ phase was prepared by induction melting in an Ar atmosphere, followed by annealing in vacuum at 950°C for 68 h. The ingot was ground to a fine powder under 500 μm.
- The soft magnetic phase (NC 100.24 commercial iron powder – Höganäs product) was pre-milled with benzene for 4 h.
- The hard/soft $Nd_2Fe_{14}B$ + 10% wt. α-Fe nanocomposites were obtained by dry-ball high-energy milling of the starting powders.
- The milled samples were annealed in an Ar atmosphere at 700°C for 0.5, 1, 1.5, 2 and 2.5 min. and quenched in air or 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 a non-magnetic polymer using an extraction magnetometer at 300 K in applied fields up to ±10T.

Results and Discussions

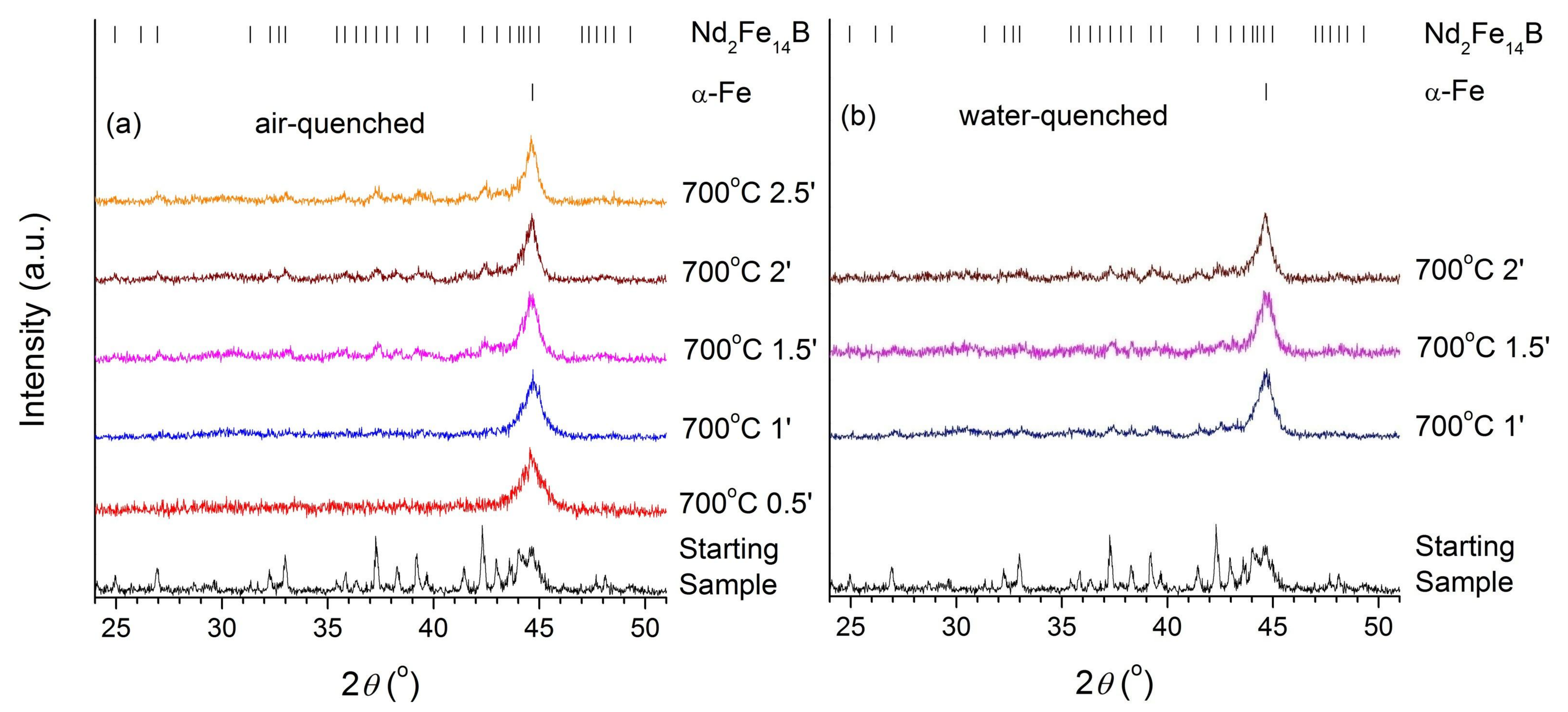


XRD patterns for the ***starting and as-milled*** $Nd_2Fe_{14}B$+10%Fe samples.

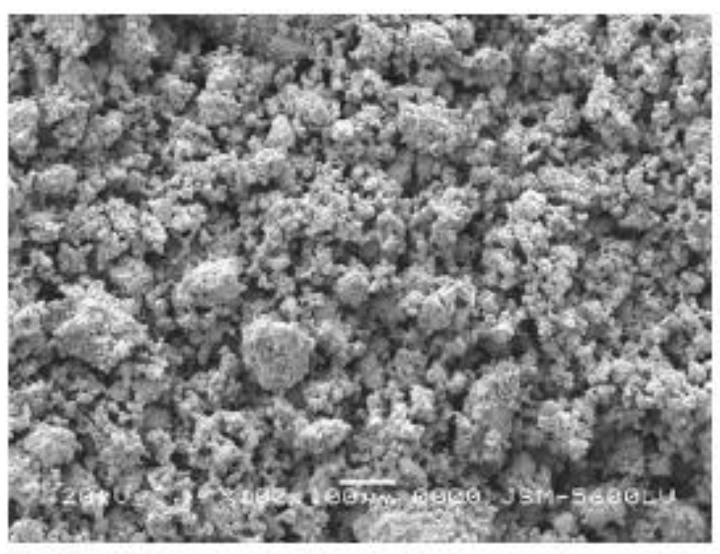
The width of the diffraction peaks increases with milling time, the characteristic peaks of the hard magnetic phase disappearing after 6 h milling time.

The $Nd_2Fe_{14}B$ crystal structure is restored after annealing.

The characteristic peaks of the hard phase are more resolved for the air-quenched samples, however, water quenching tends to promote a faster recrystallisation.



XRD patterns for the $Nd_2Fe_{14}B$+10%Fe samples ***annealed at 700°C*** quenched in air(a) and in water (b) respectively.



Nanocomposite magnetic powders obtained by mechanical milling.

For the magnetic measurements we assumed that the samples are comprised of spherical particles blocked in the non-magnetic polymer.

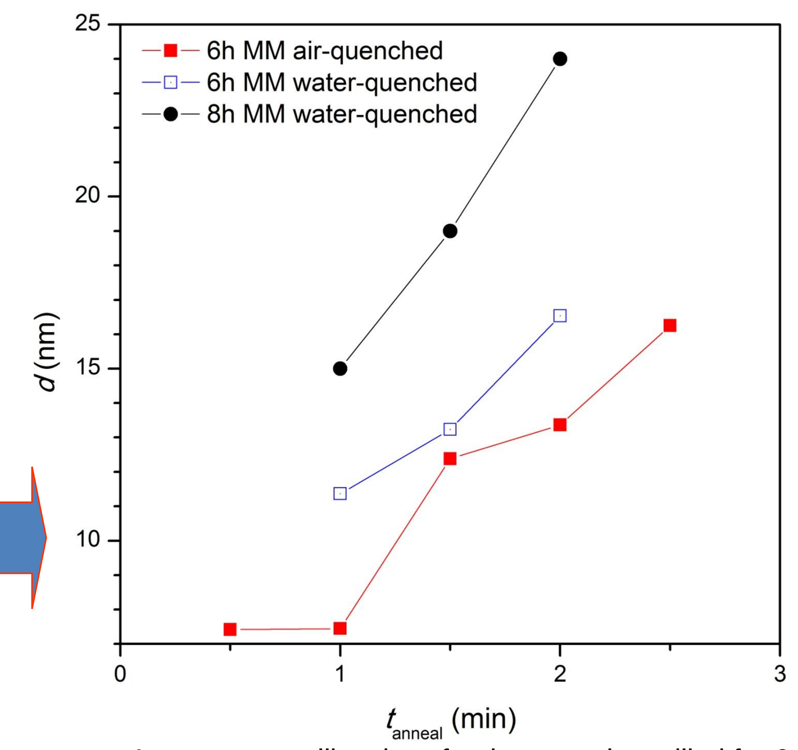
For the demagnetizing field correction we used a value of *N=1/3* for the demagnetization factor.

The crystallite sizes were determined using the Scherrer formula for the peak with 2θ=82.3° (not shown here):

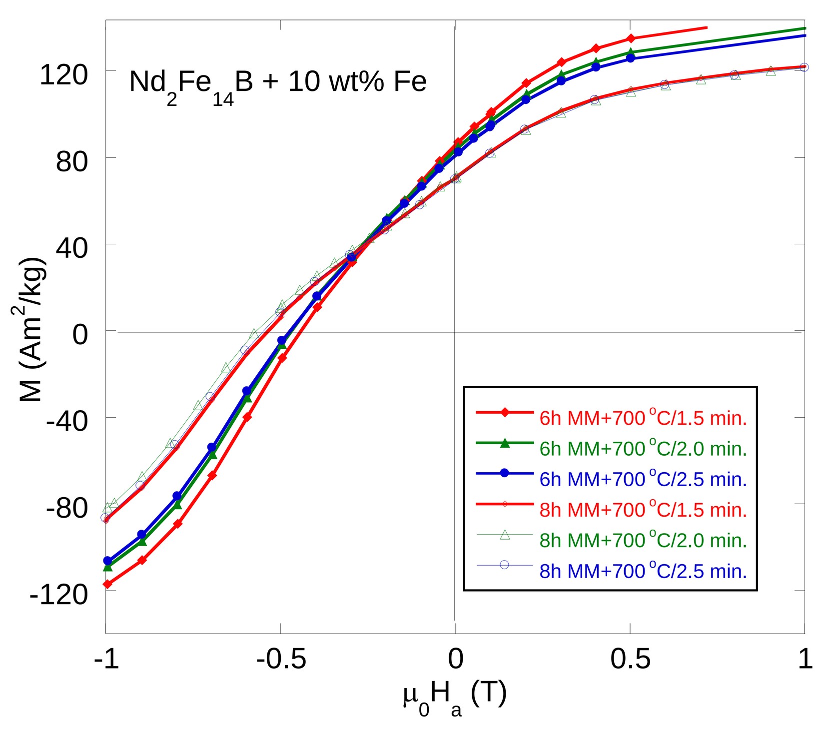
$$d = \frac{K\lambda}{\beta \cos\theta}$$

d is the average crystallite size, *K* is the shape factor, *λ* is the wavelength, *β* is the FWHM of the peak and *θ* is the Bragg angle.

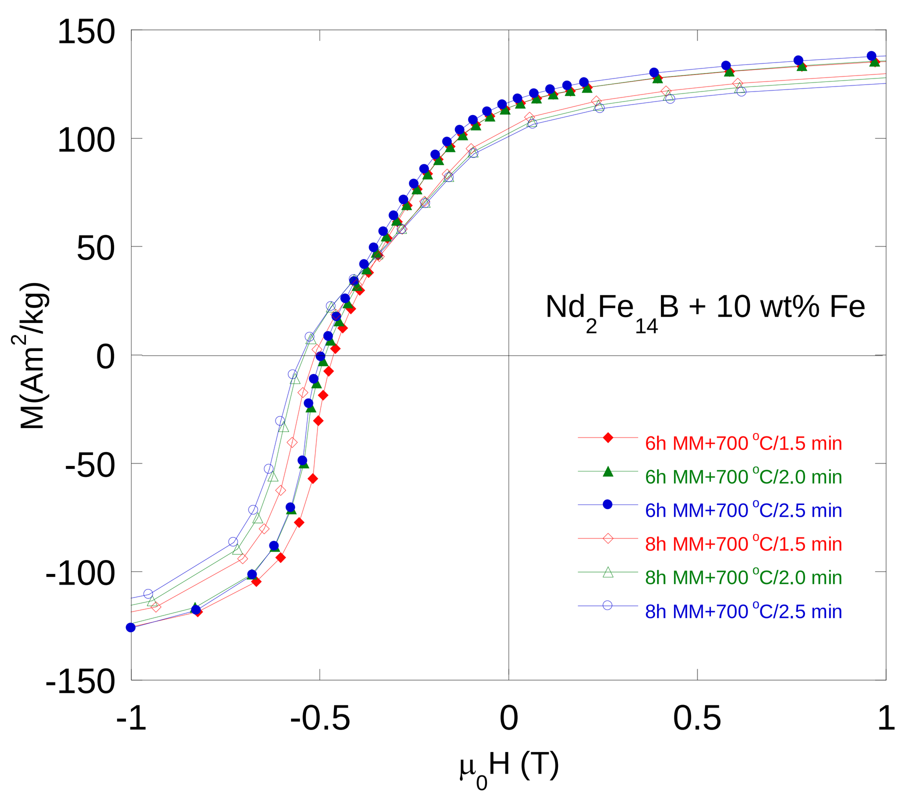
For 6 h of milling, the crystallite sizes increase with increasing annealing time for both air and water-quenched samples, however these values are slightly higher for the water-quenched samples.



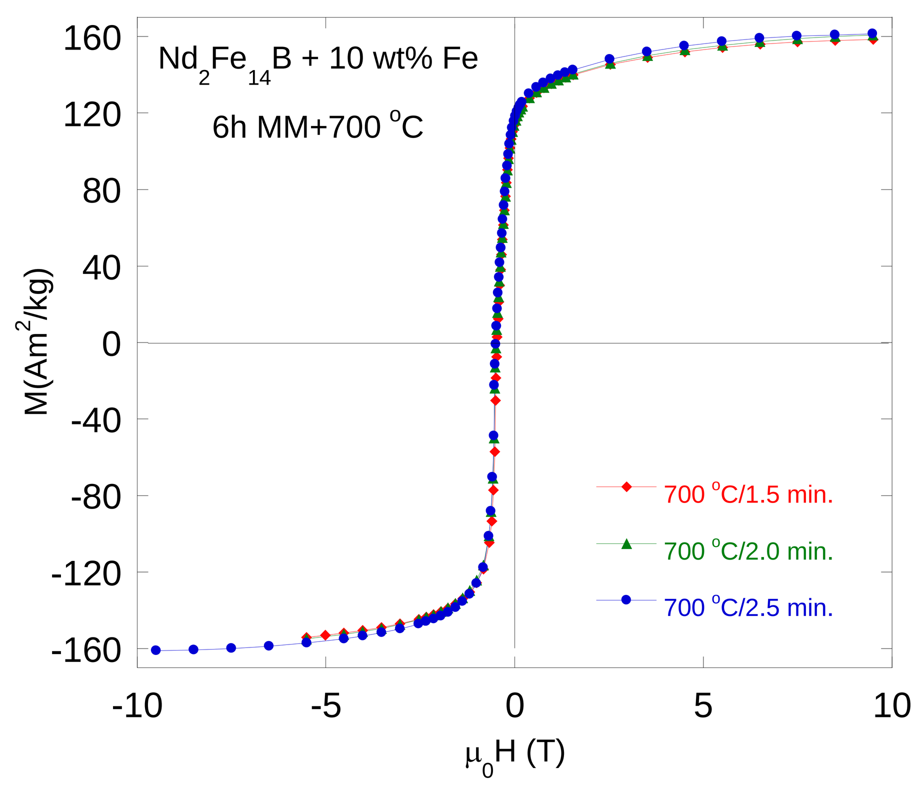
Average crystallite sizes for the samples milled for 6 and 8 h and annealed at 700 °C for different times.



As-measured demagnetization curves for $Nd_2Fe_{14}B$ + 10% wt. Fe nanocomposites milled for 6 and 8 h and annealed at 700 °C (water-quenched) for 1.5, 2 and 2.5 min respectively.

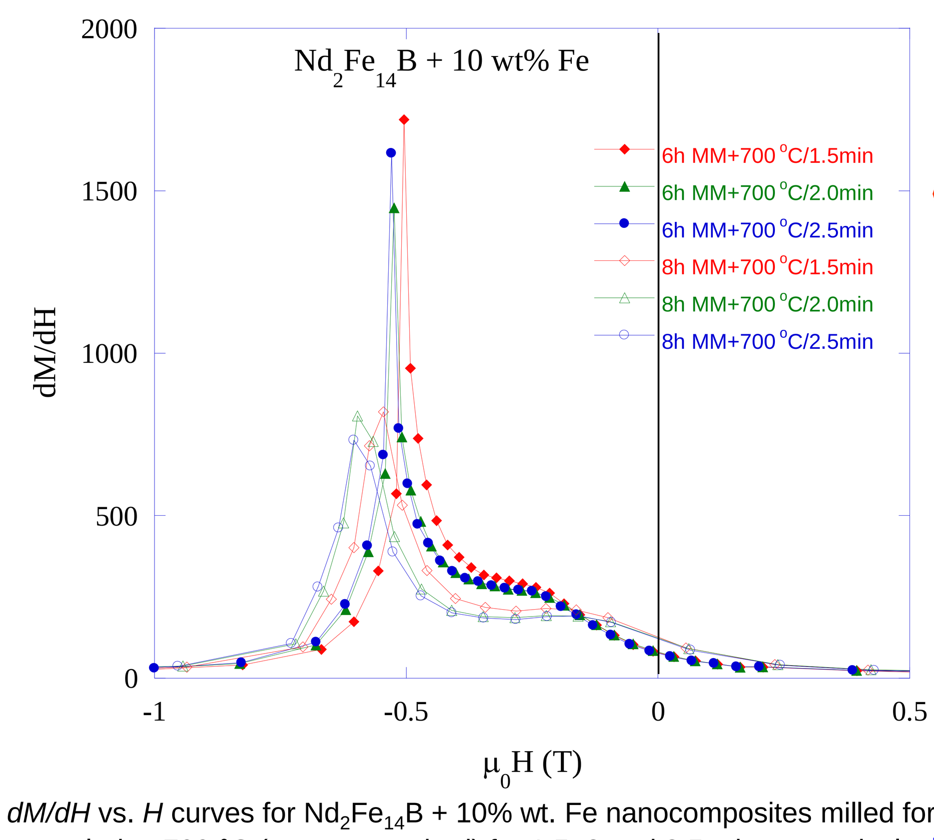


Demagnetization curves for $Nd_2Fe_{14}B$ + 10% wt. Fe nanocomposites milled for 6 and 8 h and annealed at 700 °C (water-quenched) for 1.5, 2 and 2.5 min respectively; **$H=H_a$-M/3.**



±10T magnetization curves for $Nd_2Fe_{14}B$ + 10% wt. Fe nanocomposites milled for 6 h and annealed at 700 °C (water-quenched) for 1.5, 2 and 2.5 min respectively; **$H=H_a$-M/3.**

The saturation of magnetisation is not attain for 10 T.



dM/dH vs. *H* curves for $Nd_2Fe_{14}B$ + 10% wt. Fe nanocomposites milled for 6 and 8 h and annealed at 700 °C (water-quenched) for 1.5, 2 and 2.5 min respectively; **$H=H_a$-M/3.**

The higher ratio between the high field peak and low field peak proves the better coupling for 6 h milled samples. With increasing annealing time, the width of the *dM/dH* peaks shrinks from the right side, pointing to a higher coercivity resulting from the better recrystallisation of hard phases.

Small influence of annealing time for times between 1.5 and 2.5 min.

The 6 h milled samples have a lower coercivity than the 8 h milled ones, though, they have a higher remanence.

Conclusions:

- Short time annealing restores the structure of the hard phase which was destroyed by milling. The peaks of the air-quenched samples are more resolved than the water-quenched ones, however, water quenching promotes formation of smaller Fe crystallites.
- With increasing annealing time, the width of the *dM/dH* peaks shrinks from the right side, pointing to an increasingly interphase exchange coupling.
- The width of dM/dH peaks for 6 and 8 h milled samples is smaller for 6 h of milling proving a narrower crystallites distribution.
- The 6h milled samples have a lower coercivity than the 8 h milled ones which can be explained by their higher remanence.

[1] V. Pop, S. Gutoiu, E. Dorolti, O. Isnard and I. Chicinaş, *J. Alloys Compd.*, **509**, 9964 (2011).

[2] V. Pop, S. Gutoiu, E. Dorolti, C. Leostean, O. Isnard, I. Chicinas and O. Pana, *J. Alloys Compd.*, *Article in Press, http://dx.doi.org/10.1016/j.jallcom.2013.07.194.*

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