

Influence of Synthesis Conditions on the Interphase Exchange Coupling of $\text{Nd}_2\text{Fe}_{14}\text{B}/\alpha\text{-Fe}$ Nanocomposite Obtained by Mechanical Milling

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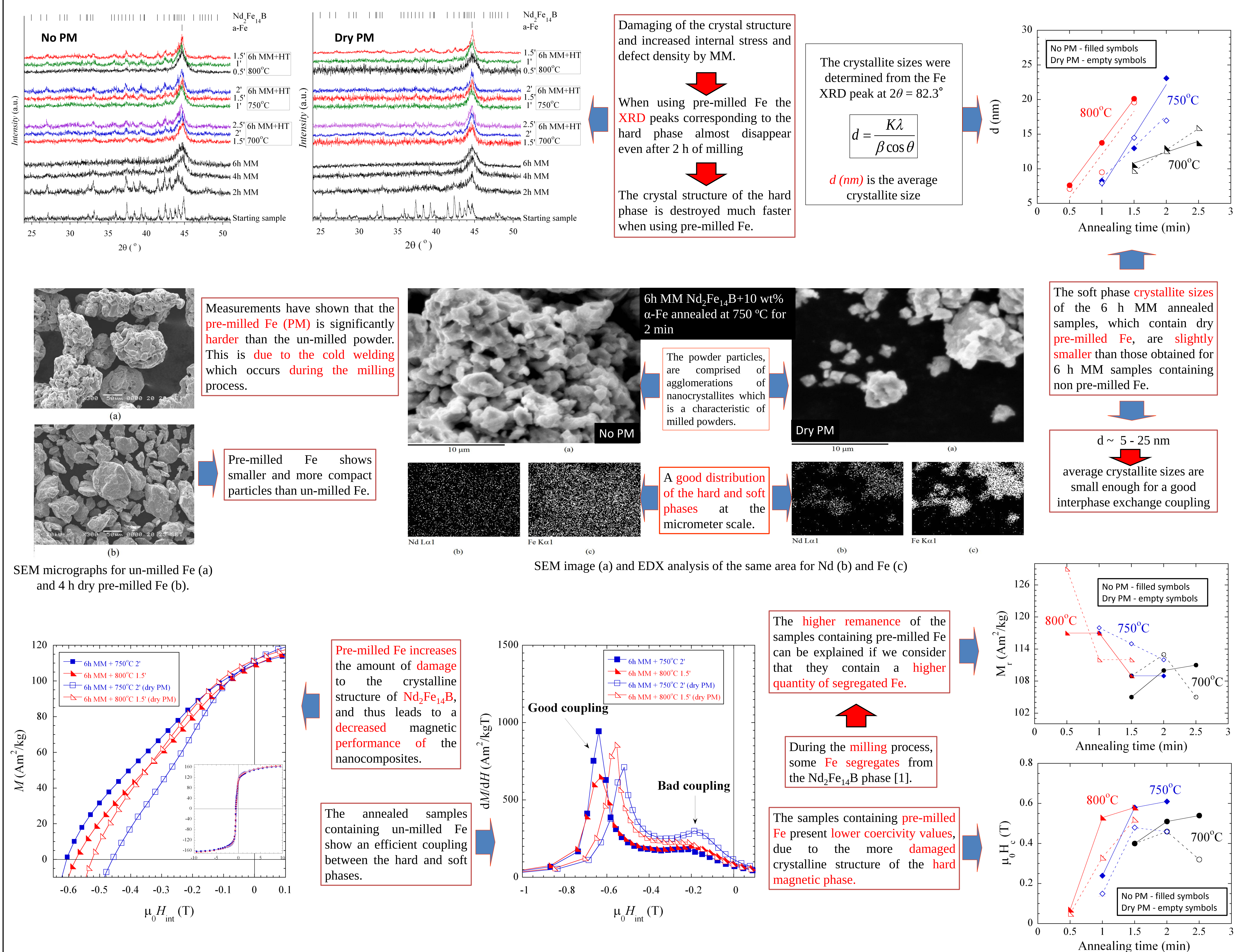
Abstract:

The effects of short time annealing and soft phase pre-milling on the structural and magnetic properties of 6 h milled $\text{Nd}_2\text{Fe}_{14}\text{B}/10\text{wt}\%$ Fe magnetic nanocomposites were investigated. The XRD peaks of $\text{Nd}_2\text{Fe}_{14}\text{B}$ progressively disappeared after milling due to crystal structure damage, this effect being more pronounced when using pre-milled Fe (PM). After annealing, the characteristic peaks of $\text{Nd}_2\text{Fe}_{14}\text{B}$ were restored with a limited growth of the $\alpha\text{-Fe}$ crystallites. The magnetic behavior was investigated using hysteresis curves and dM/dH vs. H plots. The best exchange coupling was obtained for the samples which contain un-milled Fe as the soft phase, with a maximum coercive field value of 0.61 T. The samples containing pre-milled Fe have lower coercive field values, but show a higher remanence. Taking into account the milling and annealing conditions, the $\text{Nd}_2\text{Fe}_{14}\text{B}/\alpha\text{-Fe}$ exchange coupling is analyzed.

Experimental:

- The $\text{Nd}_2\text{Fe}_{14}\text{B}$ hard magnetic phase was prepared by induction melting in a purified 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 phases consisted of NC 100.24 commercial Fe powder – Höganäs product and Fe powder milled for 4 h under Ar in a high-energy planetary mill (Fritsch Pulverisette 4) with a ball to powder weight ratio of 10:1.
- The $\text{Nd}_2\text{Fe}_{14}\text{B}$ powders were mixed with the two soft phases in a weight ratio of 90% $\text{Nd}_2\text{Fe}_{14}\text{B}$ +10% Fe. The two mixtures were dry-milled 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 °C for 0.5-2.5 min and were quenched in water.
- X-Ray diffraction measurements were performed on a Brüker D8 Advance diffractometer using $\text{Cu K}\alpha$ radiation.
- Scanning electron microscopy (SEM) and X-ray microanalysis studies were performed on a Jeol-JSM 5600 LV microscope equipped with an energy dispersive X-ray EDX spectrometer (Oxford Instruments Inca 200 software).
- The mechanical hardness of the Fe powders was measured using an AFM equipped with a XE 70 TD 21562 Sapphire Cantilever nanoindenter with a diamond tip, the data being interpreted using the Oliver and Pharr model.
- Magnetic measurements were carried out on powder fixed in epoxy resin at 300 K in applied fields up to $\pm 10\text{T}$. Assuming isolated spherical magnetic particles we used a demagnetization factor of 1/3 for magnetic data.

Results and Discussions:



Conclusions:

- XRD and SEM-EDX investigations have pointed out that while soft phase pre-milling may lead to slightly smaller $\alpha\text{-Fe}$ crystallites, this results in a more pronounced damaging of the hard phase crystal structure during milling.
- The magnetic behaviour of the 6 h MM short time annealed samples showed an efficient soft/hard exchange coupling.
- It was shown that coercivity increases with both annealing temperature and time due to the progressive restoration of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ crystal structure, with a maximum coercive field of 0.61 T being obtained for the sample annealed at 750 °C for 2 minutes.
- It was shown that Fe pre-milling weakens the soft/hard exchange coupling and decreases coercivity, however, Fe pre-milling increases remanence values.