Effect of Milling Conditions on the Microstructure and Interphase Exchange Coupling of Nd$_2$Fe$_{14}$B/α-Fe Nanocomposites

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Abstract: The effects of milling conditions on the microstructure and interphase exchange coupling of Nd$_2$Fe$_{14}$B/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$_2$Fe$_{14}$B crystal structure during milling was addressed by using different milling conditions. Nd$_2$Fe$_{14}$B+10vol% 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$_2$Fe$_{14}$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$_2$Fe$_{14}$B crystal structure during milling. The best exchange coupling was obtained for the samples annealed at 800 °C for 1.5 minutes, with a maximum coercive field of about 0.65 T. The Nd$_2$Fe$_{14}$B/α-Fe exchange coupling was analyzed as a function of milling and annealing conditions.

Experimental:
- The Nd$_2$Fe$_{14}$B hard phase was prepared by induction melting in an Ar atmosphere, followed by annealing in vacuum at 950 °C for 72 h. The ingot was ground to a fine powder under 500 µm. The soft magnetic phase is α-Fe, a commercial Fe powder – Höganäs product.
- The Nd$_2$Fe$_{14}$B powder was mixed with the Fe in a weight ratio of 90% Nd$_2$Fe$_{14}$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) to obtain a 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 Kα 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:

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 in the crystalline structure of the hard phase.
- The best exchange coupling was obtained for the 6 h milled with Ø = 15 mm sample annealed at 800°C for 1.5 minutes with a maximum coercive field value of 0.65 T.
- The highest energy product 185 KJ/m$^3$ is comparable with the best reported values on rare-earth nanocomposites [2].

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