

## MICROSTRUCTURAL MAGNETIC PARAMETERS OF Nd-Fe-B NANOCOMPOSITES

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Samples with the composition:  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  and  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  have been prepared in form of ribbons (thickness of about 20  $\mu\text{m}$ ) by rapid quenching using the melt spinning technique. The samples obtained with amorphous structure have been subsequently devitrified by annealing at 700 °C for 5min. with the aim of inducing the magnetic hardness and obtaining, in particular, magnetically two-phase  $\alpha\text{Fe}/\text{Nd}_2\text{Fe}_{14}\text{B}$  -type systems, coupled via exchange interactions ( $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$ ) and decoupled ( $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$ ). The maximum value of the fluctuation field for both coupled and decoupled systems is  $\mu_0 S_v \cong 0.6$  mT, found at applied fields close to coercive field. The activation volume, in which the magnetic moments are reversed by the local energy fluctuation, is  $v \cong 5900$  nm<sup>3</sup> for the coupled system and  $v \cong 5200$  nm<sup>3</sup> for the decoupled system.

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### 1. Introduction

The good permanent magnetic properties of Nd-Fe-B nanocomposite alloys are generated through the magnetic hardening of the iron-based soft magnetic phase (or phases – i.e.  $\alpha\text{-Fe}$ ,  $\text{Fe}_3\text{B}$ ,  $\text{Fe}_{23}\text{B}_6$ ) by the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  hard magnetic phase. This occurs when the structure is homogeneous and refined to the nanometer scale, thus ensuring effective magnetic coupling of the grains over short distances through exchange interactions. Micromagnetic modelling [1] demonstrates the necessity for reducing the grain size of the soft magnetic phase to about double the domain wall width of the hard magnetic phase ( $\delta_{w2:14:1}=4$  nm). In practice, good hard magnetic properties are achieved for a crystalline structure refined to about 20nm. However, the macroscopic magnetic properties of nanocomposite permanent magnets, are critically related to the crystalline structure and distribution of phases and hence to the annealing parameters that generate the respective structure. A non-homogeneous structure or / and a structure with grains larger than 20 nm. exhibits poor hard magnetic properties. This tight relationship between structure and magnetic properties can be depicted through the magnetic parameters at the microstructural level that characterize the magnetization reversal process. Microstructural parameters such as: *fluctuation field* ascribed to the local magnetic field variation associated with the formation of a domain with reversed magnetization and *activation volume*, ascribed to a region with the reversed magnetization, can be derived from magnetic viscosity measurements.

In this communication we present our results related to the variation of the microstructural magnetic parameters: fluctuation field and activation volume, on the exchange coupling effect in two phase  $\alpha\text{Fe} / \text{Nd}_2\text{Fe}_{14}\text{B}$  nanocomposite alloys in form of ribbons obtained by rapid quenching from the melt.

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## 2. Experimental

$\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  and  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  amorphous ribbons with width of 2mm and thickness of about 20  $\mu\text{m}$ , have been prepared by liquid rapid quenching using the melt spinning technique. In order to induce a nanocrystalline structure, the as cast samples have been subsequently heat treated at 700 °C for 5min. in accordance with our previous results [2]. The magnetic parameters have been determined using a vibrating sample magnetometer with automatic data acquisition, that operates at a maximum applied field of 1.5T and is equipped with a controlled thermoregulator unit. Magnetic viscosity measurements were carried out on both the demagnetisation branch of the hysteresis as well as on recoil curves at zero applied field. The waiting time for the experiments was 1800 seconds.

## 3. Results and discussion

The ribbon samples have been studied previously [2] with regard to the extent of the exchange coupling of the constituent magnetic phases. It was obtained that the  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  nanocrystalline ribbons behave unitary with the grains coupled at short distances through exchange interactions whereas,  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  nanocrystalline ribbons show a two-phase type profile of the demagnetization curve in the 2<sup>nd</sup> quadrant of the hysteresis. One should mention also that both type of samples separate upon crystallization the hard magnetic  $\text{Nd}_2(\text{Fe, Co})_{14}\text{B}$  phase and soft magnetic  $\alpha\text{-(Fe, Co)}$  phase in the same volume ratio of 70 to 30 % respectively. The rest of alloying elements, Cu, Nb and Si are known to form secondary phases at boundaries of the grains of main magnetic phases in a reduced volume ratio.

Assuming that in a certain magnetic state, in the absence of the external magnetic field stimulus (external magnetic field is constant), the variation of magnetization in time is a thermally activated process, one may demonstrate that the fluctuation field  $H_{\text{fluctuation}}$  can be associated with the magnetic viscosity parameter  $S_v$  [4]. The magnetic viscosity parameter represents the ratio between

the magnetic viscosity and irreversible susceptibility:  $S_v = \frac{S}{\chi_{\text{irrev}}}$ , while the fraction's terms can be

derived from the variation of the magnetization, at constant external field, with time:  $\Delta M(t) = \text{const.} + S \ln t$  and the first derivative of the irreversible component of magnetization

with applied field  $\chi_{\text{irrev}} = \frac{d(M_r^D)}{dH}$ .

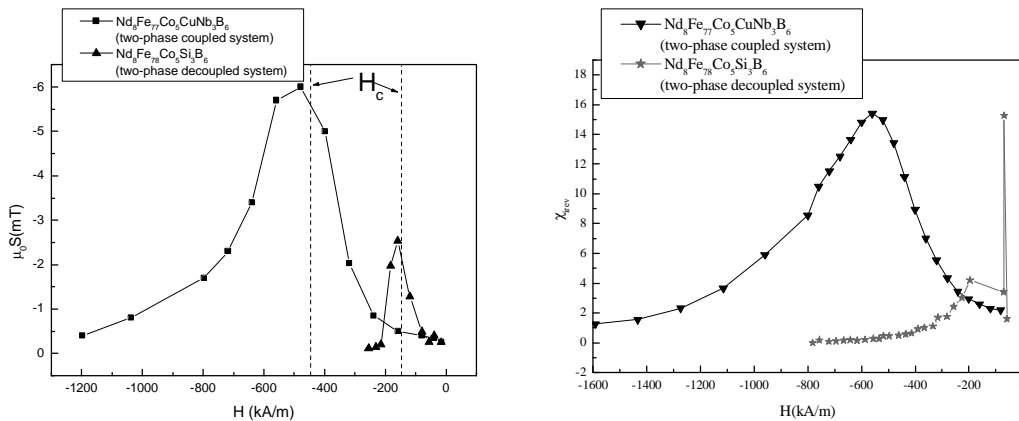


Fig. 1. Variation of the magnetic viscosity  $S$  and irreversible susceptibility  $\chi_{\text{irrev}}$  as a function of the applied field (field applied with discrete steps of 1T/min.) for nanocrystalline  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  and  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  ribbons considered as a coupled respectively, decoupled system.

Thus, by drawing the recoil curves for both samples, the irreversible susceptibility was obtained as a function of the applied field  $\chi_{\text{irrev}}(H)$  (Fig. 1). Also, for different values of the applied magnetic field, the magnetization variation in time was monitored and its slope with  $\ln t$  gave us the variation of the magnetic viscosity with the external applied field  $S(H)$  (Fig.1). Both  $S(H)$  and  $\chi_{\text{irrev}}(H)$  curves have been analysed using less square method and afterward, divided to obtain the variation of the magnetic viscosity parameter with the applied field  $S_v(H)$  that has the dimension of the fluctuation field (Fig. 2).

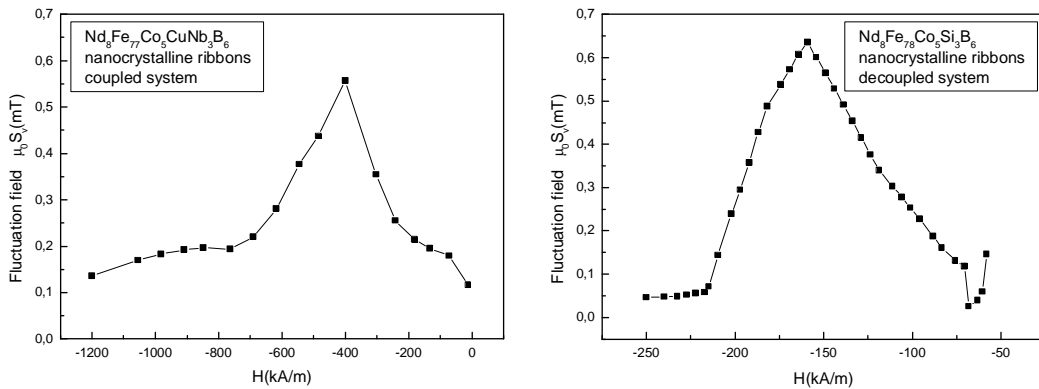


Fig. 2. Variation of the fluctuation field as a function of the externally applied magnetic field.

For both  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  and  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  samples, the variation of magnetic viscosity and fluctuation field with the applied magnetic field shows a maximum at values of the external field close to the coercive field ( $H_c = 414 \text{ kA/m}$  and  $H_c = 152 \text{ kA/m}$ ) when the magnetization vanishes [2]. The maximum value of the fluctuation field for either coupled and decoupled systems is  $\mu_0 S_v \sim 0.6 \text{ mT}$ .

We have found that the fluctuation field for investigated systems:  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  nanocrystalline samples as coupled two phase  $\text{Nd}_2(\text{Fe, Co})_{14}\text{B}/\alpha\text{-(Fe, Co)}$  system an  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  nanocrystalline samples as decoupled two phase  $\text{Nd}_2(\text{Fe, Co})_{14}\text{B}/\alpha\text{-(Fe, Co)}$  system, does not change much with increasing the temperature up to  $130 \text{ }^\circ\text{C}$  (Fig.3) comparative with single phase  $\text{Nd}_2\text{Fe}_{14}\text{B}$  system, for which we considered the data from [5].

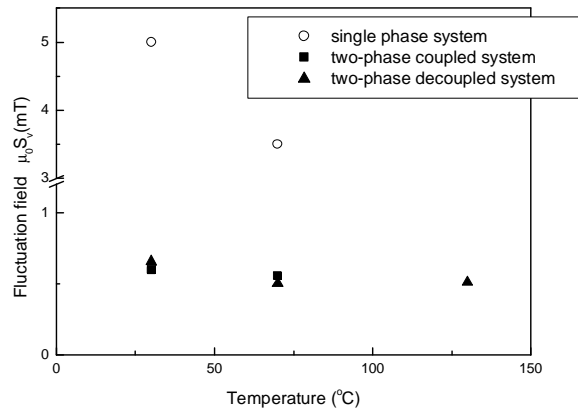


Fig. 3. Variation of the fluctuation field with temperature for coupled system (nanocrystalline  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  ribbons) and decoupled system (nanocrystalline  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  ribbons). Results for single phase system are comparatively presented (from [5]). The error in the calculation of the fluctuation field at temperatures above  $100 \text{ }^\circ\text{C}$  is  $\sim 20\%$ .

The activation volume of magnetization reversal has been calculated, equalizing the thermal energy with the magnetostatic energy of the reversed domain, with the expression  $v = \frac{\mu_0 k_B T}{J_s (\mu_0 S_v)}$

where  $J_s$  is the magnetic saturation polarization and  $S_v$  is the magnetic viscosity parameter. The results for the variation of the activation volume with temperature for the investigated systems are depicted in fig.4. We have obtained higher values of the activation volume for two-phase samples as compared with the single phase system. At room temperature, the activation volume in which the magnetic moments are reversed by the local energy fluctuation, is  $v \cong 5900 \text{ nm}^3$  for the coupled system and  $v \cong 5200 \text{ nm}^3$  for the decoupled system, while data from [5] indicate  $v \cong 600 \text{ nm}^3$  for single phase system. Considering that the activation volume is spherical one obtains that the radius of this volume is about  $r \cong 11 \text{ nm}$  for both coupled and decoupled two-phase systems while for the single phase one is about  $r \cong 5 \text{ nm}$ .

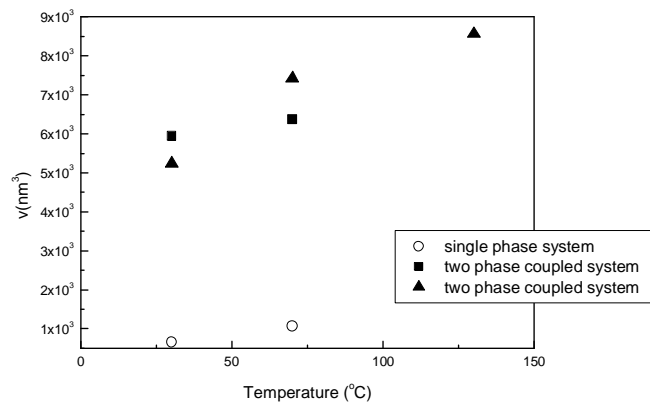


Fig. 4. Activation volume as a function of temperature for coupled system (nanocrystalline  $\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  ribbons) and decoupled system (nanocrystalline  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  ribbons). Results for single phase system are comparatively presented (from [5]). The error in the calculation of the fluctuation field at temperatures above  $100 \text{ }^\circ\text{C}$  is  $\sim 20\%$ .

#### 4. Conclusions

$\text{Nd}_8\text{Fe}_{77}\text{Co}_5\text{CuNb}_3\text{B}_6$  and  $\text{Nd}_8\text{Fe}_{78}\text{Co}_5\text{Si}_3\text{B}_6$  nanocrystalline ribbons have been studied as magnetically two-phase  $\alpha\text{Fe}/\text{Nd}_2\text{Fe}_{14}\text{B}$ - type systems coupled via exchange interactions and respectively, decoupled. The maximum value of the fluctuation field for both coupled and decoupled systems is  $\mu_0 S_v \cong 0.6 \text{ mT}$ , found at applied fields close to coercive field. The activation volume, in which the magnetic moments are reversed by the local energy fluctuation, is  $v \cong 5900 \text{ nm}^3$  for the coupled system and  $v \cong 5200 \text{ nm}^3$  for the decoupled system, much larger than for the single phase system ( $v \cong 600 \text{ nm}^3$ ).

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