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# Annealing dependence of magnetic interactions in  $Y\text{Co}_5(70\% \text{wt}) + Y_2\text{Co}_{17}(30\% \text{wt})$  nanocomposite powders

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#### **Abstract**

X-ray diffraction (XRD) and DC-magnetization measurements were used to study the microstructure and magnetic properties of  $YCo<sub>5</sub>(70%wt)$  $+ Y_2$ Co<sub>17</sub>(30%wt) nanocomposite powders prepared by mechanical milling followed by annealing at 800 °C. XRD data demonstrate that the annealing time *t* has different effects on the average grain size  $\langle D \rangle$  of the two phases: for  $YCo<sub>5</sub> \langle D \rangle$  stays almost constant at 14 nm for all values of t (1.5 min < t < 5.5 min), whereas for  $Y_2Co_{17}$   $\langle D \rangle$  increases with increasing t from 12 nm to 24 nm. This grain size increase in the soft phase influences the hard magnetic properties of the nanocomposite. Indeed, hysteresis loops  $(H_{\text{max}} = 70 \text{ kOe})$  show that both the coercivity  $H_C$  and the remanence  $M_r/M_{\text{max}}$  depend on the annealing time, with the sample annealed for 1.5 min exhibiting the highest values:  $H_C = 12.0$  kOe and  $M_r/M_{\text{max}} = 0.69$ . As indicated by  $\delta M$  plot and magnetization reversal nucleation field analysis this behavior can be attributed to a grain-sizedependent interplay between the exchange and the dipolar magnetic interactions. © 2007 Elsevier B.V. All rights reserved.

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## **1. Introduction**

Nanocomposite materials of a hard and a soft magnetic phase have attracted much attention due to their considerable potential for permanent magnet applications [\[1\].](#page--1-0) Key to the functionality of such magnetic systems is a strong exchange coupling of the magnetic moments across the grain boundaries between the hard and the soft phases, which leads to an enhanced remanence, significantly higher than in an isotropic single-phased hard magnetic material. To achieve an optimal exchange coupling, it was proposed that the average grain size in *both* phases needs to be in the 10–15 nm range [\[2\].](#page--1-0) This microstructure favors the enhancement of the remanent magnetization and induces other "good" magnetic properties such as a high reversible maximum magnetization, a typical singlephase demagnetization curve, and a substantial recoil permeability. On the other hand, large grain sizes (*>* 20 nm) are likely to favor demagnetizing long-range dipolar interactions which worsen the magnetic properties of the nanocomposite. Therefore, the ability to understand and eventually control the processing factors responsible for the average grain size of the two phases (which, in turn, determine the macroscopic magnetic behavior) is an essential step toward the rational design of these functional materials. To achieve this goal it is necessary to carry out quantitative investigations that address not only the grain size—magnetic property relationship, but also the influence of the two-phase microstructure on the microscopic magnetic arrangements and interactions that are ultimately responsible for the magnetic properties of the nanocomposite.

Here we present a study of the *microstructure and magnetic properties* of  $YCo<sub>5</sub>(70\%wt) + Y<sub>2</sub>Co<sub>17</sub>(30\%wt)$  nanocomposite powders prepared by mechanical milling and annealing at 800 ◦C *as a function of the annealing time*. We used XRD to de-

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Fig. 1. X-ray diffraction patterns of  $YCo<sub>5</sub>(70%wt) + Y<sub>2</sub>Co<sub>17</sub>(30%wt)$ nanocomposite powders annealed at 800 ◦C for: (a) 1.5, (b) 3.5, and (c) 5.5 min. The vertical bars represent the Bragg reflection markers for the two phases.

termine the average crystallite size of each phase independently and DC-magnetization to measure the coercivity *(HC)* and the remanence *(Mr/M*max*)* of the nanocomposite. In addition, *δM* plot and magnetization reversal nucleation field analyses were carried out to reveal the dependence of the magnetic interactions on the annealing time.

## **2. Experimental details**

Alloys with nominal composition  $YCo<sub>5</sub>$  and  $Y<sub>2</sub>Co<sub>17</sub>$  were prepared by arc melting high-purity elements in an Ar atmosphere. The ingots were re-melted 4 times to ensure homogeneity. Subsequently, the as-cast ingots were coarsely pulverized and a 3 g mixture with a composition of 70%wt of  $YCo<sub>5</sub>$  and 30%wt of  $Y<sub>2</sub>Co<sub>17</sub>$  was prepared. The mixed powders were mechanical milled for 4 h under Ar atmosphere using a SPEX 8000 ball mill with a ratio of powders to balls of 1 : 8. The as-milled amorphous material was annealed in high vacuum at  $800^{\circ}$ C for 1.5, 3.5, and 5.5 min, followed by quenching in water. For each sample the oxygen content was checked by energy dispersive spectroscopy (EDS) and found to be below 2%wt. XRD data were collected on finely ground powders using a Siemens D5000 diffractometer equipped with graphite monochromator ( $Cu$ – $K_{\alpha}$  radiation). DC-magnetization measurements were carried out in fields up to 70 kOe, at room temperature, on a Quantum Design Physical Property Measurement System (PPMS).

### **3. Results and discussion**

Fig. 1 shows the XRD patterns of  $YCo<sub>5</sub>(70\%$  wt) +  $Y_2Co_{17}(30\%$  wt) nanocomposite powders annealed at 800 °C for (a) 1.5, (b) 3.5, and (c) 5.5 min. All the observed peaks could be indexed to either the hexagonal  $YCo<sub>5</sub>$  phase (PDF #17-078) or the rhombohedral  $Y_2Co_{17}$  phase (PDF #18-434). For each of the three data sets the full widths at half maxi-



Fig. 2. Annealing-time dependence of average grain size in each of the two phases of  $YCo<sub>5</sub>(70%wt) + Y<sub>2</sub>Co<sub>17</sub>(30%wt)$  nanocomposite powders.

mum (FWHM) of  $YCo<sub>5</sub>$  and  $Y<sub>2</sub>Co<sub>17</sub>$  Bragg peaks were used in the Scherrer equation [\[3\]](#page--1-0) to estimate the average particle size  $\langle D \rangle$  of the two phases that make the nanocomposite. The FWHMs were obtained by fitting the corresponding peak profiles to a combination of Gaussian and Lorentzian functions. Interestingly, it was found that the annealing time *t* has different effects on the grain size of the two phases. As shown in Fig. 2, the grain size of the hard phase  $(YCo<sub>5</sub>)$  stays almost constant for all annealing times, whereas for the soft phase  $(Y_2Co_{17})$  the grain size increases with *t*, from 12 nm at  $t = 1.5$  min to 24 nm at  $t = 5.5$  min. To determine the effect of this annealing-induced increase of the soft phase grain size on the hard magnetic properties of the nanocomposite we carried out DC-magnetization vs. field measurements on the three samples annealed for different times. [Fig. 3](#page--1-0) shows the virgin magnetization curve and the hysteresis loop corresponding to the sample annealed for 1.5 min. The virgin curve indicates a pinning type magnetization mechanism [\[4\]](#page--1-0) similar to the one previously reported for single-phase  $YCo<sub>5</sub>$  nanopowders [\[5\].](#page--1-0) Magnetic saturation was not observed for the maximum applied field  $(H_{\text{max}} = 70 \text{ kOe})$ , as expected for nanostructured magnetic materials [\[6\].](#page--1-0) The maximum magnetization,  $M_{\text{max}} = 81 \text{ emu/g}$  is comparable to the one previously reported for  $YCo<sub>5</sub>$  nanopowders [\[5\],](#page--1-0) which suggests that the contributions to  $M_{\text{max}}$  from the easy-axis (YCo<sub>5</sub>) and the easy-plane  $(Y_2Co_{17})$  phases are similar. Also, the hysteresis loop reveals a high coercivity value,  $H_C = 12.0$  kOe and an enhanced remanence,  $M_r/M_{\text{max}} = 0.69$ . It is important to point out that the maximum magnetization  $M_{\text{max}}$  is different from the extrapolated saturation magnetization  $M<sub>s</sub>$  (that can be calculated from the hysteresis loop), where  $M_s > M_{\text{max}}$ . However, since for a given sample both  $M_{\text{max}}$  and  $M_r$  depend on the maximum applied field *H*max, and the same *H*max has been used for all samples, we employed  $M_r/M_{\text{max}}$  (and not  $M_r/M_s$ ) to compare the remanent magnetization of nanopowders subjected to different thermal treatments. The high coercivity observed in [Fig. 3](#page--1-0) for the sample annealed for 1.5 min is consistent with the microstructure of this nanocomposite where the grain size of

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