University of Nebraska - Lincoln

DigitalCommons@University of Nebraska - Lincoln

David Sellmyer Publications

Research Papers in Physics and Astronomy

4-15-1996

Magnetic hardening of melt-spun nanocomposite Nd₂Fe₁₄B/Fe magnets

I. Panagiotopoulos

Department of Physics and Astronomy, University of Delaware, Newark, Delaware

L. Withanawasam

Department of Physics and Astronomy, University of Delaware, Newark, Delaware

A.S. Murthy

Department of Physics and Astronomy, University of Delaware, Newark, Delaware

George C. Hadjipanayis

University of Delaware, hadji@udel.edu

E.W. Singleton

University of Nebraska - Lincoln

See next page for additional authors

Follow this and additional works at: <https://digitalcommons.unl.edu/physicssellmyer>

Part of the [Physics Commons](#)

Panagiotopoulos, I.; Withanawasam, L.; Murthy, A.S.; Hadjipanayis, George C.; Singleton, E.W.; and Sellmyer, David J., "Magnetic hardening of melt-spun nanocomposite Nd₂Fe₁₄B/Fe magnets" (1996).

David Sellmyer Publications. 88.

<https://digitalcommons.unl.edu/physicssellmyer/88>

This Article is brought to you for free and open access by the Research Papers in Physics and Astronomy at DigitalCommons@University of Nebraska - Lincoln. It has been accepted for inclusion in David Sellmyer Publications by an authorized administrator of DigitalCommons@University of Nebraska - Lincoln.

Authors

I. Panagiotopoulos, L. Withanawasam, A.S. Murthy, George C. Hadjipanayis, E.W. Singleton, and David J. Sellmyer

Magnetic hardening of melt-spun nanocomposite $\text{Nd}_2\text{Fe}_{14}\text{B}/\text{Fe}$ magnets

I. Panagiotopoulos, L. Withanawasam, A. S. Murthy, and G. C. Hadjipanayis

Department of Physics and Astronomy, University of Delaware, Newark, Delaware 19716

E. W. Singleton and D. J. Sellmyer

University of Nebraska, Lincoln, Nebraska 68588-0111

Coercivity optimization studies were done on melt-spun nanocomposite $\text{Nd}_4\text{R}_2\text{Fe}_{87-x}\text{NbT}_x\text{B}_6$ ($\text{R}=\text{Nd}, \text{Y}, \text{Dy}$; $\text{T}=\text{Ag}, \text{Cu}$) isotropic ribbon samples. The maximum attainable coercivities, after adjusting the annealing time, were found to be very sensitive to the annealing temperatures. The optimum magnetic properties [$H_C=3.9$ kOe, $(\text{BH})_{\text{max}}=10$ MGOe] were obtained by annealing at 750–775 °C for a few minutes. Optimization by flash annealing gave similar results. Microstructural studies show that the grain size is greater than the theoretically predicted grain size for optimum coupling between the hard and the soft phase. With the annealing conditions used, $\text{Nd}_4\text{Dy}_2\text{Fe}_{87}\text{NbB}_6$ samples gave moderate coercivities and in $\text{Nd}_4\text{Y}_2\text{Fe}_{87}\text{NbB}_6$ samples the coercivity was reduced more than the expected reduction in the anisotropy field due to the presence of Y. © 1996 American Institute of Physics. [S0021-8979(96)37708-2]

INTRODUCTION

Nanocomposite magnets consisting of a fine mixture of magnetically soft and hard phases can lead to large reduced remanences (M_r/M_s), relatively high coercivities, and high energy products due to the exchange coupling between the hard and soft phases.^{1–3} Our previous studies in Nd–Fe–B ribbons showed a reduced remanence of 0.78 and a coercivity of 2.7 kOe resulting in an energy product of 14.45 MGOe.⁴ However microstructural studies in these samples show that the grain size is larger than the optimum size predicted by theoretical calculations^{1–3} to be of the order of the domain wall width of the hard phase (~ 10 nm).

In this article we present data on optimization studies of $\text{Nd}_4\text{R}_2\text{Fe}_{87-x}\text{NbT}_x\text{B}_6$ ($\text{R}=\text{Nd}, \text{Y}, \text{Dy}$; $\text{T}=\text{Ag}, \text{Cu}$) isotropic ribbon samples. To increase the coercivity Nb and other elements (Ag, Cu) are introduced in order to prevent grain growth and help achieve small grain sizes. Substitution of Nd with other rare earth elements influences the magnetic properties in two ways: it changes the anisotropy of the hard phase which is supposed to affect the optimum grain size and therefore the maximum coercivity and also raises the crystallization temperature of the amorphous alloy.⁵

EXPERIMENT

$\text{Nd}_4\text{R}_2\text{Fe}_{87-x}\text{NbT}_x\text{B}_6$ ingots were prepared by arc-melting the constituent elements in an argon atmosphere. Pieces of these ingots were melt-spun from a quartz tube having an orifice diameter ~ 1 mm at high wheel speeds (60 m/s). These overquenched ribbon samples were then annealed at different times and temperatures to optimize the magnetic properties. After annealing, the samples were quenched in liquid nitrogen. Some of these samples were also flash annealed in high vacuum at currents of 0.2–1 A for time intervals 5–240 s. The properties of the annealed rib-

bons are very sensitive to the current. After finding a suitable annealing current, more fine optimization could be achieved by varying the annealing time. Hysteresis loops were measured in a vibrating sample magnetometer (VSM) with a maximum field of 20 kOe. The field was applied in the plane of the ribbons and along the ribbon axis to minimize demagnetizing effects. Transmission electron microscopy (TEM) studies were carried out using a Jeol JEM-2000FX microscope.

RESULTS

The magnetic properties were optimized by annealing for various time intervals at various temperatures. The effects of the annealing conditions to the magnetic properties are shown in Figs. 1–3 for $\text{Nd}_6\text{Fe}_{87}\text{NbB}_6$, $\text{Nd}_6\text{Fe}_{86.9}\text{Ag}_{0.1}\text{NbB}_6$, and $\text{Nd}_6\text{Fe}_{86.9}\text{Cu}_{0.1}\text{NbB}_6$ ribbon samples, respectively. When annealed at 650 °C the magnetic properties are far from op-

FIG. 1. Coercivity as a function of annealing time at various annealing temperatures for a $\text{Nd}_6\text{Fe}_{86.9}\text{NbB}_6$ sample.

FIG. 2. Coercivity as a function of annealing time at various annealing temperatures for a $\text{Nd}_6\text{Fe}_{86.9}\text{Ag}_{0.1}\text{NbB}_6$ sample.

timum; at this temperature the crystallization does not lead to the formation of the 2:14:1 phase. At 700 °C the optimum coercivity (~ 3.5 kOe) is reached after 1–2 h of annealing for all the samples. At temperatures 750–775 °C the optimum annealing time is reduced to few minutes and coercivities ~ 3.9 kOe are obtained. These differences in coercivities can be attributed to the different dependence of the nucleation and growth rates on the annealing temperature⁶ resulting in different kinds of microstructure. For even higher temperatures the optimum annealing time would probably be < 1 min. For this reason flash annealing was done in one of the samples. The optimum annealing time found is of the order of only 15–20 s (Fig. 4) and the optimum value of coercivity is slightly higher than that obtained by conventional annealing on the same sample.

The grain size of these optimized $\text{Nd}_6\text{Fe}_{86.9}\text{Ag}_{0.1}\text{NbB}_6$ ribbons was of the order of 30–50 nm which is greater than the size predicted by theory for optimum exchange coupling (Fig. 5). The critical dimension of the soft phase below which the soft and hard phases are optimally coupled is predicted by theory to be of the order of the Bloch wall width of the hard phase δ given by

$$\delta = \pi(A/K)^{1/2},$$

FIG. 3. Coercivity as a function of annealing time at various annealing temperatures for a $\text{Nd}_6\text{Fe}_{86.9}\text{Cu}_{0.1}\text{NbB}_6$ sample.

FIG. 4. Coercivity as a function of annealing time for flash annealed ribbons. (a) $\text{Nd}_6\text{Fe}_{86.9}\text{Ag}_{0.1}\text{NbB}_6$ at a current of 0.75 A. (b) $\text{Nd}_4\text{Dy}_2\text{Fe}_{87}\text{NbB}_6$ at a current of 0.45 A. (c) $\text{Nd}_4\text{Y}_2\text{Fe}_{87}\text{NbB}_6$ at a current of 0.60 A.

where A is the exchange stiffness constant and K is the first order anisotropy constant of the hard phase. The ratio of the actual to the optimum grain size defines the ratio H_C/H_K (Refs. 1 and 4), where H_K is the anisotropy field of the hard phase.

By substituting Nd with Dy, which has higher anisotropy field, one would expect to get higher coercivities. The anisotropy constant K of Dy on the other hand is close to that of Nd so that the critical dimension for the soft phase is not expected to be very different. However in the $\text{Nd}_4\text{Dy}_2\text{Fe}_{87}\text{NbB}_6$ sample only moderate coercivities were obtained as shown in Fig. 4 where H_C is plotted as a function of annealing time. This can be attributed to the fact that Dy increases the crystallization temperature of the 2:14:1 phase⁵ requiring annealing conditions unfavorable to keeping the grain size of the soft phase close to the optimum size.

When substituting Nd with the nonmagnetic Y, the anisotropy of the hard phase is reduced. This of course is expected to lower the attainable coercivity but on the other hand δ and consequently the optimum grain size, will now be increased to values closer to the actual one. As shown in Fig. 4 for the $\text{Nd}_4\text{Y}_2\text{Fe}_{87}\text{NbB}_6$ sample H_C is reduced by more than the expected reduction in anisotropy when substituting 1/3 of Nd atoms by Y. In both cases it seems that the effect of the substitutions on the kinetics of the crystallization process does not permit a direct interpretation of the data in terms of

FIG. 5. TEM micrograph of a flash annealed $\text{Nd}_6\text{Fe}_{86.9}\text{Ag}_{0.1}\text{NbB}_6$ sample.

the expected changes in the anisotropy of the hard magnetic phase.

CONCLUSION

The optimum magnetic properties in $\text{Nd}_6\text{Fe}_{86.9}\text{T}_{0.1}\text{NbB}_6$ samples were obtained by annealing at 750 °C for a few minutes. Flashed annealed samples for shorter times gave similar results. However microstructural studies show that the grain size in these samples is greater than the theoretically predicted grain size for optimum coupling between the hard and the soft phase. Therefore one can expect even better properties in these samples by proper control of the crystallization procedure that would lead to smaller grain sizes.

ACKNOWLEDGMENTS

This work was supported by the DOE DE-FG02-86ER45262 and DE-FG02-90ER45413.

¹E. F. Kneller and R. Hawig, IEEE Trans. Magn. **27**, 3588 (1991).

²R. Skomski and J. M. D. Coey, Phys. Rev. B **48**, 15812 (1993).

³T. Schrefl, H. Kronmüller, and J. Fidler, J. Magn. Magn. Mater. **127**, L273 (1993).

⁴L. Withanawasam, G. C. Hadjipanayis, and R. F. Krause, J. Appl. Phys. **75**, 6646 (1994).

⁵L. Withanawasam, A. S. Murthy, G. C. Hadjipanayis, and R. F. Krause, J. Magn. Magn. Mater. **140–144**, 1057 (1995).

⁶V. E. Martin, J. Bernaldi, J. Fidler, F. Cebollada, and J. M. Conzalez, J. Alloys Compounds **191**, 127 (1993).