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Magnetic hardening in rapidly quenched Fe–Pr and Fe–Nd alloys

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We report studies of high-field magnetization and thermomagnetic effects in rapidly quenched and heat treated alloys based on Fe–Pr and Fe–Nd. Coercivities up to ~40 kOe and large energy products result from the precipitation of a finely dispersed crystalline phase. Studies of varying the alloy composition and heat treatment are reported.

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INTRODUCTION

Recent research on rapidly quenched and heat treated alloys based on Fe and Pr has shown that giant coercivities (H_c) and large energy products $(BH)_m$ can be obtained. Our work was stimulated by the discovery of Cornelison *et al.*[1,2] that $Pr_{56}Fe_{30}Ga_{14}$ had a coercivity of 60 kOe at 100 K and that this magnetic hardness resulted from a phase separation into Fe-rich and Fe-deficient regions.[3] Hadjipanayis *et al.*[4] increased the Fe content in this series and heat treated the alloys to study the effects of crystallization on the magnetic hardness. The result was that though the remanent moment (M_R) increased considerably on adding Fe, H_c decreased after crystallization and the hard magnetic properties disappeared. However, subsequent studies of Fe–Pr-based alloys containing metalloids including B, Si, and mixtures did produce surprisingly good properties.[5] For example, in $Pr_{14}Fe_{71}B_{15}$ values of $H_c \sim 8$ kOe and $(BH)_m \cong 8.5$ MGOe were obtained after a heat treatment at 750 °C, and transmission electron microscope studies showed a very fine precipitate structure of dimension below 100 Å. Even better properties were obtained in rapidly quenched and heat treated $Fe_{76}Pr_{16}B_5Si_3$ for which H_c values between 5 and 20 kOe and $(BH)_m \simeq 12$ MGOe were obtained.[6] Studies on related materials have also shown high coercivities and energy products. Koon and Das[7] reported a value of $H_c = 9$ kOe on melt-spun and heat-treated FeBTbLa and Croat[8] has achieved $(BH)_m \simeq 3$–4 MGOe in $R_{40}Fe_{60}$ alloys where R = Pr, Nd, and Sm.

The purpose of the present work was to explore more fully the effects of alloying, both with respect to Fe composition in a given series and variation of rare-earth element, and also to study the effects of changing the temperature and length of heat treatment. The alloys studied were of the form $Fe_xR_yM_z$ where R = Pr or Nd, M = B, Si, C, Al, or mixtures, and $74 \leqslant x \leqslant 82$, $14 \leqslant y \leqslant 16$, $4 \leqslant z \leqslant 10$.

EXPERIMENTAL METHODS

Alloys of the desired compositions were prepared by arc melting the pure constituents several times. This was followed by a homogenization at 1100 °C for several hours. The melt-spinning technique was used to rapidly quench the alloys into ribbons. Before annealing, the samples were wrapped in Ta foil and placed in a quartz tube which was evacuated and backfilled with argon. Low temperature and high field magnetization measurements were made with a vibrating sample magnetometer (VSM) in a superconducting solenoid to 80 kOe. Also VSM measurements to 17 kOe were made in conventional magnets at room temperature and above. In selected cases microstructure studies were performed with a Philips 400 TEM.

RESULTS AND DISCUSSION

The as-quenched samples are in general magnetically soft. Upon heating structural charges occur as is shown by thermomagnetic data, $M(T)$, taken in a small field. As already reported for $Fe_{76}Pr_{16}B_5Si_3$,[6] $M(T)$ decreases smoothly towards zero with a Curie temperature of the as-quenched phase of about 160 °C. However, this behavior is not general. In samples such as $Fe_{82}Pr_{12}B_4Si_2$ and $Fe_{83.3}Nd_{12.5}B_{4.2}$ there is a lower temperature Curie point at around 100 °C and a higher one at 360 °C. That is, the as-cast phase contains two or more phases. In other compositions such as $Fe_{78}Pr_{16}B_{4.6}Si_{1.4}$ and $Fe_{78}Nd_{10}Pr_{2.5}(BSi)_{9.5}$ we have observed no magnetic transitions below 350 °C. Thus it appears that the microstructure of the as-quenched state is very sensitive to compositions and probably to quench rate. In some cases the as-quenched samples have been shown by x-ray diffraction to consist of a mixture of an amorphous phase and a tetragonal phase identified as $Fe_{20}R_3B$.[6,9] This phase appears to have a Curie temperature of about 350 °C.

Magnetic hardening occurs in these alloys after heat treatments at around 700 °C. Figures 1–3 show some examples of hysteresis loops for alloys of various compositions at several temperatures. Fields up to 80 kOe were used at temperatures below 160 K, but measurements in our high-temperature VSM were limited to 17 kOe. It is obvious that most of the loops are minor ones, particularly for the 300 K data. Some interesting features of the data are as follows. (1) In the cases of FePr and FeNd shown in Figs. 1 and 2 there is a general trend in which the loops at low temperature are constricted suggesting a two magnetic phase system. At intermediate temperatures (~160 K) the loops show "bulges" along the field axis leading to H_c values of 30–40 kOe. At 300 K $H_c \simeq 10$ kOe but, again, these are only minor loops. (2) There is a much larger temperature dependence of the magnetization for the FePr alloy than the FeNd alloy (Figs. 1 and 2). This might be associated with the longer anneal time in the Nd alloy. (3) The loop for the FeNdBSi sample of Fig. 3 is quite different from the corresponding Pr alloy of Fig. 1. In

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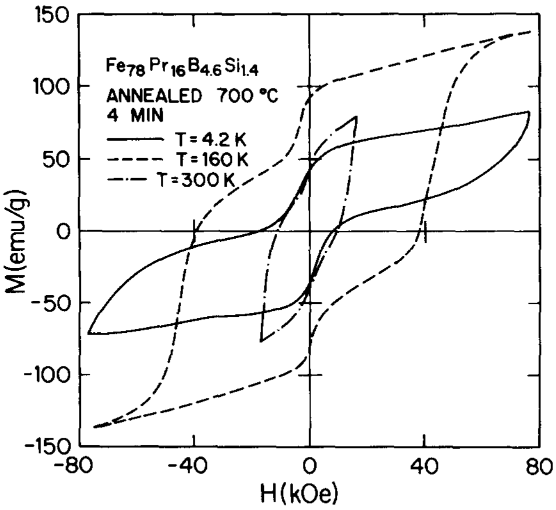

FIG. 1. Hysteresis loops for $\mathrm{Fe_{78}Pr_{16}B_{4.6}Si_{1.4}}$ at several temperatures.

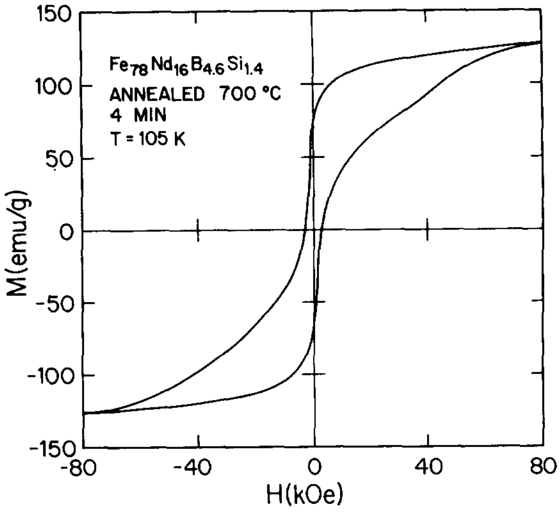

FIG. 3. Hysteresis loop for $\mathrm{Fe_{78}Nd_{16}B_{4.6}Si_{1.4}}$ at 105 K.

addition there is relatively little temperature dependence of the loops in this alloy: $H_c(T = 4.3\ \mathrm{K}) = 9.7$ kOe and there is a smooth decrease to 5.7 kOe at 105 K. (4) Given a moment (extrapolated to $H = 0$) of 150 emu/g for the $\mathrm{Fe_{76}Nd_{16}B_8}$ sample at 4.3 K (Fig. 2) and assuming that each Fe atom has a moment of $2.2\mu_B$, the average moment on the Nd atoms is $\bar{\mu}_{\mathrm{Nd}} = 1.67\mu_B$. Since gJ for $\mathrm{Nd^{3+}}$ is 3.27, this suggests the net moment on the Nd subnetwork, in whatever phases, is parallel to the Fe moments but may be spread out into a hemispherical fan with average moment $gJ/2$. (5) Corresponding samples containing rare-earth or transition metals without orbital angular momentum (Gd or Y) are not magnetically hard. For example, $\mathrm{Fe_{76}Y_{16}B_8}$ when annealed for 30 min at 700 °C yielded $H_c \approx 2$ kOe for temperatures between 4.2 and 300 K and a moment (extrapolated to $H = 0$) of 147 emu/g at 4.2 K. Thus the hard properties are intimately connected with the strong magnetocrystalline anisotropy of the light rare-earth ions.

Figure 4 shows the temperature dependence of H_c and the magnetic remanence M_R for $\mathrm{Fe_{76}Nd_{16}B_8}$. An increase of H_c (and M_R) with increasing temperature is seen, at least up to 160 K. Similar behavior was seen in rapidly quenched PrFeGa^2 alloys which are known to contain two magnetic phases. Above a high temperature T_0 which is not yet known for $\mathrm{Fe_{76}Nd_{16}B_8}$, H_c should decrease with T because of thermal activation of particles or domain walls over energy barriers. As T is lowered below T_0 there is insufficient thermal energy to aid the field in moving "frozen" domains, which then do not participate in the magnetization reversal process. Thus the loops narrow and the remanence decreases.

TEM studies have shown that the heat treated FePr samples have a very fine precipitate structure of spherical particles with dimensions below 100 Å. These particles are likely to have the tetragonal $\mathrm{Fe_{20}Pr_3B}$ structure[9] which accounts for the high anisotropy. Limited studies of the effect of heat treatment variables have shown the magnetic hardness to be a sensitive property of the anneal temperature but a rather weak function of the time of anneal.

These new alloys are of great interest as permanent magnet materials. Further studies with higher magnetic fields and with differing alloy compositions are needed to understand more fully the relationship between magnetic hardness and microstructure.

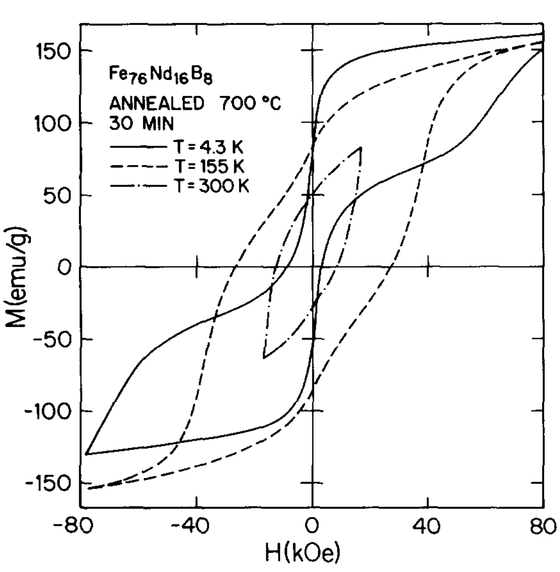

FIG. 2. Hysteresis loops for $\mathrm{Fe_{76}Nd_{16}B_8}$ at several temperatures.

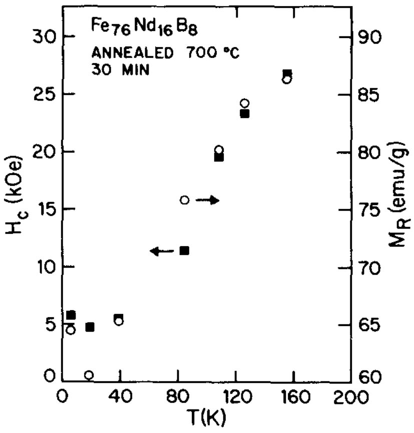

FIG. 4. Temperature dependence of apparent coercive force and remanence measured in $H_{\max} = 80$ kOe.

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[1] S. G. Cornelison, D. J. Sellmyer, and G. Hadjipanayis, J. Appl. Phys. **52**, 1823 (1981).

[2] S. G. Cornelison, D. J. Sellmyer, J. G. Zhao, and Z. D. Chen, J. Appl. Phys. **53**, 2330 (1982).

[3] J. G. Zhao, S. G. Cornelison, G. Hadjipanayis, and D. J. Sellmyer, J. Non-Cryst. Solids **55**, 203 (1983).

[4] G. C. Hadjipanayis, S. H. Wollins, R. Hazelton, K. R. Lawless, and D. J. Sellmyer, J. Appl. Phys. **53**, 7780 (1982).

[5] G. C. Hadjipanayis, R. C. Hazelton, K. R. Lawless, and D. J. Sellmyer, J. Magn. Magn. Mater. (in press).

[6] G. C. Hadjipanayis, R. C. Hazelton, and K. R. Lawless, Appl. Phys. Lett. **43**, 797 (1983).

[7] N. C. Koon and B. N. Das, Appl. Phys. Lett. **39**, 840 (1981).

[8] J. J. Croat, IEEE Trans. Magn. **MAG-18**, 1442 (1981).

[9] H. H. Stadelmaier and H. K. Park, Z. Metallkd. **72**, 417 (1981).

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