

MAY 18 1998

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Extrinsic Curie Temperature and Spin Reorientation Changes in Nd₂Fe₁₄B/ α -Fe Nanocomposites

CONF-980802--

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Abstract:

The Curie temperatures and spin reorientation temperatures of a series of four melt-spun nanocomposite materials comprised of Nd₂Fe₁₄B and varying amounts of α -Fe were measured using independent techniques. The phase constitution and grain size was assessed with synchrotron x-ray diffraction; the Curie temperatures were measured by differential thermal analysis (DTA) and *dc* SQUID magnetometry in the temperature range $375 \text{ K} \leq T \leq 800 \text{ K}$, whereas the spin reorientation transition temperature was determined from *ac* susceptibility measurements taken in the range $10 \text{ K} \leq T \leq 300 \text{ K}$. The Curie temperature increases with increasing excess iron content, resulting in a 18° enhancement over the Curie temperature of pure Nd₂Fe₁₄B for 27 wt% excess α -Fe. The spin reorientation temperatures are depressed from the single-crystal value by an average of 10 degrees. Both anomalous effects are attributed to intergranular exchange coupling present in the alloys, although the effects of uncompensated stress between the constituent phases cannot be ruled out. The experimental results suggest that while the Curie temperature of the Nd₂Fe₁₄B phase may be extrinsically enhanced significantly beyond the bulk value, possibly extending the range of applications of this compound, the anisotropy may be simultaneously lowered, impeding the attainment of high coercivities in these alloys.

Introduction:

It is a prescript that the local environment surrounding an atom determines the electronic properties of that atom and of the material comprised of such atoms. It has long been known that the surfaces of magnetic materials can exhibit significantly different properties than their bulk counterparts. Crittenden and Hoffman [1] found that the magnetization of polycrystalline Ni films of thickness 50 nm was reduced from the bulk value by as much as 40%. Measurements performed on thin films of Co, Ni and Fe have shown that the Curie temperature may be hundreds of degrees lower than the respective bulk values [2, 3], while a highly enhanced Curie temperature was found for thin films of Ni-Al [4], Co-Ni and Ag-Ni [5]. The importance of the surface is not limited to materials in thin film form: the concept may also be applied to bulk materials comprised of nanoscale grains, which are characterized by an extremely large proportion of interphase grain boundary area. Such a microstructure may confer properties that are significantly different from those exhibited by their bulk, large-grained analogs, such as strength, ductility, thermal expansion and specific heat [6, 7].

In this work we explore some of the changes found in selected magnetic properties of nanocomposite phase constituents that differ from those found in the bulk, large-grained state. These properties, the Curie temperature T_C and the spin reorientation temperature T_S , are conventionally considered to be intrinsic parameters; *i.e.*, inherent to the compound itself and therefore not affected by the microstructure. The data described in this paper are intriguing not only from a fundamental science viewpoint, but also from a practical standpoint. The results of this work suggest the mechanism for the elevation of the Curie temperature of the high-coercivity Nd₂Fe₁₄B phase responsible in nanocomposite exchange-spring alloys may also be responsible, in part, for the low coercivities found in these materials. Both results impact the applications of these alloys.

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Experimental:

Measurements were performed on four meltspun nanocomposites consisting of Nd₂Fe₁₄B (2-14-1) and α-Fe that differ in the amount of α-Fe present. The influence of the amount of excess iron, present as α-Fe, on the Curie temperature T_C and the spin reorientation temperature T_S of the Nd₂Fe₁₄B compound present in the two-phase exchange-coupled nanocomposite was examined. The microstructure and phase constitution of the alloys were characterized by synchrotron x-ray diffraction. The behavior of the Curie temperature of Nd₂Fe₁₄B in two-phase melt-spun nanocomposites was investigated as a function of composition by two different, independent methods: differential thermal analysis (DTA) and temperature-dependent magnetic measurements. The spin reorientation transition was examined on thermally-demagnetized specimens using *ac*-SQUID magnetometry.

The alloys were made from commercial-grade materials using standard melt-quenching techniques. The nominal starting compositions given in Table I, with the composition denoted by both the stoichiometry and the wt% enrichment of iron in the alloy. The alloys are identified by their excess iron enrichment δ, defined as Nd₂Fe_{14+δ}B. The overquenched ribbons were annealed for four minutes at 690°C to optimize their hysteretic magnetic properties. Synchrotron x-ray diffraction was performed at the National Synchrotron Light Source, Brookhaven National Laboratory on powdered samples with radiation of wavelength 0.90 Å ≤ λ ≤ 1.18 Å, to verify the phase composition and the average grain size of the alloys. The chosen x-ray wavelengths are well away from the absorption edge of iron, and thus the production of fluorescence is avoided. The peaks of the diffraction spectrum were fit to pseudo-Voigt functions using a step-scan data peak fitting program, and the determined 2θ values were entered into a least-squares fitting program to calculate the lattice parameters. In all cases only two phases were found, Nd₂Fe₁₄B and α-Fe; it is estimated that synchrotron x-ray diffraction would detect minor phases of 0.1 - 1.0 wt % [8]. The crystallite sizes are determined from the half-width of the Bragg peak at the half-maximum intensity position, corrected for the intrinsic broadening of the synchrotron beam, using the Scherrer formula. The lattice parameters and the average grain sizes found for each phase within the samples are included in Table I.

Stoichiometry	Excess Fe (wt%/vol%)	Starting Composition (RE = rare earth)	Lattice Parameters (Å)	Grain Sizes (Å)	Remanence Enhancement (B _r /M _s)
Nd _{2.39} Fe ₁₄ B _{0.95} (δ=0)	0/0	RE = 30 wt% B = 0.9 wt% Fe = 68.6 wt%	2-14-1: a = 8.804 ± 0.002 c = 12.261 ± 0.005	2-14-1: ≈ 340	0.53
Nd ₂ Fe _{18.6} B _{2.38} (δ=4.6)	19.0/18.4	RE = 21.3 wt% B = 1.90 wt% Fe = 76.8 wt%	2-14-1: a = 8.791 ± 0.001 c = 12.170 ± 0.002 α-Fe: a = 2.86	2-14-1: ≈ 525 α-Fe: ≈ 175	0.52
Nd ₂ Fe _{21.2} B _{1.65} (δ=7.2)	27.4/26.2	RE = 19.4 wt% B = 1.2 wt% Fe = 79.4 wt%	2-14-1: a = 8.787 ± 0.002 c = 12.178 ± 0.003 α-Fe: a = 2.87	2-14-1: ≈ 340 α-Fe: ≈ 290	0.56
Nd ₂ Fe _{23.3} B _{1.45} (δ=9.3)	32.2/31.3	RE = 18 wt% B = 0.99 wt% Fe = 81.0 wt%	2-14-1: a = 8.805 ± 0.002 c = 12.214 ± 0.005 α-Fe: a = 2.88	2-14-1: ≈ 235 α-Fe: ≈ 180	0.60

Table 1: Alloy Characterization. δ characterizes the iron enrichment, defined as Nd₂Fe_{14+δ}B.

Measurements to determine the Curie temperature transition in the nanocomposite alloys were performed with a TA Instruments Model SDT 2960 Simultaneous DTA/TGA instrument. Separate samples were examined in the temperature range $375 \text{ K} \leq T \leq 800 \text{ K}$ to examine the $\text{Nd}_2\text{Fe}_{14}\text{B}$ Curie temperature and in the range $375 \text{ K} \leq T \leq 1200 \text{ K}$ to examine the $\alpha\text{-Fe}$ Curie temperature, using a ramp rate of $20^\circ/\text{min}$. Two separate 100-mg quantities of sample per temperature region were used. Each sample was cycled up and down in temperature three times, to yield a total of six Curie temperature measurements per composition, with an associated error in T_C determined by the standard deviation of the six measurements ($< 1\%$). The measurements were performed in an atmosphere of high-purity Ar with 1% hydrogen. The addition of 1% hydrogen was found to be necessary in order to limit the weight gain of the samples via oxidation during the DTA measurement to under 0.2%. There is no indication of hydrogen uptake in the alloys during measurement. The Curie temperature transition produces a change in the slope of the DTA curve with both heating and cooling, as shown in Fig. 1a). The first heating cycle always yields a differ-

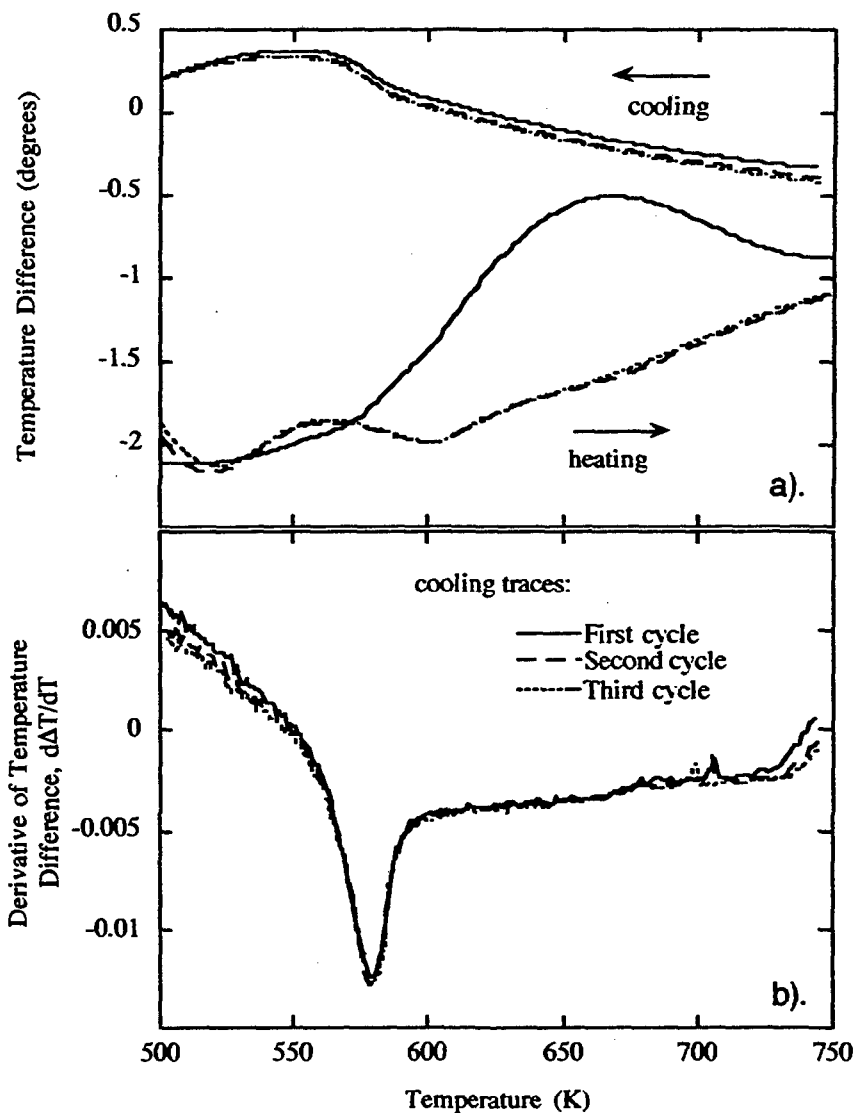


Figure 1. (a) DTA heating and cooling traces of the $\delta = 0$ sample. (b). Temperature derivatives of the DTA cooling traces of the $\delta = 0$ sample. The curves' minima are identified with the Curie temperature.

ent curve than that observed in subsequent cycles. The Curie temperatures were determined as the minimum in the temperature derivative of the DTA cooling curve, Fig. 1b). Magnetic measurements to determine the Curie temperature T_C and the spin reorientation temperature T_S were performed with a Quantum Design SQUID MPMS magnetometer. Powdered samples were packed into silica tubes of *i.d.* = 1.8 mm, held in place by silica wool and silica rods and sealed under a vacuum of $P \approx 1 \times 10^{-6}$ torr [9]. DC magnetic data were obtained in the temperature range $400 \text{ K} \leq T \leq 650 \text{ K}$ to investigate the Curie temperature, and ac magnetic susceptibility data were obtained in the range $10 \text{ K} \leq T \leq 300 \text{ K}$. Spin reorientation measurements were done on thermally demagnetized samples, as the data collected from magnetized samples was especially noisy and ambiguous. The degree of exchange enhancement present in the samples was evaluated via the room temperature remanence ratios B_r/M_s , obtained under maximum applied field of 5 T (Table I). The *dc* data were corrected for demagnetization effects, and the saturation magnetization M_s was obtained by extrapolating the high-field portion ($H_{\text{int}} \geq 20,000 \text{ G}$) of the demagnetization curve out to infinite applied field.

The determination of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ Curie temperature T_C using magnetic measurements must be done with care, as the $\text{Nd}_2\text{Fe}_{14}\text{B}$ nanocrystals are subjected to a local field emanating from both the α -Fe as well as the externally-applied measuring field. It is necessary to approach a zero-field determination of the Curie temperature, analogous to the conditions of the DTA measurements. This task was accomplished by utilizing the procedure of Weiss and Forrer [10]. The spontaneous magnetization of the sample at a specific temperature in zero field was determined from the linear extrapolation of temperature T data plotted against H_{appl} for a constant value of magnetization M , obtained from M vs. T curves from various applied fields, $5 \text{ T} \leq H_{\text{appl}} \leq 1 \text{ T}$. The temperature-dependent spontaneous magnetization of α -Fe is subtracted from the sample's zero-field spontaneous magnetization. The square of the zero-field spontaneous magnetization M thus obtained is plotted against T and linearly extrapolated to $M = 0$ to obtain the Curie temperature. Illustrations of this procedure are available in reference 11. Comparison of two separate measurements performed on the same sample yield a deviation of 0.5%. Room-temperature demagnetization curves measured before and after the elevated-temperature M vs. T measurements demonstrated that hysteretic properties such as coercivity, remanence and magnetization at 5T exhibited at most a 5% change, indicating that the phase constitution and nature of the samples was not altered in a significant way during the course of the measurement.

The spin reorientation transition temperature was determined from ac susceptibility measurements obtained in an applied dc field of 0 G and a driving field of 3.5 G at 20 hz. The samples were studied under zero applied field, to provide a maximum susceptibility signal (trapped flux in the superconducting magnet provides a remanent field less than 4 G.) The ac susceptibility data obtained from the nanocomposite samples was compared with that obtained from an oriented single crystal of $\text{Nd}_2\text{Fe}_{14}\text{B}$, to assist in the data interpretation. The minimum in the temperature derivative of the in-phase (real) portion of the ac susceptibility data is widely associated with the spin reorientation temperature in $\text{RE}_2\text{Fe}_{14}\text{B}$ -based alloys [12, 13].

Results:

Table 2 displays the Curie temperatures determined by both thermal and the magnetic methods; Fig. 3 illustrates the data in Table 2 graphically. The differences between the temperature scales in Fig. 3 should be emphasized. A clear increase in the Curie temperature of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase with increase in the amount of excess iron is demonstrated for both experimental techniques. The Curie temperatures of the α -Fe phase are unchanged with composition, within experimental uncertainty. The Curie temperature found for the nominally single-phase $\text{Nd}_2\text{Fe}_{14}\text{B}$ ($\delta=0$) sample from both methods, $T_C \sim 575 \text{ K}$, is in good agreement with that found by other researchers [14], while the 2-14-1 phase present in the sample

Stoichiometry	T_C : $Nd_2Fe_{14}B$ (dc magnetic measurements; error estimated as $\pm 0.5\%$)	T_C : $Nd_2Fe_{14}B$ (DTA measurements)	T_C : α -Fe (DTA measurements)	T_S : $Nd_2Fe_{14}B$ (ac magnetic measurements)
$Nd_{2.39}Fe_{14}B_{0.95}$ ($\delta=0$)	573.0 K	576.0 ± 0.36 K	(N/A)	120 K
$Nd_2Fe_{18.6}B_{2.38}$ ($\delta=4.6$)	589.0 K	572.4 ± 1.10	1042.6 ± 0.2 K	127 K
$Nd_2Fe_{21.2}B_{1.65}$ ($\delta=7.2$)	597.0 K	579.4 ± 1.52	1042.6 ± 0.2 K	124 K
$Nd_2Fe_{23.3}B_{1.45}$ ($\delta=9.3$)	590.0 K	586.2 ± 0.35	1043.0 ± 0.3 K	122 K
$Nd_2Fe_{14}B$ single crystal	N/A	N/A	N/A	133 K

Table 2. Measured Curie temperatures (T_C) of the constituent phases in the nanocomposite alloys and spin reorientation temperatures (T_S) of the $Nd_2Fe_{14}B$ phase.

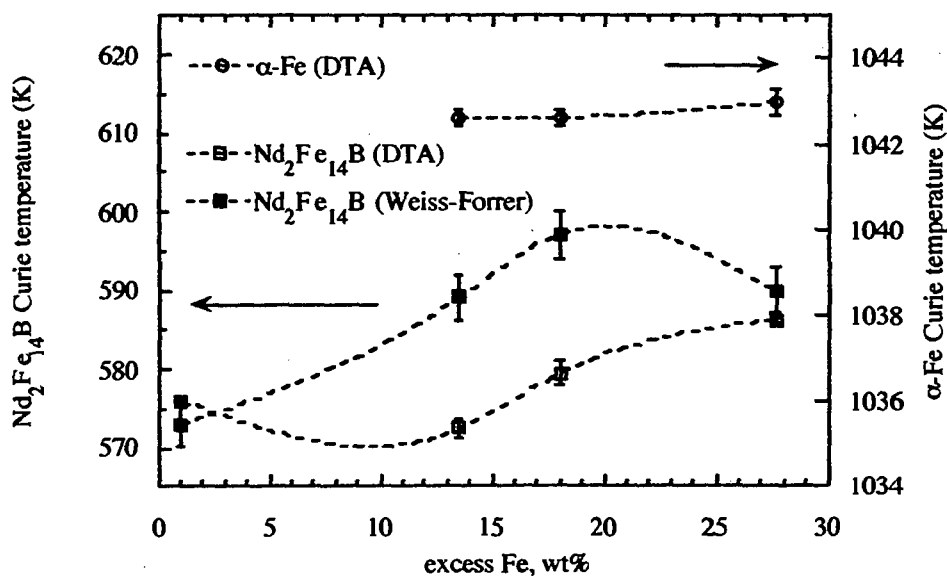


Figure 2: Curie temperature determinations of the constituent phases in the nanocomposite alloys. Lines drawn between data points to guide the eye.

with the most excess iron ($\delta = 9.3$) exhibits an approximate 2% increase in the Curie temperature over that of the nominally pure sample. Depending upon the method used, the maximum Curie enhancement is between 11 and 22 degrees. The average Curie temperature found for the α -Fe phase, 1043 K, is in excellent agreement with the literature value of 1044 K and shows essentially no variation with amount of 2-14-1 content. At present the differences between the information produced by the two techniques employed in this work are not understood, but it is recognized that the Curie temperature determined by the Weiss-Forrer method is highly dependent upon the magnitude of the moment of the second phase subtracted from the measurements. Additionally, the measurement time scales of the two methods are not the

same. For these reasons we believe that the DTA data is more reliable than the *dc* magnetic data in the determination of the Curie temperatures of the phases present in the samples.

The interpretation of *ac* susceptibility data from magnetic materials is often not straightforward because of their dependence on sample shape, microstructure, domain structure and lattice impurities [15]. As stated previously, however, the spin reorientation temperature is routinely determined as the minimum in the temperature derivative of the in-phase susceptibility. The normalized temperature derivative of the in-phase (real) part of the *ac* susceptibility data from representative samples is shown in Figure 3; results for all specimens are quantified in Table 2. Data obtained from a Nd₂Fe₁₄B single crystal specimen, measured along the (001) direction, is included in Figure 3. In general the spin reorientation transition is rather sharp for the single crystal but is broad for the nanocomposite samples, with associated spin reori-

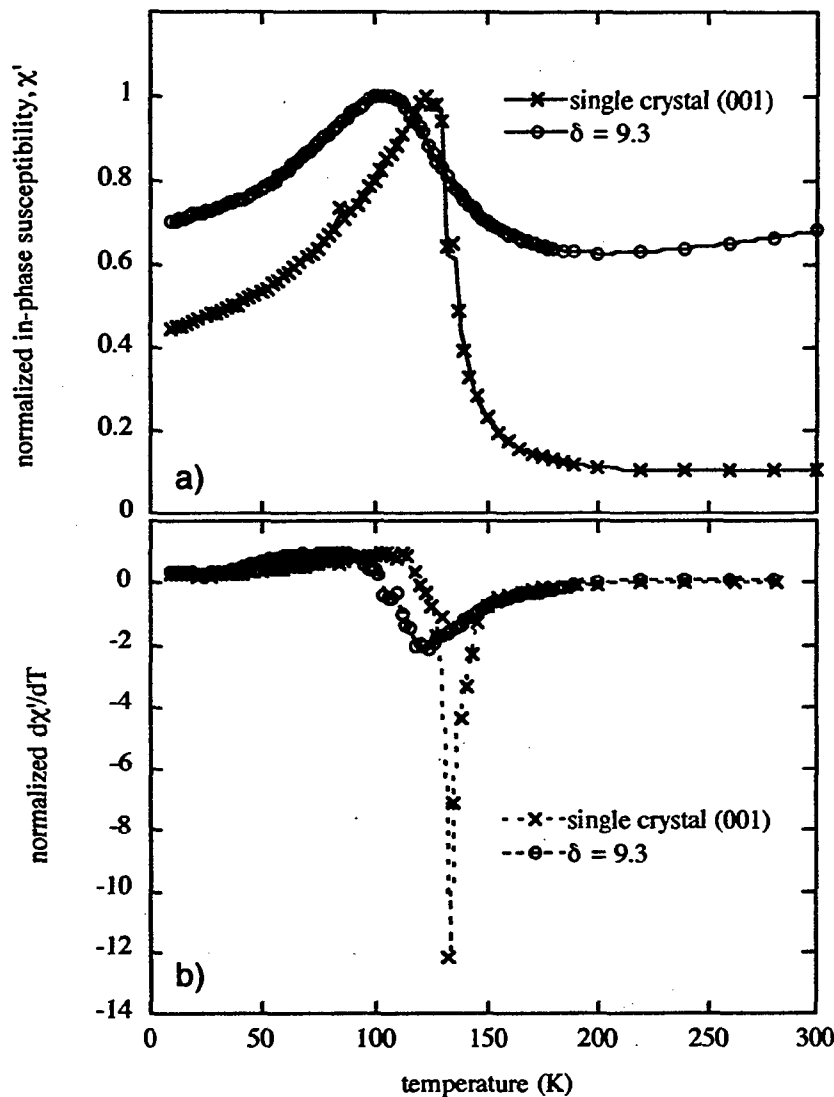


Figure 3. a). Normalized in-phase (real) data of the *ac* susceptibility for a single crystal sample (measured with field parallel to (001) direction) and the nanocomposite sample with the greatest amount of excess iron ($\delta=9.3$). b). Normalized temperature derivatives of the in-phase data in a)., above. The minimum in the temperature derivative is associated with the spin reorientation temperature.

entation temperatures depressed by an average of 10 degrees from that found for the single-crystal sample.

Discussion:

It will be assumed that the only significant difference among the samples of this study is the amount of excess α -Fe present in the microstructure. The data in Table 1 indicates that the lattice parameters and microstructure of all samples are similar, although not identical. The grain sizes of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase are in the range 25 - 50 nm, and are always larger than those of the α -Fe phase, which is smaller than 30 nm in all instances. There is no obvious correlation between the amount of excess iron and the precise scale of the resultant microstructure. Similarly, there is no obvious trend in the lattice parameters of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase with excess iron content, although the lattice parameter of the α -Fe phase appears to increase with increasing α -Fe content beyond the JCPDS reference lattice parameter value for α -Fe of $a = 2.8664 \text{ \AA}$. However, the measured Curie temperature of the α -Fe phase confirms its identity. The broadness of the spin reorientation transitions makes it difficult to assign an uncertainty in the determined T_S . However, previous investigations into the spin reorientation temperatures of Pr-substituted $(\text{Nd}_{1-x}\text{Pr}_x)_2\text{Fe}_{14}\text{B}$ melt-spun alloys [16] indicate that the depression in the nanocomposite T_S relative to the single-crystal value is not attributable to chemical differences, as the depression is very slight for low values of Pr content, as might be found in commercial Nd powder. Thus it may be hypothesized that α -Fe crystallites are dispersed moderately evenly throughout the material's matrix for all samples, and that the main difference amongst the samples is the amount of interphase boundary region.

The elevated Curie temperatures T_C and depressed spin reorientation temperatures T_S of $\text{Nd}_2\text{Fe}_{14}\text{B}$ measured in the nanocomposite samples indicates that the nanoscaled phase is behaving in a significantly different manner than its bulk counterpart. It is postulated that interplay of electron exchange across $\text{Nd}_2\text{Fe}_{14}\text{B}/\alpha$ -Fe interfaces underlies the alteration of the magnetic properties measured in this study, although possible effects such as interstitial occupancy of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ lattice and unrelieved internal stresses due to rapid solidification must be considered. It is unlikely that interstitial occupancy of extra iron within the $\text{Nd}_2\text{Fe}_{14}\text{B}$ lattice accounts for the observed increase in the Curie temperature: not only is there too much iron for such incorporation, but many researchers have concluded that $\text{Nd}_2\text{Fe}_{14}\text{B}$ is essentially a line compound, with little non-stoichiometry present [17]. While Fe interstitial occupation of the 2-14-1 lattice may be largely ruled out as the cause of the increased Curie temperatures observed in this study, the same may not be said of residual interphase interface stress. P. Gorria *et al.* [18] recently reported an increase of approximately $50^\circ/\text{GPa}$ in the Curie temperature of FeZrBCu-based amorphous alloy with increasing applied tensile stress. This effect is apparently very sensitive to the chemical constitution of the alloy, as the opposite behavior is observed in analogous FeNb-based alloys. In any case, the report of Gorria *et al.* lacks a clear description of the nanostructure of the alloy which is crucial to an analysis of their results; the samples in this study have been annealed and therefore possess an internal stress state that is lower than that of amorphous alloys.

The two constituent phases present in these alloys are indeed moderately exchange coupled, as indicated by the room-temperature remanence ratios, Table I, and by previous work [19]. It appears that the active exchange coupling field energy emanating from the robust ferromagnetic phase with atomic proximity to the incipiently-transforming ferromagnetic phase prevails over the ambient thermal randomizing energy, producing an effective increase in the Curie temperature. The experimental data of this study suggest that the larger the amount of interphase interfacial coupling, the better the transforming ferromagnetic phase is able to resist the Curie transition. Presumably, the increase of the Curie temperature of the matrix phase must depend upon the overall grain size, the topology of the ferromagnetic phase and on the ratio of the interphase surface area to the volume ratio of the grains the lower T_C phase. The work of Wang and Mills [20] support this supposition. They performed calculations of an increase in the Curie temperature of one

ferromagnetic component of heterogeneous, ferromagnetic, model thin film system with Ising character using Landau-Ginzburg theory. Depending upon the thickness and periodicity of the modeled systems, they calculate a 0.4 % to a 4.4 % increase in the Curie temperature of the lower-transition temperature phase. This result is consistent with the experimental data. A decrease in the Curie temperature of the higher transition-temperature component also results from the model calculations.

The observed depression in the spin reorientation temperatures of the nanocomposites may also be attributed to the exchange coupling present in the system. Herzer [21, 22] provides a possible explanation for the observed behavior of the spin reorientation transitions measured in this study. The spin reorientation transition in Nd₂Fe₁₄B is a manifestation of temperature-induced anisotropy changes. Herzer concludes that in nano-scaled systems with an average grain size smaller than the ferromagnetic exchange length, L_{ex}^0 :

$$L_{ex}^0 = \left(\frac{A}{k_1} \right)^{\frac{1}{2}} \quad (1)$$

(where A is the exchange stiffness of the matrix compound and k_1 is the first-order anisotropy constant), the magnetization vector cannot follow the randomly-oriented easy axis of each individual grain. This forms mesoscopic interaction domains that possess a greatly reduced effective anisotropy that is equal to the average vectoral sum of the anisotropies of the constituent grains. The reduced spin reorientation temperatures reflect a reduced anisotropy produced by the intergranular exchange coupling, while the broadness of the transition suggests a magnetically inhomogeneous microstructure with varying degrees of intergranular coupling. It is expected that the relatively low anisotropy of the α -Fe phase will further degrade the overall nanocomposite anisotropy; however, it is unclear at present if this prediction is borne out in the T_s data (Table 2).

Reports of changes in the magnetic properties due to a given materials system's microstructure are appearing more frequently in the literature. Recent studies performed by Hernando and coworkers [23, 24] on Fe-rich partially-devitrified soft magnetic nanocomposite alloys of FeZrBCu and FeNbBCu have demonstrated the presence of an elevated Curie temperature for the intergranular amorphous matrix adjacent to α -Fe precipitates. Dahlgren and Grössinger *et al.* [25, 26] have found anomalies in the magnetic properties of alloys similar to those studied here, and Chen, Daniil and Hadjipanayis [27] have found an enhancement on the order of 100 degrees in the Curie temperature in the Re₂M₁₇-type hard magnetic phase of a Sm₂(Fe,Co)₁₅Cr₂C₂/(Fe,Co) nanocomposite material.

In addition to the scientific interest of this work, there is significant technological potential to be explored. It is believed by many researchers in the field that "exchange-spring" nanocomposites will constitute the next-generation permanent magnet [28, 29]. A major disadvantage of magnets based on the Nd₂Fe₁₄B composition is their low Curie temperature, which limits their use to applications that operate at temperatures $T < 150$ °C. To overcome this limitation elements such as Co, Si and Ga are often substituted into the 2-14-1 lattice, a substitution which can result dilution of the magnetization and/or reduction of the coercivity [14]. The results of this work suggest that it may be possible to engineer Nd₂Fe₁₄B-based magnets with enhanced Curie temperatures that possess a very high magnetization both due to the α -Fe content and to the chemically undiluted 2-14-1 phase. At the same time it is recognized that, in practice, exchange-spring magnets always possess a low coercivity, even though theoretical calculations indicate that ideal exchange-spring magnets should possess coercivities on the order of 25% of the theoretical coercive limit $2K_1/M_s$ [30]. The spin reorientation measurements in this study may provide some additional insight into this discrepancy. It is expected that exchange-induced reductions in the anisotropy fields of

nanoscale magnetic materials will lead to reduced coercivities, either by lowering the energy barrier for coherent rotation, or by providing regions of reduced anisotropy for the nucleation of reversed domains. It is hoped that the results of this study will assist in the further elucidation of the interplay between microstructure and magnetic properties in this class of magnets.

Acknowledgements:

We are grateful to B. Hoermann, Northwestern University, and to K. Gallagher, Carnegie Mellon University, who performed the differential thermal analysis measurements. Research performed under the auspices of the U.S. D.O.E., Division of Materials Sciences, Office of Basic Energy Sciences under contract No. DE-AC02-98CH10886, and carried out in part at the National Synchrotron Light Source, Brookhaven National Laboratory, which is supported by the U.S. D.O.E., Divisions of Materials and Chemical Sciences. We are grateful to P. Canfield of Ames Laboratory/Iowa State University, for supplying the Nd₂Fe₁₄B single crystal.

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M98005715



Report Number (14) BNL--65211
CONF-980802--

Publ. Date (11) 199805
Sponsor Code (18) DOE/ER, XF
UC Category (19) UC-404, DOE/ER

DTIC QUALITY INSPECTED 1

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