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Correlating structure with ferromagnetism in melt-spun $Gd_{100-x}Fe_x$

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ABSTRACT

X-ray diffraction and transmission electron microscopy measurements of melt-spun $Gd_{100-x}Fe_x$ ($0 \le x \le 40$) and inert-gas condensed/compacted samples ($3.8 \le x \le 12.7$) reveal a structure of crystalline hcp-Gd grains surrounded by a non-crystalline $Gd_{1-x_{eff}}Fe_{x_{eff}}$ phase, where $x_{eff} > x$ is the effective iron concentration within the amorphous region. The two-phase structure is responsible for an unusual dependence of the coercivity on temperature in which non-zero coercivity is observed above the hcp-Gd T_c with a peak near 320 K. The coercivity decreases as the hcp-Gd grains order, then increases with decreasing temperature. This behavior is explained by the presence of magnetically correlated Fe-rich regions.
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1. Introduction

Structural inhomogeneity fundamentally changes magnetic behavior [1,2]. Cooperatively interacting groups of spins can form clusters (magnetic and/or structural), with interactions between clusters modifying phase transitions otherwise dominated by exchange [3,4]. Defects and chemical disorder can alter magnetic reversal processes, producing significant changes in properties such as the coercivity [5]. Two-phase nanostructures may have enhanced coercivity and remanence ratios [6,7]. Tailoring the properties of magnetic materials requires fundamental understanding of the correlation between the structure of materials and their magnetic properties.

The 30% difference in the atomic sizes of the elements in the $Gd_{100-x}Fe_x$ system (the Goldschmidt radius of Gd is 1.80 Å, while the value for Fe is 1.26 Å) causes Gd and Fe to segregate due to low mixing enthalpies in the Gd-rich and Fe-rich portions of the phase diagram [8,9]. $Gd_{100-x}Fe_x$ is a ferromagnetic amorphous alloy for $40 \le x \le 76$ with Curie temperatures T_C ranging from 370 K to 600 K [10–13]. At the Gd-rich end of the phase diagram, Gd segregation creates a two-phase structure consisting of hcp-Gd grains surrounded by non-crystalline Gd–Fe. This paper examines the structural and magnetic properties of melt-spun $Gd_{100-x}Fe_x$ alloys with $x \le 40$, which have not been as well studied as alloys with higher Fe concentrations.

2. Experimental procedure

Nanostructured $Gd_{100-x}Fe_x$ ($0 \le x \le 40$) samples were prepared by melt spinning. Gadolinium (99.9%) and iron (99.97%) pieces were arc melted in argon multiple times to produce homogeneous ingots. The ingots were melt spun in an argon atmosphere onto a copper wheel rotating at a tangential wheel speed of 40 m/s.

Inert-gas condensed/compacted (IGCC) samples were made by adding Fe chips to Gd sputtering targets and sputtering in Ar pressures ranging from 0.09 to 0.50 torr. The clusters produced were collected on a liquid–nitrogen-cooled coldfinger and transported to a compaction chamber under vacuum. The clusters were compacted at 2.0 GPa for 24 h to produce compacts.

Pieces of melt-spun ribbon were mounted with silicone grease on a low-background holder for X-ray diffraction (XRD) while IGCC samples were measured in a hermetically sealed X-ray diffraction holder on a zero-background quartz disk. Transmission electron microscopy (TEM) samples of the melt-spun ribbons were

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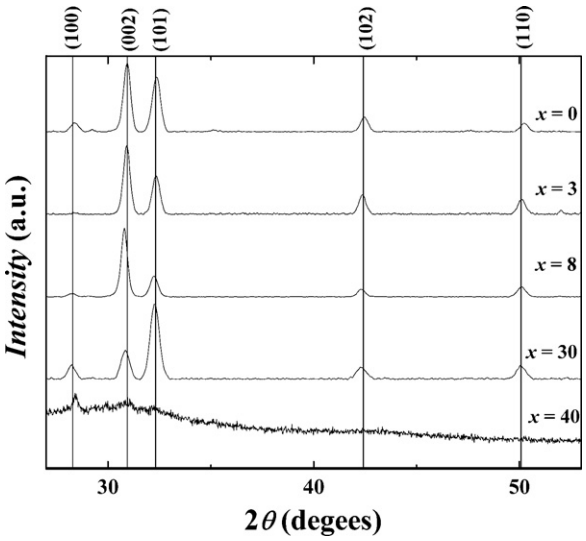

Fig. 1. X-ray diffraction patterns for $x = 0, 3, 8, 30$ and 40. The vertical lines represent the peak positions of hcp-Gd.

prepared by gluing ribbon pieces to Cu grids and then ion milling. A JEOL 2010 and Technai G^2 F20, both operating at 200 kV, were used for TEM/STEM studies.

The magnetic measurements were performed with a Quantum Design MPMS XL-7 SQUID magnetometer and a Quantum Design PPMS system with the ACMS option. Sample masses were typically a few milligrams. The melt-spun samples consisted of a single piece of ribbon with the longest dimension along the direction of the applied magnetic field in order to minimize the demagnetization factor.

3. Results

3.1. Structural properties

X-ray diffraction patterns are shown in Fig. 1 for melt-spun samples with $x = 0$, 3, 8, 30 and 40. Solid vertical lines indicate the expected peak positions for bulk hcp-Gd. The diffraction pattern of the $x = 40$ sample is distinct, having a large amorphous background and very weak, broad hcp-Gd peaks. No crystalline Fe or Gd–Fe alloy peaks are observed in any of the samples.

Transmission electron microscopy shows (Fig. 2) that the structure consists of crystalline grains mixed with a second phase appearing in the grain boundaries. An integral-breadth analysis of the hcp-Gd (1 0 1) peak yields a grain size of approximately 25 nm for samples with $x \leq 30$, and Warren-Averbach analysis shows no significant strain broadening. TEM shows irregularly shaped regions with average grain sizes ~100 nm; however, the larger structures are likely composed of smaller coherently diffracting crystalline areas, as suggested by the contrast observed within the grains at the ~20 nm scale in Fig. 2a. Nano-beam electron diffraction done on samples with $x \leq 30$ indicates that the grains are crystalline or polycrystalline (Fig. 2b) and that the intergranular second phase is much less ordered (Fig. 2a). TEM of the $x = 40$ sample (Fig. 2c) shows it to be mostly amorphous with very small (≤5 nm) crystalline regions dispersed throughout the sample. These results are consistent with the X-ray diffraction patterns of Fig. 1.

Energy dispersive X-ray spectroscopy (EDS) was performed using a scanning transmission electron microscope. The high-angle annular dark-field image for the $x = 20$ sample (Fig. 3a) reveals atomic-number contrast consistent with Gd-rich grains having a higher average atomic number and a non-crystalline Gd–Fe phase with a lower average atomic number. Gd and Fe concentrations were determined along the 50 nm-long arrow in Fig. 3a starting at the right (within a grain), passing through a non-crystalline region, and ending in a second grain. Fig. 3b (elemental atomic percent as a function of position) shows that the Fe concentration is high-

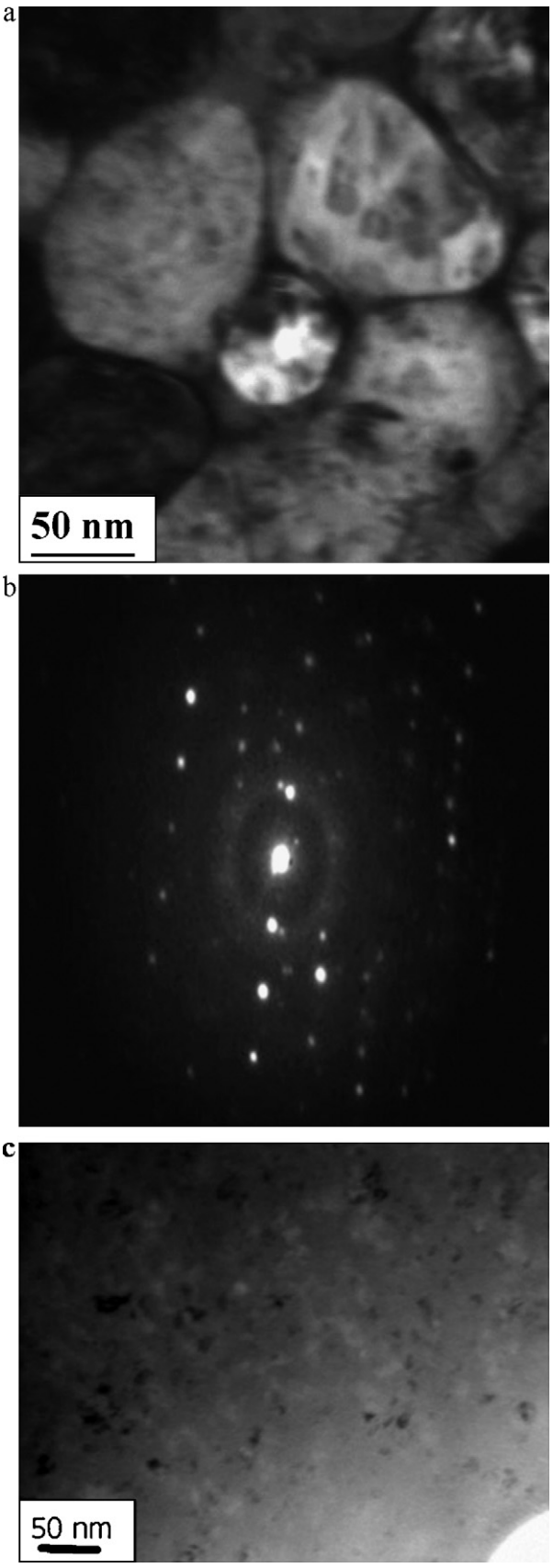

Fig. 2. (a) A transmission electron microscope image of the $x = 8$ sample. (b) Nanobeam electron diffraction pattern of the grain interior showing its crystallinity. (c) Bright field TEM image of $x = 40$ showing crystallites in an amorphous matrix.

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