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Magnetic properties of rapidly quenched and annealed Fe₁₀RTi and related alloys

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Magnetic measurements are reported for iron-rich ternary alloys with the following compositions: Fe₁₀Nd_xDy_{1-x}Ti (0 < x < 1), Fe₁₀SmM (M = Ti, V, and Mo), and Fe₁₀RV (R = Y, Gd, and Dy). The samples were prepared by splat cooling or melt spinning and selected samples were heat treated. The results of x-ray diffraction, electron microscopy, and thermomagnetic measurements on the Fe₁₀Nd_xDy_{1-x}Ti series are presented and indicate that the rapidly quenched alloys are nearly single phase and become multiphase upon heating. Magnetic measurements on the other two series show that the substitution of V for Ti increases the saturation magnetization.

I. INTRODUCTION

The search for novel permanent magnet materials has recently focused on ternary alloys containing iron, a rare earth, and a transition metal. Interest in these materials was sparked by the discovery of Fe₁₄Nd₂B several years ago.¹ Alloys of the 14:2:1 type have relatively large coercivities (though not larger than Sm-Co based magnets) and large energy products.

Recently Ohashi *et al.*² discovered another iron-rich compound, Fe₁₀SmTi, with an anisotropic structure. Their results indicate that single-phase alloys with this 10:1:1 stoichiometry can only be produced with heavy rare earths. If a light rare earth is used the alloy becomes a phase mixture of the 10:1:1 phase with α -Fe and Fe₂R, or Fe₃R.

In this work we investigate the use of rapid-quenching techniques in the production of single-phase 10:1:1 alloys, and the possibility that these techniques could be used to stabilize single-phase alloys containing light rare earths. In addition, the effects of substituting V and Mo for Ti were studied.

II. EXPERIMENTAL METHODS

Rapidly quenched samples of Fe₁₀Nd_xDy_{1-x}Ti (x = 0.0, 0.33, 0.60, 1.0), Fe₁₀SmM (M = Ti, V, and Mo), and Fe₁₀RV (R = Y, Gd, and Dy) were prepared by splat-cooling or melt-spinning mixtures containing the appropriate weight of each element.

Measurements were made on the rapidly quenched alloys and selected samples were subjected to a heat treatment at 800 °C for 1 h in a pure argon atmosphere.

The crystal structure of the Fe₁₀Nd_xDy_{1-x}Ti series was investigated by CuK α x-ray diffraction and by electron microscopy with a transmission-electron microscope (TEM), and thermomagnetic data on this series were obtained with a high-temperature vibrating-sample magnetometer (VSM). The moment of each sample was measured in a constant 1.3-kOe field while the temperature was slowly raised to 650 °C, held constant for 20 min, and then slowly lowered to room temperature.

This VSM was also used to obtain room-temperature

hysteresis loops to 8 kOe for each series. Some additional measurements to 80 kOe at 100 K were made in a low-temperature VSM.

III. RESULTS AND DISCUSSION

Representative results of the x-ray diffraction, electron microscopy, and thermomagnetic measurements for the Fe₁₀Nd_xDy_{1-x}Ti series are shown in Figs. 1, 2, and 3, respectively. The top half of Fig. 1 shows x-ray diffraction data for an as-quenched sample with x = 0.0. All of the lines can be indexed according to the tetragonal structure proposed by Ohashi *et al.*² However, electron diffraction patterns (Fig. 2) in several grains of an annealed Fe₁₀DyTi sample are inconsistent with this structure; rather, they are generally consistent with a ThMn₁₂ structure with c = 4.8 Å, a = 8.3 Å (c/a = 0.58). The x-ray data can also be indexed successfully with this latter structure. In addition, Fig. 2(b) shows satellites that suggest a structural modulation along the c

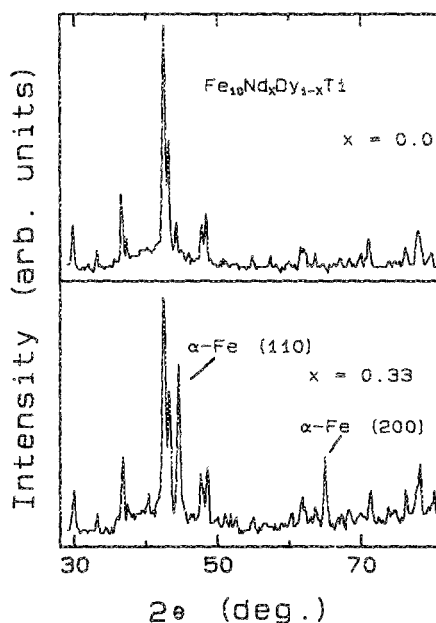
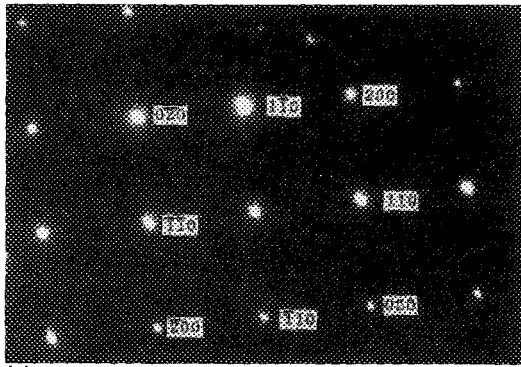
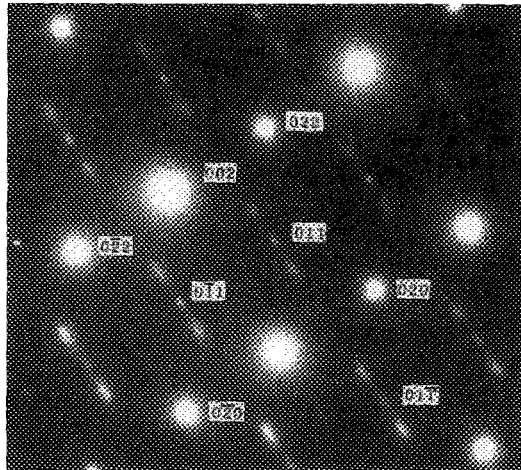


FIG. 1. X-ray diffraction patterns for x = 0.0 and 0.33 (CuK α radiation).



(a)



(b)

FIG. 2. Electron diffraction data for heat-treated $\text{Fe}_{10}\text{DyTi}$. (a) shows fourfold rotation symmetry about $[00\bar{1}]$. In (b) the beam is along $[100]$, and a modulation of about 12 \AA along the c direction is seen.

axis with a period of about 12 \AA . Thus, it is possible that the rapidly quenched material has a relatively simple tetragonal structure and that annealing produces a rather complex structure. Further work is necessary to determine whether the as-quenched material also has this structural modulation.

The absence of $\alpha\text{-Fe}$ peaks in the x-ray pattern for $x = 0.0$ indicate that the sample is single phase in the as-quenched state. Preliminary x-ray data (not presented here) for as-quenched $\text{Fe}_{10}\text{NdTi}$ suggest that it too is single-phase.

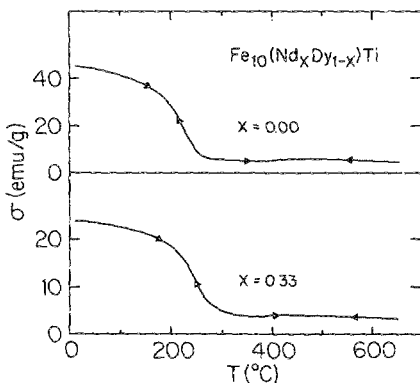


FIG. 3. Thermomagnetic data at $H = 1.3 \text{ kOe}$ for $x = 0.0$ and 0.33 .

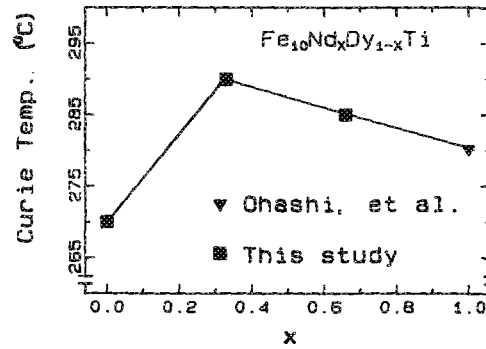


FIG. 4. Curie temperature vs composition for the $\text{Fe}_{10}\text{Nd}_x\text{Dy}_{1-x}\text{Ti}$ series.

The bottom half of Fig. 1 shows diffraction data for the heat-treated sample with $x = 0.33$. As indicated in the figure, the material contains a substantial amount of $\alpha\text{-Fe}$. All of the heat-treated samples studied (including $x = 0.0$) show this iron precipitation upon annealing.

The thermomagnetic data in Fig. 3 indicate the presence of a high-temperature magnetic phase above the Curie point of the tetragonal phase. The x-ray data indicate that this secondary phase is $\alpha\text{-Fe}$.

Magnetization data at $H = 8 \text{ kOe}$ for this series are summarized in Figs. 4 and 5. Figure 4 shows that the Curie temperature T_c for the heat-treated samples rises from 270°C for $x = 0.0$ to 290°C for $x = 0.33$ and then diminishes with increasing x .

The hysteresis measurements for this series indicate that the largest room-temperature coercivity is 630 Oe and occurs in samples with $x = 0.0$. There was no significant difference between the hysteresis loops for as-quenched and heat-treated samples. TEM measurements indicate that the typical grain size for the $x = 0.0$ sample is about 4000 \AA . This indicates that despite the rapid quenching, the grain size is so large that there is little magnetic hardening due to microstructure.

Measurements to 80 kOe show that for $x = 0.0$ the average moment per iron is about $1.8 \mu_B$, assuming the structure is ferrimagnetic, i.e., the Dy moment opposes the Fe moment. Similar results are found for $x = 1.0$ assuming a ferromagnetic structure for the Nd.

Table I summarizes the results of the magnetic measurements. These data indicate that Fe_{10}SmV has the largest magnetization of the Fe_{10}SmM materials studied. Hystere-

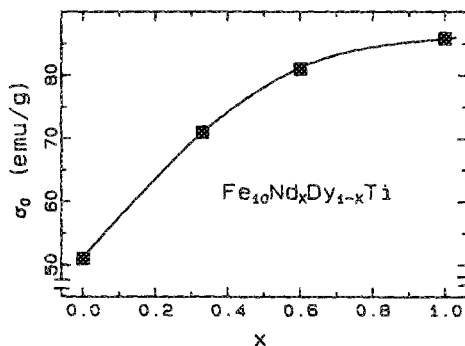


FIG. 5. Magnetization at $H = 8 \text{ kOe}$ vs composition for the $\text{Fe}_{10}\text{Nd}_x\text{Dy}_{1-x}\text{Ti}$ series.

TABLE I. Magnetization at 8 kOe for the Fe₁₀SmM and Fe₁₀RV series.

Sample	σ_0 (emu/g)
Fe ₁₀ SmV	98.6
Fe ₁₀ YV	94.8
Fe ₁₀ SmTi	86.6
Fe ₁₀ GdV	70.0
Fe ₁₀ SmMo	68.3
Fe ₁₀ DyV	64.2

sis measurements show that Fe₁₀GdV has the highest coercivity of all the samples studied, 1.2 kOe.

Preliminary x-ray data on the series indicate that the as-quenched Fe₁₀SmM and Fe₁₀RV series are single phase. The precipitation of α -Fe upon heat treatment has also been observed in these series.

IV. CONCLUSION

This study indicates that rapidly quenched Fe₁₀RM alloys have structures that are generally consistent with the

ThMn₁₂ structure. However, some evidence has been obtained for a modulation of about 12 Å along the *c* axis, which requires further study. Annealing these samples at 800 °C produced multiphased mixtures containing α -Fe. TEM measurements indicate that despite rapid quenching the alloys are rather coarse grained. This may explain the small coercivities observed for both as-quenched and annealed samples.

ACKNOWLEDGMENTS

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¹See K. H. J. Bushow, *Mater. Sci. Rep.* **1**, 1 (1986), and references therein.
²K. Ohashi, T. Yokogama, R. Osugi, and Y. Tawara (unpublished).