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Magnetic and structural studies in Sm-Fe-Ti magnets

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The effects of vanadium substitution on the magnetic properties of Sm-Fe-Ti-V melt-spun ribbons and the magnetic properties and microstructure of sintered Sm-Fe-Ti based magnets are reported. The highest coercivity, $H_c = 10.65$ kOe, was obtained in a heat-treated melt-spun $\text{Sm}_8\text{Fe}_{75.5}\text{Ga}_{0.5}\text{Ti}_8\text{V}_8$ alloy. This is the highest value of the coercivities reported in alloys with the ThMn_{12} -type structure. Bulk magnets were prepared by the usual powder metallurgy technique. However, their highest coercivity (~ 2 kOe) was much less than that of the corresponding ribbons. The low coercivity of sintered magnets has been attributed to their nonuniform microstructure.

I. INTRODUCTION

The discovery of Nd-Fe-B magnets^{1,2} having excellent hard magnetic properties has stimulated search for new ternary rare-earth-iron based compounds. Several ternary compounds with composition $\text{SmFe}_{12-x}\text{Ti}$ based on the ThMn_{12} -type structure, where $T = \text{Ti, V, Cr, Mo, W}$ or Si and $x = 1.2$ or 2 , have been found to have a relatively high anisotropy, saturation magnetization, and Curie temperature³⁻⁵ that make them good candidates for permanent magnet development. Recently, high coercivities have been obtained in ribbon samples,⁶⁻⁸ with a maximum value of 9.8 kOe.⁸ Based on the reported high anisotropy field of these systems, there is still the possibility for obtaining higher coercivity by further substitutions in Sm-Fe-Ti-V alloys. Very few reports exist on sintered Sm-Fe-Ti magnets.⁸ This is because it is difficult to obtain a high coercivity in sintered magnets.

In this paper we report the results of vanadium substitution on the magnetic properties of Sm-Fe-Ti-V melt-spun ribbons, and we present preliminary results of the magnetic properties and microstructure of sintered Sm-Fe-Ti-V magnets. This would hopefully help us understand the lack of magnetic hardening in bulk magnets.

II. EXPERIMENT

Several alloys with composition $\text{Sm}_8\text{Fe}_{76-y}\text{M}_y\text{Ti}_8\text{V}_8$ and $\text{Sm}_8\text{Fe}_{76}\text{Ti}_{8-z}\text{M}_z\text{V}_8$, where $M = \text{Cr, Mn, Ga, Zr, Nb, Mo, W}$, and $0 < y, z < 2$ were prepared by arc-melting starting materials of 99.9% purity under argon atmosphere. Pieces of ingots were made into ribbons by the melt-spinning technique. Magnetic hardening was obtained in the ribbons by heat-treating the samples at a temperature in the range 800 – 850 °C for times ranging from 5 to 150 min. Sintered magnets were prepared using the usual powder metallurgy technique.² It was necessary that an excess amount of Sm be incorporated into the alloy in order to compensate for the

vaporization of Sm during alloying, sintering, and heat treating because Sm has a high vapor pressure. Magnetic properties were measured using a vibrating sample magnetometer (VSM) with applied fields of up to 17.7 kOe. The phases present were determined by x-ray diffraction and thermomagnetic analysis. A Jeol 100C scanning transmission electron microscope (STEM) equipped with an energy dispersive x-ray analyzer (EDXA) was used to study the grain structure of the sintered magnets.

III. RESULTS AND DISCUSSION

A. Melt-spun magnets

In a previous paper¹⁰ we showed that crystallized Sm-Fe-Ti ribbons have the ThMn_{12} -type structure when annealed above 800 °C, but for lower annealing temperatures some alloys have the TbCu_7 -type structure. The higher annealing temperatures also were used in this study to obtain a high coercivity. Samples annealed at 800 °C exhibit a higher coercivity than at 850 °C. However, longer times are needed at the lower annealing temperatures to achieve the optimum

FIG. 1. X-ray diagram of $\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$ ribbons heated at 800 °C for 60 min.

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TABLE I. Magnetic properties of Sm-Fe-Ti based ribbons annealed at 800 °C.

Composition	H_c (kOe)	M_s (G)
$\text{Sm}_8\text{Fe}_{83}\text{Ti}_9$	5.6	806
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$	10.2	664
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_7\text{Zr}_1\text{V}_8$	10.25	674
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_6\text{Zr}_2\text{V}_8$	9.4	...
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_7\text{Nb}_1\text{V}_8$	9.4	608
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_7\text{Mo}_1\text{V}_8$	8.1	596
$\text{Sm}_8\text{Fe}_{76}\text{Ti}_7\text{W}_1\text{V}_8$	8.0	599
$\text{Sm}_8\text{Fe}_{75.5}\text{Ga}_{0.5}\text{Ti}_8\text{V}_8$	10.65	692

coercivity. The x-ray diffraction pattern of $\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$ ribbons annealed at 800 °C for 60 min is shown in Fig. 1. Nearly all peaks could be indexed with the ThMn_{12} structure except one peak which comes from a secondary bcc phase with a lattice constant close to $\alpha\text{-Fe}$. In an attempt to obtain higher coercivities several substitutions have been carried out on Sm-Fe-Ti-V alloys. Samples with composition $\text{Sm}_8\text{Fe}_{76-y}\text{Ti}_8\text{M}_y\text{Ti}_8\text{V}_8$ and $\text{Sm}_8\text{Fe}_{76}\text{Ti}_{8-z}\text{M}_z\text{V}_8$ where $\text{M} = \text{Cr, Mn, Ga, Zr, Nb, Mo, W}$, and $0 < y, z < 2$ were made. Table I shows the coercivity and saturation magnetization M_s , for Sm-Fe-Ti-V ribbons annealed at 800 °C for 60 min. The magnetic data show that an increase in coercivity can be obtained by replacing Fe with a small amount of Ga or by replacing Ti with a small amount of Zr. Annealed $\text{Sm}_8\text{Fe}_{76}\text{Ti}_7\text{Zr}_1\text{V}_8$ and $\text{Sm}_8\text{Fe}_{75.5}\text{Ga}_{0.5}\text{Ti}_8\text{V}_8$ ribbons have a coercivity of 10.25 and 10.65 kOe, respectively. To our knowledge, these are the highest values among all the reported values of coercivity in melt-spun ribbons with the ThMn_{12} -type structure. A typical magnetic hysteresis loop for these samples is shown in Fig. 2.

B. Sintered magnets

Alloys with composition $\text{Sm}_9\text{Fe}_{83}\text{Ti}_8$ and $\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$ were sintered at 1100 °C for 60 min and quenched in argon

FIG. 2. Magnetic hysteresis loop of $\text{Sm}_8\text{Fe}_{75.5}\text{Ga}_{0.5}\text{Ti}_8\text{V}_8$ ribbons annealed at 800 °C for 60 min.

FIG. 3. Thermomagnetic data of $\text{Sm}_9\text{Fe}_{83}\text{Ti}_8$ magnets sintered in (a) vacuum, (b) argon gas, and (c) Sm atmosphere.

gas, and then were annealed at 730 °C for 40 min. Thermomagnetic data of sintered $\text{Sm}_9\text{Fe}_{83}\text{Ti}_8$ magnets under various sintering conditions are shown in Fig. 3. Samples sintered in Sm atmosphere appear to have nearly a single phase with the ThMn_{12} -type structure. But the 1:12 phase is a minority phase in the other two samples where $\alpha\text{-Fe}$ is predominant. This result is attributed to the large Sm losses during sintering which make it difficult for the 1:12 phase to be formed.

Figure 4 shows the magnetic hysteresis loops of $\text{Sm}_9\text{Fe}_{83}\text{Ti}_8$ and $\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$ magnets sintered in Sm atmosphere. The former magnets have a coercivity of only 800 Oe, while the latter have a coercivity of about 2 kOe. These values are far less than those of the corresponding ribbons,^{3,8} which are 5.6 and 9.8 kOe, respectively. The large differences are attributed to the different microstructure of the two magnets. The microstructure of ribbons consists of fine 1:12 grains with a size in the range of 300–800 Å. On the contrary the grain structure of the sintered magnets is not well developed [Fig. 5(a)]. The grains are not well-defined polygons like in other sintered magnets. The Sm-Fe-Ti-V

FIG. 4. Magnetic hysteresis loops of sintered $\text{Sm}_9\text{Fe}_{83}\text{Ti}_8$ and $\text{Sm}_8\text{Fe}_{76}\text{Ti}_8\text{V}_8$ magnets.

FIG. 5. SEM studies showing the grain structure of (a) $\text{Sm}_{0.5}\text{Fe}_{8.5}\text{Ti}_8$ and (c) $\text{Sm}_{0.5}\text{Fe}_{7.6}\text{Ti}_8\text{V}_8$ magnets.

magnet with the highest coercivity appeared to have a more uniform microstructure [Fig. 5(b)]. All sintered magnets were found to contain at least four phases (Fig. 6); the 1:12 phase, $\alpha\text{-Fe}$, a Sm-rich-Fe-Ti phase and another Sm-rich phase possibly a samarium oxide. In the magnets with a nearly single 1:12 phase, the 1:12 grains had a size in the range of 8–15 μm . Additions of extra samarium were found to lead to Sm-rich regions near the grain boundaries or to Sm-rich grains squeezed between the 1:12 grains.

IV. CONCLUSIONS

Coercivities exceeding 10 kOe have been obtained in melt-spun Sm-Fe-Ti-V based magnets with the highest value of 10.65 kOe observed in $\text{Sm}_8\text{Fe}_{78.5}\text{Ga}_{0.5}\text{Ti}_{18}\text{V}_8$. Sintered magnets with similar composition have been prepared into a nearly single 1:12 phase but their maximum coercivity was only 2 kOe. The much smaller coercivity of sintered magnets has been attributed to their microstructure which is not well developed for optimum magnetic hardening.

FIG. 6. EDXA studies in Sm-Fe-Ti-V magnets showing the composition of the various phases: (a) 1:12, (b) Sm-rich-Fe-Ti, (c) Sm-rich.

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