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Magnetic Properties of Fe-Based Binary Crystalline Alloys Produced by Vapor Quenching

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Structure and magnetic properties of Fe–M (M=Ti, Zr Hf, Nb or Ta) alloy films produced by rf sputtering haye been investigated in Fe-rich compositional ranges below 10 at%M. These alloy films have a nonequilibrium bcc phase with lattice parameters larger than that of pure Fe. They exhibit high saturation magnetization and soft magnetic properties. In particular, the bcc $Fe_{94}Zr_6$ film annealed at 673 K for 1 h exhibits a saturation magnetization of 2 T and a magnetostriction less than 1×10^{-6}.

KEYWORDS: soft magnetic material, sputter deposition, Fe-based alloy, high saturation magnetization, zero magnetostriction, nonequilibrium phase

§1. Introduction

Recent improvements in high-density magnetic recording media requires magnetic heads with high saturation magnetization.[1] Crystalline Fe-based alloy films and their compositionally modulated films such as Fe–Si and Fe–C reveal high saturation magnetization and low coercivity,[2-6] and some of them also show very small magnetostriction.[5,6]

On the other hand, the changes of magnetic properties and structure with TM (transition metals) content have been systematically studied for nonequilibrium Fe–TM alloys produced by vapor quenching.[7-11] These data suggest that nonequilibrium Fe-rich bcc alloys with high saturation magnetization are good candidates for high-density recording head material. In this respect, the structure and magnetic properties were studied for Fe–M (M=Ti, Zr, Hf, Nb or Ta) films produced by vapor quenching. This paper shows experimental results of X-ray diffraction, magnetization, coercivity and magnetostriction in as-sputtered and annealed states of the $Fe_{100-x}M_x$ films with 0 to 10 at%M.

§2. Experimental Procedures

Fe–M alloy targets were prepared from 99.9 wt%Fe and 99.0 wt%Ti, 99.6 wt%Zr, 95.0 wt%Hf, 99.9 wt%Ta or 99.5 wt%Nb by arc-melting in an argon atmosphere. The alloy films with a thickness of about 1 μm were deposited on indirect-water-cooled glass substrates by a conventional rf sputtering method. The back-pressure was about 1×10^{-4} Pa and the Ar gas pressure during sputter-deposition about 1 Pa. The rf power was 80 W. The distance between the target and substrate was 40 mm and the target diameter was 80 mm. The deposition rate was about 0.2 μm/h. The composition was determined by electron probe microanalysis while the composition of some of the films was additionally confermed by inductively coupled plasma spectroscopy. No significant difference was detected between these results.

X-ray diffraction patterns were obtained using Cu Kα radiation with a graphite monochromater. The 2θ angle was calibrated with Si powder. The magnetization at 290 K was measured using a vibrating sample magnetometer in an applied field below 0.8 MA/m (10 kOe). The coercivity was measured using a dc hysteresis loop tracer at a maximum magnetic field of 8 kA/m (100 Oe). The magnetostriction was measured in a field up to 56 kA/m (700 Oe) by an optical cantilever method.

§3. Results and Discussion

As shown in Figs. 1(a) and 1(b), the X-ray diffraction patterns of the sputter-deposited Fe, Fe–Nb and Fe–Zr alloys display only bcc diffraction peaks and the crystal growth has a preferred orientation in Fe, $Fe_{94}Nb_6$ and $Fe_{95}Zr_5$ films. The half widths of the diffraction peaks are wider than that of a bulk Fe specimen, probably because of lattice defects, internal strain, incorporated Ar atoms and small grain sizes in the sputter-deposited films.[12] The grain size of the present Fe–M films estimated by Scherrer's equation is about 10–20 nm.

Figure 2 shows the lattice parameters of the bcc phase as a function of the M content for $Fe_{100-x}M_x$ (M=Ti, Zr, Hf, Nb or Ta) films derived from X-ray diffraction measuzements. The lattice parameter increases from 0.287 to 0.29 nm with increasing the M concentration. The concentration dependence of magnetization at 290 K for the $Fe_{100-x}M_x$ films is shown in Fig. 3. The magnetization at 290 K decreases with increasing x. Assuming that these M atoms carry no magnetic moment, the magnetic moment per Fe atom can be estimated for these alloys. As shown in Fig. 4, the average magnetic moment of the Fe atoms at 290 K does not decrease below 5 at%M, even though the concentration of the nonmagnetic elements increase.

Figure 5 shows the concentration dependence of the coercivity of the bcc $Fe_{100-x}M_x$ films. The coercivity exhibits a minimum in the range of 2 to 5 at%. The minimum is probably due to very small magnetostriction, as has been observed in some bcc Fe-rich alloys.[2,3,6]

Table I summarizes the saturation magnetization, coer-

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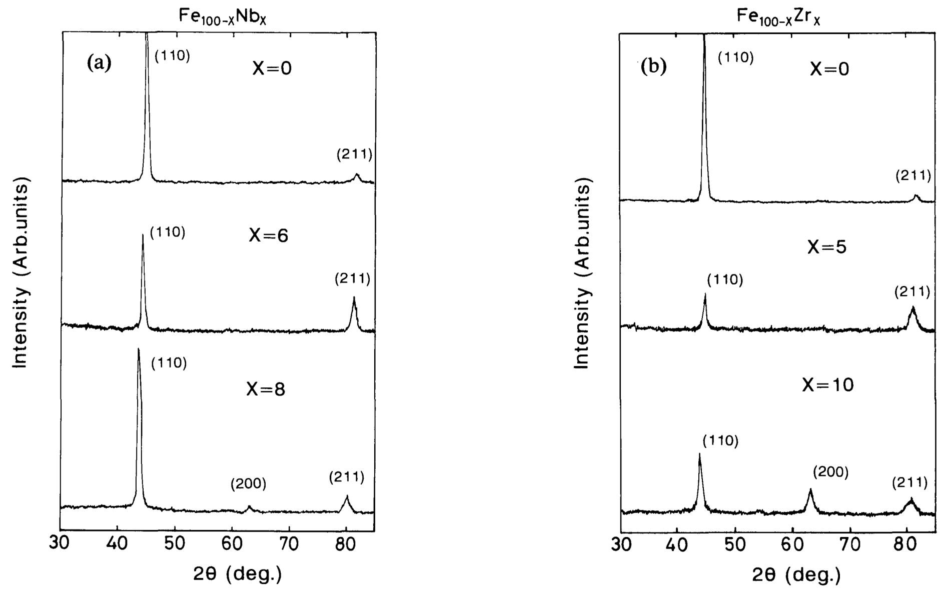

Fig. 1. (a) X-ray diffraction patterns of sputter-deposited Fe and $Fe_{100-x}Nb_x$ films. (b) X-ray diffraction patterns of sputter-deposited Fe and $Fe_{100-x}Zr_x$ films.

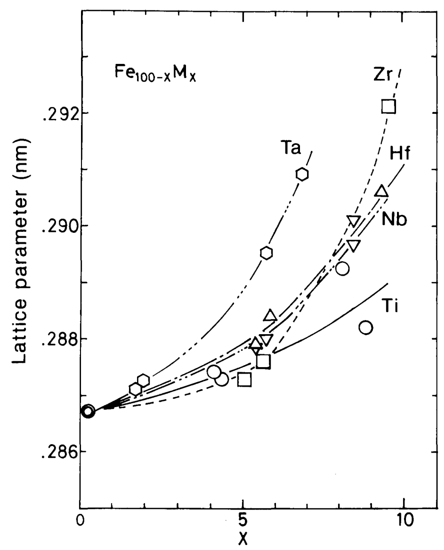

Fig. 2. Lattice parameters of bcc $Fe_{100-x}M_x$ alloys produced by rf sputtering. ◎: Fe, ○: Fe-Ti, □: Fe-Zr, △: Fe-Hf, ▽: Fe-Nb, ⬡: Fe-Ta.

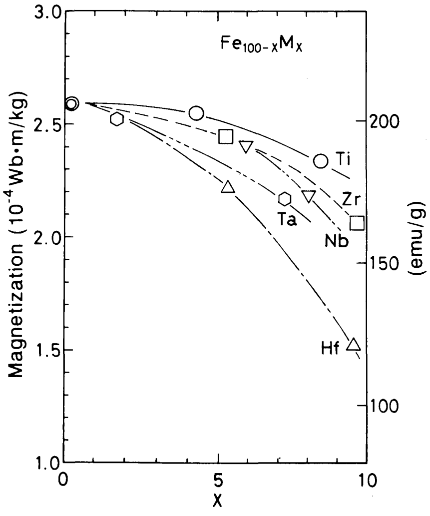

Fig. 3. Concentration dependence of magnetization at 290 K for $Fe_{100-x}M_x$ sputter-deposited films. ◎: Fe, ○: Fe-Ti, □: Fe-Zr, △: Fe-Hf, ▽: Fe-Nb, ⬡: Fe-Ta.

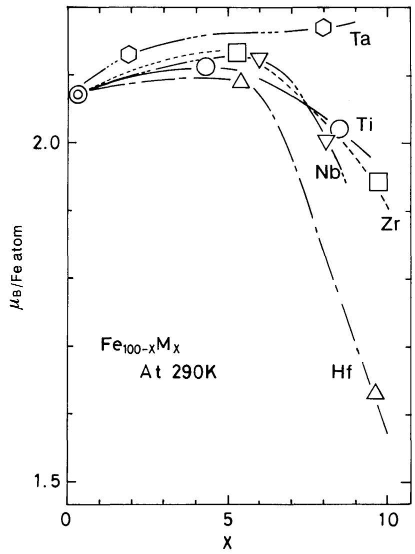

Fig. 4. Magnetic moment per Fe atom at 290 K for sputter-deposited $Fe_{100-x}M_x$ films. ◎: Fe, ○: Fe-Ti, □: Fe-Zr, △: Fe-Hf, ▽: Fe-Nb, ⬡: Fe-Ta.

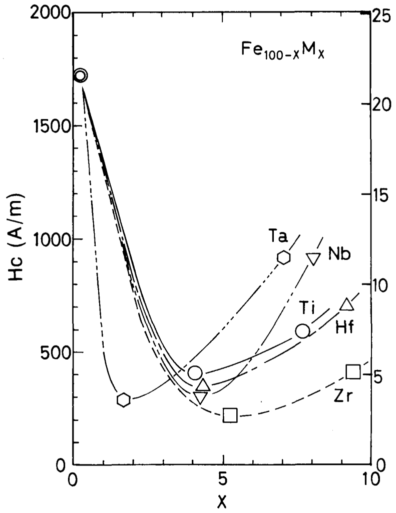

Fig. 5. Coercivity at 290 K of $Fe_{100-x}M_x$ films in as-sputter-deposited state. ◎: Fe, ○: Fe-Ti, □: Fe-Zr, △: Fe-Hf, ▽: Fe-Nb, ⬡: Fe-Ta.

Table I. Saturation magnetization, coercivity and magnetriction for Fe and Fe-M (M=Zr, Hf, Nb, and Ta) alloys as sputter-deposited.

Composition	Saturation magnetization $4\pi Ms$ (T)	Coercivity Hc (A/m)	Magnetostriction (at 5.6×10^4 A/m)
Fe_{100}	2.0	1.7×10^3	-1.1×10^{-6}
$Fe_{95}Zr_5$	2.0	2.1×10^2	$+1.9\times10^{-6}$
$Fe_{95}Hf_5$	1.9	3.5×10^2	$+2.2\times10^{-6}$
$Fe_{96}Nb_4$	1.9	3.0×10^2	-3.8×10^{-6}
$Fe_{98}Ta_2$	2.1	2.9×10^2	-5.2×10^{-6}

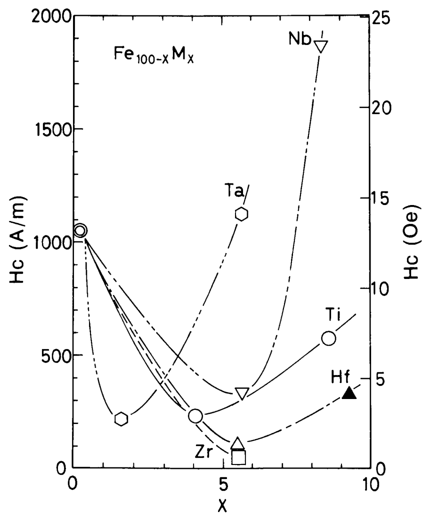

Fig. 6. Coercivity at 290 K of $Fe_{100-x}M_x$ films annealed at 673 K for 1 h. ◎: Fe, ○: Fe-Ti, □: Fe-Zr, △: Fe-Hf, ▲: Fe-Hf, (two-phase) ▽: Fe-Nb, ⬡: Fe-Ta.

civity and magnetostriction for the bcc $Fe_{100-x}M_x$ films. The $Fe_{95}Zr_5$ film seems to be the best candidate as a soft ferromagnetic material among these films, because it has the high saturation magnetization and the lowest coercivity.

In order to improve the soft magnetic properties, the $Fe_{100-x}M_x$ films were annealed at 673 K for 1 h in a zero magnetic field. The coercivity of the annealed $Fe_{100-x}M_x$ films is plotted as a function of the M content in Fig. 6. The coercivity of the annealed Fe and Fe-M films is much smaller than that of the as-sputter-deposited Fe and Fe-M alloy films, where the lowest coercivity is obtained in the region of 2 to 6 at%M, except when M=Ta.

Table II. Saturation magnetization, coercivity and magnetostriction for Fe and Fe-M (M=Ti, Zr, Hf and Ta) alloys annealed at 673 K for 1 h.

Composition	Saturation magnetization $4\pi Ms$ (T)	Coercivity Hc (A/m)	Magnetostriction (at 5.6×10^4 A/m)
Fe_{100}	2.0	1.1×10^3	-7.4×10^{-6}
$Fe_{96}Ti_4$	2.0	2.3×10^2	-4.6×10^{-6}
$Fe_{94}Zr_6$	2.0	5×10	$<1.0\times10^{-6}$
$Fe_{95}Hf_5$	1.9	1.0×10^2	$+2.3\times10^{-6}$
$Fe_{98}Ta_2$	2.1	2.1×10^2	-5.5×10^{-6}

Table II shows the saturation magnetization, coercivity and magnetostriction for the annealed $Fe_{100-x}M_x$ films. In particular, the bcc $Fe_{94}Zr_6$ film annealed at 673 K for 1 h shows high saturation magnetization, low coercivity and nearly zero magnetostriction. Further studies are in progress for Fe-Zr and Fe-Hf films made under several sputtering and annealing conditions.

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