Journal of Korean Magnetics Society, Volume 5, Number 5, October 1995

MAGNETIC PROPERTIES OF NANOCRYSTALLINE (Fe,Co)-B-Al-M (M=Nb/Mo/Ta) ALLOYS

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Abstract—Soft magnetic properties of Fe-based (Fe,Co)-B-Al-M (M=Nb, Mo or Ta) nanocrystalline alloy have been investigated. The alloy obtained directly from the rapid solidification process. Microstructure of the alloy is a mixture of ultrafine bcc Fe(Co) nanocrystallines and a small amount of retained amorphous phase. Heat treatment of as-prepared alloys improves soft magnetic properties in high frequency range. $(Fe_{.85}Co_{.15})_{70}B_{18}Al_{10}Ta_6$ alloy annealed at 500 ℃ for 1 h shows the most improved soft magnetic properties among the alloys examined. Average grain size of the nanocrystalline is about 10 nm.

Ⅰ. INTRODUCTION

It is well known that Fe-based amorphous alloy shows excellent soft magnetic properties of low coercivity, high flux density and permeability. However, the Fe-based amorphous alloy shows poor performance for the applications of high frequency devices because of large magnetostriction. Reduction of the magnetostriction of the amorphous alloy was attempted in order to improve the high frequency performance. Some of the attempts are adding additives of Mo, Nb, Cr[1], or forming very small amount of α-Fe precipitates in amorphous matrix by annealing near the crystallization temperature[2],[3].

Recently, Yoshizawa et al.[4] reported that the new soft magnetic material composed of α-Fe ultrafine grains of about 10 nm has excellent soft magnetic properties in the high frequency range. They found that the remarkable improvement of the Fe-based nanocrystalline alloy comes from the random distribution of magnetocrystalline anisotropy and almost zero magnetostriction[4],[5].

In the present work, nanocrystalline alloy of (Fe, Co)-B-Al-M(M=Nb,Mo,Ta) system was fabricated, and soft magnetic properties of the alloy were investigated for high frequency applications.

Ⅱ. EXPERIMENTAL

$(Fe_{85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$ (M=Nb, Mo, Ta) $(2 \leq x \leq 6)$ alloys were prepared by rapid solidification process with a wheel speed of 37 m/s. The width of as-prepared sample was about 1 mm, and the thickness of the alloys was about 20 μm. Saturation magnetization was measured by a vibrating sample magnetometer(VSM) within maximum applied field of 5 kOe. Hysteresis curves, power loss and ac permeability were measured by a single strip automatic ac hysteresis loop tracer. Heat treatment was done in the temperature range of 250~650 ℃ for 1 h in a vacuum of $\sim 2\times10^{-5}$ Torr. The heat treatment relieves the internal stress of as-prepared samples introduced during rapid solidification process. X-ray diffractometer was used for phase analysis and grain size determination.

III. RESULTS AND DISCUSSION

Y.S. Cho et al.[6] have reported previously that the addition of 10 at.% Al into $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{80}\mathrm{B}_{20}$ alloy causes direct precipitation of α-Fe(Co) crystallites during rapid solidification. Microstructure of the alloy is typical bcc α-Fe nanostructure with small amount of retained amorphous phase. The result suggests that Al additive raises nucleation rate of α-Fe(Co) nanocrystallites during rapid solidification of $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{80}\mathrm{B}_{20}$ alloy. Microstructure of the $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20}\mathrm{Al}_{10}$ alloy is very similar to that of K. Suzuki et al.[7]. Thus, as long as microstructure is concerned, the $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20}\mathrm{Al}_{10}$ nanocrystalline alloy is expected to have good soft magnetic properties. However, contrary to the expectation, experimental results of the alloy revealed very poor soft magnetic properties. It may be considered that the magnetostriction of about 20 ppm which is too much for high frequency applications and the internal stress formed during rapid solidication is responsible for the discrepancy.

In order to eliminate the internal stress, heat treatment is attempted, and simutaneously modified composition is proposed to suppress grain growth during the heat treatment. The modified composition is $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20-x}\mathrm{Al}_{10}\mathrm{M}_x$ (M=Nb, Mo or Ta, $2\le x\le 6$). The average grain size of as-prepared nanocrystalline $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20-x}\mathrm{Al}_{10}\mathrm{M}_x$ alloy is measured by X-ray diffraction. It turned out that the average grain size of α-Fe(Co) nanocrystallines remained almost unchanged. The result shows that Nb, Mo or Ta additive suppresses the growth of α-Fe(Co) nanocrystallines during rapid solidification. Fig. 1 shows the variation of ac permeability as a function of annealing temperature for $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20-x}\mathrm{Al}_{10}\mathrm{Ta}_x$ alloy. The ac permeability increases slowly with increasing annealing temperature. The alloy with Nb or Mo additive also shows similar behaviour. The trend strongly suggests that internal stress is relaxed with increasing annealing temperature, and the relaxation of internal stress leads to high ac permeability.

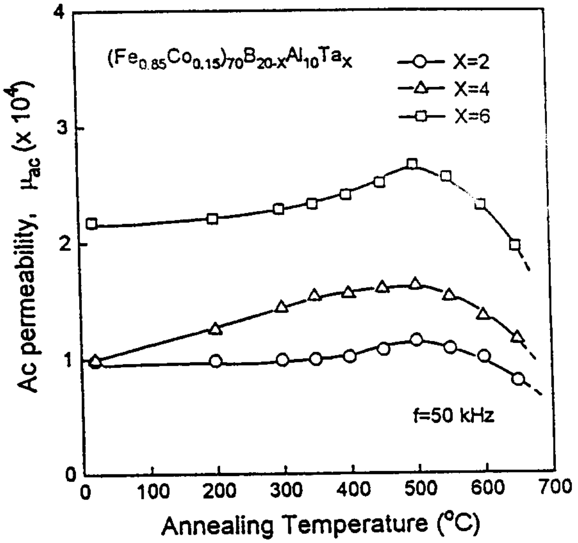

Fig. 1 Variation of ac permeability on annealing temperature for $(\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20-x}\mathrm{Al}_{10}\mathrm{M}_x$ alloys.(M=Nb, Mo, Ta, f=50 kHz, B_m= 0.2 T)

The variation of ac permeability of the modified alloy, $\mathrm{Fe}_{.85}\mathrm{Co}_{.15})_{70}\mathrm{B}_{20-x}\mathrm{Al}_{10}\mathrm{M}_x$(M=Nb, Mo, Ta), is also measured as a function of composition, and is shown in Fig. 2. The alloy with additive of Nb or Mo, ac permeabitlity decreases with increasing amount of the additive. The observation may imply that the volume fraction of retained Nb or Mo-rich amorphous phase increases with increasing Nb or Mo, and that causes incomplete relaxation of internal stress, since the Nb or Mo-rich amorphous phase has higher crystallization temperature.

On the contray, ac permeability of the alloy with Ta shows opposite trends. In order to analyze the variation of ac permeability with respect to additives, average grain size of the alloy was calculated from full width at half maximum(FWHM) of α-Fe (110) peak using Scherrer's eq.

Calculated grain size of optimally annealed alloys is shown in Fig. 3. The grain size increases with increasing amount of additives.

Fig. 4 is X-ray diffraction patterns of $(Fe_{.85}Co_{.15})_{70}B_{18}Al_{10}Mo_2$ alloy annealed at various temperature up to 600 ℃. It is found that α-Fe(Co)

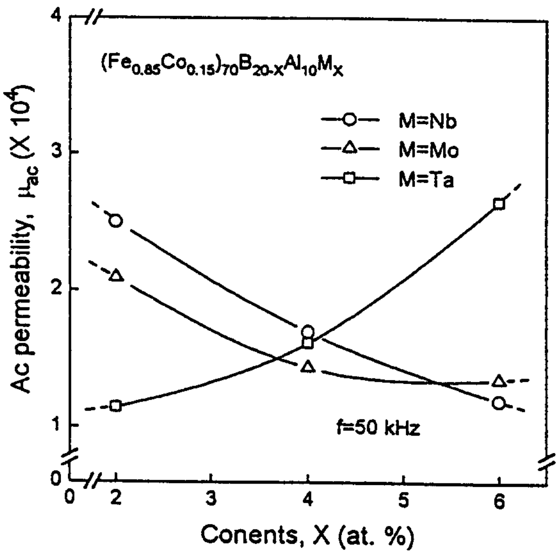

Fig. 2 Ac permeability $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$ (M=Nb, Mo, Ta) alloys(f=50 kHz, B_m= 0.2 T).

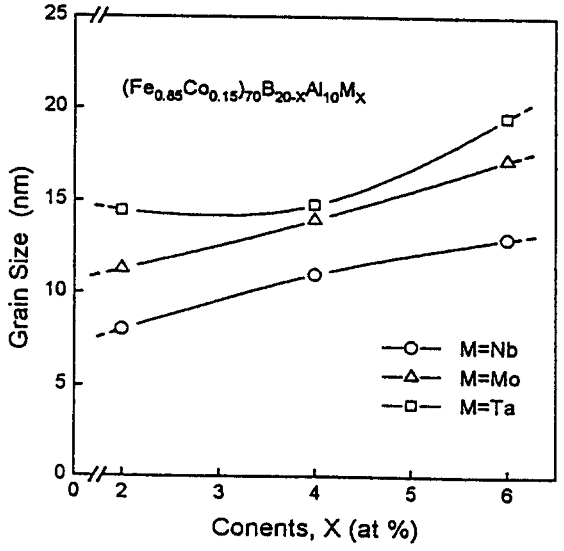

Fig. 3 Average grain size of optimally annealed $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$ (M=Nb, Mo, Ta) alloys.

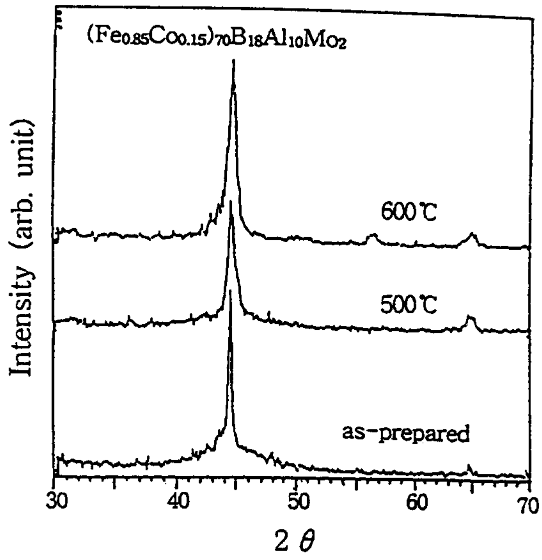

Fig. 4 X-ray diffraction petterns of $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}Mo_x$.

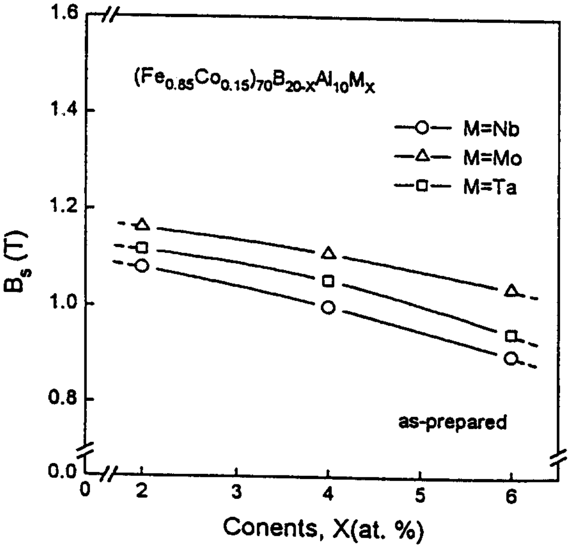

Fig. 5 Maximum flux density of as-prepared $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$ (M=Nb, Mo, Ta) alloys.

phase remains almost unchanged at the annealing temperature.Fig. 5 and Fig. 6 show the maximum flux density of $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$(M=Nb, Mo, Ta) ($2 \leq x \leq 6$) alloys.

The saturation flux density of an optimally annealed $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$(M=MoTa) nanocrystalline is higher than that of as-prepared sample.

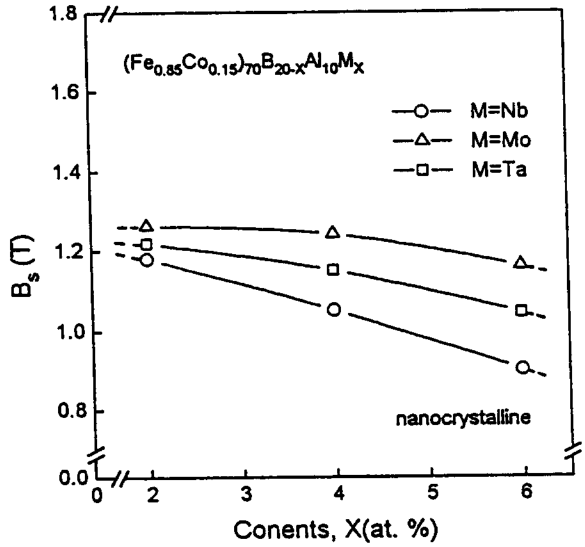

Fig. 6 Maximum flux density of optimally annealed $(Fe_{.85}Co_{.15})_{70}B_{20-x}Al_{10}M_x$ (M=Nb, Mo, Ta) alloys.

Ⅳ. CONCLUSION

Modified alloy system of (Fe, Co)-B-Al-M(M=Nb, Mo,Ta) with ultrafine microstructure was investigated. Ultrafine α-Fe(Co) nanocrystallines are formed directly by rapid solidification. Al additive plays an important role on the precipitation process of α-Fe(Co) nanocrystalline phase during rapid solidification. Additives of Nb, Mo and Ta suppress grain growth of the alloys. High frequency soft magnetic properties is improved by annealing the as-prepared (Fe,Co)-B-Al-M(M=Nb,Mo,Ta) alloys. $(Fe_{.85}Co_{.15})_{70}B_{18}Al_{10}Ta_6$ alloy annealed at 500 ℃ for 1 hour has the most improved magnetic properties among the prepared alloys. Average grain size of the alloy is about 10 nm.

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