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Microstructure and Magnetic Properties of Nanocrystalline bcc Fe-Nb-B Soft Magnetic Alloys

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Abstract ------The microstructure and the magnetic properties of melt-spun Fe-Nb-B alloy ribbons were examined. High permeability at 1 kHz above 20000 as well as saturation flux density B_s of about 1.5 T were obtained in the compositional range of 5.5 to 7.5 at % Nb and 8 to 12 at % B for nanocrystalline bcc Fe-Nb-B alloy ribbons annealed for 3.6 ks at 923 K. Soft magnetic properties substantially depend on the annealed structure which does not essentially depend on as-quenched structure for $Fe_{84}Nb_7B_9$ alloy ribbons.

I. INTRODUCTION

Recently, nanocrystalline softmagnetic materials have been developed by utilizing the first stage of crystallization process of Fe-Si-B-Nb-Cu [1], Fe-M-B (M = Zr, Nb, Hf) [2]-[5] and Fe-P-C-Ge-Cu [6] amorphous alloys. Among these alloys, the Fe-M-B nanocrystalline alloys is characterized by higher B_s even than the Fe-Si-B-Nb-Cu alloy because of higher Fe content in the alloys owing to the high glass-forming ability of M element and the formation of the nanocrystalline bcc phase without additional non-magnetic element of Cu, Si or Ge.

It has been known that soft magnetic properties depend on the grain size of the bcc phase [7], but the microstructure formed by crystallization of the amorphous phase has not been investigated in detail, furthermore, the relation between the microstructure and the soft magnetic properties is not wholly clarified. Therefore, the aim of this paper is to investigate the change in microstructure and soft magnetic properties in an annealed state as a function of the composition and the thickness of ribbons, and to study the relation between the annealed nanostructure and as-quenched microstructure for the melt-spun Fe-Nb-B alloys which are considered to be promising for practical use because the ribbon can be produced more easily in Ar atmosphere than the Fe-Zr-B or Fe-Hf-B alloys.

II. EXPERIMENTAL

Ternary alloy ingots were prepared by arc-melting mixture of pure metals in an argon atmosphere. Fe-Nb-B ribbons with a cross section of about 0.006-0.026 x 15 mm^2 were produced by a single-roller melt spinning method in an argon atmosphere.

The structures for melt spun and annealed samples were examined by X-ray diffractometry using Co-Kα radiation; An incident angle in the measurement was fixed at 2°, usually applied for deposited film samples, in order to obtain the profiles near the surface. The lattice parameter (a_0) was evaluated from the (110)bcc, (200)bcc and (211)bcc reflection peaks measured by the step scanning method. The diffraction angle was calibrated on the basis of the reflection angle of the internal standard material (pure Si powder, 640a). The average grain size (Gs) was evaluated by using Scherrer's equation from the half-width of (110)bcc reflection peak. The broadening of diffraction line due to the width of X-ray source was removed by Warren's method. Saturation flux density (B_s) under an applied field of 800 kA/m, effective permeability (μ_e) under an applied field of 0.4 A/m, coercive force (H_c) under an applied field of 8 kA/m and saturation magnetostriction (λ_s) under an applied field of 40 kA/m were measured at room temperature with a vibrating sample magnetometer, vector impedance analyzer using a ring-shaped sample with a size of 6 mm in inner diameter and 10 mm in outer diameter, dc B-H loop tracer and by a strain gage method, respectively. The ribbon surface roughness was measured by scanning transverse direction of ribbon using stylus roughness tester and represented the number of roughness above 0.5 μm in depth per 1 mm.

Microstructures and composition of the bcc and the amorphous phase for a $Fe_{84}Nb_7B_9$ ribbon annealed under an optimum condition for the achievement of soft magnetic properties were observed by high-resolution electron microscopy (HRTEM) combined with EDS and EELS analysis. HRTEM observations were performed with JEM-2010F. The electron beam for EDS and EELS analysis were 0.6 nm and 3 nm in diameter, respectively.

III. RESULTS AND DISCUSSIONS

The compositional dependence of B_s and μ_e for rapidly solidified Fe-Zr-B and Fe-Hf-B nanocrystalline alloys have already been reported [2][4]. Figure 1 shows the compositional dependence of B_s, μ_e and λ_s for rapidly solidified Fe-Nb-B alloys annealed for 3.6 ks at 873 K or 923 K, namely under the optimum conditions, along with the phase field in the melt-spun state. High μ_e values above 20000 are obtained in the range of 5.5 to 7.5 at % Nb and 8 to 12 at % B. These composition range corresponds to the phase field of either an amorphous single phase or coexistent amorphous and bcc phase in the melt-spun state and also agrees with that for formation of mostly bcc single phase. The results are similar to that of Fe-Zr-B and Fe-Hf-B alloys previously reported [2][4].

Figure 2 shows X-ray diffraction patterns for an $Fe_{85}Nb_7B_8$ alloy consisting of a bcc phase and an amorphous phase and an $Fe_{84}Nb_7B_9$ alloy consisting of a mostly single amorphous phase in an as-quenched state. Both alloys crystallized by annealing for 3.6 ks at 923 K show the high permeability as shown in Fig. 1. Therefore, it can be said that the completely single amorphous phase in an as-quenched state is not always necessary for achievement of high μ_e.

Figure 3 shows X-ray diffraction patterns of $Fe_{84}Nb_7B_9$ ribbons with various thickness in the melt-spun state (a) and annealed for 3.6 ks at 923 K (b). In the melt-spun state, the microstructures of the free surface depend on the ribbon thickness, or the cooling rate at which the samples are

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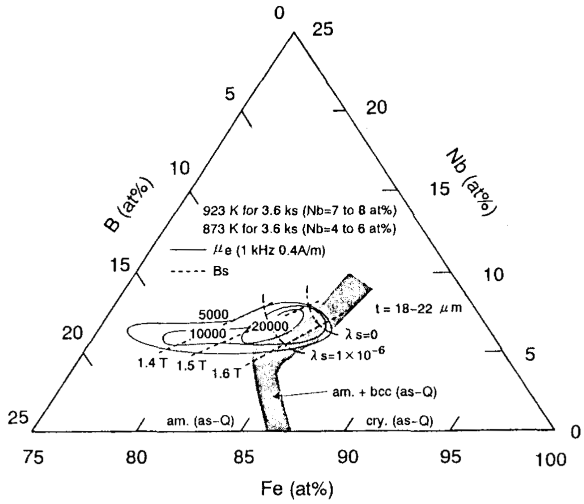

Fig. 1 Compositional dependence of saturation flux density (B_s), effective permeability (μ_e) and saturation magnetostriction (λ_s) for melt spun Fe-Nb-B alloys annealed for 3.6 ks at 873 K or 923 K. The data of the phase field in a rapidly solidified state are also shown for reference.

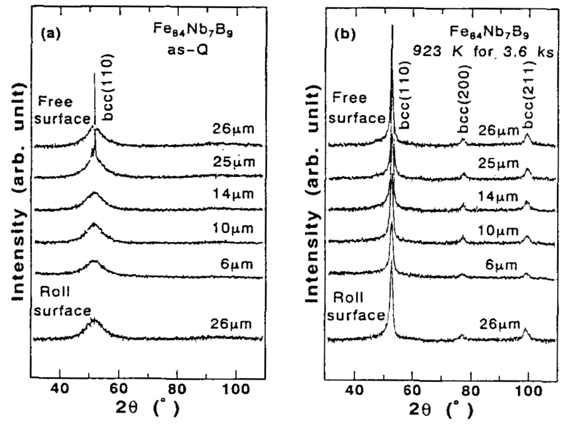

Fig. 3 X-ray diffraction patterns of $Fe_{84}Nb_7B_9$ alloys with various thickness in an as-quenched state (a) and annealed for 3.6 ks at 923 K.

solidified. The samples with 6 to 14 μm in the thickness have a mostly single amorphous phase, the samples with 25 and 26 μm in thickness consist of mixtures of an amorphous and a bcc phase. The structures of the roll surface do not depend on the ribbon thickness. The annealed structures of the free and roll surface show nearly the same X-ray diffraction patterns, are considered to not depend on the thickness.

Figure 4 shows the thickness dependence of the G_s and a_0 of the bcc phase for a melt-spun $Fe_{84}Nb_7B_9$ alloy annealed for 3.6 ks at 923 K. The G_s and the a_0 show the nearly the constant values irrespective of ribbon thickness, respectively.

The dependence of μ_e at 1 kHz, H_c and the roughness of the free surface of the ribbons on the ribbon thickness are shown in Fig. 5. The roughness of the roll surfaces for the ribbons with various thickness, which are not presented in this paper are nearly the constant. The roughness of the free surface decreases with the ribbon thickness; the μ_e increases and H_c decreases with decreasing the roughness. The similar relation between soft magnetic properties and the roughness or smoothness of the ribbon has been well known for amorphous soft magnetic alloys [8]. It is thus assumed that the changes of H_c and μ_e occur by the ribbon roughness, not by the structural change due to the difference of the ribbon thickness. Therefore, it is considered that almost the same nanocrystalline structures for the achievement of good soft magnetic properties are obtained by the annealing either the amorphous single phase or the mixture of the amorphous and the bcc phase.

Figure 6 shows a high-resolution micrograph, EDS and EELS spectra for an $Fe_{84}Nb_7B_9$ ribbon with 14 μm in thickness annealed for 3.6 ks at 923 K. The photograph reveals a detailed nanostructure consisting of a mixture of the crystalline grains and the amorphous phase surrounding the bcc grains. The EDS and the EELS spectra reveal that the concentration of Nb and B in the amorphous region (a) are

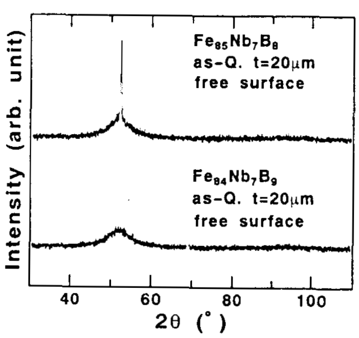

Fig. 2 X-ray diffraction patterns of an $Fe_{84}Nb_7B_9$ alloy and an $Fe_{85}Nb_7B_8$ alloy with thickness of 20 μm in an as-quenched state.

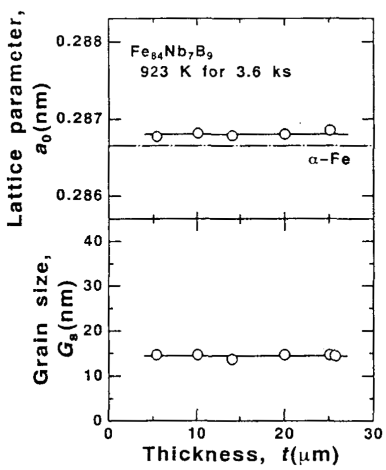

Fig. 4 Changes of lattice parameter (a_0) and grain size (G_s) as functions of ribbon thickness (t) for $Fe_{84}Nb_7B_9$ alloys annealed for 3.6 ks at 923K.

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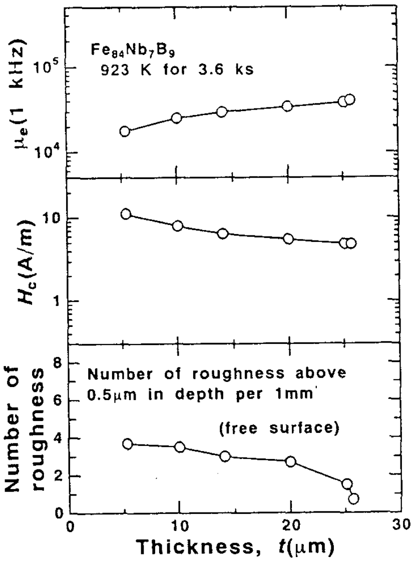

Fig. 5 Changes of effective permeability (μ_e), coercive force (H_c) and roughness of the free surface of ribbons as functions of ribbon thickness (t) for $Fe_{84}Nb_7B_9$ alloys annealed for 3.6 ks at 923 K.

considerably higher than that in the bcc region (b). As shown in Fig. 4, the a_0 and the G_s do not depend on the ribbon thickness. Since the a_0 value is well known to be changed by the amounts of solute elements [9], the amount

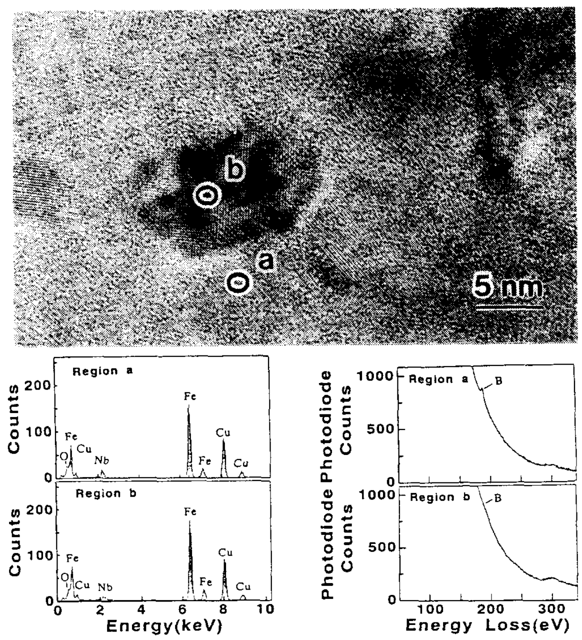

Fig. 6 A high-resolution electron micrograph of an $Fe_{84}Nb_7B_9$ ribbon annealed for 3.6 ks at 923 K. EDS spectra and EELS spectra were taken from the amorphous phase (region a) and the bcc phase (region b) in the micrograph.

of Nb and B elements in the bcc phase is considered to little depend on the ribbon thickness. Therefore, it is assumed that the amounts of Nb and B element in the residual amorphous phases are little changed for the ribbon with the various thickness.

The residual amorphous phases for the various thickness have nearly the same curie temperature (T_c), which have been reported to strongly depend on the amounts of M (M = Zr, Hf) and B elements [4][10]. The T_c is considered to be a dominant factor for good soft magnetic properties as well as the G_s for the nanocrystalline Fe-M-B alloys [4], the slight change in the T_c and the G_s results in the slight change in soft magnetic properties. From this consideration, the change in the H_c shown in Fig. 5 is supposed to be mainly due to the change in the roughness of the surfaces of the ribbon.

IV. CONCLUSIONS

The structure and magnetic properties for Fe-Nb-B alloys were examined, the results are summarized as follows.

The high μ_e at 1 kHz above 20000 as well as B_s of about 1.5 T were obtained in the compositional range of 5.5 to 7.5 at % Nb and 8 to 12 at % B for nanocrystalline bcc Fe-Nb-B alloy ribbons annealed for 3.6 ks at 923K.

Almost the same nanocrystalline structures for the achievement of good soft magnetic properties are considered to be obtained by the annealing either the amorphous single phase or the mixture of a large amount of the amorphous and a small amount of the bcc phase.

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