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IMPROVEMENT IN HIGH FREQUENCY MAGNETIC PROPERTIES OF THIN AMORPHOUS RIBBONS BY SURFACE OXIDATION

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The effects of surface oxidation on magnetic properties were investigated at high frequencies (10k-10MHz) for 7-18μm thick $Co_{70}Fe_5Si_{15}B_{10}$ amorphous ribbons with controlled domain structure. Oxidation was accelerated by acid-treatment or anodic oxidation treatment, and the insulation layers were prepared on the surfaces of the ribbons. The acid-treatment was effective in improving permeability and magnetic loss. Although the anodic oxidation treatment was effective in both making oxide layer and thinning, the magnetic properties were not improved compared with the case of the acid-treatment.

Ⅰ. INTRODUCTION

Non-magnetostrictive Co-based amorphous ribbons are of interest as core materials for new switching devices operating at frequencies above 1MHz.[1,2] Their magnetic properties, however, are strongly restricted by eddy current that increases with an increase in ribbon thickness. Therefore, a reduction in ribbon thickness is effective in improving their high frequency properties. In addition to thinning, control of the magnetic domain structure and reduction in surface roughness are excellent methods of improving high frequency properties.[3]

In high frequency above 1MHz, thinning ribbons is not necessarily effective because of eddy current flowing beyond layers. Consequently, insulation between ribbons is needed to improve high frequency magnetic properties of cores prepared from thin amorphous ribbons, and we showed that insulation between ribbons improves magnetic properties at frequencies higher than 1MHz.[3] However, the space factors of cores were not so high because the thickness of the insulation layer was roughly equal to that of the ribbons (several μm).

Recently, oxide layers were made on Fe-based soft magnetic metal particles to separate from each other and to enhance high frequency magnetic properties.[4] Thus we tried with success to make thin insulation layers by acid-treatment or anodic oxidation of ribbon surfaces and to improve high frequency magnetic properties of the cores made from the thin amorphous ribbons.

Ⅱ. EXPERIMENTAL PROCEDURE

$Co_{70}Fe_5Si_{15}B_{10}$ amorphous ribbons were prepared by the single-roller quenching technique in He atmosphere (about 250Torr) or in air. The width and the thickness of the prepared ribbons were about 3mm, and 7.1-13.5μm (in He atmosphere) and 17.6μm (in air), respectively.

The surfaces of the prepared ribbons were oxidized by acid-treatment or anodic oxidation treatment. The acid-treatment is done in 2% nitric acid solution for 30sec to accelerate oxidation. The weight change due to the acid-treatment was less than 2.5% under our experimental condition, which suggests that the ribbon thickness dose not changed remarkably by the treatment. In anodic oxidation treatment, a ribbon was dipped in 5% sulfuric acid solution, and then the D.C. voltage of 0.5V was applied between the ribbon and the tungsten cathode bar to accelerate oxidation and to thin the ribbon by electrolytic polishing.(Fig.1) The temperature of the solution and the treating time were about 27℃ and 30min, respectively.

After the surface oxidation of the ribbons, toroidal cores, 15mm in outer diameter and 13mm in inner diameter, were made from the ribbons. Subsequently, the cores were annealed in O_2 atmosphere under an applied field of 288kA/m perpendicular to the ribbon axis. This annealing induced magnetic anisotropy and lays magnetic domain walls in the transverse direction to the ribbon axis. Thus the cores were magnetized mainly by the rotation of magnetization.

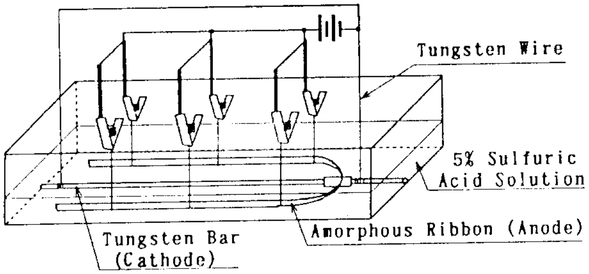


Fig.1 Schematic representation of apparatus for anodic oxidation treatment.

A small inductors were prepared from the cores, and then the complex permeability, μ'-jμ", was determined up to 10MHz by measuring its impedance with an impedance analyzer (YHP 4275A). The magnetic loss per cycle was measured up to 2MHz with a digital oscilloscope (Yokogawa DL1200). The depth profile of oxygen content was measured by ESCA.(Electron Spectrum for Chemical Analysis)

Ⅲ. RESULTS AND DISCUSSION

A. Effect of acid-treatment

Typical examples of the depth profile of oxygen content are shown in Fig.2. In the untreated ribbon, the content of oxygen is high at the surface but decreases markedly with the etching time. After etching for 5min, the oxygen content reaches a steady value. On the other hand, the oxygen content in the acid-treated ribbon decreases gradually with the etching time, and dose not reach a steady value even after etching for 60 min. This result suggests that oxide layers of the acid-treated ribbon are thicker than those of the untreated ribbons. The thickness of the oxide layers prepared by the acid-treatment is not clear at the present state, because the etching speed is not clear for our apparatus. If we assume the etching speed for Co, the etching time of 60 min corresponds to the removal of the 470nm surface layer.

The real part of the permeability at 10MHz, μ', is shown in Fig.3(a) for the cores prepared from the ribbons with and without the acid-treatment. The average thickness d was determined from their weight, length, width and density. The figure also includes μ' calculated analytically under the classical assumption that the magnetization changes by rotation. Under this assumption, μ' is expressed as

$$\mu' = \frac{\mu_{dc}}{\gamma d}\frac{[2\sinh^2(\gamma d)+2\sin^2(\gamma d)]^{1/2}}{\cosh(\gamma d)+\cos(\gamma d)}\cos\delta, \qquad (1)$$

$$\delta = \tan^{-1}\left(\frac{\sinh(\gamma d)-\sin(\gamma d)}{\sinh(\gamma d)+\sin(\gamma d)}\right), \qquad (2)$$

$$\gamma = \left(\frac{\omega\sigma\mu_{dc}}{2}\right)^{1/2}, \qquad (3)$$

where ω, d, ρ and μ_{dc} are the angular frequency, the ribbon thickness, the electrical conductivity and the D.C. permeability, respectively. In our case, μ_{dc} was determined from the experimental value ($1800\times\mu_0$-$2200\times\mu_0$) and σ was assumed to be $7.7\times10^5\Omega^{-1}m^{-1}$. The cores prepared from the acid-treated and field-annealed ribbons (*B) show higher μ' than those prepared from untreated and field-annealed ribbons (*C) at all the thickness studied. However, the measured μ' is much smaller than calculated one. This difference may be attributed to the roughness of ribbon surfaces as indicated in our previous work.[3]

Thickness dependence $\mu_0\tan\delta/\mu'$ (δ : the loss angle), which is a quantity related to the magnetic loss in a small excitation regions, is shown in Fig.3(b). $\mu_0\tan\delta/\mu'$ at 10MHz decreases drastically by the acid-treatment at large d region. This effect, however, is not so remarkable at small d region. The reason for this phenomenon is considered as follows. With decreasing d, the surface roughness of ribbons increases and then the electrical resistivity between ribbons would increase. Consequently, interlayer eddy current would

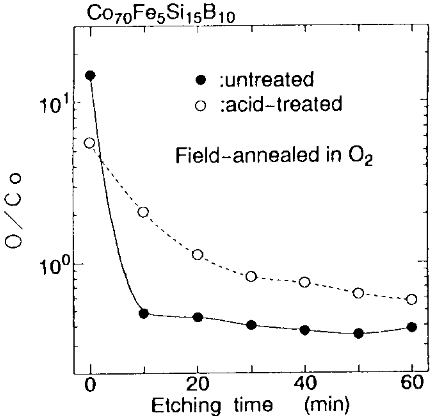
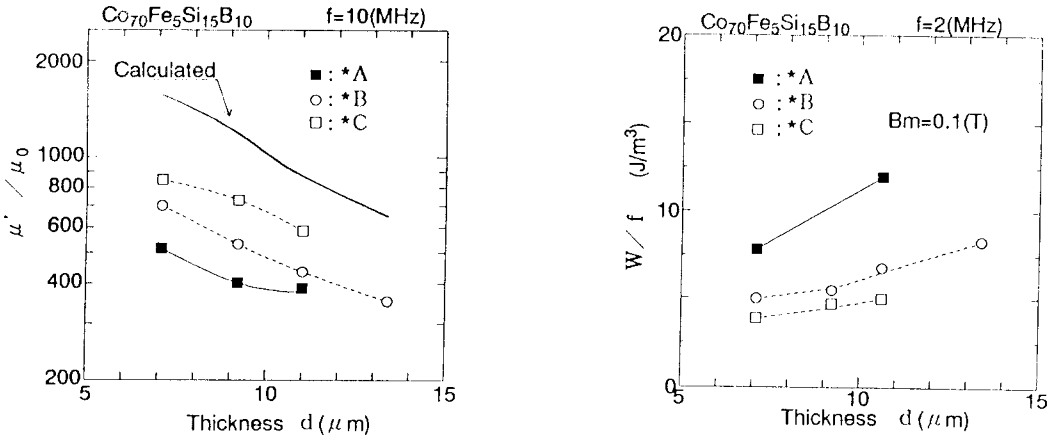
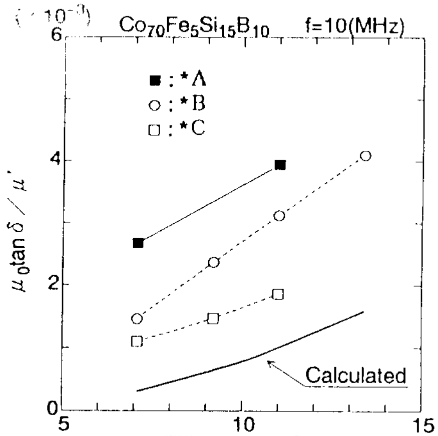


Fig.2 Depth profile of oxygen content obtained for ribbons with and without acid-treatment. Both the ribbons were field-annealed in O_2 atmosphere.



(a) μ'/μ_0 at 10MHz

(c) Magnetic loss per cycle, W/f at 2MHz



(b) $\mu_0 \tan\delta / \mu'$ at 10MHz

Fig.3 Effect of acid-treatment on permeability and magnetic loss. All the ribbons were field-annealed in O_2 atmosphere.

*A : acid-treated and unannealed
*B : untreated and field-annealed
*C : acid-treated and field-annealed

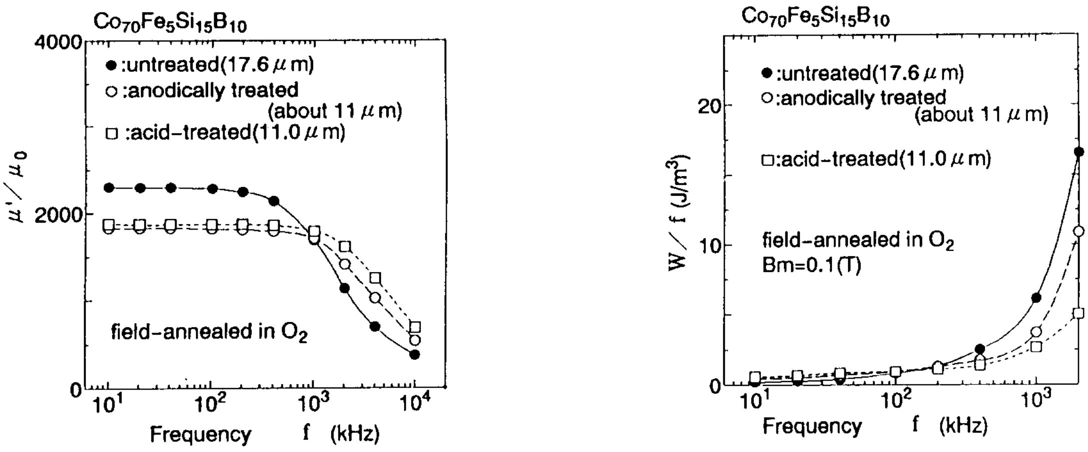
be suppressed due to poor electric contact between ribbons, even if ribbons dose not have insulated layers on their surfaces. Thus improvement by the acid-treatment would not have remarkable effect in small d region. At d=7.1μm, the measured $\mu_0 \tan\delta/\mu'$ is about three times as large as calculated one. Therefore reduction of surface roughness is expected to result in further improvement in $\mu_0 \tan\delta/\mu'$. If ribbons had smoother surface than our ribbons, the acid-treatment would be more effective than the present case.

The magnetic loss, W/f, under a large excitation condition is also improved by the acid treatment as shown in Fig.3(c). The effect, however, is not noticeable compared with the case of a small excitation.(Fig.3(b)) This difference may be attributed to the fact that oxide layers are very thin and insulation can be broken out by the large voltage between ribbons induced by large dB/dt.

B. Effect of anodic oxidation treatment

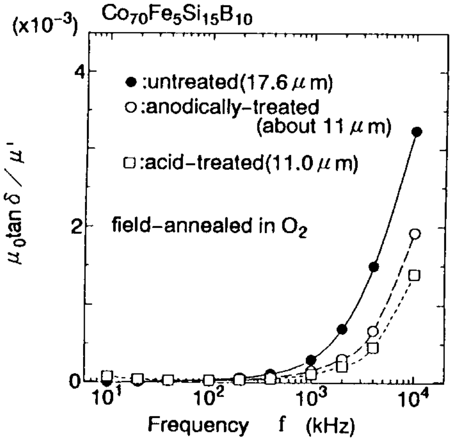
As anodic oxidation of thin ribbons made holes in their bodies, thick ribbons (17.6μm, prepared in air) were used for the experiment. After anodic oxidation treatment, the thickness of the ribbon was reduced into about 11μm. Fig.4(a) shows frequency dependence of μ'/μ_0 of the anodically treated ribbon, together with that of the acid-treated and untreated ribbons. The permeabilities of the acid-treated and anodically treated ribbons are higher than that without treatments beyond 1MHz. However, μ'/μ_0 of the anodically treated ribbon is smaller than that of the acid-treated ribbon whose thickness is roughly the same.

Frequency dependencies of μ'/μ_0 and magnetic loss per cycle, W/f were shown in Fig.4(b) and Fig.4(c). The effect of anodic oxidation treatment was not remarkable compared with that of the acid-treatment.



(a) μ'/μ_0

(c) Magnetic loss per cycle, W/f



(b) $\mu_0 \tan\delta / \mu'$

Fig.4 Frequency dependence of permeability and magnetic loss of untreated, anodically treated and acid-treated ribbons. The acid-treated ribbon has the same thickness as that of the anodically treated ribbon. All the ribbons were field-annealed in O_2 atmosphere.

IV. CONCLUSION

The effect of insulation by surface oxidation on magnetic properties were investigated for thin $Co_{70}Fe_5Si_{15}B_{10}$ amorphous ribbons in the frequency range of 10k-10MHz. The results are summarized as follows.

1. Insulation by surface oxidation accelerated by acid-treatment was effective in improving permeability and magnetic loss at all the thickness studied.(7-14μm)
2. Anodic oxidation was effective in making oxide layers and thinning. The magnetic properties, however, were not improved compared with the case of the acid-treatment.

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