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## Crystal Oriented Manganese Zinc Ferrites Made from Acicular Fine Particles

N. Hiratsuka, K. Kakizaki and M. Sugimoto\*

*Department of Functional Materials Science, Faculty of Engineering, Saitama University,  
255 Shimo-Ohkubo, Urawa 338, Japan*

*\* Department of Culture Information, Teikyo Heisei University, Uruido, Ichihara 290-01, Japan*

### Abstract

The acicular Mn-Zn ferrite fine particles composed of 65.6 mol% Fe, 21.9 mol% Mn and 12.5 mol% Zn were made. These Mn-Zn ferrite particles were etched in 1N-HCl solution for 3hrs. The fine particle was made up of several crystallites whose long axis were 0.2-1.0 $\mu$ m, and arranged to  $\langle 311 \rangle$  direction. These fine particles were pressed with applying magnetic field. The oriented Mn-Zn ferrites were prepared by sintering at 1250 $^{\circ}$ C for 4hrs. in air and then cooling in nitrogen atmosphere. The oriented Mn-Zn ferrite exhibited strong crystal orientation similar to the single crystal of it. The magnetic anisotropy constants were  $K_1=2.5 \times 10^3$  erg/cc and  $K_2=-0.2 \times 10^3$  erg/cc and the easy magnetization axis was  $\langle 100 \rangle$  direction.

### 1. Introduction

Single-crystal Mn-Zn ferrites, which have high saturation magnetization and initial permeability, are widely put into practice as high density magnetic head. However, they can't been used at high frequency of 1 MHz or above because of low resistivity. On the other hand, polycrystalline Mn-Zn ferrites, whose saturation magnetization and initial permeability are not good as those of single-crystal Mn-Zn ferrites, can improve magnetic characteristics at high frequency by forming high resistive boundary layers. We therefore prepare crystal oriented Mn-Zn ferrites whose crystallites are oriented like those of single-crystal as well as form high resistivity layers at grain boundary. The reports of the crystal oriented soft ferrites were few : the Mn-Zn ferrite was formed by using hexagonal platelet  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and  $\alpha$ -MnOOH. [1,2] In this paper, preparation procedure, crystal orientation and magnetic properties of the crystal oriented Mn-Zn ferrites are presented.

### 2. Experimental

The starting materials were acicular goethite ( $\alpha$ -FeOOH), of the order of around 3.5 $\mu$ m long axis and aspect ratio of around 35, as well as Mn(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>3</sub> and Zn(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>2</sub>. These were weighed to compose of 65.6mol% of Fe, 21.9mol% of Mn and 12.5mol% of Zn, and then well-mixed in the solvent of water and ethanol. The acicular Mn-Zn ferrite fine particles were prepared when heated at 900 $^{\circ}$ C for 4hrs. in nitrogen gas atmosphere. After these fine particles were dispersed in 70% of slurry concentration by using wet ball-milling method, they were pressed at 200kg/cm<sup>2</sup> by the wet press method being applied 12kOe(960kA/m) of magnetic field. The samples which were also pressed applying no magnetic field for comparing with above-mentioned samples. The oriented Mn-Zn ferrites were prepared when they were heated at 1250 $^{\circ}$ C for 4hrs. in air and then cooled in nitrogen gas atmosphere.

### 3. Results and discussion

Photo 1 shows SEM photograph of the acicular Mn-Zn fine particles ( $M_s=68.8$ emu/g) having about 2 $\mu$ m of long axis and about 15 of aspect ratio. Photo 2 shows SEM photograph of the fine particle which was etched in 1N-HCl for 3hrs. Several crystallites having 0.2-1.0 $\mu$ m of long axis arranged to  $\langle 311 \rangle$  in the parallel direction to its long axis.

Figure 1 shows a scheme of press and applied magnetic field direction for the cubic shaped Mn-Zn ferrite. Oriented Mn-Zn ferrite were made as follows. The pressed bodies were sintered at 1250 $^{\circ}$ C for 4 hrs. in air and then cooled in nitrogen gas.

Figure 2 shows x-ray diffraction patterns of the sample as shown in fig.1. The sample indicates intense crystal orientation :

(311) plane in (a), (100) plane in (b) and (110) plane in (c). Figure 3 shows a torque curve of the oriented Mn-Zn ferrite measured for (c) plane as shown in fig 1. The curve was distorted comparing with sine wave in the vicinity of 150° to 210°. When the curve was analyzed and calculated anisotropy constants,  $K_1$  and  $K_2$  were  $2.5 \times 10^3$  erg/cc and  $-0.2 \times 10^3$  erg/cc, respectively.

The easy magnetization axis of the oriented Mn-Zn ferrite corresponds to  $\langle 111 \rangle$  direction as a result of  $K_1 > 0$ . Toroidal cores of the oriented Mn-Zn ferrite bulk were prepared to investigate dependence of initial permeability on frequency. Three kinds of toroidal cores were prepared to press by wet method with applied magnetic field: sample (A) was pressed between same magnetic poles, sample (B) between different poles and sample (C) without magnetic field. These were sintered by the above-mentioned process. Figure 4 shows dependence of initial permeability on frequency for the samples. The sample (A) shows the highest value of initial permeability. This is because the  $\langle 100 \rangle$  direction of the easy magnetization axis arranged parallel to flux. As a result of x-ray diffraction analysis, the crystal orientation of the sample (A), (B) and (C) arranged parallel to the  $\langle 100 \rangle$ ,  $\langle 111 \rangle$  and  $\langle 110 \rangle$  direction, respectively.

4. Conclusion

- 1) The acicular Mn-Zn ferrite fine particles composed of 65.6 mol% Fe, 21.9 mol% Mn and 12.5 mol% Zn were fabricated by sintering at 900°C for 4hrs. in nitrogen gas atmosphere. The fine particles, of the order of 2µm long axis and 15 of aspect ratio, have 68.8 emu/g of saturation magnetization.
- 2) These Mn-Zn ferrite particles were etched in 1N-HCl solution for 3hrs.. The fine particle was made up of several crystallites whose long axis were 0.2-1.0µm, and arranged to  $\langle 311 \rangle$  direction.
- 3) These fine particles were pressed with applying magnetic field. The oriented Mn-Zn ferrites were prepared by sintering at 1250°C for 4hrs. in air and then cooling in nitrogen atmosphere.
- 4) The oriented Mn-Zn ferrite exhibited strong crystal orientation similar to the single crystal of it. The magnetic anisotropy constants were  $K_1 = 2.5 \times 10^3$  erg/cc and  $K_2 = -0.2 \times 10^3$  erg/cc and the easy magnetization axis was  $\langle 100 \rangle$  direction.

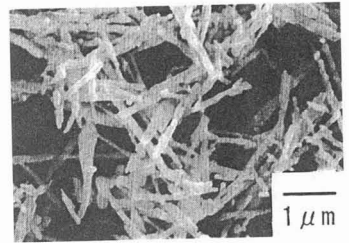


Photo.1 SEM micrograph of acicular manganese zinc ferrite particles sintered at 900°C.

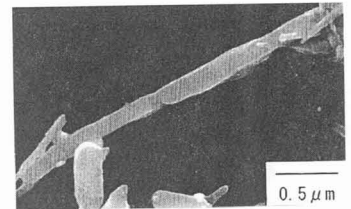


Photo.2 SEM micrograph of chemically etched acicular manganese zinc ferrite particles.

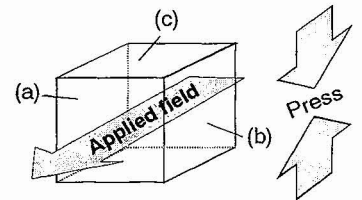


Fig.1 Schematics of the process of making crystal-oriented Mn-Zn ferrites.

References

- [1] K.Kugimiya : IEEE Trans. Mag., 10 (1974) 907.
- [2] E.Hirota : IEEE Trans. Mag., 7 (1971) 337.

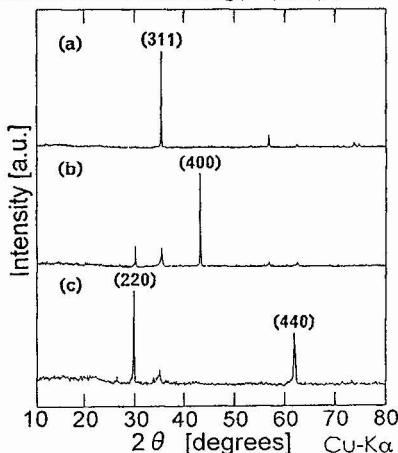


Fig.2 X-ray diffraction patterns measured in the three dimensional directions of crystal-oriented manganese zinc ferrite sintered at 1250 °C.

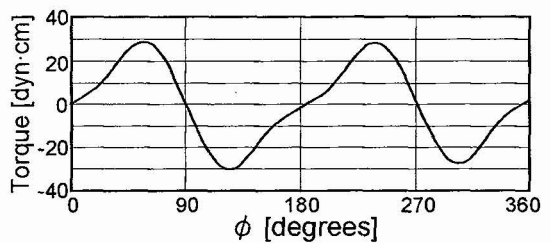


Fig.3 Magnetic torque curve of the oriented Mn-Zn ferrite measured for (c) plane as shown in fig.1.

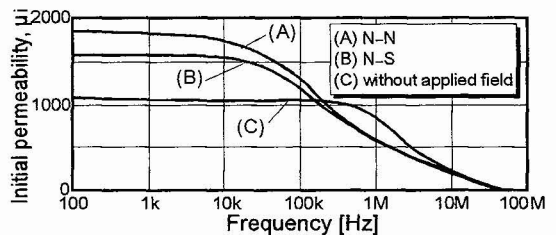


Fig.4 Dependence of initial permeability on frequency for the samples.