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Low Loss of Fine Grained Mn-Zn Ferrite

Y. Yamamoto, A. Makino and T. Nikaidou

Central Research, Alps Electric Co., Ltd., 1-3-5 Higashitakami, Nagaoka 940, Japan

abstract. Mn-Zn ferrite with grain size of $1.5\mu\text{m}$ was obtained by sintering hydrothermal-precipitated powders with uniform dispersion of additions. The fine grain size and high density were realized simultaneously by the use of fine particle ferrite powder and fine particle additives, planetary ball mill mixing of MnZn ferrite powder and additives and low temperature sintering. The core loss of a newly developed ferrite with fine grain was significantly reduced to that of a commercial ferrite.

1. Introduction

Recently, power supplies are required to reduce the volume and the thickness in compliance with miniaturization in the portable electronic equipments and the on-board power supply modules. As an increase in the driving frequency is an effective method for these purpose, core materials with low core losses at high frequency are earnestly desired. Mn-Zn ferrites generally have lower core losses at high frequency in comparison with Ni-Zn ferrites and metallic magnetic materials. We have reported [1,2] that the fining the grain size of the Mn-Zn ferrite is effective for the reduction of core losses at the frequency range from 1 to 2MHz, mainly due to the reduction of the residual loss which is a dominant part of the core losses at the high frequency. In this study, we tried to realize simultaneously the fine grain size and uniform dispersion of additions such as SiO_2 , CaCO_3 , Ta_2O_5 , TiO_2 by (1) using hydro-thermal precipitated Mn-Zn ferrite powder with 0.13mm in size and the additions with small particle sizes, and (2) mixing the powders in a planetary ball mill.

2. Experimental procedure

The MnZn ferrite powder for the sintered bodies were prepared by the hydrothermal method. A hydrothermal-precipitated ferrite powder (particle size : $0.13\mu\text{m}$) with composition of $\text{Mn}_{0.72}\text{Zn}_{0.16}\text{Fe}_{2.12}\text{O}_4$ was used as a starting material. The additions were added to the ferrite powder, mixed by planetary ball mill and this uniform mixture of the ferrite powder and the additions were calcined in a reduced atmosphere. Sample A and C were undoped and sample B and D were doped with 0.01wt% of SiO_2 (particle size : $0.012\mu\text{m}$), 0.021wt% of CaCO_3 (particle size : $0.04\mu\text{m}$), 0.038wt% of Ta_2O_5 (particle size : $0.3\mu\text{m}$) and 0.034wt% of TiO_2 (particle size : $0.5\mu\text{m}$). Sample A and B were calcined at 973K and sample C and D were calcined at 1173K. The calcined powders were milled by planetary ball mill, dried, mixed with a proper quantity of organic binder, pelletized, cold isostatically pressed at 275MPa for 180s and sintered at 1323K for 14.4ks in a reduced atmosphere. The microstructure was observed with a optical microscope. The electric resistivity (ρ) was measured by the four-point probe method. The permeability (μ' , μ'') and $\tan\delta$ were measured under a field of 0.4A/m with a impedance analyzer. The core loss (P_c) was measured with an B-H analyzer. The hysteresis loss (P_h) was determined by extrapolating the (P_c/f) -f plots to $f=0$. The classical eddy current loss (P_e) is given as equation (1) under a model of simultaneous rotation magnetization process [3].

$$P_e = (\pi^2 f^2 B m^2 d^2) / 6\rho \quad (\text{W/m}^3) \quad \dots\dots(1)$$
 where d is thickness and ρ is electrical resistivity. Residual loss (P_r) is calculated as $P_r = P_c - P_h - P_e$. The samples were ring-shaped, with outer and inner diameter of 8 and 4 mm, respectively, and a thickness of 2.0 mm. The mag-

Table 1. Densities, grain sizes, electric resistivities and core losses of sample A, B, C and D prepared by sintering at 1323K.

	Additions	Calcining temperature (K)	Relative density (%)	Grain size (μm)	Electric resistivity ($\Omega\cdot\text{cm}$)	Core loss (at 1MHz, 50mT) (kW/m^3)
Sample A	No	973	97.2	3.4	7.5	730
Sample B	Yes*	973	98.8	1.5	63	450
Sample C	No	1173	97.2	1.8	2.8	720
Sample D	Yes*	1173	98.6	1.5	135	270

*1 : $\text{SiO}_2=0.01\text{wt}\%$, $\text{CaCO}_3=0.012\text{wt}\%$, $\text{Ta}_2\text{O}_5=0.038\text{wt}\%$, $\text{TiO}_2=0.034\text{wt}\%$

netic properties of the commercial power ferrite were compared with a newly developed ferrite.

3. Results and discussion

Table 1 shows the densities, the average grain sizes, the electric resistivities and magnetic properties of the sample A,B,C and D. The increases of the ρ and the decreases of the core losses by adding Si, Ca, Ta and Ti. Figure 1 shows the frequency dependence of μ' , μ'' and the relative loss factor ($\tan\delta/\mu'$) of the doped sample D, which shows the minimum P_c , and the commercial ferrite. A noteworthy finding is that the relative loss factor in the lower frequency range of the two are in agree but in the higher frequency range that of the sample D remarkably decrease, namely, the effectiveness of the sample D for the high frequency transformer is confirmed.

To consider the reason of the superior core loss property of the sample D, the relationship of the core loss per cycle and the frequency of the two samples are plotted (Fig. 2). A mutual relationship of the two is strikingly similar to the mutual relationship of frequency dependence of the relative loss factor (Fig.1). In the lower frequency region the core losses per cycle are irrelevant to the frequency and the core loss of this frequency region are caused by hysteresis loss. An important matter is that the core loss per cycle in the lower frequency region of the two are approximately equal and the effect of the large difference of the average grain sizes of the two (sample D:1.5 μ m, commercial ferrite:9.5 μ m) is not actualized. Accordingly, this suggest that the core loss per cycle in the lower frequency region can be eliminated the effect of the grain sizes of the samples. On the other hand, in the higher frequency region the frequency dependence of the P_c for the sample D occurs in higher frequency than the commercial ferrite. This frequency region corresponds to the region of the eddy current loss or the residual loss. Therefore, the additional effect of Si, Ca, Ta and Ti is concluded as the suppressive effect of these losses in high frequency.

Table 2 shows the physical properties and core losses of the sample D and commercial ferrite. It is noticeable that the core loss in high frequency of the commercial ferrite is higher than that of the sample D, in spite of considerably higher electric resistivity than sample D. Therefore, if the core loss depend on the eddy current loss, the eddy current loss cannot be explained by the dc-resistivity originated in a grain boundary barrier. For this reason, the eddy current loss which is attributable to the bulk conductivity seems to be the most probable. In this case, it is supposed two capabilities as arusult of the dissolution of the dopant to the bulk, one is the inhibition of the hopping between Fe^{3+} and Fe^{2+} for the bulk and another is the inhibition of the resultant electrons by the thermal dissociation of the constituent oxygen of the ferrite, these two possibilities are under investigation. This estimation as described above is in harmony with the fact that the core loss of the doped sample calcined at high temperature, 1173K, is lower than that of the doped sample calcined at 973K (Table 1); It can be considered that the high temperature calcined sample has a highly inhibitory action, the above mentioned, as the increase of amount of dopants dissolution into the bulk. Furthermore, the increase of the dc-resistivity and the decrease of the core loss for doped samples of Table 1 are comprehended by considering the simultaneous occurring of the deposition / segregation of the dopant to the grain boundary and the dissolution to the bulk. On the other hand, if the core loss in the high frequency region originates in the residual loss, a model can be considered that cation vacancies and anion vacancies are created by doping and then the magnetic wall displacement or the rotation of the magnetization is pinned by these vacancies.

Fig.1 Change in permeability and $\tan\delta/\mu'$ as a function of frequency for the sample D and the commercial ferrite.

Fig.2 Change in the core loss per cycle as a function of frequency for the Sample D and the commercial ferrite.

Table 2. Comparison of physical properties and separated core losses of the sample D and commercial ferrite.

	Grain size (μ m)	Resistivity (Ω -cm)	Permeability	Core loss (1MHz, 25mT, 40°C) / kWm ⁻³			
				Total loss	Hysteresis loss	Eddy current loss	Residual loss
Sample D	1.5	135	1600	25	10	7	8
Commercial ferrite	9.5	960	2900	208	15	79	114

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