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Influence of Small Addition of Ti^{4+} Ions on the Properties of High Permeability Ferrite

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Abstract. Mn-Zn ferrites substituted with Ti^{4+} ions are now becoming commercially important. The substitution with titanium ions in Mn-Zn ferrite is recommended for ferrites with low losses and low temperature factor of initial permeability, T_k/μ_i , because of the shift of the secondary maximum of permeability, SMP, towards lower temperatures [1] and the increase of the d.c.resistivity [3,4,5].

The present paper reports the results obtained by substitution of a small amount of titanium ions to high permeability ferrite $\text{Mn}_{0.51}\text{Zn}_{0.43}\text{Fe}^{2+}_{0.06}\text{Fe}_2\text{O}_4$, by adequate reduction of Zn^{2+} content. The magnetic and electric properties improve: d.c. electrical resistivity increases while the loss factor $\text{tg}\delta/\mu_i$ and disaccommodation decrease. By addition of 0.35% by weight TiO_2 , the initial permeability, at 20°C, improves. We remark the flattening of $\mu_i(T)$ curves with the increase of content of Ti^{4+} ions and the diminution of the peak at the Curie point, facts that can be associated with two points of anisotropy compensation instead of one, the second minimum of K_1 being in the neighborhood of T_c . Structural and magnetic properties of high permeability ferrite with substitution of titanium ions confirm that Ti^{4+} ions make pairs with Fe^{2+} ions.[4, 6].

1. INTRODUCTION

High permeability ferrite is obtained in a relatively small percentage range of the Mn-Zn ferrite system [2] and the raw materials must be of adequate purity, usually a total content of impurity must be $\approx 0.1\%$ [1,2,]. On the other hand, the substitution of Ti^{4+} ions in Mn-Zn ferrite is recommended for the ferrite characterized by low losses and low T_k/μ_i , because of the increase of d.c.resistivity and the shift of SMP towards lower temperatures, $\sim 100^\circ\text{C}/1\text{mol TiO}_2$ [2, 3]. A particular feature of the substitution with titanium is that the nature of the disaccommodation process is modified, giving a pronounced improvement in the long-term stability [4]. The present paper reports the results obtained by addition of small amount of TiO_2 (0.1-1% by weight) to high permeability ferrite. For 0.35% by weight TiO_2 , initial permeability, at 20°C, improves.

2. EXPERIMENTAL

The ferrite $\text{Mn}_{0.51}\text{Zn}_{0.43}\text{Fe}^{2+}_{0.06}\text{Fe}_2\text{O}_4$ and the same ferrite with the additions of Ti^{4+} ions were prepared by the usual ceramic technique. Appropriate mixtures of commercial oxides Fe_2O_3 , ZnO , Mn_2O_3 and TiO_2 were pre-fired at 850°C , milled in demineralized water and granulated. After pressing, the toroids were all sintered at 1350°C in air and cooled in a nitrogen atmosphere with less 0.1% O_2 [7]. For quick reference, the sample without titanium ions was denoted A, the sample with 0.35%g TiO_2 was denoted A₁, the sample with 0.67%g TiO_2 was denoted A₂ and the sample with 1.02%g TiO_2 was denoted A₃. The sintered samples are in accordance with the general formula: $\text{Me}^{2+}_{1-x}\text{Fe}^{2+}_{2x}\text{Ti}^{4+}_{2-2x}\text{Fe}^{3+}_{2-2x}\text{O}_{4+y}$ where $x=0.01, 0.02, 0.03$. The initial permeability and relative loss factor were measured at 1-100 kHz using a Maxwell bridge. The disaccommodation factor was obtained from the inductance change in 10 and 100 min. after 50 Hz ac demagnetization. The d c resistivity was measured with indium-gallium contacts. The initial permeability and relative loss factor (at 10 kHz) versus temperature within the range -150° - $+150^\circ\text{C}$ were measured, using a constant temperature chamber, to ensure that the samples would be in thermal equilibrium.

3. RESULTS AND DISCUSSION

The curves $\mu_i(T)$, figure 1a, show two characteristic phenomena. The first one is the shift of secondary maximum of permeability towards lower temperatures with the increase of titanium ions amount. The shift of SMP is approximately -50°C and -60°C per 1mol TiO_2 addition. The second characteristic is the flattening of $\mu_i(T)$ curves with the increase of Ti^{4+} ions. This flattening of curves could be explained by the existence of two points of anisotropy constant compensation. We remark two minimums on the $\text{tg}\delta/\mu_i$ vs. temperature curves (fig. 1b) only for the samples of ferrites substituted with Ti^{4+} ions. These results are in accordance with the paper [5]. Stijntjes *et al* showed that in a material without magnetocrystalline anisotropy or at zero crossings of anisotropy, one expects a minimum of hysteresis loss and residual loss.

Fig.1a: Initial permeability at 10 kHz vs. Temperature

Fig.2a: SEM of ferrite with 0.35% TiO₂

Fig.1b: Loss tangent/initial permeability vs. Temperature

Fig.2b EDAX analysis of ferrite with 0.35% TiO₂

Fig. 2a shows the microstructure of high permeability ferrite with 0.35% TiO₂ and the concentration of the elements rated at Fe content (fig.2b). A uniform distribution of titanium ions on the surfaces of grains and boundaries is observed. The microstructure is characterized on the uniform pore-free crystallites. In the meantime, the d.c. electrical resistivity, the loss factor vs. frequency and the disaccommodation factor are improved by addition of TiO₂ (Table 1).

Table 1

Sample	Ti ⁴⁺ ions (x)	tgδ/μ _i × 10 ⁻⁶				ρ × 10 ⁻² [Ωm]	DAF × 10 ⁻⁶ (at 20°C)
		1kHz	20kHz	50kHz	100kHz		
A	0.00	1.6	5.0	18	32	15	4.0
A ₁	0.01	1.5	3.0	6	11	260	1.2
A ₂	0.02	2.5	5.0	8	12	500	0.8
A ₃	0.03	3.8	5.5	9	14	900	2.0

4. CONCLUSION

This study shows that the substitution of small amount of Ti⁴⁺ ions can lead to the improving of magnetic properties of high permeability ferrite. The experimental data indicate that addition of TiO₂ less than 0.5% by weight will yield optimum disaccommodation and temperature factor of μ_i and reduced loss factor tgδ/μ_i. By the addition of 0.35% TiO₂, the initial permeability at room temperature increases.

The experimental results show that Ti⁴⁺+Fe²⁺ ions contribute to the anisotropy constant K₁ with a positive and weakly dependent temperature value, in good agreement with [4, 6]. We remark the flattening of μ_i(T) curves with the increase of content of Ti⁴⁺ ions and the diminution of the peak at the Curie point, facts that can be associated with two points of anisotropy compensation instead of one, the second minimum of K₁ being in the neighborhood of T_C.

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