

RELATIONS BETWEEN MICROSTRUCTURE AND MAGNETIC PROPERTIES OF MN-ZN FERRITES FOR POWER APPLICATIONS

BY

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ABSTRACT. This report deals with the influence of the milling time of the raw powders and with the influence of the co-additives CaO and Si₂O on the microstructure and on the magnetic properties of the ferrite cores. Two categories of samples were obtained by varying the milling time and the additions. The samples differ in average grain size and porosity, therefore the electrical conductivity and magnetic properties are different for various samples. The sample with longest milling time (250 hours) and with additive presents the highest performances.

1. INTRODUCTION

Many efforts have been done for improving the power loss of Mn-Zn ferrites [1,2]. It was reported [3] that additions of CaO and Si₂O in suitable proportions to Mn-Zn ferrites resulted in much higher resistivities than when was only CaO or Si₂O added. Toolenaar [4] found that below a temperature of 1200°C the presence of silicon had no influence on the development of the fine-grained microstructure.

This paper focuses its discussion on the influence of the particle size of Mn-Zn ferrite powders and of the additives on the main magnetic properties of four ferrite core specimens prepared by high temperature sintering (1360°C).

2. EXPERIMENTAL RESULTS

In order to investigate the effect of milling time of the raw powders and of

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the additives CaO and Si₂O on the microstructure and the porosity of Mn-Zn ferrites, samples without and with additions were prepared by conventional methods. First time two raw powders with chemical compositions denoted by A and B:

Composition A: Fe₂O₃ 51.5 mol%, MnO 35 mol%, ZnO 13.5 mol%;

Composition B: Fe₂O₃ 51.5 mol%, MnO 35 mol%, ZnO 13.5 mol%,
Si₂O 0.014 wt%, CaO 0.014 wt%.

The raw materials (α Fe₂O₃, Mn₃O₄ and ZnO) were mixed in suitable proportions for 16 hours in steel ball-mill using water as the mixing medium. The mixture was dried and pre sintered at 800°C for 1.5 hours in air. The X ray analysis of the pre sintered powder contain diffraction maximum specific for spinelic phase as well as for α Fe₂O₃, Mn₃O₄ and ZnO phases.

The presintered powders were milled in water the ratio material: balls: water was 1: 2: 1.5. Four categories of sample, presented in Table 1, were obtained.

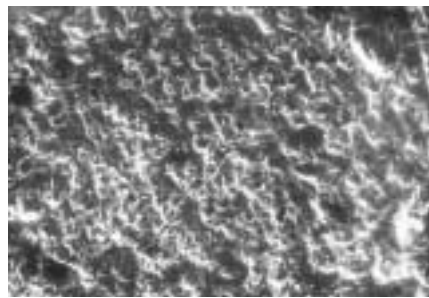
Sample	Milling time (hours)	Green density (g cm ⁻³)	Density (g cm ⁻³)	BET Ss (m ² g ⁻¹)	Contraction (%)
A0	32	2.8 ± 0.1	4.56	3.84	13.58
A1	70	2.8 ± 0.1	4.29	5.95	14.89
B0	70	2.8 ± 0.1	4.52	6.62	15.63
B1	225	2.8 ± 0.1	4.68	14.42	16.05

The ring cores with outer radius 24 mm, inner radius 12 mm and height 12.5 mm were pressed at 150 MPa and sintered 2 hours in a Riedhammer oven with protective gas atmosphere and cooled without oxygen (less of 0.1 vol%).

The microstructures of the sintered samples were observed using the optic microscope IOR MC-1. The specimens were polished with aluminium oxide and etched in a solution of 25vol% HCl conc. in 75vol% ethanol for 1 hour. Figures 1a-1d show these microstructures.

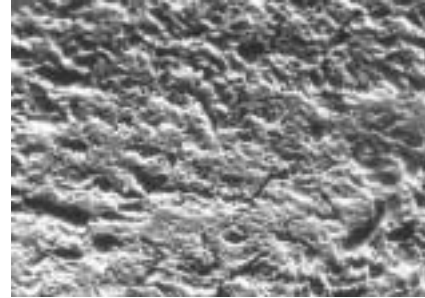
One observes that sample A0 presents pores on the surface of the particles and on the grains boundaries, as is shown in Fig.1a. The pores on the surface, having diameter of 2 - 4 μ m, are rarely and non-uniform distributed. The pores on the boundaries are uniform distributed and have sub micrometer diameters. The sample A1 obtained in the same condition by varying the milling time (see Table 1) presents a microstructure (featured in Fig.1b) characterised by low

porosity on the surface, the pores have diameter of 1 – 3 μm , and uniform distributed pores on the grains boundary.



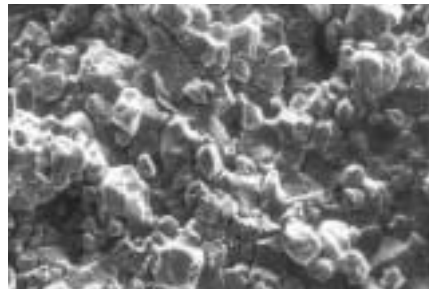
100 μm

Fig. 1a



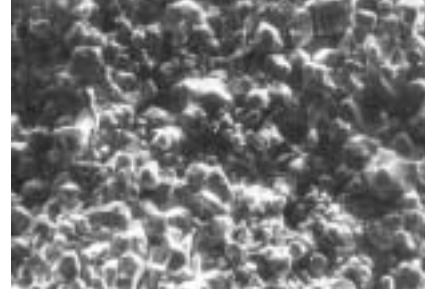
100 μm

Fig. 1b



100 μm

Fig. 1c



100 μm

Fig. 1d

Fig. 1 The microstructures: a) Sample A0, b) Sample A1, c) Sample B0, d) Sample B1.

The sample B0 was obtained by adding Si_2O and CaO to the A1 pre sintered powder and sintering in the same procedure (sample with the microstructure presented in Fig. 1c). The average grain size of the B0 sample increases. The porosity on the surface and on the boundaries increases. The pores on the grain boundaries, having diameter of 2 – 5 μm become significant. The sample B1 with the same composition was obtained by increasing the milling time, consequently the BET specific surface. The microstructure of the sample B1, featured in Fig. 1d, proves that the increase of milling time and the

additions allows lowest porosity on surface and on grain boundaries. Rarely one observes pores with sub micrometer diameters and in majority these are distributed on grain boundaries.

The microstructures explain the magnetic properties and performances of the ferrite cores. In order to obtain the hysteresis loops for all the samples a storage scope interfaced to the computer was used [9]. The hysteresis loops obtained at 150 A/m magnetic field strength for the sinusoidal waveform with the frequency ranging in 0.3 – 15 kHz were processed to determine the main magnetic characteristics. Figures 2a – d show the influence of the excitation frequency on the shape of the hysteresis loop.

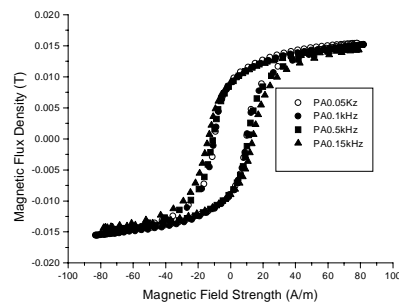


Fig. 2a

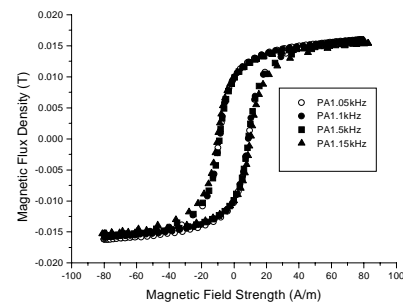


Fig. 2b

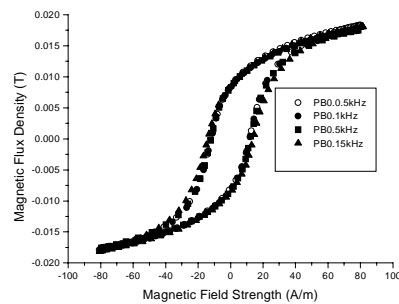


Fig. 2c

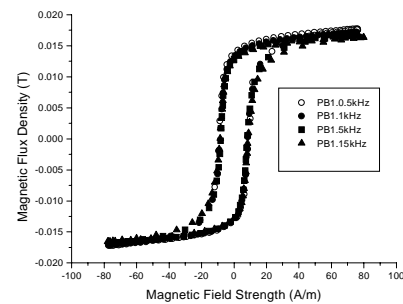


Fig. 2d

Fig. 2 The hysteresis loops for four frequencies (0.5, 1, 5, 15kHz) at 150 A/m magnetic field strength for: a) Sample A0, b) Sample A1, c) Sample B0, d) Sample B1

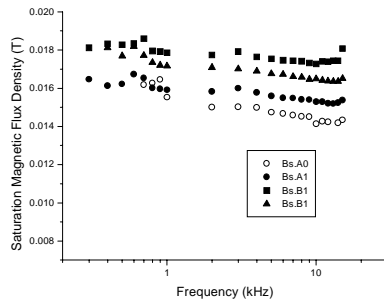


Fig. 3a

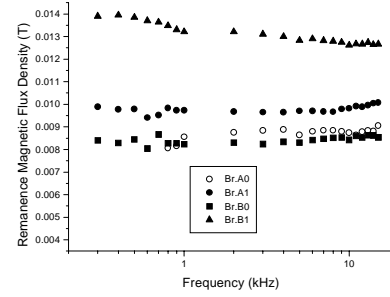


Fig. 3b

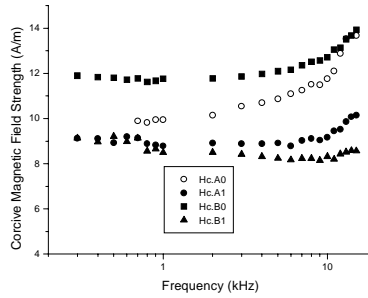


Fig. 3c

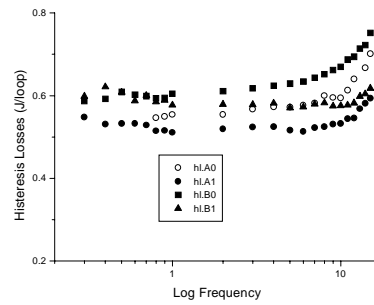


Fig. 3d

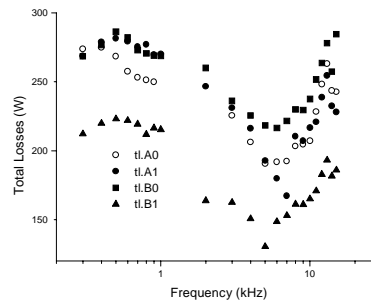


Fig. 2e

Fig.2 Magnetic properties vs. frequency for the samples: ○ Sample A0, ● Sample A1, ■ Sample B0, ▲ Sample B1; for 150A/m magnetic field strength: a) saturation magnetic flux density, b) remanence magnetic flux density, c) coercive magnetic field strength, d) hysteresis losses, e) total losses

From the hysteresis loops presented in figure 2a-d one observes that the changes with the excitation frequency of the saturation magnetic flux and

coercive field are smaller. One observes differences depending on the milling time and on the additions. The addition 0.014%wt SiO₂ and 0.200%wt CaO contributes to the decrease of the coercive magnetic field and of the saturation magnetic flux densities.

The saturation magnetic flux density at fixed magnetic field strength (150A/m) decrease with increasing the frequency (see figures 2a) as well as the remanent magnetic flux density (see figure 2b). The coercive magnetic field strength increases by increasing the frequency (see fig. 2c) and the increasing slope depend on porosity influenced by the additions. The addition increase the resistivity but as non magnetic phase lead to the drop of the saturation magnetic flux and to the increase of the coercive field by impeding the movement of the domains walls. The increase for the sample A0 is fastest while for sample B1 with longest time milling and additions the slope of the increase is slower. The hysteresis losses featured in figure 2d also increase by increasing the frequency and are low influenced by the frequency in B1 sample case. The dependence of the total losses versus frequency are a usually one as is show in fig. 2e.

3. CONCLUSIONS

The influence of time milling and of the 0.014%wt SiO₂ and 0.200%wt CaO additions on the microstructure and magnetic properties of Mn-Zn ferrite cores for power applications were investigated. The samples sintered in the same conditions at 1360°C present different behaviours determined by the differences on time milling and on the addition, which provide differences in the microstructures. The sample B1 characterised by smallest porosity on the boundary and on the surface of the grains features the highest magnetic performances.

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