

# INVESTIGATION OF POWER FERRITES

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**KEY WORDS:** power ferrites, MnZn ferrites, ferrite cores, magnetic materials, magnetic properties, switch mode power supplies, materials research, experimental results

**ABSTRACT:** The microstructure and grain boundary chemistry of MnZn ferrites for high frequency Switch Mode Power Supplies (SMPS) were investigated. Results show that the oxygen partial pressure, applied during the sintering of ferrites, is of vital importance for the microstructure development, grain boundary chemistry and magnetic properties of MnZn ferrites.

# RAZISKAVE MOČNOSTNIH FERITOV

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**KLJUČNE BESEDE:** močnostni feriti, MnZn feriti, feritna jedra, magnetni materiali, magnetne lastnosti, stikalni pretvorniki, raziskava materiala, eksperimentalni rezultati

**POVZETEK:** Raziskovali smo vpliv kemijske sestave meje med zrni in razvoj mikrostrukture MnZn feritov za močnostne aplikacije. Doseženi rezultati kažejo velik vpliv parcialnega tlaka kisika v fazi sintranja na razvoj mikrostrukture, kemijsko sestavo meje med zrni in magnetne lastnosti MnZn feritov.

### INTRODUCTION

The application of Switch Mode Power Supplies (SMPS) increases permanently and promotes the development of new materials which enable a higher energy transfer per volume unit of ferrite material, the operation of ferrite cores at higher frequencies and an efficient design of new, improved circuits.

The useful application of MnZn ferrite in SMPS is associated with long term investigations. Particularly the optimisation of the composition and the engineering of the microstructure were important in improving the ferrite quality and achieving cores with good performances. However, on a fine scale the ingredients present intentionally ( $\text{TiO}_2$ ,  $\text{SnO}_2$ ) or not ( $\text{SiO}_2$ ,  $\text{CaO}$ ) can modify the grain resistivity or segregate on the grain boundary and form isolating films which increase the bulk resistivity. In that way the eddy current losses which dominate other losses at higher frequencies can be effectively suppressed<sup>(1,2,3)</sup>.

### EXPERIMENTAL

Ferrite samples of various compositions were prepared by conventional ceramic process using chemical grade materials. A computer-controlled tube furnace was used to regulate the firing temperature and oxygen partial pressure. Ferrite toroids were sintered for 2 hours at 1200, 1300 and 1355°C with equilibrium  $p\text{O}_2$ , which enables 70, 80, 90 and 95 % decomposition of excess  $\text{Fe}_2\text{O}_3$ .

The temperature dependence of core losses was measured with a wattmeter at test frequencies 25 and 100 kHz and induction level of 200 and 100 mT. Other frequency dependent parameters were measured using the impedance analyser.

The microstructure was determined by optic microscope and grain boundaries were inspected by the use of TEM.

**Table I: Compositions of ferrites studied and sintering profiles**

A	$\text{Mn}_{0.658}\text{Zn}_{0.260}\text{Sn}_{0.010}\text{Ti}_{0.011}\text{Fe}_{2.061}\text{O}_{4\pm\gamma}$	1	$T_s=1300^\circ\text{C}$	$t = 2\text{h}$	$\text{O}_2 \rightarrow 70\%$
B	$\text{Mn}_{0.659}\text{Zn}_{0.266}\text{Sn}_{0.010}\text{Ti}_{0.011}\text{Fe}_{2.055}\text{O}_{4\pm\gamma}$	2	$T_s=1200^\circ\text{C}$	$t = 2\text{h}$	$\text{O}_2 \rightarrow 80\%$
C	$\text{Mn}_{0.659}\text{Zn}_{0.268}\text{Sn}_{0.010}\text{Ti}_{0.011}\text{Fe}_{2.052}\text{O}_{4\pm\gamma}$	3	$T_s=1355^\circ\text{C}$	$t = 2\text{h}$	$\text{O}_2 \rightarrow 95\%$
D	$\text{Mn}_{0.659}\text{Zn}_{0.271}\text{Sn}_{0.010}\text{Ti}_{0.011}\text{Fe}_{2.049}\text{O}_{4\pm\gamma}$	4	$T_s=1300^\circ\text{C}$	$t = 2\text{h}$	$\text{O}_2 \rightarrow 90\%$

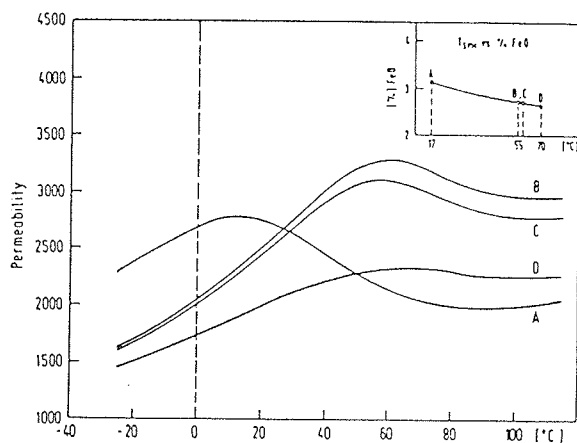


Fig. 1: Temperature characteristics of permeability

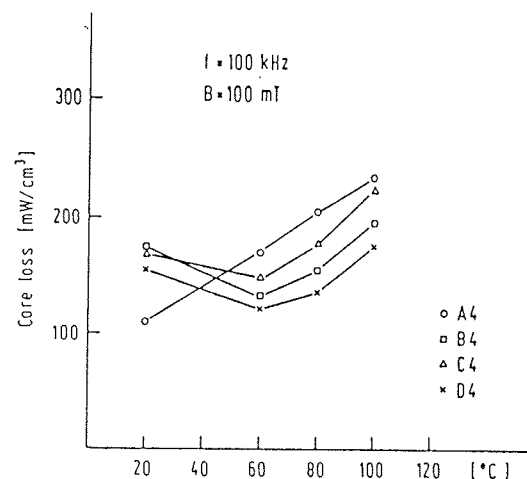


Fig. 2: Temperature characteristics of core loss

## RESULTS AND DISCUSSION

In Fig. 1 the temperature dependence of initial magnetic permeability vs. ferrite stoichiometry is shown. The compositions of ferrite studied and the sintering profiles used are presented in Table I.

The gradual increase in the FeO in samples is accompanied by a corresponding shift of  $T_{SMP}$  ( $T_{SMP}$  = temperature where the permeability exhibits the peak in magnetic permeability). At that temperature the mechanism of magnetic polarisation is at least hindered so the temperature characteristics of core loss exhibits at that temperature its minimum as can be noticed from the temperature characteristics of core losses, Fig. 2. In Fig. 3 the frequency characteristics of initial permeability vs. composition is shown. The firing cycle was identical for all samples studied. The courses of frequency characteristics of magnetic permeability for samples A, B, C and D are similar, demonstrating that the limited change in the composition of samples studied does not influence noticeably the frequency stability, provided the firing cycle and hence the microstructure and stoichiometry are identical. A slight deviation can be noticed for sam-

ples A. The microstructure inspection of samples A shows that in these samples the beginning of discontinuous grain growth can be found here and there.

On the other hand, samples A1 and A4, sintered at firing cycles 1 and 4 respectively, show different courses of frequency characteristics of electric resistivity, Fig. 4. During the firing cycle 1 higher partial pressure of oxygen was applied (Table I). Samples A1 show lower grain boundary resistivity while samples A4 exhibit higher grain boundary resistivity. At higher frequencies where the grain-boundary layer is short-circuited by the high displacement current, both curves approach to the same resistivity, that is to that of ferrite grains.

TEM examinations of samples A1 and A4 show that the segregation of ingredients in samples A4 is much more pronounced, Fig. 5b. Besides, these samples exhibit a fine grain microstructure as well, Fig. 4b. Therefore a higher number of grain boundaries and the presence of insulating layers on it due to impurities segregation

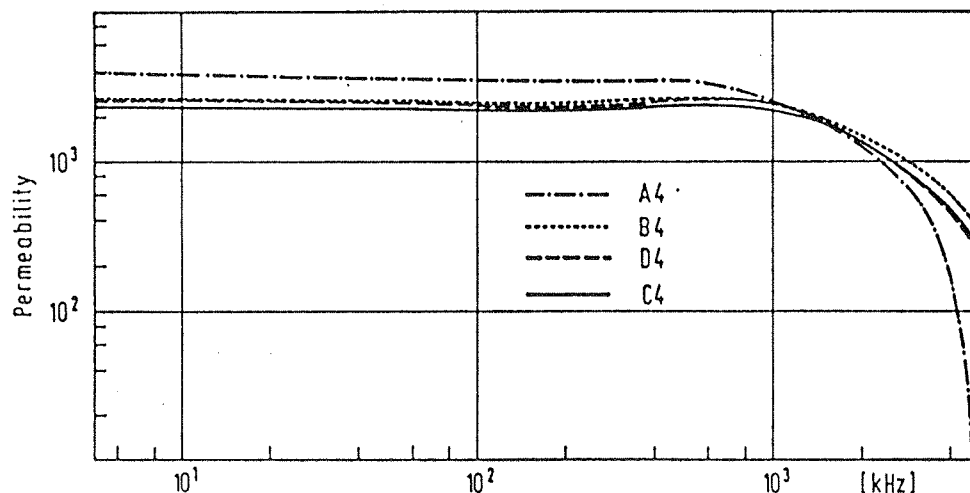


Fig. 3: Frequency characteristics of initial permeability

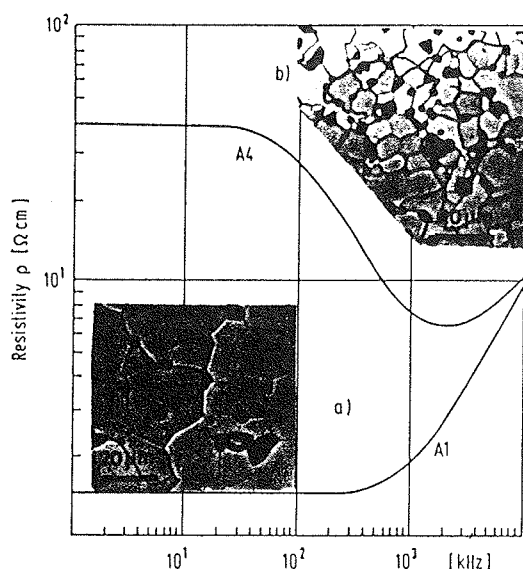


Fig. 4a,b: Frequency characteristics of resistivity

increase their grain boundary resistivity and minimises their eddy current losses.

On the other hand samples A1 exhibit lower grain boundary resistivity, and larger average grain size (Fig. 4a) while the segregation of impurities on their grain boundaries is less pronounced, Fig. 5a.

Since the sintering time of samples A using profiles 1 and/or 4 was the same in both cases one is justified to suppose that the grain boundary velocity is the most important parameter which governs the microstructure development and grain boundary chemistry and consequently eddy current losses.

In MnZn ferrites the microstructure development depends on the kinetic of grain boundary and pore motion<sup>(4)</sup>. Further, grain boundary and pore velocity depend

on the volume diffusion of vacancies, surface diffusion and vapour phase (oxygen) transport which determines the grain boundary velocity in MnZn ferrites to a great extent. Therefore the oxygen partial pressure is the essential parameter governing the intrinsic and effective grain boundary velocity in MnZn ferrites. In the case the grain boundary exceeds the pore velocity, large grains, exhibiting non-isolating grain boundaries<sup>(6)</sup> with intragranular porosity are formed. However, when the pores are attached to the grain boundaries during grain growth and a normal microstructure is formed, this is still not a guarantee for the formation of thick isolating grain boundaries, this is particularly true when relatively large grains are formed. Besides, samples A1 show large pores which increase the substantial magnetic flux density and total magnetic losses.

In samples studied the difference in the microstructure and consequently different magnetic losses are due primarily to different ambient conditions, i.e. different partial pressure of oxygen during sintering. The oxygen partial pressure and the temperature were found to be the most important parameters governing the microstructure development and grain boundary chemistry during the engineering of the performances of MnZn ferrites. Besides, a relatively high oxygen partial pressure increases the grain boundary resistivity due to the preference oxidation of ferrous ions in the grain boundary region<sup>(7)</sup> and enhance the impurities segregation on the grain boundaries<sup>(6)</sup>. However, oxygen pressure induced high grain boundary velocity may eliminate the beneficial influence of high oxygen pressure during sintering of power ferrites.

## CONCLUSIONS

The chemical composition of MnZn ferrites studied has a remarkable influence on the temperature characteristics of core losses in SMPS. Particularly, composi-

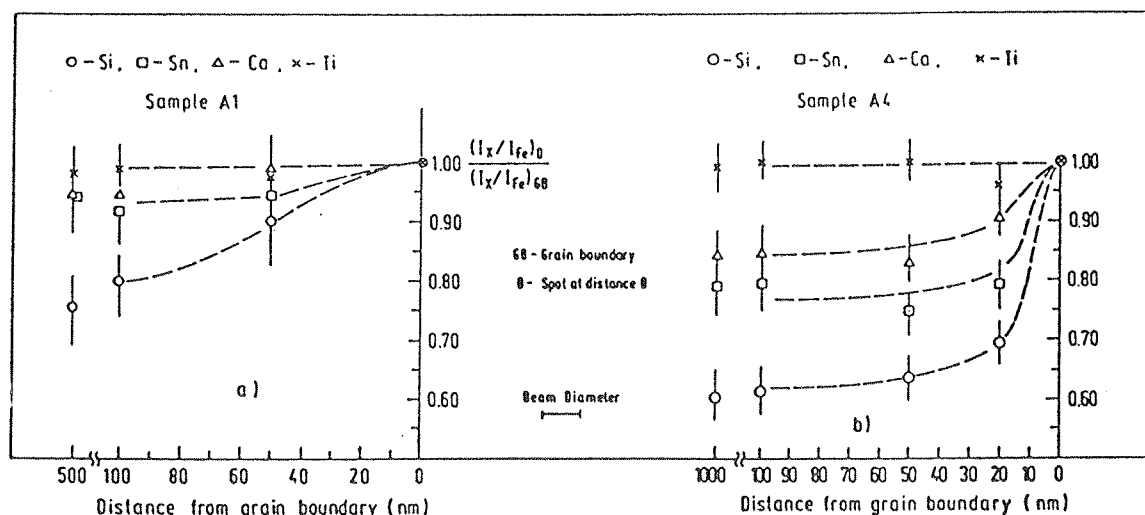


Fig. 5a,b: TEM/EDX study of the segregation of elements at grain boundaries in a - sample A1 and b - sample A4. Peak intensity ratios for different elements (scaling element is Fe), normalised to values measured at the grain boundary are plotted as a function of the distance from the boundary.

tions rich in ferrous ions modify the temperature characteristics of core losses to a great extent. The sintering parameters which keep a continuous grain boundary migration, i.e. a relatively high oxygen partial pressure, increase the average grain size and hinder the formation of insulating films on the grain boundaries that leads to high core losses.

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