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To cite this version:

HAL Id: jpa-00254816
https://hal.archives-ouvertes.fr/jpa-00254816
Submitted on 1 Jan 1997

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Effect of Doping with Nb$_2$O$_5$ on the Magnetic and Structural Properties of Mn-Zn Ferrite

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Abstract. The present paper reports the magnetic and structural properties of manganese-zinc ferrites obtained by the addition of small amount of Nb$_2$O$_5$ (0.03-0.1%g). For power applications of Mn-Zn ferrite it is necessary to have the Secondary Maximum of Permeability (SMP) and correspondingly low losses, in the domain of positive temperature, 50-100°C. The magnetic properties: initial permeability, power losses, temperature factor of permeability and $T_c$ improve by the addition of 0.03-0.07%g Nb$_2$O$_5$ to the Mn-Zn ferrite. Magnetic and structural results: XRD, SEM, EDX analysis, suggest that Nb $^+$ ions partially dissolve in the ferrite lattice, substituting Fe octahedral and loosely bounding Fe $^+$ ions.

1. INTRODUCTION

Manganese-zinc ferrites are widely used for power applications such as switched-mode power supplies, transformers, choke coils. It has been shown that the magnetic properties of Mn-Zn ferrites are dependent on the small amount of additives such as: SiO$_2$, CaO, Nb$_2$O$_5$, Ta$_2$O$_5$ [1,2,3], which affect the microstructure of the ferrites and the properties of the grain boundary.

The addition of Fe $^+$ or, and Ti $^+$ ions determines the decrease of loss factor $\tan \delta / \mu _1$ and an important shift of the secondary maximum of permeability, towards lower temperatures [4]. For power applications of Mn-Zn ferrite it is necessary to have the high $\mu _1$ and low losses in the domain of positive temperature, 50-100°C, in order to avoid overheating.

In this study, magnetic and structural properties of a powder Mn-Zn ferrite doped with Nb were analyzed in dependence on the Nb$_2$O$_5$ content. The magnetic properties $\mu _1$, $M_s$, $T_c$, power losses are improved by the doping with 0.03-0.07%g Nb$_2$O$_5$. The additions of Nb$_2$O$_5$ above 0.1%g establish structural and microstructural inhomogeneity.

2. EXPERIMENTAL PROCEDURE

Mn-Zn ferrite with the basic formula Mn$_{0.716}$Zn$_{0.277}$Fe$_{0.006}$O$_5$ (for quick reference the sample was denoted by a) and with various amounts of Nb$_2$O$_5$: 0.03%g (sample denoted b), 0.05%g (sample denoted c), 0.07%g (sample denoted d), 0.1%g (sample denoted e) were prepared by conventional ceramic technology. The mixture of raw materials ($\alpha$-Fe$_2$O$_3$, Mn$_3$O$_4$, ZnO with and without the amount of Nb$_2$O$_5$) was prefired for 1h at 1000°C. The presintered powder was milled to obtain a specific area of 2.5m$^2$g$^{-1}$ (BET method). The samples, toroidal shapes, were sintered in a tunnel-type kiln with protective gas atmosphere at 1370°C and cooled in a nitrogen atmosphere with less 0.1%O$_2$. Various magnetic properties such as initial permeability, initial permeability vs. temperature within the range 10-250°C, saturation induction, power losses at 200mT and 16 kHz were measured at these samples with and without niobium ions.

The structural parameters were determined by XR analysis using Seifert Debyeflex Diffractometer. The microstructure was observed with Electron Microscope Philips CM30. The concentration of various atoms within the grains and near the grain boundaries (-1.5µm-1.5µm) was analyzed by EDAX at 25 keV.

3. RESULTS AND DISCUSSION

Initial permeability at 10 kHz versus temperature is shown in Fig.1. The secondary maximum of permeability, SMP, is found to be shifted more and more towards lower temperatures, as the Nb content increases (0.03-0.07%g Nb$_2$O$_5$). However, for the addition of 0.1%g Nb$_2$O$_5$, SMP shifts in the opposite direction, to higher temperatures, behind the 95°C, corresponding to Mn-Zn ferrite without Nb$_2$O$_5$. The doping with Nb $^+$ ions produces the increase of $T_c$ ($5^\circ$C/300ppm Nb$_2$O$_5$), perhaps because of their influence on the Fe $^+$ ions distribution in sublattices. Table I gives the relative power loss, $P_L$, $B_s$ at $H$=3000Am$^{-1}$ and the temperature factor of $\mu _1$, $\alpha _T$, for the range 20-70°C.
The XRD patterns (Fig.2) for the samples \((a, b, c, d, e, \ldots)\) show a cubic symmetry of the spinel structure with the calculated values of elementary cell parameter, \(a_0\), in the range: 0.8435-0.8498 nm. We can remark a slightly modification, up to 0.17\%, of \(a_0\) for the 0.03-0.07\% Nb_2O_5 concentration, correlated with a continuous decrease of \([311]\) X-ray peak intensity. This behavior can be explained as a perturbation produced by the presence of Nb ions on the substitutional or interstitial positions in the host lattice. A significant decrease in lattice parameter, about 0.74\%, was evidenced only as a result of increasing of Nb_2O_5 concentration to 0.1\%. The \(a_0\) diminution can be attributed to Nb_5+ ion segregation out from the host lattice to grain boundaries, in concordance with the EDAX results. In the same time the \([311]\) peak intensity increase at a value similar to that for undoped sample, revealing a good formed crystalline structure. This is in concordance with SEM micrographs of sample with 0.1\% Nb_2O_5 that shows the crystallites with 50-70 \(\mu\)m diameter and many trapped pores, the beginning of discontinuous grain growth.

The atomic concentrations were obtained assuming that the elements are distributed uniformly within the analyzed area. The figure 3 show that Nb atoms are enriched near the grain boundary, but over a long distance (1.5\(\mu\)m).

The magnetic properties, the shift of SMP for Mn-Zn ferrite with 0.03-0.07 \% Nb_2O_5 towards lower temperatures and XRD analysis suggest that Nb_5+ partially dissolve in the ferrite lattice, substituting Fe octahedral, and loosely bound Fe_3+ ions. The changes in the microstructure and EDX analysis for Mn-Zn ferrite with 0.1\% Nb_2O_5 suggests that above this content, Nb_5+ ions are segregated to the grain boundaries. This effects are reflected in the magnetic properties.

The experimental data indicate that the samples with less 0.07\% Nb_2O_5 has a high initial permeability, good temperature factor and reduced power losses.

References