

# Analysis of power loss in vanadium doped nickel-zinc ferrites

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Nickel-zinc ferrite system,  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4 + x \cdot \text{V}_2\text{O}_5$ , with different vanadium additions from 0.0 wt% to 1.5 wt% in steps of 0.3 wt% has been prepared by conventional ceramic technique. The samples were sintered at 1210 °C for 4 hours in air atmosphere followed by natural cooling. The power loss and microstructures of these materials are examined. Microstructures of the samples reveal that vanadium additions resulted in fine grain structures with no significant variation in grain size with vanadium concentration. The power loss was measured in the frequency range from 100 kHz to 10 MHz under different exciting flux densities from 5 mT to 30 mT, and was analyzed as a function of frequency, composition and maximum exciting flux density. The hysteresis and eddy current losses were separated and analyzed as a function of frequency. The materials have displayed low power losses up to 3 MHz, thus making them suitable for power applications up to this frequency. The hysteresis loss is predominant loss mechanism in the lower frequencies approximately below 200 kHz, and for frequencies above 200 kHz the eddy current loss increases gradually with increasing frequency and becomes predominant in the power loss. The results are explained in terms of the compositional and microstructural modifications brought about by the vanadium additions.

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## 1. Introduction

Ferrites are useful electromagnetic materials at high frequencies. The magnetic behaviour of ferrites linking the extrinsic properties such as permeability, microstructure and power loss was widely studied with respect to composition, impurity levels and sintering conditions [1-3]. Fine grained high density materials with high magnetization and high resistivity are desirable for high frequency performance. Thus, efforts were made to enhance the density while retaining the fine grain size by introducing small quantity of sintering aids or by altering the sintering schedules [4-5]. These sintering aids often improve electric resistivity by contributing grain boundary component substantially, but cause to degrade magnetic performance because of secondary phase imperfections and pinning the movement of domain walls. Therefore, the type and amount of impurity that is required to obtain desired properties for a given application has become extremely selective.

Mn-Zn ferrites are usually used as core materials in the high frequency switching power supplies [6]. But, these materials exhibit high power losses when the operating frequencies go beyond 1 MHz. This is particularly due to the high n-type electrical conductivity negated between the  $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$  pairs in these materials. Ni-Zn ferrites with high saturation magnetization are an alternative as they possess relatively higher resistivities. Therefore, among all the compositions of a Ni-Zn ferrite system, the composition that results highest room temperature saturation magnetization,  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$  is

considered as basic composition for the present study. Since additions of high valences are reported to improve the core losses in Mn-Zn ferrites [7], it is aimed at studying additions of vanadium ions in the above Ni-Zn ferrite composition. This paper reports and analyzes the influence of vanadium additions in Ni-Zn ferrites on the permeability and power loss at high frequencies.

## 2. Experimental details

Ni-Zn ferrites with the general formula  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4 + x \cdot \text{V}_2\text{O}_5$ , where  $x$  varies from 0.0 to 1.5 wt% in steps of 0.3 wt%, have been prepared by conventional ceramic technique. Sintering of the samples was done at 1210 °C for 4 hours in air atmosphere. After the soaking temperature, the furnace was switched off and the samples were allowed to cool naturally. X-ray diffraction patterns of the samples confirm cubic spinel structures in all the samples. No extra lines were observed to represent any of the starting materials. Micrographs are taken on fractured samples using Philips XL-50 microscope. Permeability and power loss measurements are made on toroidal samples from 100 kHz to 10 MHz using Iwatsu B-H Analyzer. Hysteresis loops were measured on toroidal samples using hysteresisgraph. Magnetization and resistivity measurements were made on cylindrical samples by VSM and standard two-probe method, respectively.

### 3. Results and discussion

Hysteresis loops of vanadium doped samples for applied drive magnetic field amplitude at 5 kHz are shown in Fig. 1. The loops have almost similar coercivities for all the samples, but the saturation magnetic flux density varies with the composition. The saturation magnetization data is listed in Table 1 along with other parameters. Typical micrograph of V2(0.3wt%) sample is shown in Fig. 2. It can be seen from the micrograph that there are large number of intergranular bridges with discontinuous grain growth and the pores are mainly centered between the grains. The vanadium pentoxide with low melting point acts as a sintering aid and promotes densification provided the material is fired at a higher temperature. In order to avoid rapid grain growth and intragranular pores, the materials in the present study were fired at 1210 °C only. As a result, it seems that the grain growth is slow. However, as the concentration of vanadium increases, despite the slow grain growth, increasing number of pores would be trapped within the grains as evidenced by the micrograph. The slow grain growth in the system may be due to melting of  $V_2O_5$  which forms a liquid film at grain boundaries and thereby inhibits the grain growth [8]. This argument holds good for the whole range of concentrations supported by a marginal increase in grain size, as listed in the Table 1.

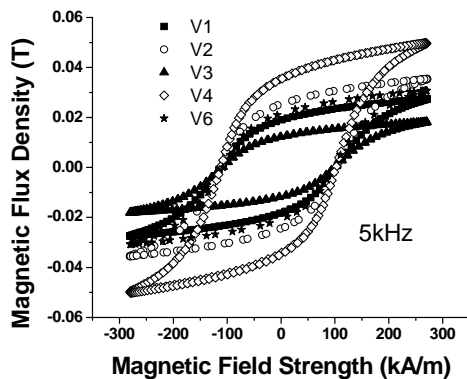


Fig. 1. Hysteresis loops of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + x V_2O_5$  samples at 5 kHz.

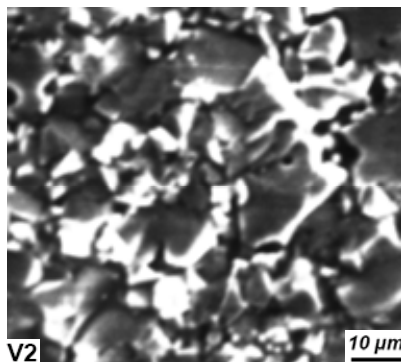


Fig. 2. Typical micrograph of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + 0.3 \text{ wt}\% V_2O_5$  (grain size=4.9  $\mu\text{m}$  with open pores).

Typical frequency dependence of complex permeability from 100 kHz to 10 MHz for the sample of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + 0.3 \text{ wt}\% V_2O_5$  is shown in Fig. 3. The real part of the permeability is stable up to 7 MHz beyond which it decreases steeply while accompanying a corresponding increase in imaginary part of the permeability to obtain peak around 10 MHz. Variation of relative permeability with vanadium additions at the frequency of 1 MHz is shown in Fig.4. The applied magnetic field was set as 0.1 Oe in making both the complex permeability and the relative permeability measurements. The vanadium additions have consistently resulted in a slight decrease in relative permeability up to 1.2 wt% concentration, and thereafter a marked decrease can be observed.

Table 1. Grain size, saturation magnetization and power loss of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + x V_2O_5$ .

Sample wt%	Grain size, $\mu\text{m}$	Ms, emu/g	Power loss, $\text{kW/m}^3$	
			at 500 kHz	at 1 MHz
0.0	4.9	78.5	30.62	89.44
0.3	4.9	78.0	35.09	97.93
0.6	4.9	78.3	35.08	106.24
0.9	5.2	77.9	38.98	119.17
1.2	5.9	77.1	37.29	113.83
1.5	7.8	75.5	42.13	130.1

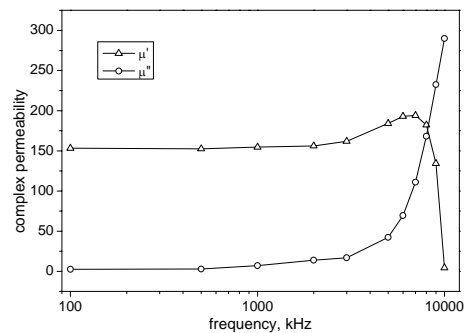


Fig. 3. Typical frequency response of complex permeability of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + 0.3 \text{ wt}\% V_2O_5$ .

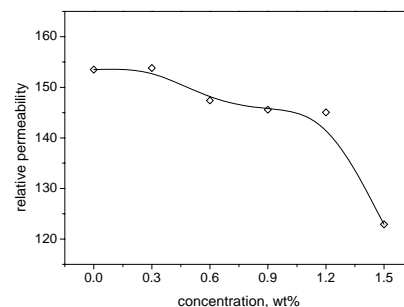


Fig. 4. Variation of relative permeability,  $\mu_a$  with additive concentration,  $x$  at 1 MHz.

Variations of power loss ( $P_{cv}$ ) with exciting flux density at 1 MHz is shown in Fig. 5. The power loss is small for all the samples at low flux densities, while it increases rapidly as the exciting flux density increases. Typical frequency dependence of power loss for a flux density of 10 mT for  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + 0.3 \text{ wt\% } V_2O_5$  is shown in Fig. 6. The power loss is very small up to 3 MHz, beyond which it starts to increase and after 5 MHz there observed a steep increase. In the inset of Fig. 6, variation of hysteresis and eddy current losses as a function of frequency for that sample is shown. The Hysteresis loss is predominant loss mechanism in the lower frequencies approximately below 200 kHz, and for frequencies above 200 kHz the eddy current loss increases gradually with increasing frequency and becomes predominant in the power loss. It should be noted that the power loss remains low at 1 MHz for the exciting condition of  $B_m=10 \text{ mT}$  as depicted in Fig. 6. The power loss has remained low for all the samples up to 3 MHz beyond which it has been observed to increase rapidly. The samples with either zero or small quantities of vanadium additions have low losses.

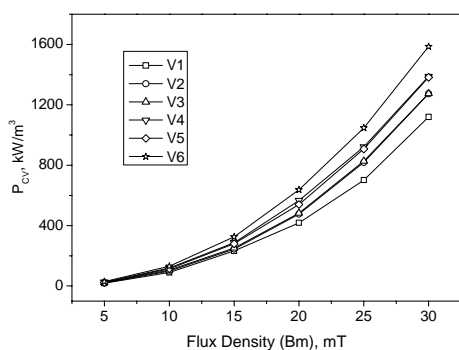


Fig. 5. Variation of power loss ( $P_{cv}$ ) with flux density of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + x V_2O_5$ .

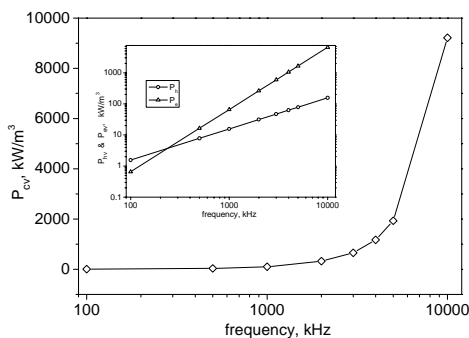


Fig. 6. Fig. 7. Frequency dependence of power loss ( $P_{cv}$ ) of  $Ni_{0.65}Zn_{0.35}Fe_2O_4 + 0.3 \text{ wt\% } V_2O_5$  Inset: Frequency dependence of  $P_h$  and  $P_e$ .

The frequency response of permeability shows a typical resonance character [9], which may be due to reversible displacement of domain walls and also due to rotation of magnetization dipoles inside the domains. Phase difference between the applied field and the magnetization occurs normally due to the damping of either spin motions or domain walls. The observed low values of resistivity are in support of the damping at high frequencies. If there is no damping, the imaginary part is zero for all frequencies except at resonance frequency. But, magnetic resonances appear as  $\mu'$  slightly increases to have small peak before decreasing to a low or even a negative value as the frequency approaches resonance limit and  $\mu''$  has a maximum and sharp peak near the resonance frequency. The  $\mu''$  values for all the samples in the present study are marked with maximum values good enough for resonance.

The relative permeability variations can be explained by the microstructural changes brought about by the addition of vanadium ions in these ferrites. Because of its low melting point, the vanadium oxide melts at grain boundaries and initially acts as grain growth inhibitor. Usually at low doping levels, the vanadium ions penetrate in the lattice inducing slow modifications of the microstructures marked by fine grains and grain boundary melts as secondary phases. These imperfections tend to pin the domain walls from bulging, thereby having bad influence on the magnetic performance of the ferrite. At higher concentrations, as the grain size increases, besides grain boundary phases, a large number of small closed pores are being trapped inside the grains. These pores and secondary phases have demagnetizing influences and lower the magnetization as well as permeability [10, 11]. The observed lower values of permeability and magnetization at higher concentrations of vanadium doped samples are in agreement with the above considerations.

Variation of power loss can be explained as follows: Since the basic composition in the present study is a high room temperature saturation magnetization ferrite among the entire Ni-Zn series, only additions of vanadium ions to this composition, without altering the magnetic cations directly, are not likely to bring out major changes in the density of magnetic ions and their environment. However, due to their incorporation particularly in small quantities, they are likely to enter the lattice and convert some  $Fe^{3+}$  ions in to  $Fe^{2+}$  ions to maintain the charge balance, and thereby indirectly alter the magnetic environment slightly. In the present system, the addition of vanadium ions slightly degraded the magnetization with increase in vanadium concentration. Also, despite the slight increase in grain size, the microstructures of these ferrites due to their secondary phase imperfections and porosities contributed very little to improve the initial permeability. As a result, the power loss has been observed to be slightly more for the vanadium containing samples compared to the undoped sample. However, optimum sintering schedule in combination with minor compositional modifications in these ferrites with very small quantities of vanadium ions would help to obtain better densities and higher resistivities [11], which could probably decrease

power loss considerably and push up resonances further by a few megahertz.

#### 4. Conclusions

Vanadium additions in small quantities in nickel-zinc ferrites seem to produce better low power losses. Because of its low melting temperature, the  $V_2O_5$  forms a thin grain boundary film, which impedes the grain growth and indirectly controls the grain size to the small size of the grains is an important parameter for power applications. More vanadium brings (perhaps due to the acceleration of the grain growth) gives rise to a number of small closed pores inside the grains in microstructures and affects the high frequency magnetic behaviour. Optimum sintering with small amount of vanadium in high magnetization basic ferrite composition would be interesting for further studies.

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