

INFLUENCE OF V₂O₅ ADDITION ON THE MAGNETIC AND ELECTRICAL PROPERTIES OF IRON-DEFICIENT NI-ZN FERRITE

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ABSTRACT

The influence of V₂O₅ addition on the magnetic and electrical properties of iron deficient ferrites with composition: Ni_{0.65}Zn_{0.35}Fe_{1.96}O_{4+x}V₂O₅ have been investigated, where values of x (in mol unit) are 0, 0.002, 0.004, 0.006, 0.008 and 0.01. The sintered density, saturation magnetization, Curie temperature of the samples increase up to V₂O₅ (x=0.004) and then decrease. The addition of V₂O₅ to the composition of ferrite increases the anion vacancy concentration, prohibit the discontinuous grain growth, which in turn enhances the density. The increment of saturation magnetization is due to the enhancement of resultant sublattice magnetic moment and density. Upto V₂O₅ (x=0.004), V₂O₅ goes into solid solution with the ferrite matrix and increases the Curie temperature. This is probably due to ions occupy some octahedral sites (B-sites) of the ferrite lattice by V⁵⁺, as a result some Fe³⁺ ions may come out from octahedral to tetrahedral sites (A-sites) thereby increasing A-B interaction and hence the Curie temperature. The dc resistivity of all the samples increases with the increment of V₂O₅ in the compound.

INTRODUCTION

Nickel-zinc ferrites are extensively used as core materials for low frequency electronic components such as inductors, radio antenna, television line scanning transformers, picture tube deflection yokes and coils in filter circuits. A small amount of an additive which forms a liquid phase at sintering temperatures can have a great influence on the magnetic and electrical properties of ferrites. Such an additive generally lowers the sintering temperature, i.e., promotes the densification at lower temperatures. Additive oxides of transition elements such as V₂O₅, or MoO₃, melt around 700°C and form liquid phases at commonly used sintering temperatures of Ni-Zn ferrites. Saturation magnetization, Curie temperature and initial magnetic permeability are the important magnetic properties of these core materials. Densification of Ni-Zn ferrites in presence of V₂O₅ has been studied earlier^(1,2). Magnetic and electrical properties of Ni-Zn ferrite⁽³⁾ and Li-Zn ferrite^(4,5) have been found to improve remarkably with the addition of V₂O₅. It is observed that in Li-Zn ferrites, a part of the added V₂O₅ goes into solid solution with the ferrite matrix. Incorporation of V⁵⁺ ions into the spinel ferrite introduces many complexities including creation of Fe²⁺ ions and precipitation of Fe₂O₃ as a second phase. The present work aims at investigating the influence of low melting V₂O₅ on the densification, magnetic and electrical properties of iron deficient Ni-Zn ferrites.

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EXPERIMENTAL

Ferrite pellet and toroid samples were prepared from Fe_3O_4 (a beach sand mineral) and commercial grade NiO, ZnO and V_2O_5 by solid state reaction method of the following iron deficient composition $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_{1.96}\text{O}_{4+x}\text{V}_2\text{O}_5$, where x (in mol unit) = 0, 0.002, 0.004, 0.006, 0.008 and 0.01.

Ingredient oxides were weighed and mixed homogeneously by wet ball milling with the help of a centrifugal ball mill, dried and pressed in a die to form big pellets for presintering. The pellets were presintered in air at 1000°C for two and half hours for ferritization. The presintered pellets were broken into pieces and wet milled for 8 hours to get a fine reactive powder for densification, mixing with required quantity of V_2O_5 addition in each composition. After drying the milled powder, it was granulated with the help of PVA solution and then compacted into small pellet and toroidal form at a pressure of 1-2 tons/cm². The pellets and toroids were sintered at 1250°C for two hours in normal atmosphere to get final densification. The rate of heating was around $200^\circ\text{C}/\text{hr}$ and the furnace was allowed to cool down slowly to room temperature to avoid any micro-crack in the samples.

The surfaces of the sintered samples were polished and their densities (d_s) were measured. Single phase spinel structure of the sintered samples were confirmed by means of X-ray diffraction using $\text{CuK}\alpha$ radiation (PHILIPS X' Pert PRO x-ray Diffractometer). The lattice parameter (**a**) of the samples were calculated from X-ray analysis data. Saturation Magnetization (M_s) was measured in a vibrating sample magnetometer (Model No VSM 02, Hirstlab, England) and the Curie temperature (T_c) of the samples were determined from permeability versus temperature curves at 100 kHz with an accuracy of $\pm 1^\circ\text{C}$. Magnetic initial permeability (μ') and Q- factor were measured using an impedance analyzer (HP 41921A). The dc resistivity (ρ) of the samples were measured with the help of an electrometer (6514 System Electrometer) using two point Silver paste contact. Photo micrograph of the polished and etched surface of the samples were taken by an optical microscope of magnification 400.

RESULTS AND DISCUSSION

Fig. 1 shows the variation of sintered density (d_s) and lattice parameter (**a**) for samples with increasing concentration of V_2O_5 addition. A significant increase of the density is observed upto V_2O_5 addition, $x=0.004$ and then decreases with increasing amount of V_2O_5 . The inclusion of V_2O_5 to the composition of iron deficient Ni-Zn ferrites increases the anion vacancy concentration, prohibit discontinuous grain growth which in turn enhances the density. The decrease in density beyond V_2O_5 , $x=0.004$ content is due to the segregation of second phase rich in vanadium⁽³⁾. The lattice parameter remains almost unaffected by the addition of V_2O_5 as shown in Fig. 1. V^{5+} ions have a smaller ionic radius (0.59 Å) than Fe^{3+} ions (0.64 Å), its incorporation into lattice results in the creation of some Fe^{2+} ions which have an ionic radius of 0.74 Å. This

presumably results in no change in the lattice parameter when V_2O_5 goes into solid solution in the ferrite.

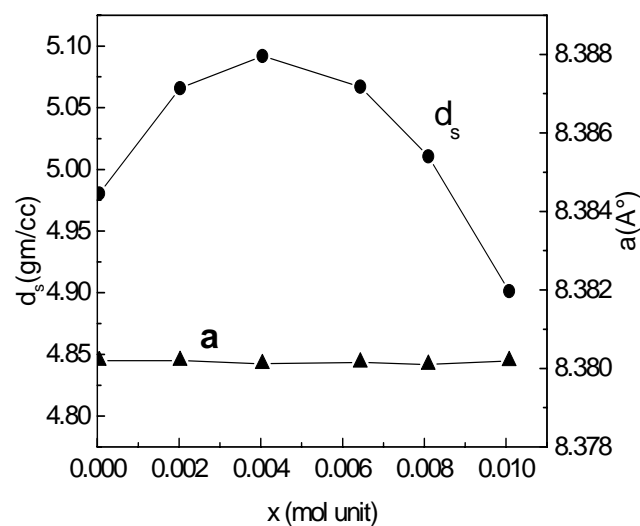


Fig.1 Variation of d_s and a on V_2O_5 addition

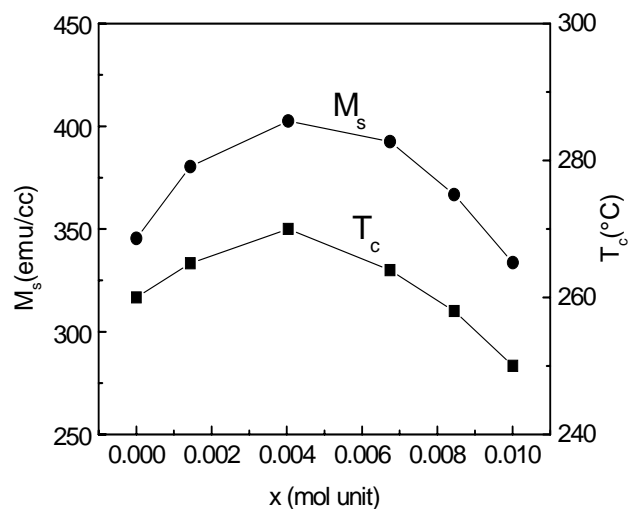


Fig.2 Variation of M_s and T_c on V_2O_5 addition

The variation of saturation magnetization (M_s) and Curie temperature (T_c) is shown in Fig. 2. An increment in the saturation magnetization up to V_2O_5 , $x=0.004$ is due to the increase of resultant sublattice magnetic moment as well as density. It is observed from this figure that up to $x=0.004$, V_2O_5 goes into the solid solution with ferrite matrix and increases the Curie temperature. This is probably due to some of V^{5+} ions occupy octahedral (B-site) sites of the ferrite lattice, as a result some of Fe^{3+} ions would be pushed off from B to A (tetrahedral) sites thereby increasing A-B interaction and hence

the Curie temperature. As the amount of V_2O_5 is increased the V^{5+} ions occupy the electro-statically stable A sites, weakening the A-B interaction and the Curie temperature decreases⁽³⁾.

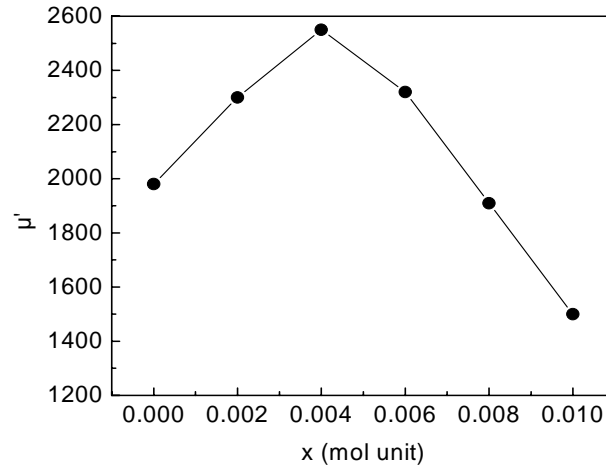


Fig.3 Variation of μ' at 100 kHz on V_2O_5 addition

The variation of initial magnetic permeability (μ') at 100 kHz of the ferrite system with increasing concentrations of V_2O_5 is shown in Fig. 3. An appreciable increase in the permeability up to V_2O_5 , $x=0.004$ can be observed from the figure and then decreases. Such an increase in permeability can not be attributed to densification alone. This increment is thus the reflection of the effect of V_2O_5 content on the microstructure of the material as shown in Fig. 4.

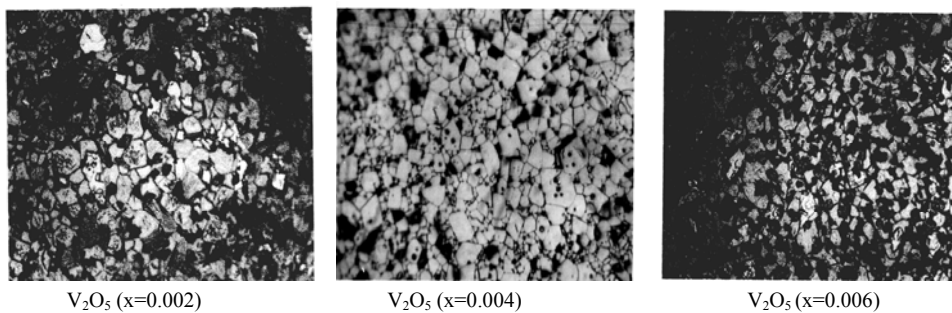


Fig. 4 Microstructure of Ni-Zn ferrite samples with V_2O_5 addition

For this composition of ferrite, as V_2O_5 content is increased, the grain size first increases presumably due to the creation of oxygen vacancies and the consequent increase in the rate of diffusion of oxygen ions, and then decreases as V_2O_5 forms a second phase. The second phase which appears at higher V_2O_5 content as shown in Fig 4 forms a nonmagnetic discontinuity around the ferrite grains and contribute towards the decrease in permeability.

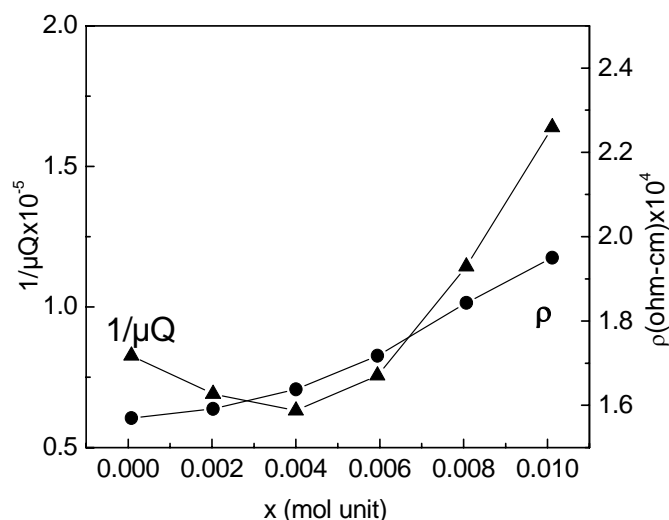


Fig.5. Variation of $1/\mu Q$ and ρ on V₂O₅ addition

The variation of normalized magnetic loss factor ($1/\mu Q$) and dc resistivity (ρ) is shown in Fig.5. The loss factor first decreases, reaches a minimum and then increases with increasing addition of V₂O₅. It suggests that the increment of loss factor at higher concentration of V₂O₅ is mainly due to the drastic decrease in permeability at these concentrations of V₂O₅. The dc resistivity of the samples increases almost linearly with the increasing concentration of V₂O₅. With the addition of V₂O₅, V⁵⁺ ions occupy B-sites as mentioned earlier which limit the degree of Fe²⁺ to Fe³⁺ electron hopping conduction resulting in increase in resistivity.

CONCLUSION

A small addition of V₂O₅ (up to 0.4 mol%) increases the sintered density of iron deficient Ni-Zn ferrites, while higher concentrations of V₂O₅ decrease the density. A small addition of V₂O₅ increases saturation magnetization, Curie temperature, initial magnetic permeability and decreases the magnetic loss factor. So, it can be concluded that the addition of 0.4 mol % V₂O₅ in iron deficient Ni-Zn ferrite will be helpful to obtain good quality core materials to use in low frequency electronic components.

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