

Effect of iron content on permeability and power loss characteristics of $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$

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Abstract. Substituted lithium ferrites having the chemical formula, $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$, with different iron (metal) contents (2, 4, 6, 8 and 10) in wt% have been prepared by solid-state technique. Complex permeability and power loss of all samples have been measured by network analyser in the frequency range of 50–5000 kHz. Magnetic properties like saturation magnetization, coercivity, retentivity have been measured by vibrating sample magnetometer (VSM). The permeability of cadmium doped lithium ferrites exhibited higher values than zinc doped lithium ferrites. The power loss of cadmium doped lithium ferrites is lesser as compared to zinc doped lithium ferrites in the frequency range of 50–5000 kHz and at flux density of 10 mT. The behaviour of power loss with flux density has been found near about same for both series. Magnetic and power loss behaviour of the samples suggest that a small amount of Fe content can improve the properties of ferrite samples for microwave devices.

Keywords. Solid state technique; complex permeability; power loss; flux density.

1. Introduction

Lithium based ferrites have been used for cathode materials in lithium ion batteries (Dlpeeiotor and Dethackeray 1986), microwave applications (Nutan *et al* 2005) and memory core (Venudhar *et al* 2002), because of their high Curie temperature, high magnetization, excellent squareness of $M-H$ curve and good magnetic properties (Cho *et al* 1999; Yue *et al* 1999). In response to the current demand for size reduction of electronic devices, the development of compact and efficient switched-mode power supplies has received considerable attention. One way to achieve this is to use high switching frequencies which require low power loss cores to afford the transformation of high power (Verma *et al* 2006). The main types of losses encountered in ferrites are the eddy current loss, hysteresis loss and residual loss. Consequently the requirements of a power ferrite are high resistivity to keep the eddy current low, high permeability to reduce the residual losses, which consist mainly of resonance–relaxation losses. Additive materials can affect the magnetic properties, electrical properties and microstructure of ferrites by different mechanisms (Shokrollahi and Janghorban 2007; Verma *et al* 2008). Some additives such as V_2O_5 , Bi_2O_3 , etc act as grain growth accelerator by different mechanism like increasing the pore mobility due to the creation of excess cation vacancies. Other types of additives such as SiO_2 , CaO , etc can create an electrical insulating film

around the grain and increase the resistivity of materials (Verma *et al* 2007). In the completely inverse ferrites such as NiFe_2O_4 , large moments of the two Fe^{3+} sublattices cancel each other and no advantage is taken for the potential Fe^{3+} moment. In other ferrites like lithium ferrites in which Fe^{3+} on the two sublattices are disproportionate the large moment is used. Of course, these effects are not chemical (i.e. they are not related to Fe_2O_3 content) but crystallographic (i.e. they are related to lattice site distribution). Actually, with great variety of possible chemistries of spinel ferrites, Fe_2O_3 content of the finishing ferrite is varied least of all of the metal ions since it is pegged at 50 mole percent by the spinel formula ($\text{MO}\cdot\text{Fe}_2\text{O}_3$). In most commercially important MnZn ferrite materials, the starting mix may contain slightly > 50 mole percent Fe_2O_3 . The purpose of the extra iron is to improve the magnetic properties by the formation of Fe^{2+} ions.

Two compositions of substituted lithium ferrite is selected for our work because of their magnetic and dielectric properties (Bellad *et al* 2000; Yusoff and Abdullah 2003). This paper brings out the effect on magnetic properties and power loss of doping of iron metal by 2, 4, 6, 8 and 10 wt% in $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$.

2. Experimental

Cadmium and zinc doped lithium ferrites, $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ (LiCd ferrite) and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$ (LiZn ferrite) were prepared by conventional solid-state sintering technique from high purity oxides and carbonates of

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Li_2CO_3 , CdO , ZnO and Fe_2O_3 . According to the stoichiometric compositions, $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$, specified molar ratio of the iron oxide, lithium carbonate, cadmium oxide, zinc oxide were milled by wet grinding in power ball mill (RETCH, pm 400). Ball milled powder was pre-sintered at 750°C for 10 h. The pre-sintered powders were mixed with iron metal powder in 2, 4, 6, 8 and 10 wt% to form two different batches of LiCd and LiZn ferrites followed by final sintering at 1050°C for 5 h. Heating and cooling rates were controlled at $5^\circ\text{C}/\text{min}$. The structural characterization of samples was carried out by X-ray diffraction (XRD Rigaku Miniflex II, step size = 0.02°) technique using $\text{CuK}\alpha$ radiation (wavelength, $\lambda = 1.5406 \text{ \AA}$). A scanning electron microscope (SEM LEO 440) was used to observe microstructure details of the samples. Permeability values were measured by LCR meter (Agilent 4284 and 4285). Power loss values were measured by a B-H analyser (IWATSU, SY 8232). For permeability measurements, toroids were used having outer and inner radius 6 and 3 mm, respectively. Magnetic measurements were performed for all samples at room temperature by plotting $M-H$ curves using vibrating sample magnetometer (VSM, Lake Shore 7304).

3. Results and discussion

Figures 1(a) and (b) show X-ray diffraction of iron metal doped LiCd and LiZn ferrites. The powder X-ray diffraction patterns exhibited that all the samples were identified as a single phase of cubic spinel structure.

Figure 2 shows scanning electron micrographs of lithium cadmium and lithium zinc ferrites with variation of iron content by wt%. We observed some improvement in grain size with the variation of iron content in both soft ferrites. The average grain size of pure $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{3.35}\text{O}_4$ improved

from $4 \mu\text{m}$ with iron doping of 4 wt%. But in the case of $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$, there are two kinds of grain formation in pure sample as shown in figure 2(d). The average grain size of bigger and smaller grains of about 14 and $2 \mu\text{m}$ are observed. On putting extra iron of 4 wt% in LiZn ferrite the grain size of smaller grains increases to $3 \mu\text{m}$ and these decrease to $< 2 \mu\text{m}$ in case of 8 wt% sample.

Initial permeability response curves for LiCd and LiZn ferrites with different variation of iron in the frequency range 10^5 – 10^8 Hz are shown in figure 3. The frequency response of permeability shows a typical relaxation character, which may be due to reversible displacement of domain walls and also due to rotation of magnetization dipoles inside the domains. It is observed that the value of initial permeability (97) of pure lithium ferrite cadmium ferrite increases with iron concentration and attains a maximum value (137) for 4 wt% iron doped sample and starts to decrease with further increase in iron content. A similar behaviour is also found in case of lithium zinc ferrite as shown in figure 3(b). The permeability response for different compositions can be defined by the following relation (Huang *et al* 1994):

$$\mu_i \propto \mu_0 M_s^2 D_m / [K_1 + (3/2)\lambda_s \sigma] \beta^{1/3} \delta,$$

where μ_i is the initial permeability, M_s and D_m are the saturation magnetization and average grain size, respectively. K_1 is the magneto-crystalline anisotropy, λ_s the saturation magnetostriction constant, σ the inner stress, β the volume concentration of impurity and δ the thickness of the domain wall.

The relationships between exciting flux densities and power loss P_{cv} at 50 kHz of both series are shown in figure 4. Power loss values increase obviously with exciting flux density for both samples. It is well known that Pa can be divided into hysteresis loss (Ph), eddy current loss (Pe) and residual loss (Pr). At high frequencies hysteresis

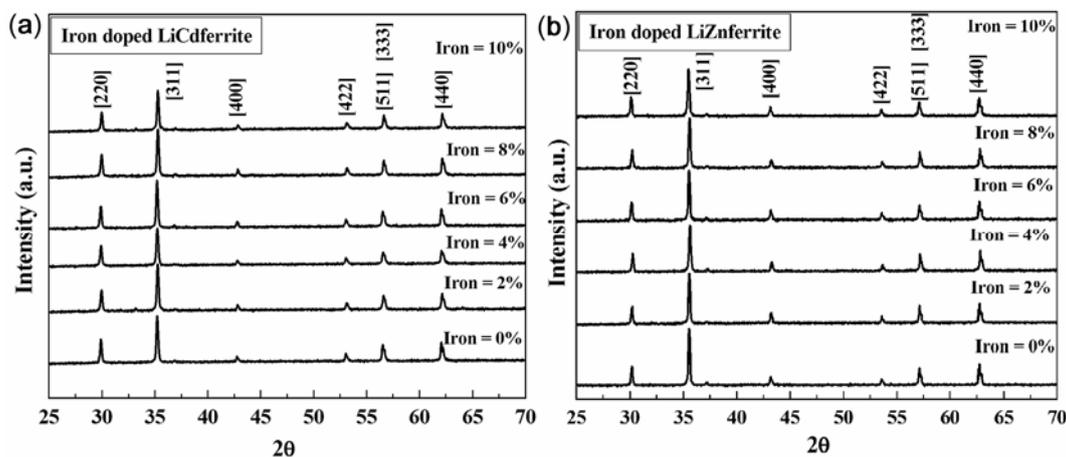


Figure 1. (a) X-ray diffraction pattern of iron doped lithium cadmium ferrite and (b) X-ray diffraction pattern of iron doped lithium zinc ferrite.

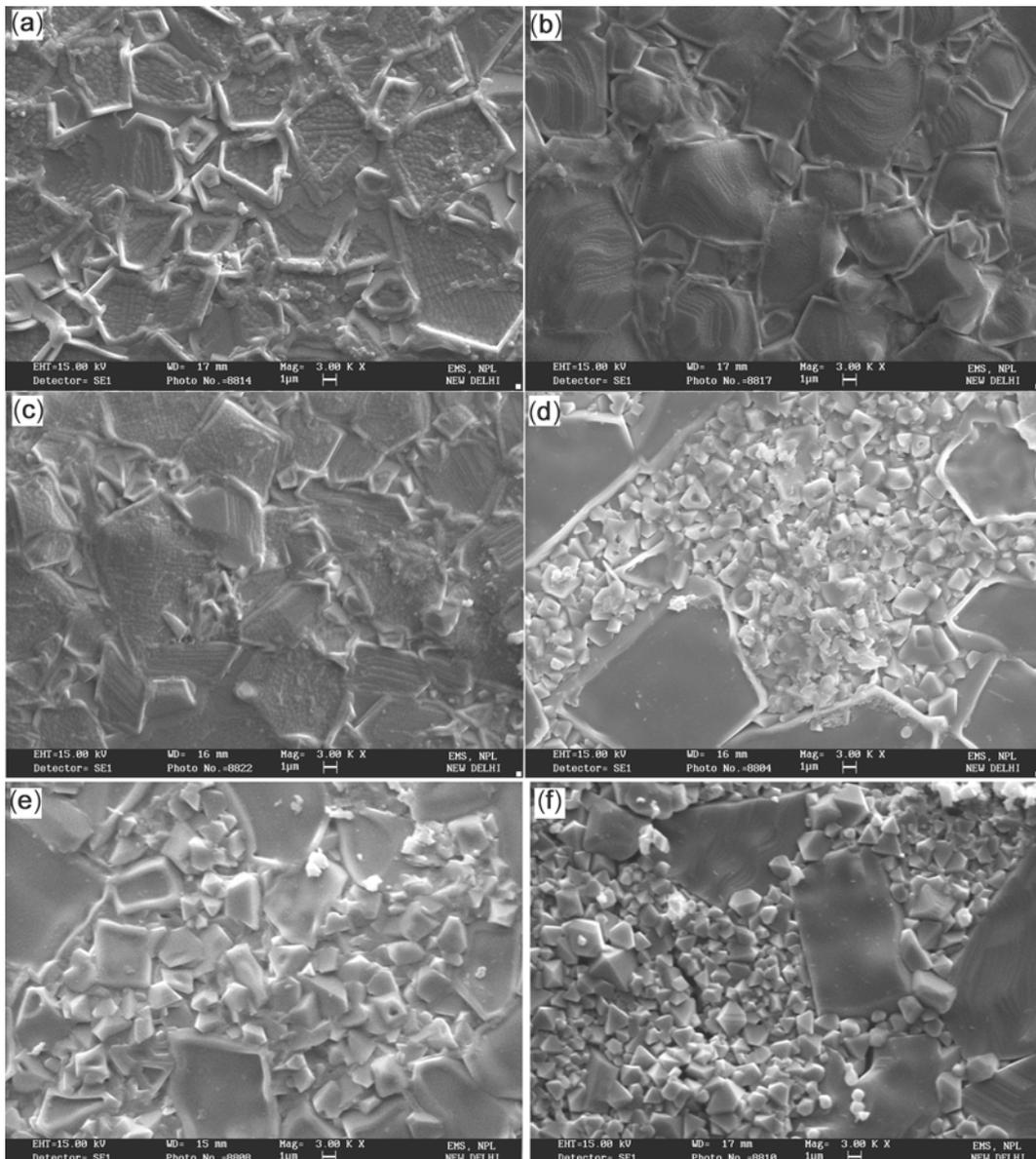


Figure 2. SEM micrographs of iron doped lithium cadmium ferrite (a) pure, (b) 4 wt%, (c) 8 wt% and lithium zinc ferrite (d) pure, (e) 4 wt% and (f) 8 wt%.

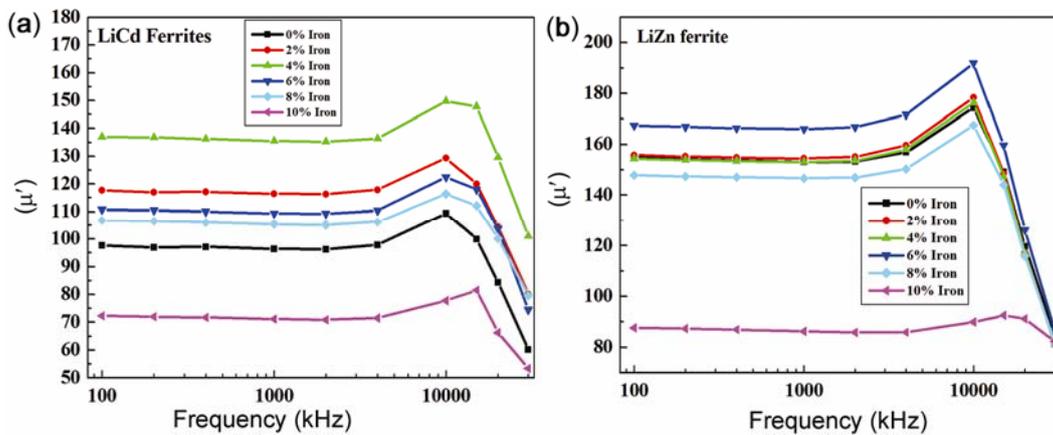


Figure 3. Permeability vs frequency of (a) $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and (b) $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$ with different contents of iron.

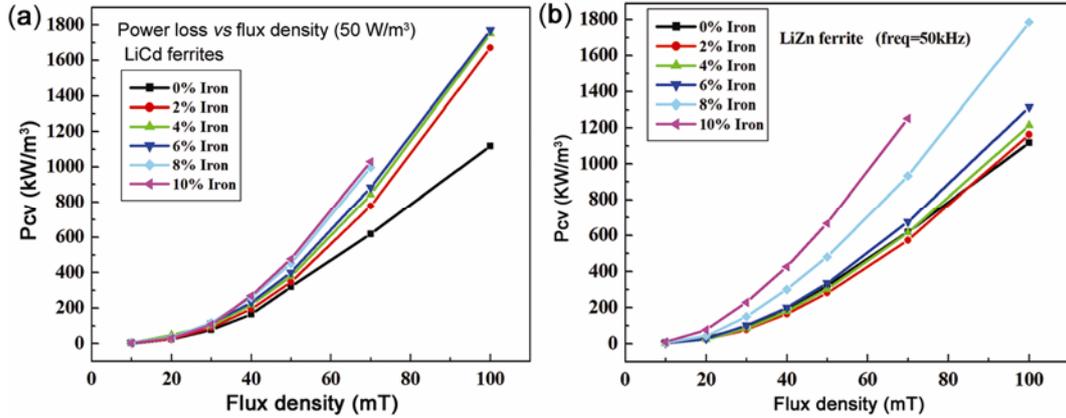


Figure 4. Power loss vs flux density at 50 kHz of (a) $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and (b) $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$ with different contents of iron.

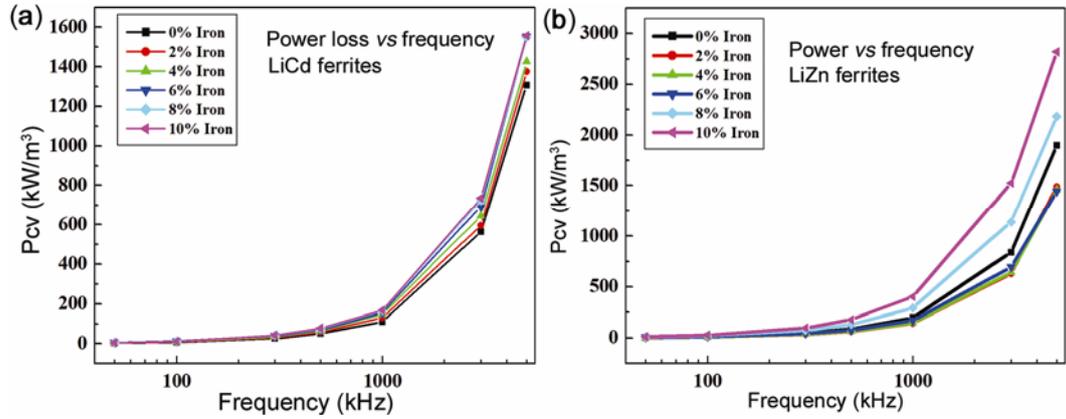


Figure 5. Power loss vs frequency at 10 mT of (a) $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and (b) $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$ with different contents of iron.

loss is prominent in ferrites. Hysteresis loss can be expressed by (Hendricks *et al* 1991)

$$P_h = f\phi HdB,$$

where f is the frequency, H the magnetic field strength and B the magnetic induction. Magnetocrystalline anisotropy, magnetostriction, volume fraction of inclusions (pores, impurities and defects) and saturation magnetization are some properties of materials which influence the power loss of materials.

Low-hysteresis loss ferrites should have a high density, with the remaining porosity being located at the grain boundaries so as not to impede domain wall movement. Large and thin grain boundaries are advantageous for reduced interference with domain wall movement. Eddy current loss can be expressed as (Hendricks *et al* 1991):

$$P_e = CB^2f^2d^2/\rho,$$

where C is the proportionality constant, B the flux density, f the frequency, ρ the resistivity and d the thickness of the material. Eddy current loss can be reduced by increasing the resistivity of the polycrystalline ferrite by increasing grain boundary resistivity.

Frequency dependence of power loss for both series in frequency range 100 kHz–5 MHz at the exciting condition of $B_m = 10$ mT are shown in figures 5(a) and (b). The power loss has remained low for all the samples up to 1 MHz beyond which it has been observed to increase rapidly.

4. Conclusions

We find that some extra amount of iron in $\text{Li}_{0.35}\text{Cd}_{0.3}\text{Fe}_{2.35}\text{O}_4$ and $\text{Li}_{0.35}\text{Zn}_{0.3}\text{Fe}_{2.35}\text{O}_4$ seems to have produced better permeability and microstructure. These ferrites have low power loss up to 1 MHz frequency and therefore these ferrites are suitable for power applications in this frequency range.

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