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Comparative Studies of NiMgCuZn composite ferrites with Equimolar (NiCuZn+MgCuZn) Nanocomposite ferrite useful for Microinductors Applications

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Abstract

In this paper, we report a comparative study on structural, magnetic, electric properties and stress sensitivity of the $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ with equimolar nano composite ferrite $0.5((Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4)+(Mg_{0.25}Cu_{0.25}Zn_{0.5}\ Fe_2O_4))$ prepared by conventional double sintering technique, with a view to develop a ferrite composition for its use as core material for microinductor applications. The structural properties were estimated from X-ray diffraction patterns which confirms the formation of single phase cubic spinel structure and the grain size was estimated using SEM micrographs. Initial permeability measurements of samples were carried out in the temperature range of 30-400 ^{0}C at 10^0C temperature intervals, it was noticed that the initial permeability of $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ sample is drastically reducing when compared to equimolar composite ferrite. The effect of the external applied stress on the open magnetic circuit type coil with these ferrites was studied by applying uniaxial compressive stress parallel to magnetizing direction and the change in the inductance was measured. The variation of ratio of inductance ($\Delta L/L$)% with external applied compressive stress was examined.

Keywords: Microinductor applications, Magnetic properties, X-Ray diffraction, SEM Patterns, Ferrites, Stress Sensitivity..

Introduction

A considerable amount of research in recent years has focused on NiCuZn and MgCuZn ferrites, because of their excellent properties which possesses innumerable electronic applications, communication industries also having much importance in microindcutors, both have similar electromagnetic properties which are suitable for core materials in microinductors, with the advantage that they are economic [1] and easy to synthesize. Especially they are potential candidate materials for multilayer chip inductors with high performance and low cost. Microminiaturization of electronic circuits especially in the fields of mobile communication and information technology demands electronic components with very small size [2]. Multilayer chip inductor (MLCI) as key component of electronic devices confronts new challenges [3]. Better magnetic properties, especially required high initial permeability for reducing the number of layers of multilayer chip inductors (MLCI) minimizing the capacity between the layers and realizing the miniaturisation [4]. So far, NiCuZn and MgCuZn ferrites have been used extensively for the production of the MLCI [5] due to their better magnetic properties and low temperature sintering [6-7]. However, they suffer from stress sensitivity [8-10]. It is believed that increase of initial permeability can be obtained by decreasing magnetostriction constant [4,11]. As the magnetostriction constant of MgCuZn ferrite is lower than that of NiCuZn ferrite [5], it is also expected that the multilayer chip inductor (MLCIs) using MgCuZn ferrites [4] would show higher magnetic properties [9-10,12] making further miniaturization possible than that of NiCuZn ferrites, compared to that of NiCuZn ferrites one can realize at low cost MLCIs with MgCuZn ferrites. The growing interest during the past decade is essentially due to the possibility of a reduction in manufacturing cost on account of energy savings.

We aimed to produce low-cost ferrite materials according to the desired application with easy and synthesis technique. In search of the suitable ferrite materials for microinductor applications, assuming that the ferrite composites would yield enhanced magnetic properties. Initially, composite ferrites were prepared in this laboratory by choosing NiCuZn and MgCuZn ferrite compositions. As reported earlier these composites [10] showed high initial permeability, fairly high resistivity and stress insensitivity, As the part of continuation, the two compounds can be successfully incorporated into a single composite, it is expected that the composites might have interesting electrical as well

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as magnetic properties, it's very interesting to study the electromagnetic properties of composition dependence by considering the stoichiometric formula of each composition ($Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4$ and $Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4$) and the resulting sample was ($Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$) . So, In view of this, an attempt has been made to develop ferrite material suitable for MLCI and studies on magnetic, electrical and stress sensitivity has been investigated and the results are discussed and compared with the equimolar composite.

Experimental

In previous research we studied the magnetic, electrical and stress sensitivity properties of composite ferrite (X) $Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4$ + (1-X) $Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4$ in which X varies from 0.0 to 1.0 [10] which predicts that both the end compositions i.e., pure ferrite's have almost equal initial permeability values also undergoes stress insensitivity and the equimolar composite ferrites i.e., (0.5) [($Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4$) + ($Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4$)] exhibits high initial permeability value of μ_i = 9619. So, keeping in point of view, we thought of investigating the properties of $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}$ Fe_2O_4 (generic formula has taken from the end compositions ferrites i.e., $Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4$ and $Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4$) in the present study an attempt is made on these ferrites composites, to investigate their electrical properties as well as thermoelectric effect studies were carried out with an intention of developing a ferrite composition suitable for multilayer chip inductors and the results are reported in this paper.

Preparation of ferrites:

The composite ferrite was prepared by choosing the stiochiometric of each composition i.e., ($Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4$ and $Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4$) and the resulting sample with the generic formula $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ were prepared employing double conventional sintering method using analytical grade NiO, MgO, CuO, ZnO and Fe_2O_3. These oxides were weighed and intimately mixed in stoichiometric proportions. These constituents were then milled in a planetary ball mill (RETSCH PM-200) in acetone medium using polyethylene jars with iron balls (φ = 5mm) for 24 hrs and then dried in an oven. The ground powders were presintered at 800 ^{o}C for 4hrs in the form of cakes in alumina crucibles. After presintering, these cakes were crushed and ball milled once again for 8 hrs to obtain fine particle size and finally, these powders were sieved to get uniform particle size.

The presintered green powders were mixed with 2% polyvinyl alcohol as a binder and were compacted in the form of torroids of 12 x 10^{-3} m outer diameter (O.D) ., 8 x 10^{-3} m inner diameter (I.D) and 4 x 10^{-3} m height (h), also in the form of pellets of 10 x 10^{-3} m diameter and 2 x 10^{-3} m thickness and cylinders of diameter 10 x 10^{-3} m and length nearly 20 x 10^{-3} m long using an isostatic press at 150 MPa. These compacted bodies were finally sintered at 1050 ˚C for 2 hrs in a programmable furnace and were cooled to room temperature at the rate of 80 ^{o}C /hr. Sufficient care was taken to avoid the zinc loss during the sintering process by placing these compacted pellets, cylinders and torroids in a closed alumina crucible with excess of ZnO rich atmospheric powder. All the samples were structurally characterized using Philips high resolution x-ray diffraction system (Model PW-1710 X-ray diffractometer) with monochromatic CuK_α radiation at room temperature.

Microstructures of sintered samples were recorded with the help of using SEM (Philips XL30ESEM) Scanning electron microscope instrument Thermally etched samples were taken for making the SEM specimens. The samples were mounted on brass studs with double sided adhesive tape and coated with Au-Pd alloy of 120-150 Å thickness of argon ambient atmosphere of 8-10 Pascal. Then the sample is scanned for various surfaces and the best photographs were recorded.

Experimental details:

Initial permeability

The initial permeability, μ_i of these ferrite torroids were evaluated using the standard formulae from the inductance measurements carried out at 1 KHz using computer controlled impedance analyzer (Hioki Model 3532-50 LCR HiTester, Japan). These measurements were carried out in the temperature range of 30 to 400 ˚C using the formula [9].

$$L = 0.0046N^2 * \mu_i * h * \log_{10}\left(\frac{D_1}{D_2}\right) \quad \ldots. (1)$$

Where D_1 and D_2 are the inner and outer diameter of the torroid in inches, L is the inductance in microhenrys, h is the height of the torroid in inches and □i is the initial permeability of the core and N is the number of turns.

Stress sensitivity studies

In order to study the effect of external stress, uniaxial compressive stress parallel to the magnetizing direction was applied to the cylindrical shaped ferrite cores using uniaxial press system. The stress magnitudes were varied from 0 to 10 MPa. The change in inductance was measured using the above mentioned LCR HiTester

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by employing 100-120 turns coil wound on each cylinder.

DC electrical conductivity

DC electrical conductivity measurements were carried out with laboratory designed cell having guarded ring facility in addition to the two probe method [13].

It consists of an electrically heated furnace, a digital DC micro voltmeter and a transistorized power supply unit as a source of DC voltage. Silver paste was applied to both the surfaces of the pellet to obtain good ohmic contacts. The pellet was mounted in a sample holder consisting of two silver rods in which the sample can be sandwiched with the help of screws. The cell was placed in a tube furnace. A calibrated cromel-alumel thermocouple was used to measure the temperature of the tube furnace. The measurements of conductivity were carried out by measuring the current at a constant voltage of 5 volts from a regulated power supply, room temperature resistivity was determined by the relation.

$$\rho = \left(\frac{\pi r^2}{t}\right)\left(\frac{V}{I}\right) \qquad \text{..... (2)}$$

and the electrical conductivity (σ) of MgCuZn ferrites under investigation has

been computed using the formula.

$$\rho = \left(\frac{It}{VA}\right) \qquad \text{..... (3)}$$

Where, t is the thickness of the sample in cm , r is the radius of the disc in cm, I is the current passing through the specimen in amperes, A denotes the area of a cross section of the sample in cm^2 and V is the voltage applied of the specimen in volts.

AC electrical conductivity

The AC electrical conductivity (σ_{AC}) of these ferrites was evaluated using the following standard relation.

$$\sigma_{ac}(\omega) = \frac{Z''}{(Z'^2 + Z''^2)} X \frac{t}{A} \qquad \text{..... (4)}$$

Where"t" is the thickness of the pellet and A is the area of a cross-section of pellet.

Using Eq. (4), the AC conductivity, σ_{AC} of these ferrites was evaluated from the real (Z') and imaginary parts (Z'') from directly obtained impedance values at 1 kHz employing a computer controlled low frequency impedance analyzer (Hioki 3532-50 LCR Hi-Tester) in the temperature range 30 to 490 ºC. The measurements of Z' and Z'' were also made at different frequencies in the range 100 Hz to 1 MHz at room temperature and σ_{AC} was evaluated.

Thermo-electric power

Thermo-electric power measurements were made in the temperature region 30 to 360ºC by the differential method similar to that of Reddy et.al., [14] with a few modifications. It contains a main furnace assembly, DC micro- voltmeter, sample holder, and digital micro-voltmeter. The sample holder consists of a small independent heater in intimate contact with the top electrode that facilitates the setting of the temperature of the upper electrode at any desired value. The mechanical assembly of thermo-electric power set-up has the provision to mount the pellet between the top and bottom electrodes with a spring loading arrangement. Before loading the sample into the sample holder, the surfaces of pellet were coated with silver paste for good ohmic contact. The entire mechanical assembly was placed inside a tube furnace for the measurement of thermo-electric power. The details of the furnace and the temperature controller assembly were described elsewhere [15]. Employing the arrangement a temperature difference of 10 K was produced across the pellet with the help of a micro furnace fitted to the sample holder assembly. The sample was then heated, and thermo-emf was recorded at different temperatures. In the measurement of the thermo-emf, the micro-voltmeter gives a positive deflection by connecting its positive terminal to the hot end of the sample. This happens in case of an N-type charge carrier. This situation reverses when the charge carriers are of P-type.

Thermo-electric power was determined by using the relation.

$$\alpha = \left(\frac{\Delta V}{\Delta T}\right) \qquad \text{…. (5)}$$

Following convention adopted by Lal et.al., [16].

The sample was maintained at a given temperature for about 15 minutes for attaining thermal equilibrium. The thermo-emf measurements were made after the attainment of thermal equilibrium. The temperature was measured using calibrated chromel-alumel thermocouple.

Results and Discussion

Structural and Microstructures of the NiMgCuZn ferrite:

Fig.1 (a), shows the typical XRD pattern of $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$

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sintered at 1050 ºC. The formation of all the peaks i.e., (220), (311) (222) (400) (422) (511) and (440) is the evidence that the sintered samples contain only spinel cubic structure, no second phase was detected. Fig.1 (b) shows equimolar composite sintered at 1250 ºC confirms the formation of single phase cubic structure, which also shows that the (222),(400) (422) (511) and (440) peaks which belong to octahedral and tetrahedral sites are maximum suppressed in the equimolar composition of ferrite composite i.e $0.5((Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4)+ (Mg_{0.25}Cu_{0.25}Zn_{0.5}\ Fe_2O_4))$ when compared to $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$, also it can be noticed that the intensity of (311) peak is maximum in equimolar composition when compared.

The interplanar distance d (Å) were calculated using Bragg's law. The lattice parameter "a" was calculated using the relation [17].

$$\left(\frac{1}{d^2}\right)=\left(\frac{h^2+k^2+l^2}{a^2}\right) \qquad \text{..... (6)}$$

Where "a" is the lattice constant, (hkl) are the Miller indices and "d" is the interplanar distance. The lattice parameters have been determined using the method of least squares to an accuracy of ± 0.007Å.

The calculated lattice parameter identified the samples to be cubic spinel. The crystalline sizes for each composition are calculated from XRD line width of the (311) peak using Scherrer formula [18]. The values of the particle size, lattice constant (a), measured density (ρ_m) and the X-ray density (ρ_X) as reduced from the X-ray data are given in Table.2. The measured density (ρ_m) is determined using the formula.

$$\left(\rho_m=\frac{m}{r^2h}\right) \qquad \text{..... (7)}$$

Where " m" is the mass, "r" the radius and "h" is the height of the samples.

The X-ray density of the prepared samples was calculated from the relation.

$$\left(\rho_x=\frac{8M}{Na^3}\right) \qquad \text{..... (8)}$$

where M is the molecular weight of the samples, N is the Avogadro's number and "ä" is the lattice constant.

It can be noticed from Table.2 which shows the variation of the lattice parameters of the both the end products, equimolar composite and $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ the lattice parameter exhibit a decrease in $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ when compared to equimolar composition, it can be explained on the basis of the relative ionic radius of ions [19-20]. The ionic radius of Mg^{2+}= 0.65 Å, which has smaller radius when compared to Ni^{2+} = 0.72 Å, Cu^{2+} = 0.72 Å, Zn^{2+}= 0.74 Å and Fe^{2+}= 0.64 Å which are larger in radius may cause a decrease of lattice constant may be logically attributed due to the difference in the ionic radius, which results in the reduction of the unit cell.

The variations of X-ray density and measured density are given in Table.2. It can also be observed from Table.2 the mass density is slightly higher than the X-ray density of the sintered samples, This may be due to the existence of pores formed during the sintering process, also one can notice that mass density (ρ_m) of $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ is greater than the equimolar composite.,

Percentage porosity (P) of the samples was calculated from ρ_X and ρ_m

values using the expression [21].

$$P=\left(1-\frac{\rho_m}{\rho_x}\right) \qquad \text{..... (9)}$$

Grain size is the more important parameter affecting the magnetic properties of ferrites. It is well known that ferrites with high density and larger average grain size posses high initial permeability.

(b) *Scanning electron micrographic (SEM) studies*

The scanning electron micrograph for equimolar composite and $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ are shown in Fig.2, which shows a systematic grain structure having clear grain boundaries indicating a microstructure, having high density and having large grain size. But it is interesting to note that the morphology of grains is different in both the compositions. However, the initial permeability's values obtained are fairly high. This may be attributed to the initial permeability being a result of the easy reversal of domain wall displacement in the direction of the applied magnetic field, the greater the number of domain walls, the higher the initial permeability.

Magnetic properties:

Initial permeability is known to be one of the most sensitive magnetic properties of ferrites, and also it is a very sensitive parameter that depends on various factors like external stress, grain size, temperature, and method of preparation, etc., an

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important requirement for MLCI fabrication is the low sintering temperature of the ferrite materials. For MLCI applications, the temperature dependence of initial permeability is important. The initial permeability (μ_i) of low-temperature sintered $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ along with equimolar composite and its ends products at room temperature are shown in Table.3.

The variation of initial permeability (μ_i) with temperature $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ is shown in Fig.3, the initial permeability increases with an increase in temperature; these phenomena were considered to be due to a faster decrease in the anisotropy field rather than a decrease in a saturation moment [22]. It has also been reported that initial permeability increases with an increase in temperature by showing a sharp fall in (μ_i), the value just before reaching the Curie temperature [23]. The variation of initial permeability with temperature shows the presence of two slopes before reaching the Curie temperature (T_C). The presence of two slopes can be explained on the assuming contribution of μ_{rk} and μ_w to μ_i i.e., (1) resulting from spin rotation (μ_{rot}) (2) resulting from domain wall motion/displacement (μ_w). The domain walls normally remain pinned to the grain boundary when subjected to a small magnetic field. The initial permeability is mainly due to reversible motion of the domain walls Globus [24] assumed that the permeability due to the wall motion is likely to be dependent on the grain size, while the permeability contribution due to spin rotation was assumed to be independent of grain size.

Now, the contribution to (μ_i) by (μ_{rot}) is a slow and temperature- independent process, whereas the contribution due to (μ_w) is a relatively quick and temperature-dependent process, which is again a characteristic of the materials with the addition of composition distinct change in (μ_i) versus temperature.

Thus, the variation of initial permeability of $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ was influenced not only microstructures, but also by magnetostriction, inner stress and other factors. The high initial permeability in equimolar composite may attributed to the interaction between A_1B_2 and A_2B_1 ions on tetrahedral and octahedral sublattices in the spinel structure of the composites.

Electrical properties:

The DC electrical resistivity is an important property of low temperature sintered for MLCI applications. The resistivity of the ferrites was known to depend on the purity of the starting materials, sintering temperature and sintering time.

The room temperature DC resistivity's for $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ and equimolar composite ferrite is found to be the order of 10^9 to 10^8 Ω-Cm, which is the good requirement of MLCS. AC conductivity also shows a similar trend. The room temperature values of DC resistivity (ρ_{DC}), DC conducitivity (σ_{DC}), AC resistivity (ρ_{AC}), AC conductivity (σ_{AC}) along with seebeck coefficient values for all the compositions are presented in the Table.3.

Hall effect and Thermo-electric properties are widely used in the interpretation of the conduction mechanism in semiconductors. In case of low mobility materials such as ferrites, it's sometimes difficult to measure the Hall effect in such cases the thermo-electric power measurement is the only alternative. The sign of the thermo emf gives the vital information about the type of conduction in semiconductors, whether it's P or N type. Another important significance of thermo emf is that it enables one to calculate the values of Fermi- energy that help in the determination of the various regions, viz., Impurity conduction, impurity exhaustion and intrinsic conduction regions of the semiconductor. As can be observed from the values of Table.3. All the compositions exhibt positive Seebeck Coefficient. Showing that they behave as P- type materials.

Stress sensitivity studies:

Fig.4 shows the graphically represented data of the ratio of inductance change (ΔL/L) % as a function of applied compressive stress for equimolar composite and $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$. An examination of the Fig.4 indicates that in $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ (ΔL/L)% increases up to a certain applied stress and thereafter, it remains constant and found to be less stress sensitive. In all the samples studied in this series the (ΔL/L) % values are positive in the entire region of the applied stress applied. Exactly, similar behaviour was noticed by [9-10, 25].

These variations of permeability with applied compressive stress can be attributed to the magnetostrictive contributions of varied amounts of nickel (λ_s = -26 x 10^{-6}), and iron (λ_s = -19.5 x 10^{-6}) present in these samples [26]. For small compressive stresses, the stress raises initial permeability with negative magnetostriction and for large tensile stresses the permeability decreases [27].

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Conclusions

The attempt is made in order to achieve high initial permeability, to develop a soft magnetic ferrite useful for microinductor applications, and is compared with equimolar composite ferrite. Thus low temperature sintered $Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$ samples also possess good electromagnetic properties, as well as fine - grained microstructures, making them suitable materials for MLCIs having high performance and low cost.

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Conflict of Interests Section

The authors declare that there is no conflict of interests regarding the publication of this article

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Comparison of the Results

S.No	Properties	Compositions		Conclusions
		$Ni_{0.175}Mg_{0.125}Cu_{0.15}Zn_{0.55}Fe_2O_4$	$0.5((Ni_{0.35}Cu_{0.05}Zn_{0.6}Fe_2O_4)+(Mg_{0.25}Cu_{0.25}Zn_{0.5}Fe_2O_4))$	
1.	Average grain size 't'(μm)	6.51	4.11	greater
2.	Porosity	0.082	0.072	Slightly increases
3.	Lattice constant A (Å)	8.408	8.414	Slightly decreased
4.	X-ray density (ρ_x) gm/cc	5.013	5.062	Slightly decreased
5.	Measured density(ρ_m) gm/cc	5.426	4.978	Greater
6.	Initial Permeability (μ_i)	2580	9619	Drastically fallen down
7.	Curie Temperature T_c(°c)	150	150	equal
8.	ρ_{DC} at RT Ω-Cm	3.694×10^{8}	3.316×10^{9}	Slightly variation in order
9.	ρ_{AC} at RT Ω-Cm	2.303×10^{7}	1.676×10^{7}	Slightly changed
10.	σ_{DC} at RT $\Omega^{-1}Cm^{-1}$	2.707×10^{-7}	3.016×10^{-10}	Decreases
11.	σ_{AC} at RT $\Omega^{-1}Cm^{-1}$	4.342×10^{-6}	5.967×10^{-8}	Decreases
12.	Seebeck Coefficient α μV/K	+1681	+2180	Decreases
13.	Stres sensitivity Studies (ratio of inductance change (ΔL/L)) %	Positive and low stress sensitivity	Positive and low stress sensitivity	Responses in the same manner