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Preparation and Characterization of Mn Doped NiCuZn ferrite.

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Abstract: The ferrite composition $[\text{Ni}_{0.25-x}\text{Mn}_x\text{Cu}_{0.2}\text{Zn}_{0.55}]\text{Fe}_2\text{O}_4$ with values $x = 0.00, 0.05, 0.1$ were synthesized by auto combustion method. XRD analysis of prepared ferrite powder shows the cubic spinal structure. The crystallite size of prepared ferrite is varied from 22.3 to 39.3 nm the resultant powder were calcined at 650 °C/2hr and the pressed ferrite were sintered at 950 °C/4hr the initial permeability, dc resistivity were measured with frequency range 100Hz to 5MHz. The permeability is found to be increase up to $x=0.1$ and dc resistivity was decreased with Mn Substitution. The very high permeability in the composition $x=0.1$ was due to better densification and lower magnetostriction constant. The lattice parameter are also slightly increases from $x=0$ to $x=0.1$. The composition is better than the NiCuZn based material. It is useful for Multilayer chip inductor.

Keywords: auto combustion method, ferrites, calcination, XRD, permeability.

INTRODUCTION

Today's age is the information technology age. The electromagnetic components are more and urgently demanded having small size, low cost and high efficiency. The ferrites are used in SMDS, the M.L.C.I. is component which is widely used in electronic product such as video camera, note book. Cellular phone and computer. NiCuZn ferrites have been dominated material for M.L.C.I. due to its better magnetic properties at higher frequency and low sintering temperature. The Mn-Cu-Zn ferrite is also pertinent magnetic material for wide application of its high resistivity, Curie temperature and environmental stability. To reduce the number of layers in M.L.C.I. high permeability material is

require. Decreasing magnetostriction constant the initial permeability can be increased. The magnetostriction constant of Mn-Cu-Zn is lower than NiCuZn¹⁻⁶. Here expectation is that by adding the Mn the electromagnetic properties of NiCuZn ferrite are improved.

EXPERIMENTAL

The analytical grade magnesium nitrate $[\text{Mn}[\text{NO}_3]_2 \cdot 6\text{H}_2\text{O}]$, zinc nitrate $[\text{Zn}[\text{NO}_3]_2 \cdot 6\text{H}_2\text{O}]$, copper nitrate $[\text{Cu}[\text{NO}_3]_2 \cdot 6\text{H}_2\text{O}]$, iron nitrate $[\text{Fe}[\text{NO}_3]_3 \cdot 9\text{H}_2\text{O}]$ and citric acid $[\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}]$ were used to prepare $[\text{Ni}_{0.25-x}\text{Mn}_{0.20}\text{Cu}_{0.20}\text{Zn}_{0.55}]\text{Fe}_2\text{O}_4$ with $x=0.0, 0.05, 0.1$ by autocombustion method. The metallic nitrates and citric acid were dissolved in de-ionized water and mixed in 1;3 m ratio of nitrate to citric acid. The solution was heated to transfer it in to gel. Then the dried gel burnt in self propagating combustion manner until all gel was completely burns out to form fully loose powder. The burnt precursor powder were calcined at $650^\circ\text{C}/2\text{hr}$. the calcined powder were granulated by using PVA as binder. It is uniaxial pressed at 1.5 to 2 ton $/\text{cm}^2$ to form pellet and toroidal specimen. These specimens were sintered at $950^\circ\text{C}/4\text{hr}$. in air atmosphere the sintered ferrites were characterized with respect to phase identification, crystallite size, and lattice parameter determined using X-ray diffraction with $\text{CuK}\alpha$ radiation $[\lambda = 1.5406\text{\AA}]$. Bulk density were measured by using formula, $\text{dx} = \text{ZM}/\text{Na}^3$. For obtaining the value of permeability, the inductance was measured with L-C-Q-R meter. The resistivity was measured on pellet samples by applying silver electrodes on the surface.

RESULTS AND DISCUSSION

The XRD pattern is shown in **Fig.1**. It shows that the sintered ferrite powder are in crystallite state and it is observed that, it contains cubic spinal ferrite phases similar to JCPDS card No. 03-0864, there is no second phase is detected by XRD. The broad peak in XRD pattern indicates that the fine crystallite size of the ferrite particles.

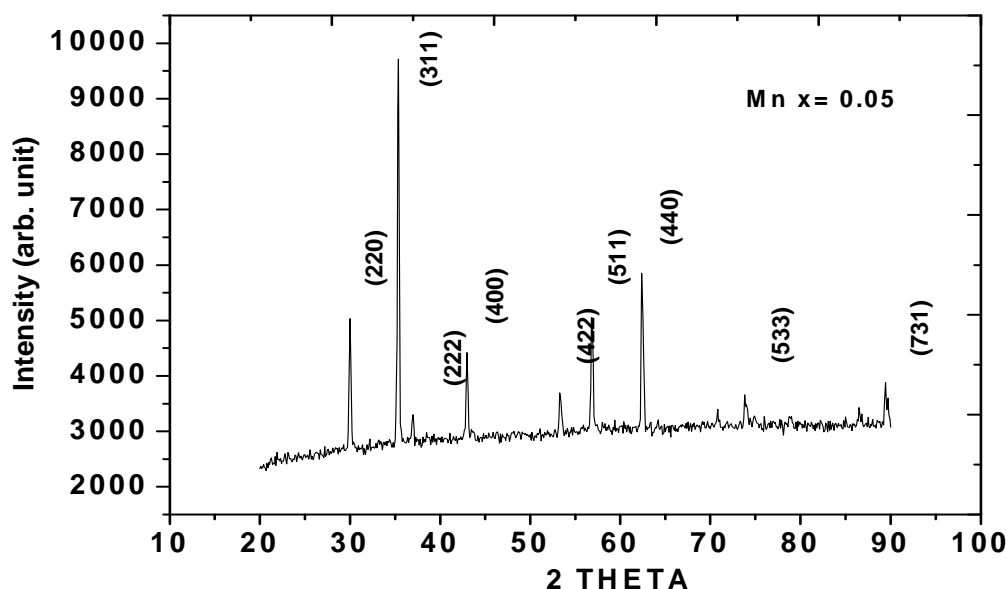


Fig. 1: X-ray diffraction pattern of typical Mn doped ferrite.

The crystallite size was calculated by using scherer formula. The crystallite size of ferrite was observed to vary from 22.3 to 39.3 Nm. When burnt ashes were sintered at 950 °C/4hr there is the noticeable effect on crystallite size. The crystallite size was increased by addition of Mn. the crystallite size, lattice parameter of the sintered ferrite is shown in Table-1, along with their bulk density, permeability. Dc resistivity and grain size by SEM.

Table-1: Various parameters for $[\text{Ni}_{0.25-x}\text{Mn}_x\text{Cu}_{0.20}\text{Zn}_{0.55}] \text{Fe}_2\text{O}_4$ ferrite.

Mn[x] contant	Bulk Density gm/cc	Crystallite Size nm	Lattice ParameterÅ	Permeability At 200 Hz	D.C. Resistivity Ωcm [at480°C]	Grain size from SEM [nm]
0.00	6.17	22.28	8.342	205	4.57	813
0.05	6.63	38.81	8.403	208	4.50	870
0.1	6.91	39.28	8.407	270	4.41	930

In the present work bulk density increases with Mn addition. The lower bulk density of NiCuZn at $x=0$ is due to the absences of Mn in the composition. The crystallite size in the sintered body also shows the composition with $x=0$ have lower grain growth due to lower amount of liquid phase formation compared with composition containing both Ni and Mn. there is no noticable change is observed in lattice parameter of the ferrite in different composition as radia of $\text{Mn}^{+2} = \text{Ni}^{+2}$

The permeability is large at $x=0.1$ composition of Mn-Cu-Zn ferrite. It is primarily attributed to increase in bulk density. It is seen that the ferrites with large bulk density. Large grain size by sem posses higher permeability. The high initial permeability was observed⁷ at composition $x=0.1$.¹

The permeability can be calculated by using inductance and following formula.

$$\mu_i/0.0046\text{N}^2.\text{h}.\log_{10}[\text{d}_2/\text{d}_1]$$

Where; L-Inductance in micro hennerly, d_1 -inner diameter, d_2 -outer diameter of toroid,
h-height in inches.

However the initial permeability of composition. $x=0$ is lower than the composition $x=0.1$.this may be attributed due to lower grain size and absence of mn in composition.

The **Fig.2** shows the frequency dependency of permeability.

In $[\text{Ni}_{0.25-x}\text{Mn}_x\text{Cu}_{0.2}\text{Zn}_{0.55}]$

Fe_2O_4 the permeability is stable in frequency range 100Hz to 1Mhz.its dispersion occur above 1Mhz .

It is known that the high frequency dispersion are associated with domain wall dynamics. The increase in frequency dispersion by Mn containt shows that the critical field decreases due to Mn incorporation⁸⁻⁹. The curie temperature of the ferrite composition is higher at $x = 0$ and it is decreases by addition of Mn. the Curie temperature are high at $x = 0$ because Ni is present in said composition and Curie temperature of Ni is more than Mn.

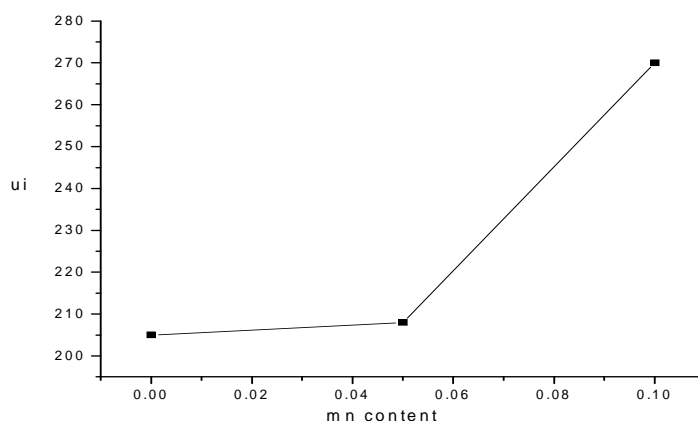
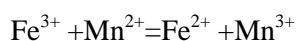


Fig-2: Permeability v/s Mn content.

Dc electrical resistivity: The dc electrical resistivity is the important property of sintered ferrite for MLCI application, because resistivity remarkably affects the electroplating of devices. Fig shows the variation of dc resistivity versus Mn contain [x] for samples at 480 °C. It can be seen that resistivity is decreased by the addition of Mn.

This decrease in resistivity may be attributed to the fact that in the case Mn-doped NiCuZn ferrite. The conduction mechanism in ferrite is considered as the electron hopping between Fe^{2+} and Fe^{3+} . In the B sites of the Mn doped NiCuZn ferrite are occupied by Ni^{2+} , Fe^{3+} and Cu^{2+} ions. Obviously more the Fe^{2+} ions content the higher conduction and consequently decrease in resistivity. Therefore observed decrease in dc resistivity with increase of Mn content may be attributed to the presence of increased Fe^{2+} ions. In NiCuZn Mn doped ferrite the following equilibrium may exist during the sintering¹⁰⁻¹²



With increasing Mn content, more Fe^{2+} are formed, resulting in increasing the probability of electron hopping and decreasing resistivity as shown in **Fig-3**

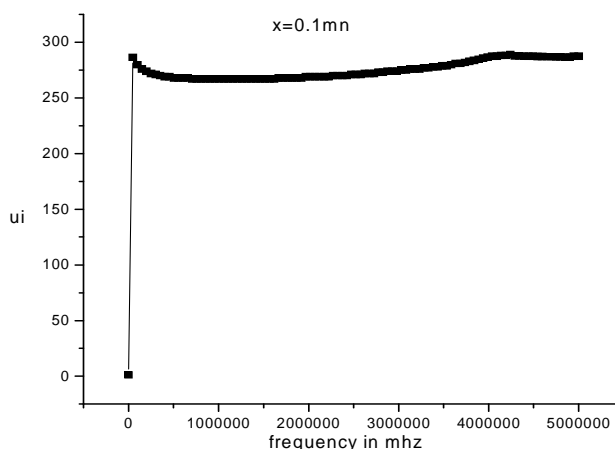


Fig. 3: Plot of frequency dependency of permeability

CONCLUSIONS

The experimental results given above indicate that the Mn addition in NiCuZn ferrite has significant role in improvement of electrical and magnetic properties. The frequency and temperature affects the electrical and magnetic properties of Mn-Cu-Zn ferrite. With increasing Mn content, the initial permeability increases and dc resistivity decreases up to $x=0$. The crystallite size and bulk density are increased with mn addition. The obtained results provide important information for improving significant properties of NiCuZn ferrite for MLCI and SMDs.

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