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STUDY OF MICROSTRUTURE AND TEMPERATURE DEPENDENT LOW FREQUENCY AC PROPERTY OF $Ni_{0.46}Zn_{0.54}Ca_{0.05}Fe_2O_4$ FOR MECHATRONICS AND ELETRONIC DEVICE APPLICATION

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ABSTRACT
By solid state reaction method $Ni_{0.46}$ $Zn_{0.54}Ca_{0.05}$ Fe_2O_4 compound was prepared. By XRD the crysta structure was revealed and by SEM the microstructure was studied. At different temperatures the ac parameters such as conductivity and capacitance were studied from 1 KHz to 6 MHz. Q factor was measured to find the specific frequency range for switching property. All these experimental results are found to be useful in fabrication of future MEMS technology since the industry is taking new approaches for miniaturizing circuit elements for control and mechatronic applications. The minute addition of Calcium enhanced the electrical properties required for such applications.

Keywords: Ni Zn Ferrite Composites, Control System, Mechatronic Device Material, SEM, XRD

1. INTRODUCTION
Nickel Zinc Ferrite solid composites are the well known spinel ferrites. These composites prepared by different techniques and with different levels of doping are widely been used in the soft ferrite industry. Ni Zn Ferrite composites have become a major concern of research for several decades for their extensive uses in high-frequency transformers and inductors [1], signal transformer, power transformer, pulse transformer, input/output filter chokes [2], deflecting yokes and flyback transformer for TV receivers, antenna rods for radio set [3] and recording heads [4]. Recent interest is focused on the development of miniaturization of electronic components to downsize various electronic packages and thus to reduce the size/weight ratio. Although high-frequency Mn Zn ferrite is used in most cases to develop mini dc–dc converters and inductors [5] for higher electric resistance exceeding $10^7\Omega$ m, Ni Zn ferrite core is a good competitor in this field. Electronic and magnetic applications sensitive to eddy current losses and usable at moderate permeability, Ni Zn ferrite offers better performance than Mn Zn ferrites. For commercial EMI suppression Ni Zn ferrite composites are already in the market. [6] Another use of it is magnetic switching where it shows much better performance than electrically switched systems. All these inventions and researches are very important for control system and mechatronic applications. Ni Zn Ferrite is spinel type soft magnetic material combining tetrahedral A site and octahedral B site in AB_2O_4 crystal structure. It consists of a combination of normal and inverse spinels which has very important magnetic properties for applications above 1MHz. [7] In this paper we investigated the ac properties at different temperatures and micro structure of the $Ni_{0.46}Zn_{0.54}Ca_{0.05}Fe_2O_4$

2. EXPERIMENTAL PROCEDURE
$Ni_{0.46}Zn_{0.54}Ca_{0.05}Fe_2O_4$ sample was synthesized using standard solid state reaction technique using powder NiO, ZnO , CaO and Fe_2O_3 as raw materials from Baker, Germany. Stoichiometric amount of required powders were pressed into disk shaped and toroid shaped samples. The samples were sintered at 1200^oC. The temperature ramp was at 5^0C/ min for both cooling and heating. The samples were of two sizes: sample A was a solid flat disc and sample B was a toroid. Microstructural properties have been investigated by XRD and SEM. X ray diffraction was carried out with a Philips X-'pert automatic powder diffractometry system using Cu-K_α radiation. The Philips XL30 Scanning Electron Microscope with an accelerating voltage of 15 KV was used for SEM imaging. Conductivity and capacitance were measured t 25^0C, 50^0C, 75^0C temperatures at low frequency range (typically from 100 KHz to 6 MHz

with an Agilent low frequency impedance analyzer 4291A model where the circuit was in series connection with the material under test.

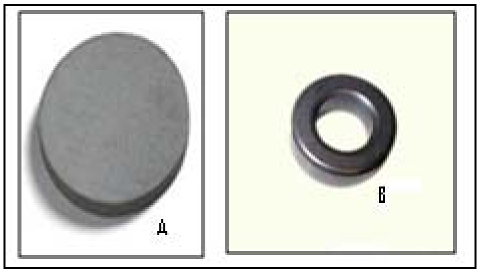

Fig 1. Prepared samples A and B

Table 1: Dimensions of the samples

Sample	A	B
Thickness (mm)	2.92	3.0
Outer diameter (mm)	11.3	15.0
Inner diameter (mm)	6.05	-

3.1 MICROSTRUCRE STUDY

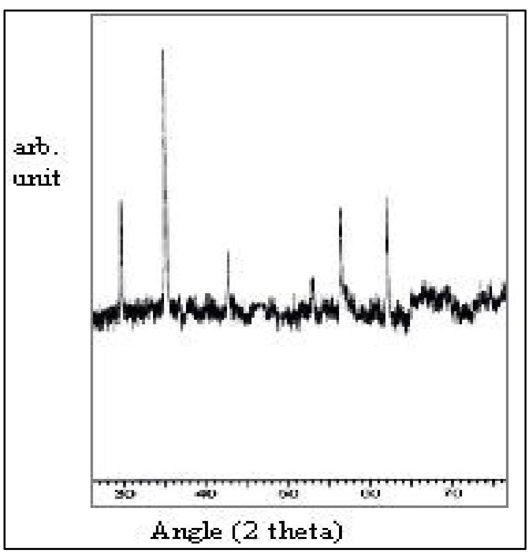

Fig 2. XRD of $Ni_{0.46}$ $Zn_{0.54}Ca_{0.05}$ Fe_2O_4

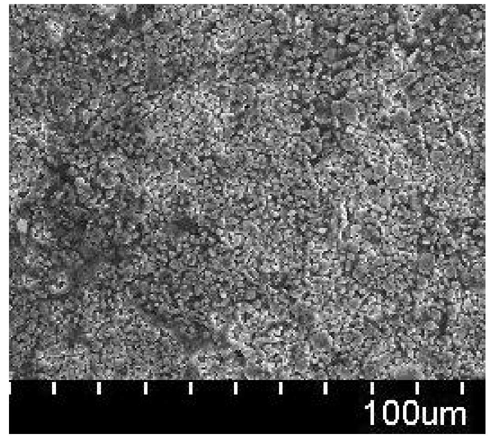

Fig 3. SEM image at 100 µm resolution

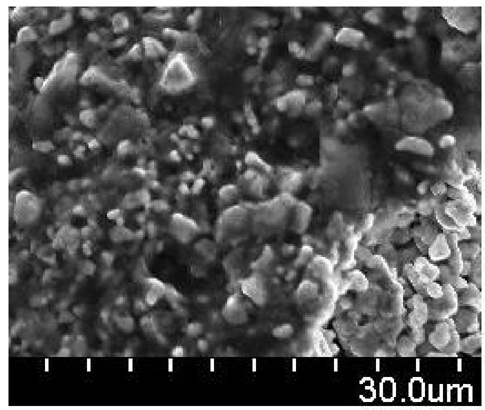

Fig 4. SEM image at 30 µm resolution

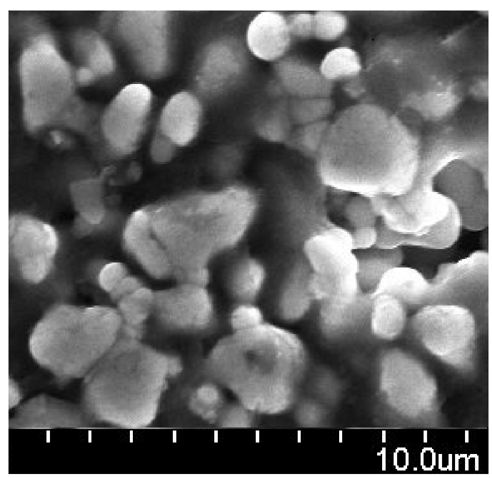

Fig 5. SEM image at 10 µm resolution

3.2 XRD RESULT

X-ray diffraction (XRD) pattern showed the polycrystalline phase and formation of spinel crystal structure. The angles of the peak values correspond to (111), (222), (220), (440) planes due to the atoms present at octahedral and tetrahedral sites. Because the ionic radii of Ca^{2+} ions are greater than that of Ni the replacement of Ni by Ca^{2+} ions is expected to increase the lattice constant of ferrite. [8] Redistribution of tetra- and octahedral sites are evident from the increase of intensities of (111) and (222) peaks and decrease of (220) and (440) peaks. By applying scherrer formula the size of the crystallite size is measured and found around 8.42^0 A.

The calculation was made as followings:

$$d = \frac{0.9\,\lambda}{\beta \cos\theta} \quad (1)$$

Where, d= crystalline diameter
λ = wavelength of the XRD = 1.56^0 A
θ= angle of the highest peak = 18^0
β = half line width of the maximum peak

3.3 SEM IMAGE

Scanning electron microscope is a powerful tool of studying microstructure. In our case SEM was done at 15 K Electron Volt and at three different resolutions 100μm, 30μm and 10μm. From these images the grain size was measured. The average grain size was about 5 micro meter. The images showed inhomogenity in the crystal structure. The black spaces are the voids created during solidification.

4. CONDUCTANCE MEASUREMENT

Frequency dependent ac parameters such as conductivity and capacitance were measured at 25^0C, 50^0C, 75^0C temperatures at low frequency range with an Agilent low frequency impedance analyzer 4291A model. The flat disc sized sample was in series circuit during the measurement. Joule heating was done by variable external voltage. By calibrating the voltmeter with the thermocouple the temperature was measured. The specimen sample A was subjected to high pressure to make better contact in the circuit. Temperature dependent conductance was measured at 25^0, 50^0 and 75^0 C. It is found that with the increasing temperature the conductivity decreases. The difference between 50^0 C and 75^0 C is slight but these two show big differences with the room temperature measurement. The conduction process in ferrites with excess ferrous content (*n*-type semiconductors) is usually attributed to electronic jumps between Fe^{2+} and Fe^{3+} in the octahedral sites. [9] The electrical conductivity of these materials can be approximately described by:

$$\sigma = (ne^2 l^2\, \upsilon_0/kT)\exp(-E/kT) \quad (2)$$

Where, *n* = density of charge carriers,
e = electron charge,
l = length of a jump (usually assumed to be 0.3 nm, representing the distance between the octahedral positions),
υ_0 = characteristic frequency (10^{12} –10^{14} s^{-1}),
k = Boltzmann constant,
T = absolute temperature,
E =activation energy of the conduction process (“electron hopping”)

The addition of calcium in the Ni Zn ferrite composite enhances resistance and impedance by introducing glassy behavior in the grains boundaries. From the graphs (Fig 6-8) of our experiment frequency dependent conduction behavior was revealed which is necessary for finding the eddy current losses in the system. Each ferrous ion can be considered as a donor containing an extra electron, which jumps to the adjacent ferric ion easily and constitutes the electronic conduction process. The variation of temperature changes dc conductivity. The oxidation of divalent iron reduces the donor concentration in the material such that the electrical resistivity is increased. The grain boundary resistivity is, generally, expected to be higher than the bulk resistivity. [10] Thus the material preparation techniques have a great role in determining the ac parameters and in a rich area for experimental researchers.

5. CAPACITANCE MEASUREMENT

The capacitance of the sample increased with the increasing temperature. All the graphs (Fig 9-11) showed exponential decay of the capacitance with the increasing frequency. These curves showed the dynamic behavior of charge concentration in the composite with varying frequency at three different temperatures.

6. Q FACTOR MEASUREMENT

In control system and mechatronics remote magnetic switching to avoid the wiring and reduce size of the components often spinel ferrite is a good choice. To make band pass filter and tuning operated circuit, oscillating circuit and varactors Quality factor measurement is an important aspect. In our experiment Q factor was measured in LRC precision meter 4285 A. Various type of band pass filters can be designed from 0.3 to 0.7 MHz frequency range.

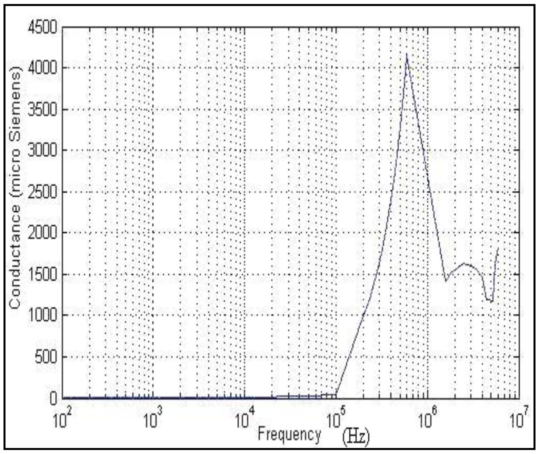

Fig 6. Frequency vs Conductivity at room 25^0 C

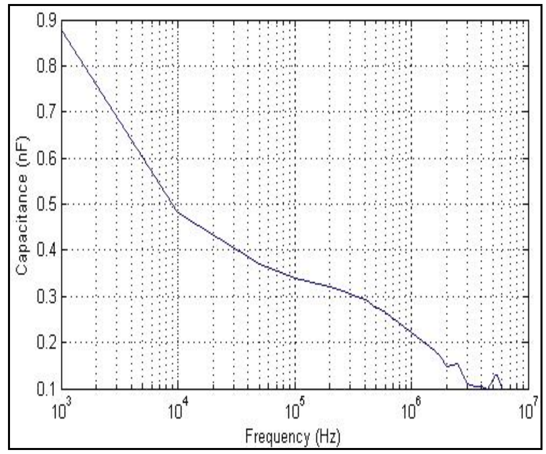

Fig 9. Frequency vs Capacitance at room 25^0 C

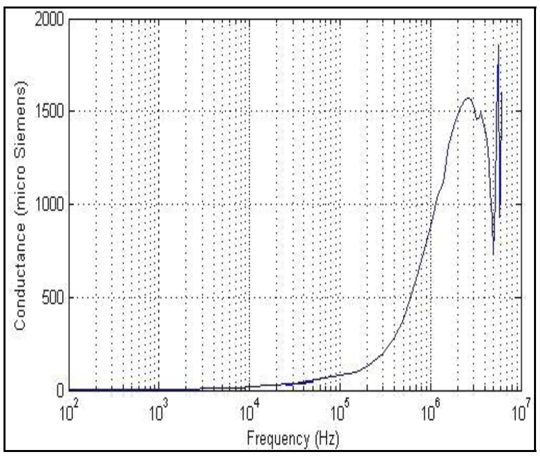

Fig 7. Frequency vs Conductivity at room 50^0 C

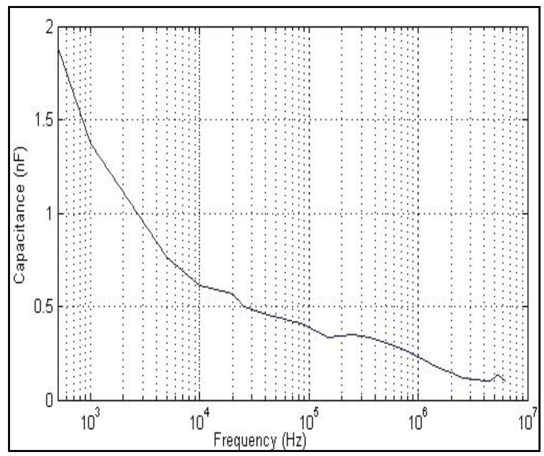

Fig 10. Frequency vs Capacitance at room 50^0 C

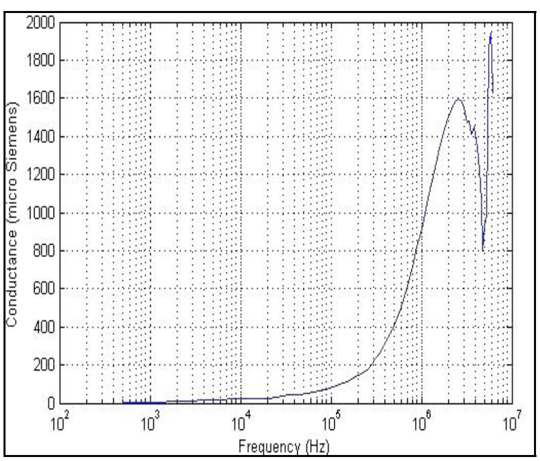

Fig 8. Frequency vs Conductivity at room 75^0 C

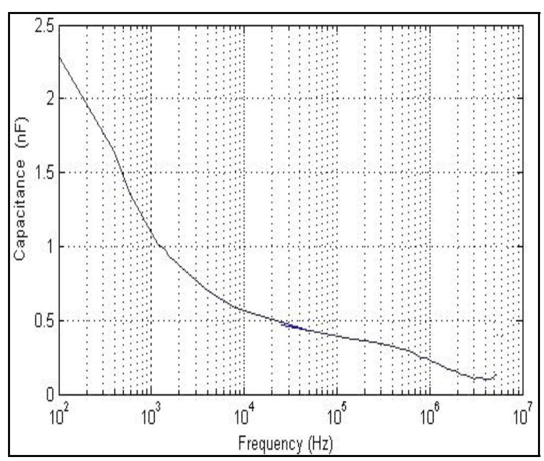

Fig 11. Frequency vs Capacitance at room 75^0 C

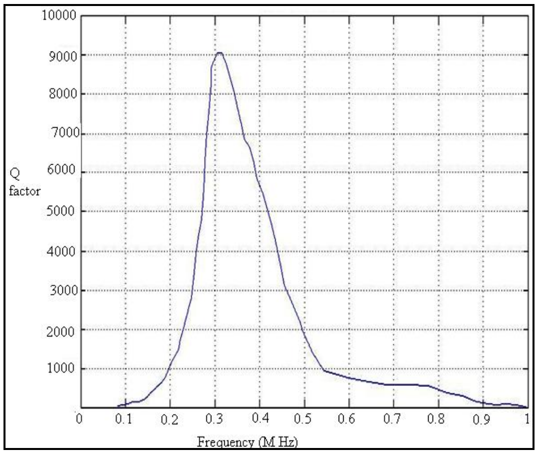

Fig 12. Q factor vs frequency at 25^0 C

7. CONCLUSION

The study of $Ni_{0.46}$ $Zn_{0.54}Ca_{0.05}$ Fe_2O_4 shows that samples prepared by solid state reaction techniques is appropiate for practical applications in control system and mechatronics application. The results found in this experiment is well suited with the theoretical study of spinel ferrites. The studied ac characteristics are important for designing miniature circuits at varying temperatures and recent MEMS technology.

8. REFERENCES

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9. NOMENCLATURE

Symbol	Meaning	Unit
d	Crystallite diameter	(m)
θ	Angle of peak	(^{0})
β	Half line-width of peak	(rad)
λ	Wavelength of the XRD	(m)
T	Absolute temperature	(K)
n	density of charge carriers	(C/m^3)
l	the length of a jump	**(m)**
E	Activation energy of conduction process	**(J)**
υ_0	Characteristic frequency	**(s^{-1})**
C	Capacitance	(F)
G	Conductance	(S)
Q	Quality factor	Dimensi-on less

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