

Executive Summary of the Report

Ni-Zn ferrites are technologically important as they are useful for making antenna rods, inductors, transformers cores, electronic instruments and digital computers. Therefore it was decided to select Ni-Zn ferrite system. Substitution of Zinc lowers the Curie temperature, however it increases the magnetization up to 50% substitution of Zn^{2+} . Elevated firing temperatures give rise to formation of Fe^{2+} ions which degrades the magnetic properties at high frequencies. In order to localize Fe^{2+} ions, we decided to substitute by TiO_2 in the ferrites. However, the solubility of Ti^{4+} is high in Ni-Zn ferrites.

To investigate the effects of these additives it was proposed to undertake the following experimental work, Preparation and characterization of ferrite compositions $Ni_{0.55}Zn_{0.45}Ti_xFe_{2-x}O_4$ ($x = 0, 0.005, 0.01, 0.015, 0.02, 0.025$ and 0.03) Microstructure determination and relation of microstructure with the bulk magnetic properties. Experimental work on variation of permeability, susceptibility and loss factor

The general formulae of the ferrite system is as follows $Ni_{0.55}Zn_{0.45}Ti_xFe_{2-x}O_4$ ($x = 0.005, 0.01, 0.015, 0.02, 0.025$ and 0.03) AR grade oxides of NiO , ZnO , TiO_2 , and Fe_2O_3 were used for the preparation of various compositions of the ferrite. These oxides were weighed in the required mole proportions using a single pan balance having least count 0.001 gm and mixed thoroughly in the agate-morter in acetone for about 2 hrs. The mixture was sieved using a sieve of mesh size 200 micron. The mixture of each composition was preheated in platinum crucible at $55^{\circ}C$ for 8 hrs. achieve homogenization of the end product. The mixture was cooled in the furnace atmosphere (air) at the rate of $80^{\circ}C/hr$. The temperature of the furnace was measured with the

help cromel Alumel thermocouple . $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ($X= 0.0 - 0.03,$) composition were successfully prepared by a standard ceramic route.

X-ray powder diffraction spectra were used to determine the lattice constants and to establish the single phase nature of the ferrites. We have carried out XRD studies on the system. $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ($x= 0.005, 0.01, 0.015, 0.02, 0.25$ and 0.03). The diffraction lines have been indexed and indices were tallied with those expected for spinel structure. The reflection observed are (111), (220), (311), (400), (422), (511), and (440). These correspond to the allowed values of reflection for cubic spinel structure.

SEM photographs the grain size D_m is calculated as follows :-

- i) Drawing a diagonal on the photograph.
- ii) Measuring the maximum unidirectional particle size in the vertical direction against diagonal.
- iii) Averaging the maximum unidirectional particle size.

From SEM photographs it is seen that the grains are well define. The grain growth is continuous and there are no exaggerated grains. The grains are pore free. The grain growth are increasing up to $X = 0.015$ addition of TiO_2 while beyond increase of TiO_2 the grains growth decreases.

The permeability as a function of frequency at room temperature was measured over the frequency range from 100 Hz to 1MHz for the $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ($x= 0.0$ to 0.03). The initial permeability was calculated from the low field inductance measurements with toroidal core of 75 turns . In all the samples it is seen that with increase of Ti^{4+} the permeability decreases. Initial permeability is one of the properties which can be very dependent upon the method of manufacture. The occurrence of dispersion an absorption at frequencies in the region of 1 MHz. In the composition of $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ($x = 0, 0.005, 0.01, 0.015, 0.02, 0.025$

and 0.03) the Loss Factor decreases with frequency up to 100 KHz. Above this frequency the Loss Factor increases. The variation of μ_1 with frequency up to 1 MHz for the compositions of $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ($x = 0, 0.005, 0.01, 0.015, 0.02, 0.025$ and 0.03) Show that in all the composition decreases by large amount at low frequency while μ_1 the dispersion of is small on high frequency side. The low frequency dispersion is attributed to domain wall motion.

Bulk magnetic behavior is generally comprehended in terms of domain structure. Temperature variation of a.c. magnetic susceptibility and hysteresis provides useful data on the domain structure. A crystalline ferrite material- may contain , Multi-Domain (M.D.), Single Domain(S.D.) and) Super paramagnetic (S.P.). From $X_{ac} - T$ curve shows that, the composition contains M.D. Particles, Iso-tropic peak for the materials in the M.D. state provided the materials has a temperature at which magnetocrystalline anisotropy is zero, and the $X_{ac} - T$ does not show variation in the temperature region of inversion.

Ferrites have vast applications from microwaves to radio frequencies. So it is important to study their dielectric behavior at different frequencies . Ferrites have a very low conductivity. Which is one of important the considerations for microwave applications. The order of the magnitude of conductivity greatly influences the dielectric and magnetic behaviour of ferrites. The dielectric properties of these materials are very sensitive to the method of preparation, the sintering temperature, the sintering atmosphere and the amount and type of substitution. We have carried out measurements of capacitance ‘C’ resistance ‘R’ and loss tangent $\tan \delta$ directly in the frequency range 100 Hz to 1MHz. The measurements in the range 1 MHz to 5 MHz were carried out on LCR meter. The addition of $\text{Ni}_{0.55}\text{Zn}_{0.45}\text{Ti}_x\text{Fe}_{2-x}\text{O}_4$ ferrite the dielectric constant ϵ decreases while the resistivity ρ_{dc} increases, the loss tangent tends to decrease. All the sample exhibit dispersion due to Maxwell Wagner interfacial polarization^[1] in agreement with Koops

phenomenological theory. The variation of $\tan \delta$ with frequency , it is seen that with increase of frequency the loss tangent $\tan \delta$ decreases. The dielectric constant and resistivity decrease with increase of frequency. Thus the decrease of $\tan \delta = \epsilon'' / \epsilon'$ appears to be due to increase of ϵ'' . Thus it is concluded that, addition of Ti^{4+} leads to increase of dielectric losses. It is seen that the loss factor goes on increasing with the addition of Ti^{4+} in $Ni_{0.55}Zn_{0.45}Ti_xFe_{2-x}O_4$. The increase of loss factor suggests that substituting cations offer increasing impedance to the domain wall motion.

The Fourier transform infrared spectra of sintered powder were recorded in the range 800 cm^{-1} – 400 cm^{-1} . The infrared spectra of sintered powder shows two absorption bands. The first band observed at frequency of 568 to 565 cm^{-1} (v_1) is attributed to the stretching vibration of tetrahedral group $Fe^{3+}-O^{2-}$ and second band observed at frequency of 466 - 476 cm^{-1} . The force constants for tetrahedral (k_t) and octahedral (k_o) sites have been determined. And hence the elastic constants such as stiffness constant (C_{11}, C_{12}), Young's modulus (E), Bulk modulus (K), rigidity modulus (G) and Poisson's ratio (σ) can be determined from infrared spectral analysis.

In the present study a simple and inexpensive method called standard ceramic technique was used for preparation of $Ni_{0.55}Zn_{0.45}Ti_xFe_{2-x}O_4$. For preparation of microwave absorber, $Ni_{0.55}Zn_{0.45}Ti_xFe_{2-x}O_4$ ferrite powder was used. The microwave absorption properties of the samples were measured in the frequency range 200 KHz to 3000 MHz. Scattering parameters S_{11} and S_{12} of developed ferrite were measured over a frequency range 2.3 to 2.5 GHz. All samples show absorption in the frequency range 2.0 to 2.1 GHz. A good quality absorber should have a large reflection coefficient.