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Dielectric, Modulus, Impedance Spectroscopy and Conductivity Properties of Mg-Substituted Cu-Zn-Al Nanoferrites with Structural Rietveld Refinement

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Abstract::Nanocrystalline ferrites are needed in many electronic applications instead of powder with grain in the nanometric range that is the most valuable objectives of the present research. In the present research, $Mg_xCu_{0.65-x}Zn_{0.35}Fe_{1.925}Al_{0.075}O_4$ ($0.0 \le x \le 0.5$. in the stages 0.1) nanoparticles were prepared annealed at 700 °C from notable Sol-gel synthesized techniques. It is revealed in many previous cases that suitable doping in ferrites showed improved characteristics like enhance resistivity and magnetization, reduce dielectric loss etc. Analyzing XRD data, the structure of every composition comply with the cubic spinel structure. By the implementation of XRD data, structures have been analyzed through Rietveld Refinement technique using Full prof suitable software. After getting good chi^2 value different parameters have been determined. These include crystallite size, cation distribution and electron density. Moreover, bond structures are found by using the output of Rietveld Refinement procedure. It is depicted from electronic modulus study that dielectric loss tangent reduces exponentially with increasing frequency and remains constant at higher frequency. With the arisen frequency the electrical properties of grain and grain boundaries have been measured from the complex impedance study. In addition of Mg^{2+} content, both the resistance increases of grain and grain boundary. The σ_{ac} has been raised through the increasing frequency.

Keywords: A-Mg-Cu-Zn-Al ferrites, B-XRD, C-Dielectric properties, D-Cole-Cole plot, E-AC Conductivity.

Ferrite nanoparticles

Spinel ferrite nanoparticles have been the subject of considerable interest in the recent years due to their potential technological applications in various fields such as high-density data storage compartment, ferrofluid, spintronics, sensors, magneto caloric refrigeration, heterogeneous catalysis, hydrogenation flourishes, magnetically guided drug delivery, magnetic fluid hyperthermia procedure, magnetic resonance imaging etc.

Nanosized ferrite particles exhibit the unusual magnetic and electric properties which are not observed in the bulk material, e.g. single domain behavior, superparamagnetism, enhance electrical resistivity and reduced Curie temperature, and magnetization.

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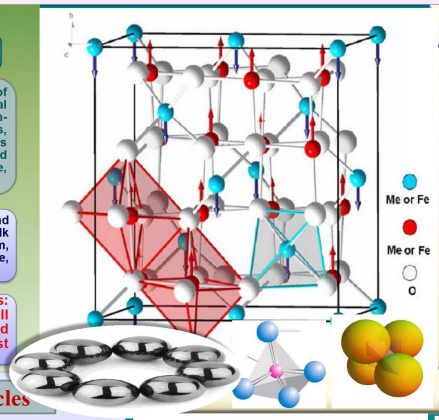
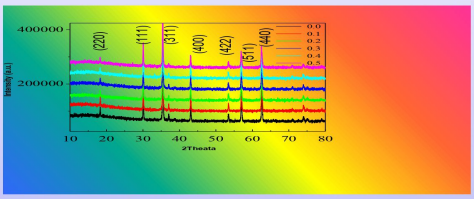


Synthesis of Ferrite Nanoparticles

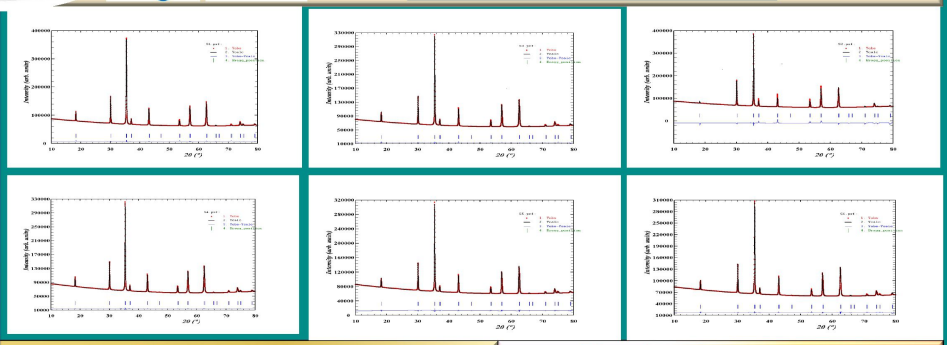
- $Mg_xCu_{0.65-x}Zn_{0.35}Fe_{1.925}Al_{0.075}O_4$ (x = 0.1, 0.2, 0.3,0.4 and 0.5) samples were prepared by Sol-Gel Auto Combustion Technique.
- Appropriate amount of high purity powders of $Mg(NO_3)_2.6H_2O$, $Cu(NO_3)_2.3H_2O$, $Fe(NO_3)_3.9H_2O$, $Al(NO_3)_3.9H_2O$,, $Zn(NO_3)_2.6H_2O$, have been mixed thoroughly. Mixing was performed with pure ethanol in a glass beaker. p^H of the mixed solution always maintained at 7 by adding ammonia solution.
- The mixture was placed in a constant temperature bath at 70 °C, followed by an ignition to get dried gel. The dried gel was then placed in an oven at fixed temperature 300 °C to form fluffy loose powders. Finally, these fluffy powders were annealed at 700 °C for 5 hours to get the desired Mg-Cu-Zn-Al nanoferrite for further characterization.
- The heat treated powders then grinded and pressed into disk-shape for measurements of transport properties such as dielectric constant, ac conductivity and dielectric losses.
- Analysis of complex impedance and electric modulus spectra (Cole-Cole plot) of different compositions were investigated.

XRD of Ferrite Nanoparticles

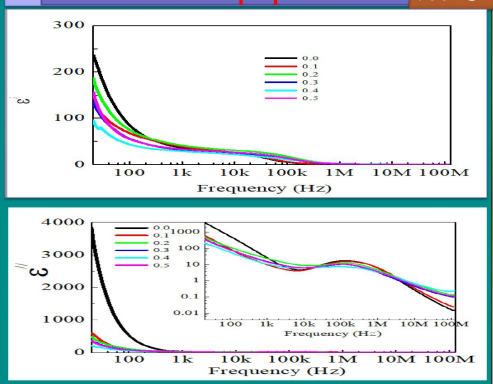


Single phase structure formation is one of the prime deterministic conditions for solid state materials as well as ferrites. Figure shows the XRD patterns of various $Mg_xCu_{0.65-x}Zn_{0.35}Fe_{1.925}Al_{0.075}O_4$ annealed at 700 °C .The most intense peaks are indexed as (1 1 1),(2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) in all specimens were found to mach well with the spinel ferrite structure for each composition and no traces of raw materials were found ,thereby confirming that the chemical reaction completed.

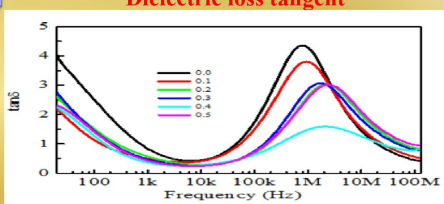
Structural Analysis (Rietveld Refinement)



Dielectric properties 700 °C

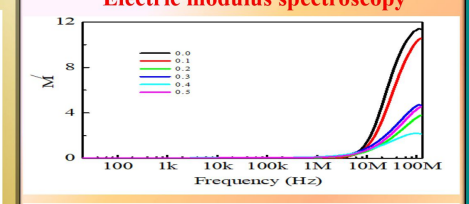


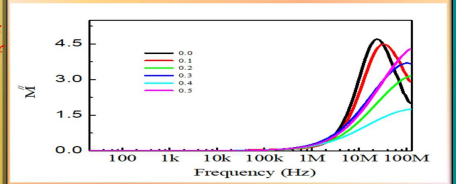
Dielectric loss tangent



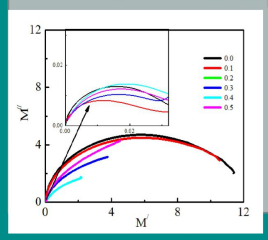
The dielectric constant show normal behavior whereas the dielectric loss shows abnormal behavior exhibiting loss peaks as a function of frequency which can be explained on the basis of electron hopping mechanism. Dielectric loss tangent remarkably lower for smaller nanoparticles and from modulus plot it is clear that the transition of long range to short range hopping frequency increases with Mg content.

Electric modulus spectroscopy

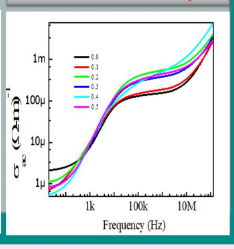




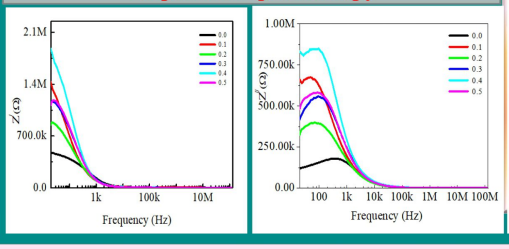
Modulus (Cole-Cole plot)



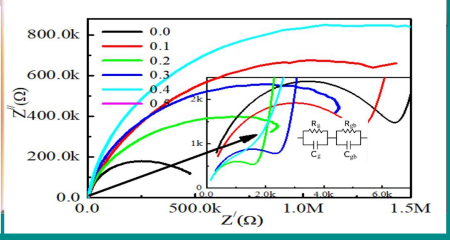
Ac conductivity



Impedance spectroscopy



Complex impedance spectroscopy



Conclusions

- Nanoparticles of pure and Mg substituted Cu-Zn-Al have been successfully synthesized by sol gel technique.
- The single phase cubic spinel of all synthesized Nano-crystalline samples of $Mg_xCu_{0.65-x}Zn_{0.35}Fe_{1.925}Al_{0.075}O_4$ at 700 °C has been confirmed by XRD analysis.
- Rietveld Refinement has found good fitting values (χ^2 = 1-2).
- The crystallite size determined from XRD data is found in the range of (14-21) nm for 700 °C.
- The value of dielectric constant at lower frequency is comparatively lower for nanoparticles than that of bulk samples due to the reduction of space charge polarization.
- The dielectric loss tangent decreases remarkably for synthesized nanoparticles.
- The modulus analysis has established the possibility of hopping mechanism for electrical transports processes in the nano particle system.
- The real part of impedance shows usual behavior with frequency and it increases with increasing Mg content.
- The Nyquist plot indicate the possible contribution of the nano grain and grain boundaries with Mg content and both grain-grain boundary contribution increased as reported .
- Short processing time of gel formation, homogeneity, well-crystallized particles and nano size are achievement of the present research.

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