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The study of dielectric properties of ZCF using XRD and synthesis of Zn-Fe spinal using CCP techniques

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Abstract – *In this work, a study of dielectric properties of ZCF using XRD and synthesis of Zn – Fe spinal. XRD profiles are used to confirm the single phase spinal Fe^{3+} formation. The sample prepared through CCP have solidification > 75 % making these materials suitable for sensing applications. The dielectric loss, dielectric constant and AC conductivity are analyzed as a function of frequency, temperature, and composition using IS. Overall, dielectric behavior has been studied through temperature and frequency variations of different parameters. The making of the testing sample using CCP techniques.*

Keywords – *Impedance spectroscopy (IS); Chemical co precipitation (CCP); Dielectric conductivity, $ZnCr_xFe_{1-x}O_4$ (ZCF, x= 0.5), Impedance analyzer (IA).*

I. INTRODUCTION

Nano crystalline Fe^{3+} materials have achieved excellent physical and dielectric properties as compared to their bulk complement. Properties of Fe^{3+} materials are highly influenced by mass nature of grains, grain boundaries, and solidification, composition and preparation techniques. An amazing characteristic of spinal Fe^{3+} is that their composition and properties can be strongly modified depending foremost the applications keeping the basic crystalline structure unaltered. Effect of preparation technique on structural, micro structural and dielectric parameters has been calculated and reported.

I. EXPERIMENTAL PROCEDURE

II. SYNTHESIS OF $ZNFE_2O_4$ AND $NFE_{1.5}CR_{0.5}O_4$ BY CHEMICAL COPRECIPITATION

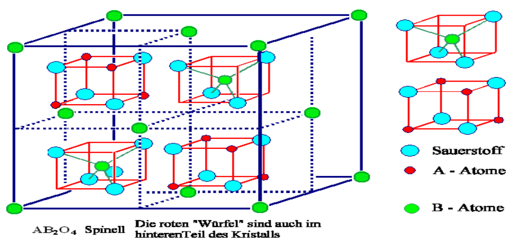

Figure 1 Spinal crystal structure

$ZnFe_2O_4$ and $ZnFe_{1.5}Cr_{0.5}O_4$, $Fe3^+$ were synthesized via CCP technique by taking $ZnCl_2$, Cr $(NO_3)_3.9H_2O$ and Fe $(NO_3)_3.9H_2O$ as starting chemicals. Metal salts were in use in stoichiometric ratio 2:5 and their identical solutions were prepared in distilled water using magnetic stirring. $C_{18}H_{34}O_2$ was added to avoid agglomeration of particles and to defend the particles from impressive oxygen. NH_3 solution was added drop wise under constant stirring for the precipitation of Fe^{3+}.

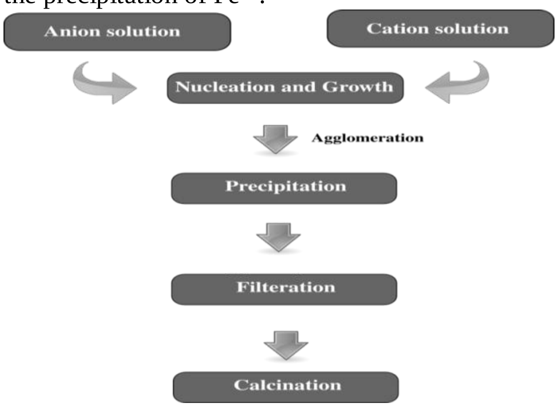

Figure 2 Micro and nano particles synthesis flow chart of CCP method.

I. XRD, D.C. AND DIELECTRIC MEASUREMENTS

As obtained powder samples were first annealed at 350°C and then characterized for phase classification using an X-Ray Diffract meter in 2θ range of 20–70° (at 2°/min). In each fine powdered sample (~0.255 g) was used to prepare cylindrical pellet (13 mm) by putting it in the die under a constant pressure (40 MPa) for 5 min. To avoid outcome of polarization, we applied direct voltage and a clasp switch was used to measure the current in both directions and then averaging was done. Dielectric capacities of freshly prepared pellets were carried out on an IA.

I. RESULTS AND DISCUSSION

XRD analysis of taster Z-CCP, ZC-CCP after calcinations at 350°C (Fig. 3) clearly indicates single phase structure of prepared Fe^{3+}. For the duration of calcinations reaction involving different outrider takes place to obtain the final product. For total reaction and uniform assimilation, it is mandatory to diffuse unlike outrider through extended distances among themselves and therefore higher calcinations temperature is essential in solid state reaction method. In CCP method originator powder keep higher particle than in other technique. As higher particles keep larger surface area and hence react at lower temperatures. Particles of taster prepared via CCP technique particles are spherical and cubic correspondingly.

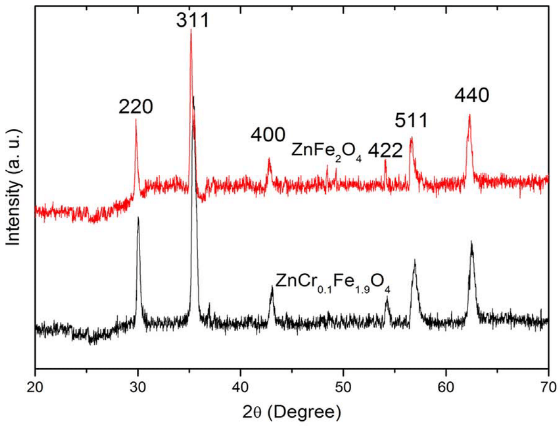

Figure 3 XRD pattern of Z-CCP and ZC-CCP calcites at 350 ℃

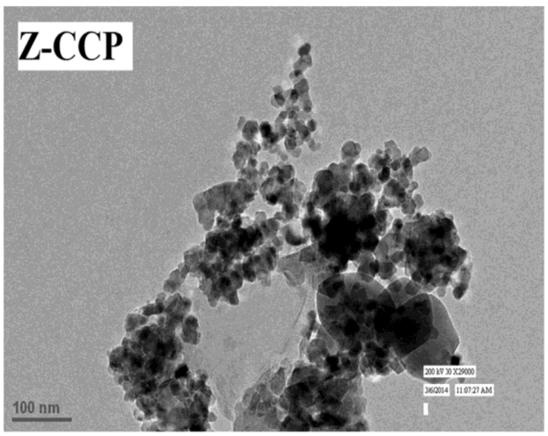

Figure 4 HRTEM micrographs of Z-CCP

II. DIELECTRIC PROPERTIES

i. Dielectric constant

Deviation of dielectric constant (ε′) (for Z-CCP) with frequency at 250°C is given away in Fig.5. It is observed that all these compositions demonstrate the normal dielectric behavior. The high value of dielectric constant at low frequencies is maybe on the basis that of a rate of space charge polarization on account of contact with two differently conducting surface areas. An initial rise in frequency results in reduced mobility of electrons in among Fe^{2+} and Fe^{3+} ions which eliminate the surface charge polarization and at sufficiently high frequencies, electronic actions are unable to synchronize with changing AC field which reduces the dielectric constant. It is observed that with Cr^{3+} substitution, dielectric constant decreases due to a stable oxidation state of Cr^{3+} ions as chromium ions do not take part in conduction but back the electron ireful among Fe^{2+} and Fe^{3+} ions. An analogous type of variation with preparation techniques was observed in Cr^{3+} substituted samples. Fig. 6 shows the temperature dependence of ε′ (at 1 MHz) for ZC prepared by CCP techniques. Raise in ε′ with increasing temperature as clear from Fig. 6 may be mostly due to rise in electron ireful among Fe^{2+} and Fe^{3+} ions.

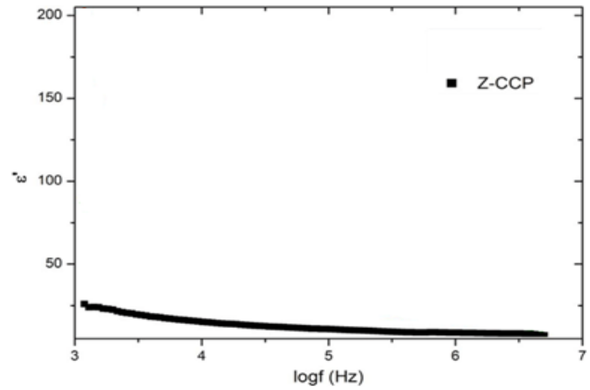

Figure 5 Variation of dielectric constant (**ε**') of Z-CCP

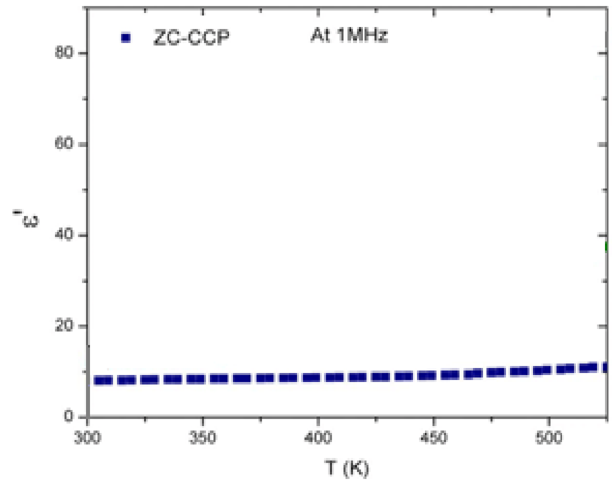

Figure 6 Variation of **ε**' with temperature for ZnCrxFe2-xO4 (x=0.5) sample prepared by CCP at 1 MHz.

ii. DIELECTRIC LOSS

Fig. 7 shows the variation of dielectric loss with frequency for Z-CCP samples at 250°C. The high value of tan δ at low frequencies is endorsed to the high resistivity of grain boundaries which are more effective at lower frequencies. It is because the high resistivity of grain boundaries requires more energy for electron exchange among Fe^{2+} and Fe^{3+} ions so maximizing energy loss. Different to this, at higher frequencies lesser energy is required for electron exchange which minimizes the energy loss. Trends in dielectric loss are analogous to that of a dielectric constant which can be explained in the same manner as that of dielectric constant. There is a decrease in a dielectric loss with Cr^{3+} replacement which can be explained on the same basis as that of dielectric constant. Fig. 7 shows the temperature dependence of tan δ at 1 MHz for ZCF (x=0.5) sample prepared by CCP techniques. The analysis shows that tan δ rises with rising in temperature which is mostly due to improving ireful of charge carriers.

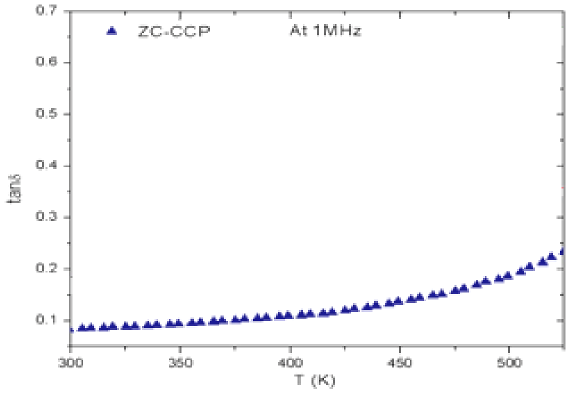

Figure 7 Variation of dielectric loss (tan**θ**) with temperature for $ZnCr_xFe_{2-x}O_4$ (x=0.5) sample prepared by CCP techniques at 1 MHz.

CONCLUSIONS

XRD pattern of nano crystalline Fe^{3+} samples prepared by CCP techniques confirmed single phase cubic spinal structure formation. Solidification of Fe^{3+} sample synthesized by CCP method was found > 75%, as a result, these materials are good for sensing applications. Dielectric constant and dielectric loss were found to rise with rising in temperature. The fe^{3+} sample prepared through CCP method were found to have a small value of dielectric loss and AC conductivity, production these materials appropriate for use in microwave devices.

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