

Effect of sintering on structural and electrical properties of co-precipitated Mn-Zn ferrites

S. Mehmood, F. Zahra and M. A. Rehman

Applied Thermal Physics Laboratory, Department of Physics,
COMSATS Institute of Information Technology, Islamabad 44000, Pakistan

E-mail: marehman@comsats.edu.pk

Abstract. Mn-Zn ferrite is one of the important class of soft ferrites. These are famous for possessing high initial permeability. In the present work, we have studied the effect of sintering on Mn-Zn nano particles. The particles were synthesized using co-precipitation method. The structural characterization of the prepared sample after each sintering step were done by using XRD. The XRD analysis showed the spinel structure. The electrical properties were studied as a function of temperature. It was observed that dielectric constant, loss tangent and AC conductivity varies with respect to temperature. The prepared composition is useful in microwave devices.

1. Introduction

Ferrites nanoparticles are ceramic ferromagnetic materials. These materials are famous due to their wide field of technological applications in microwave setup, TV, and radio sets [1]. Mn-Zn ferrites belong to the soft ferrites. These are renowned for their utilization in modern electronic devices such as EMI suppression, switch mode power supplies and data transmission circuitry [2,3]. There are number of methods by means of which these nano ferrite particles can be prepared such as co-precipitation, hydrothermal and sol-gel mechanical method [4,5]. In order to control the structural and electrical properties of ferrites there are certain important factors, one of them is the method of preparation [6] and the other one is sintering temperature [7]. By controlling these parameters the structural and the electrical properties of a material can be changed.

In the present work $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles were synthesized by using co-precipitation method. The effect of sintering on the structural and the electrical properties of $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ nanoparticles were studied as a function of temperature. The prepared sample was sintered from 350 °C to 500 °C with the step of 50 °C for 2 hours. For a fixed frequency of 1MHz and the temperature ranged from room to 500 °C. AC electrical properties including dielectric constant (ϵ'), loss tangent ($\tan\delta$) and AC conductivity (σ_{AC}) were measured.

2. Experiment

Mn-Zn ferrite of composition $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ was prepared by the co-precipitation method. The precursors of iron nitrate nonahydrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, zinc nitrate hexahydrate $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and manganese nitrate tetrahydrate $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ were weighed in stoichiometry. These precursors were dissolved in de-ionized water through magnetic stirring. The molarity of the solution was kept 0.4M. The solution of NaOH was used to start the process of precipitation. These solutions were heated for 30 minutes using hot plate magnetic stirrer. The temperature was kept constant at 75 ± 5 °C



and the pH was set to 12.5-13. Reaction vessel was covered with a plastic cover to diminish the evaporation of the solution. Finally the de-ionized water were used for washing the precipitates. The particles were dried in an oven at 105 °C and then grinded into fine powder. Pellets were prepared and then sintered at different temperatures.

3. Results and discussion

3.1. Structural analysis

The X-ray diffraction (XRD) of the prepared pellets was done by using an X' pertPro X-ray diffractometer with CuK α radiation ($\lambda=1.5406\text{\AA}$). The analysis of as prepared and sintered pellets were studied. By using the two probe method the dielectric constant (ϵ), loss tangent ($\tan\delta$) and AC conductivity (σ_{AC}) were studied. The XRD pattern of the prepared sample sintered at different sintering temperature were studied as shown in Figure 1.

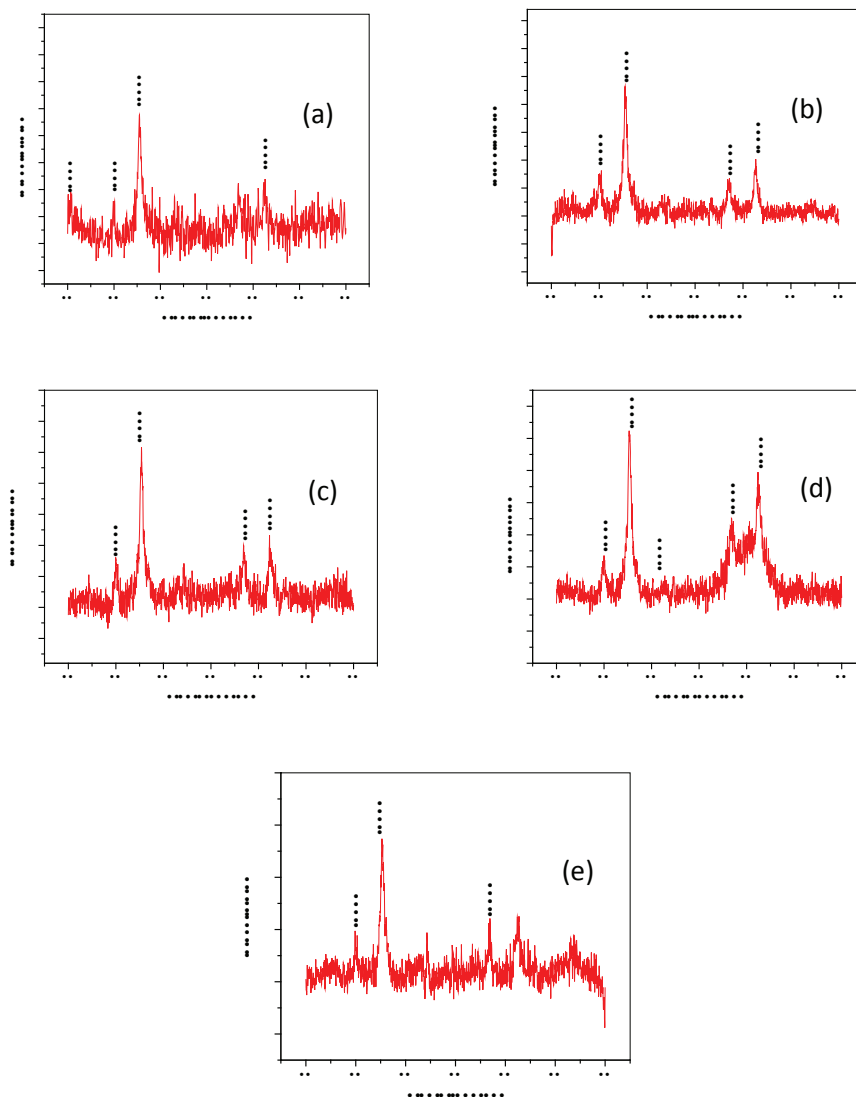


Figure 1. XRD pattern of Mn-Zn ferrite (a) as prepared, sintered at (b) 350 °C (c) 400 °C (d) 450°C (e) 500 °C

The prepared sample showed the spinel structure [8-9]. The sintering temperature was from 350 °C to 500 °C with the step difference of 50 °C. It was observed that by increasing the sintering temperature the lattice constant and the crystallite size of the sample were increased. The lattice constant varies from 8.38Å to 8.42Å. The maximum crystallite size of 19nm was observed at the sintering temperature of 450 °C.

3.2. Electrical properties

The number of grain boundaries would increase if the grain size is small. By increasing the sintering temperature the grain growth increased and the porosity decreased. Due to that the charge carriers faced less pores and the resistivity decreased. In Figure 2, the variation in dielectric constant (ϵ') as a function of temperature is shown.

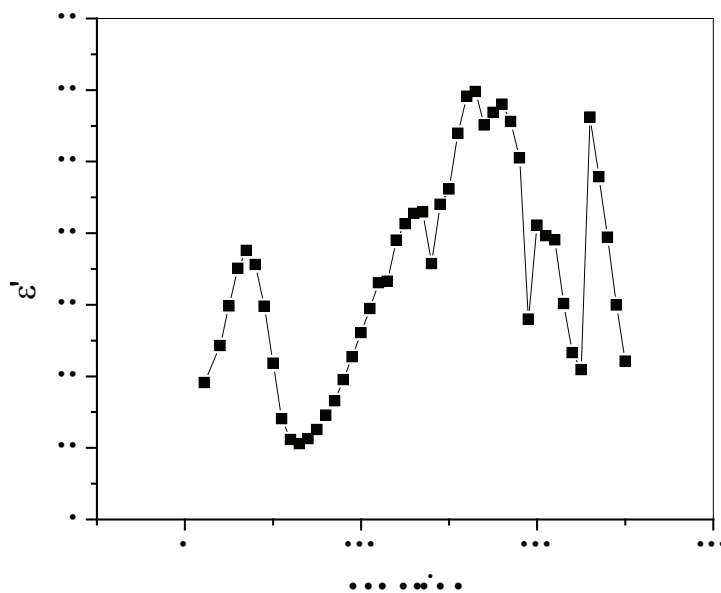


Figure 2. Variation in Dielectric constant (ϵ') as a function of temperature for a fixed frequency of 1 MHz.

The frequency was kept constant at 1MHz and the temperature ranged was from room to 500 °C. It was observed that the dielectric constant of the fully sintered sample increased by increasing temperature but after reaching at a certain value it decreased. The further increase in the value of the dielectric constant was observed at the Curie temperature. The Curie temperature for the prepared composition was 380K [10]. The similar variation of the dielectric constant with respect to temperature was reported earlier [11]. The variation of the dielectric constant due to temperature in the prepared composition is an important characteristic of a disordered perovskite structure with a diffuse phase transition. Due to the disordered arrangement of the cations on one or more crystallographic sites the microscopic heterogeneity in the composition created as a result of which different local Curie points generated [12-13].

In Figure 3, the variation in loss tangent ($\tan \delta$) is shown. It was observed that the loss tangent remained constant for the smaller value of temperature however, for the higher values of temperature it increased. This is due to the thermally activated hopping process. Dielectric properties of any material are strongly dependent on polarization. By increasing temperature interfacial polarization of the material increased which in return increased the value of loss tangent and the dielectric loss.

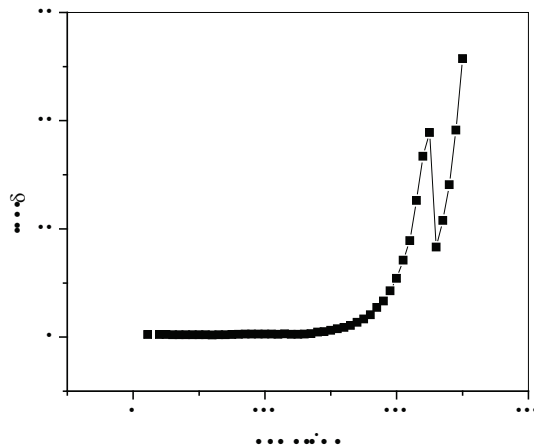


Figure 3. Loss tangent ($\tan \delta$) as a function of temperature for a fixed frequency of 1 MHz

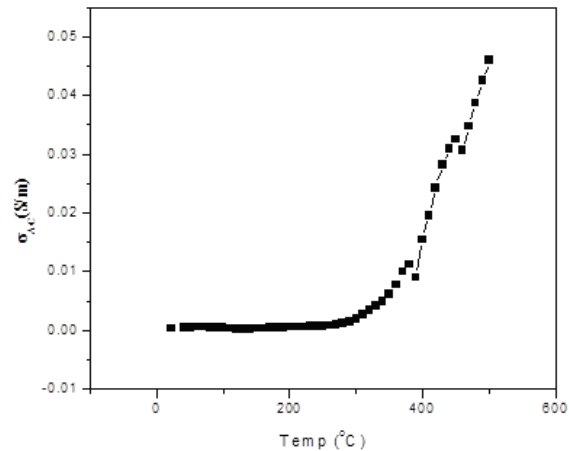


Figure 4. AC conductivity (σ_{AC}) as a function of temperature for a fixed frequency of 1 MHz

In Figure 4, AC conductivity (σ_{AC}) is shown as a function of temperature. The conductivity of a prepared material increased by increasing temperature. This is in accordance with the ion hopping mechanism. Temperature increased the mobility of the charge carriers and as a result conductivity also increased.

4. Conclusions

Phase pure $Mn_{0.5}Zn_{0.5}Fe_2O_4$ was successfully synthesized using co-precipitation method. Increase in the sintering temperature (350 °C to 500 °C with the step of 50 °C for 2 hours) affected the structural as well as the electrical properties. The XRD showed that the crystallite size were in nano meter range for both as prepared and the sintered samples confirming the selection of suitable temperature range for sintering. More variation in the ϵ' at higher temperatures showed the disordered perovskite structure of the prepared samples. The microscopic heterogeneity in the prepared composition was observed which results in the formation of local Curie points. The $\tan \delta$ and the σ_{AC} remained constant for the smaller values of temperature however at higher values these increased, which is in accordance with thermally activated hopping process. The prepared composition can be used in high frequency device applications.

5. References

- [1] Elhiti M A 1999 *J. Magn. Magn. Mater.* 192 305
- [2] Tomar M S, Singh S P, Perez O P, Guzman R P, Calderon E, Ramos C R 2005 *J. Microelectronics*, 36 475
- [3] Arulmurugan R, Vaidyanathan G, Sendhilnathan S, Jeyadevan B 2006 *J. Magn. Magn. Mater.* 298 83
- [4] Taketomi S, Ozaki Y, Kawasaki K, Yuasa S, Miyajima H 1993 *J. Magn. Magn. Mater.* 122 6
- [5] Mergen A, Qureshi A 2009 *J. Alloys Compd.* 478 741
- [6] Barakat M M, Henaish M A, Olofa S A, Tawfik A 1991 *J. Therm. Anal.* 37 605
- [7] Verma A, Goel T C, Mendiratta R G 2000 *Second International Conference on Processing Materials for Properties: The Mineral, Metal & Material Society* pp. 493-497.
- [8] Nasir S, Anis-ur-Rehman M 2011 *Phys. Scr.* 840 25603.
- [9] Jacob B P, Thankachan S, Xavier S, Mohammed E M 2013 *J. Alloys Compd.* 578 314
- [10] Lan N T, Hien T D, Duong N P, Truong D V 2008 *J. Korean Physic. Soc.* 52 1522
- [11] Olofa S A 1994 *J. Magn. Magn. Mater.* 131 103
- [12] Yadav K L, Choudhary R N P 1994 *Mater. Lett.* 19 61
- [13] Bera S, Choudhary R N P 1995 *Mater Lett.* 22 197