Structural and Physical Properties of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ Prepared by Combustion Technique

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Abstract: ***The nanocrrystalline $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrite have been successfully synthesized by combustion technique. The observed particle size of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ is found to be 9 nm. Disc- and toroid-shaped samples are prepared and sintered at various temperatures (1150, 1200, 1250, 1300 and 1350ºC) in air for 1 hour. Structural and surface morphology are studied by X- ray diffraction (XRD) and high resolution optical microscope, respectively. The initial permeability of this composition is characterized by high frequency (10 kHz-120MHz) complex permeability measurements. The influence of microstructure and sintering temperatures on the initial permeability of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrite will be discussed in this paper.***

Keywords: Combustion, XRD, Initial permeability, Nanocrrystalline

1. Introduction

Ferrites are mixed metal-iron oxides adopting a variety of structures which have been used as ceramic ferromagnetic materials in the electronic industry for more than fifty years [1]. Ferrite has tetrahedral A-site and octahedral B-site in AB_2O_4 crystal structure. Various cations can be placed in A-site and B-site to tune its magnetic properties. Depending on A-site and B-site cations, it can exhibit ferromagnetic, anti ferromagnetic, spin (cluster) glass, and paramagnetic behavior [2, 3]. Spinel ferrites are mixed metal-iron oxides ferrites display improved properties by virtue of their unique electronic and crystalline structure which may be harnessed for various applications such as inductors, transformers, antennas, deflection yokes, choke coils, recording heads, electromagnetic interference (EMI) and power transformer [4, 5-9]. The Mn-Zn ferrites adequately suit these demands [10]. Recently, in our laboratory, Hossain et. al [11] have studied grain size dependent permeability of $Ni_{0.5-x}Mn_xZn_{0.5}Fe_2O_4$ ferrites prepared by the standard solid state reaction technique. It was reported that preparation condition of the samples and substitutions of Mn have an influence on the magnetic properties of these ferrites.

2. Materials and Method

2.1. Sample preparation

The analytical grade of $Li(NO_3)_2$, $MnCl_2.4H_2O$, $Cu(NO_3)_2.3H_2O$ and $Fe(NO_3)_3.9H_2O$ are weighted according to the stoichiometric amount and dissolved in ethanol. The mixture was placed in a magnetic stirrer at 70ºC, followed by an ignition, the combustion takes place within a few seconds and fine nanosized powders were precipitated. These powders were crushed and ground thoroughly. The fine powders of the composition are then calcined at 900°C for 5 h for the final formation of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrites nano-particles. Then the fine powders are granulated using Polyvinyl Alcohol (PVA) as a binder and pressed into disk- and toroid-shaped samples. The samples are sintered at various temperatures (1150, 1200, 1250, 1300 and 1350ºC) in air for 1 hour. The temperature ranges for sintering are 5˚C/min for heating, and 10ºC/min for cooling.

3. Result and Discussion

3.1. X-ray Diffraction analysis

The X-ray diffraction (XRD) was performed to verify the formation of spinel structure of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrites, The XRD of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrites sintered at 1200˚C in air for 1h are shown in Figure 1.

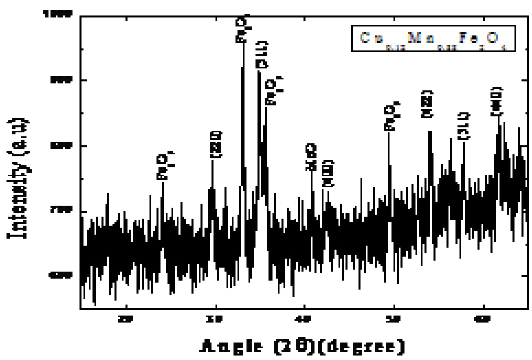

Figure 1: The X-ray diffraction patterns for $Cu_{0.12}Mn_{0.88-2}Fe_2O_4$

The results indicated that the material have a well defined single crystalline phase and formation of cubic spinel structure. Analyzing the XRD patterns it is observed that the positions of the peaks comply with the reported value [12] and some traces of raw materials (Fe_2O_3 and MnO) were found. The average particle size was estimated by using Debye-Scherrer [13] formula from the broadening of the highest intensity peaks (311) of XRD patterns

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(1)}$$

Where D is the average particle size, λ is the wavelength of the radiation used as the primary beam of Cu K_α (λ=1.54178 Å), θ, is the angle of the incident beam in degree and β is the full width at half maximum (FWHM) of the fundamental reflection (311) in radian of the FCC ferrites phase. Debye-Scherer's formula assumes approximation and gives the average particle size if the grain size distribution is narrow and strain induced effects are quite negligible.

3.2. Microstructures and initial permeability

The optical micrographs of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ is shown in Figure 2, sintered at 1150, 1200, 1250, 1300 and 1350˚C, respectively. Average grain sizes (D) of the sample is determined from optical micrographs by linear intercept technique [14].

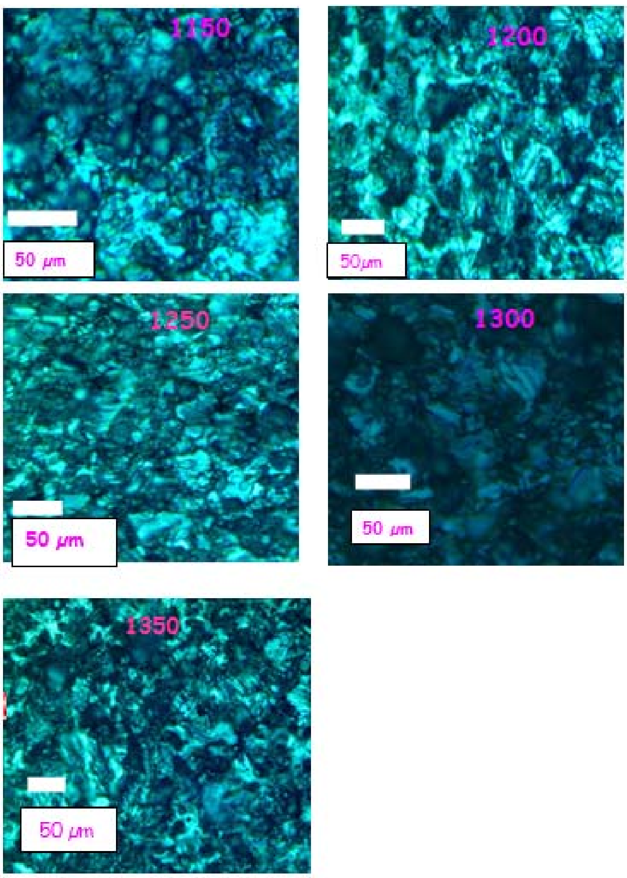

Figure 2: Optical Micrograph of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ sintered at various temperatures

The values of D for various sintering temperature of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ is presented in Table 1.

Table 1: The initial permeability and average grain size of the $Cu_{0.12}Mn_{0.88}Fe_2O_4$ sample sintered at various temperatures.

Sintering temperature Ts (ºC)	*Average Grain Size (µm)*	*Initial permeability (at 10 KHz)*
1150	29	30
1200	36.9	33
1250	26.48	17
1300	32	28
1350	56.67	136

The grain size is significantly dependent on sintering temperature of the sample. It is known that, the mobility of domain walls is greatly affected by the microstructure of ferrites. Therefore, in the present case, variation of the initial permeability may be influenced by its grain size. Initial permeability fairly remain constant up to 1250ºC, (figure 3) Beyond 1250ºC permeability increases greatly because, larger grain size favor domain wall mobility, giving rise to this high permeability. Therefore, the high values of initial permeability (136) in the present sample can be attributed to the high grain sizes (for D=56.67µm) of the sample.

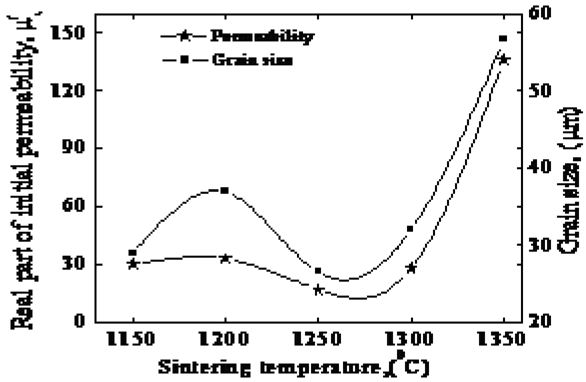

Figure 3: Sintering temperature dependant permeability and grain size of $Cu_{0.12}Mn_{0.88}Fe_2O_4$

4. Conclusion

The $Cu_{0.12}Mn_{0.88}Fe_2O_4$ nanoparticles have been successfully synthesized by the combustion technique. Nanoparticles size has been determined from the broadening of the highest intensity peaks (311) of XRD patterns by using Debye-Scherrer formula. The observed particle size is 9 nm. The XRD patterns confirm that the sample are single phase and form cubic spinel structure. The real part of the initial permeability increases with increasing sintering temperature. With increasing sintering temperature initial permeability of $Cu_{0.12}Mn_{0.88}Fe_2O_4$ ferrite increases by 77%.

5. Suggestions for future work

Magnetization and Neél temperature can be measured for characterization of its magnetic properties. Sintering additives like *Bi_2O_3* and *V_2O_5* can be mixed to promote densification and getting better result in lower sintering temperatures to magnetic materials.

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