STUDY ON STRUCTURAL AND ELECTRICAL PROPERTIES OF MAGNESIUM FERRITE PREPARED BY CO-PRECIPITATION METHOD

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Abstract

Ferrite materials with tunable electrical and magnetic properties are potential candidates for modern technological applications. The magnesium ferrites; $MgFe_2O_4$ having excellent combination of magnetic and dielectric properties can particularly be used for high-frequency applications. The synthesis conditions such as sintering and composition can manage the properties of these materials. On this background, the magnesium ferrite has been prepared by employing the co-precipitation method. The X-ray diffraction (XRD) patterns of prepared samples confirm the formation of a single phase cubic spinel structure. The crystallite size and lattice parameters of the sample have been calculated from XRD data. The FTIR study has been confirmed the presence of the functional group of O-H band and Fe-O band. Scanning Electron Microscopy (SEM) has been employed to observe the morphological features of $MgFe_2O_4$. Response of capacitance has been studied and the dielectric constants show the variation at relatively low frequency and both become stable at relatively higher frequency ranges.

Keywords: $MgFe_2O_4$, co-precipitation, capacitance, XRD, SEM, FTIR

Introduction

Ferrites include a wide range of materials with various crystal structures, compositions and applications. They are ceramic materials, dark gray or black in appearance and very hard and brittle. Spinel ferrites have interesting magnetic and electrical properties. Nowadays, these materials are largely synthesized in nanometric scale for new and improved properties.

The magnetic and electrical properties of spinel ferrites can be tailored for specific device applications by choosing the cation type and cation distribution between tetrahedral (A) and octahedral (B) sites of the spinel lattice. Moreover, the preparation conditions, sintering temperature, sintering time and the method of preparation are other important parameters in the synthesis route. Magnesium ferrites are suitable materials for miniaturizing the size of antennas, along with enhanced properties. The variations of thermal treatment, type of precursors, molar ratio and synthesis route affect the properties of magnesium ferrite and its area of applications.

Experimental Procedure

Magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$, iron nitrate nonahydrate $Fe(NO_3)_3.9H_2O$ and sodium hydroxide NaOH are used as raw materials. Aqueous solutions of analytical graded $Mg(NO_3)_2.6H_2O$, $Fe(NO_3)_3.9H_2O$ have been mixed by magnetic stirring. To obtain smaller size, narrow size distribution and chemically homogeneous ferrite particles, the precipitating reagent (NaOH) has been mixed quickly into the metal solutions. The pH of the solution is maintained at 12.5. For the transformation of metal hydroxides into ferrites, the temperature of the solution has been maintained at 80°C for 40 minutes with constant stirring. Then the solution has been filtered with filter paper and repeatedly washed with distilled water. The washed powder was dried in an electric oven at 100°C for 3 hours to remove water content. The dried powder has been grinded with A-gate mortar and calcined at 900°C for 3 hours. After

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calcination, the phase identification has been conducted by the RIGAKU MULTIFLEX X-ray diffractometer.

Fourier Transform Infrared Spectroscopy (FTIR) spectra of $MgFe_2O_4$ have been recorded in the range 4000-400cm^{-1}. After the samples have been synthesized, the morphological features of the samples have been studied by Scanning Electron Microscope (SEM). The grain size, shape and homogeneity of the samples have been estimated from the SEM images. Again, the sample is made into pellets and final- sintered at 1200°C, 1250°C, 1300°C, 1350°C and 1400°C for 3 hours. After sintering, the phase identification has been conducted by the RIGAKU MULTIFLEX X-ray diffractometer and the morphological features of the samples have been studied by Scanning Electron Microscope (SEM). Dielectric measurements have been done by Fluke LCR meter-189 in the frequency range of 1 kHz to 1 MHz.

Results and Discussions

Structural Analysis of X-ray Diffraction (XRD)

The X-ray diffraction (XRD) spectra of the synthesized samples are taken by the RIGAKU MULTIFLEX X-ray diffractometer. The XRD spectra show that all samples of $MgFe_2O_4$ are formed spinel phase cubic structures. The data has been collected in a 2θ range from 10°C to 70°C. The lattice parameter of $MgFe_2O_4$ sample has been calculated from X-ray lines broadening of the reflections of the (220), (311), (222), (400), (422), (440) and (511) peaks. XRD spectrum of $MgFe_2O_4$ pre-sintered at 900ºC and final-sintered at 1200ºC, 1250ºC, 1300ºC, 1350ºC and 1400ºC are shown in Figure 1 to Figure 6. XRD data of $MgFe_2O_4$ pre-sintered at 900ºC is shown in Table 1 and variations of average lattice constant and average crystallite size with final-sintering temperature are shown in Table 2.

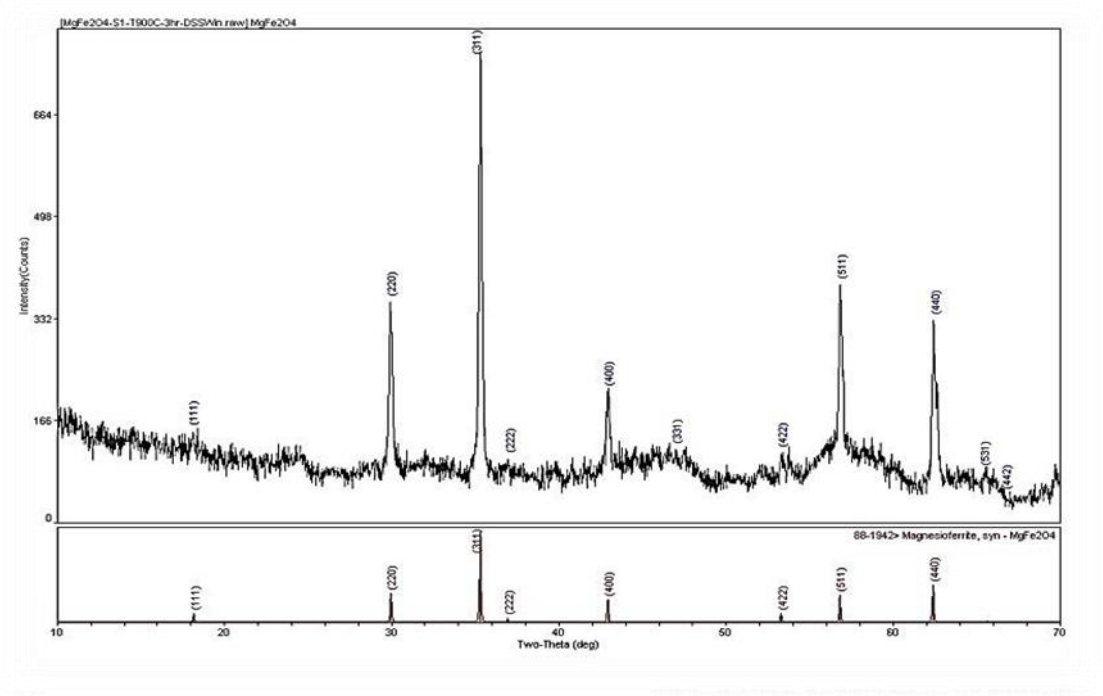

Figure 1 XRD spectrum of $MgFe_2O_4$ pre-sintered at 900ºC

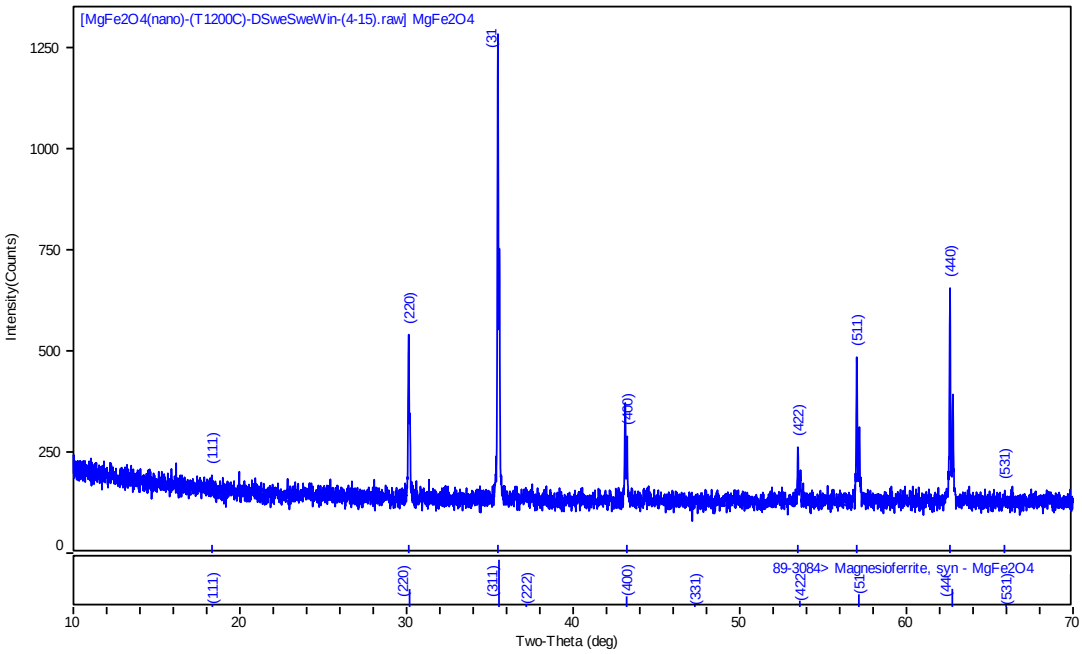

Figure 2 XRD spectrum of $MgFe_2O_4$ final-sintered at 1200ºC

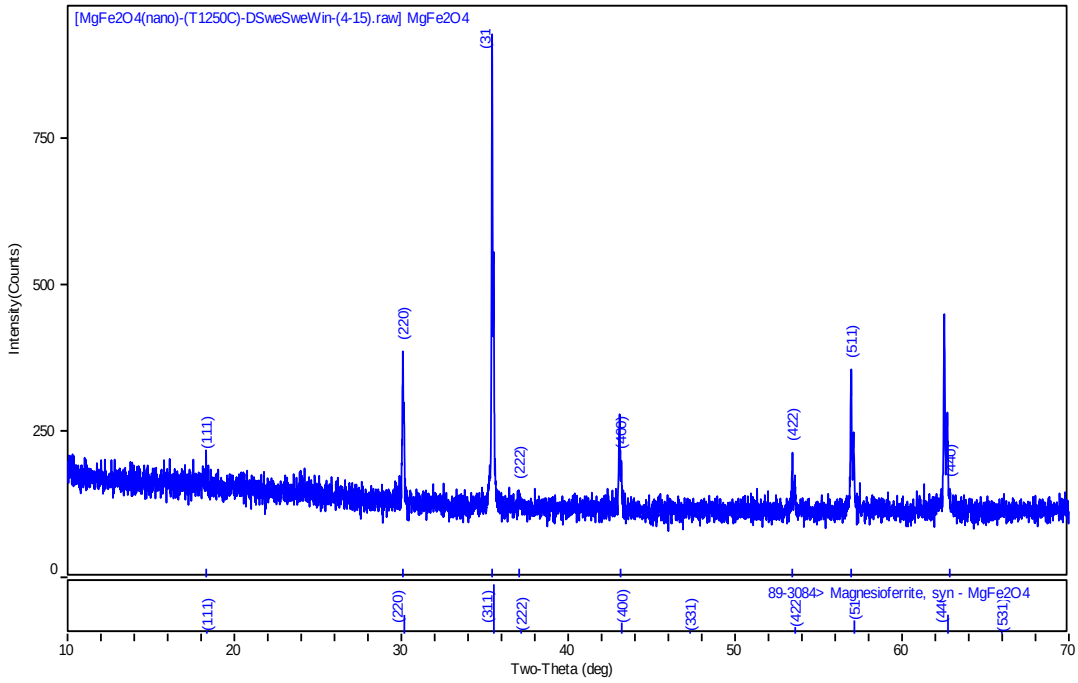

Figure 3 XRD spectrum of $MgFe_2O_4$ final-sintered at 1250ºC

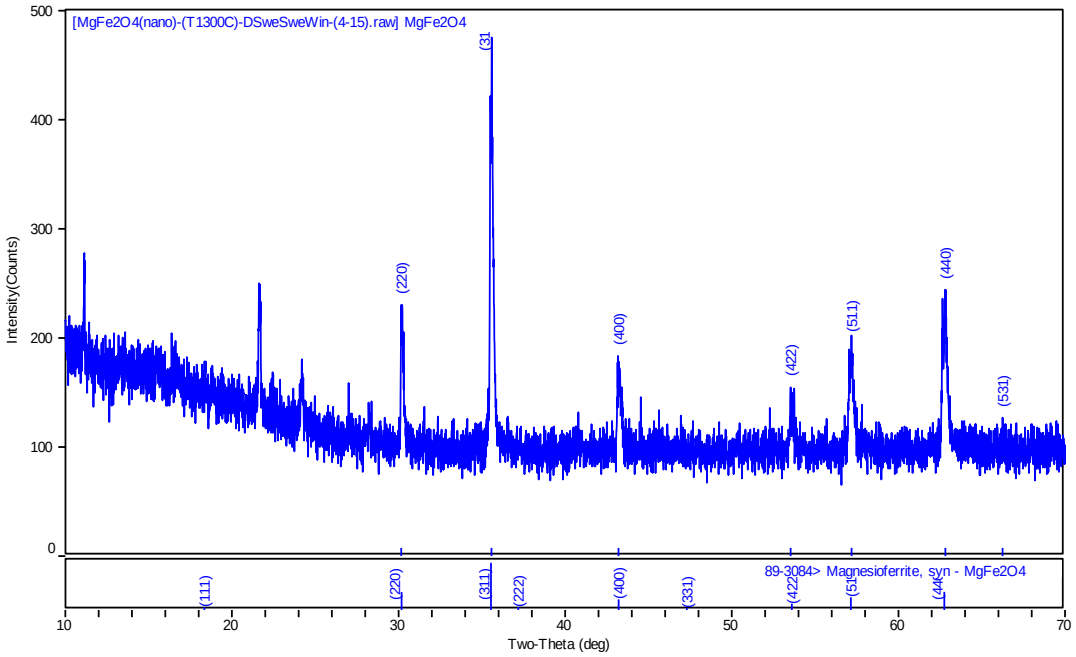

Figure 4 XRD spectrum of $MgFe_2O_4$ final-sintered at 1300ºC

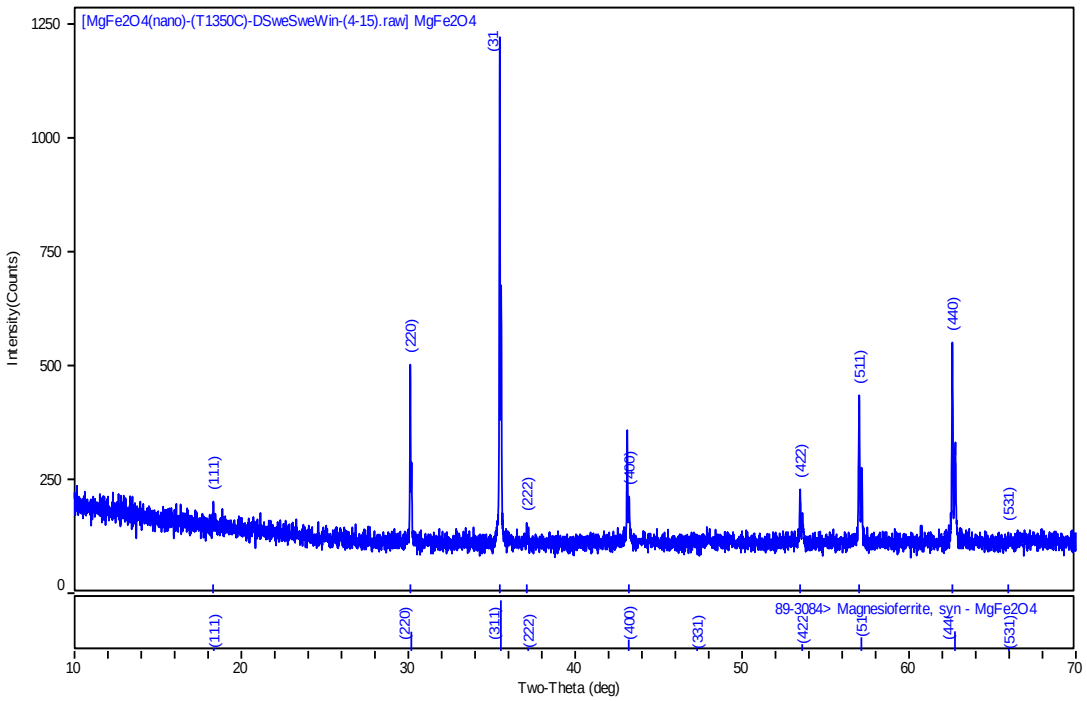

Figure 5 XRD spectrum of $MgFe_2O_4$ final-sintered at 1350ºC

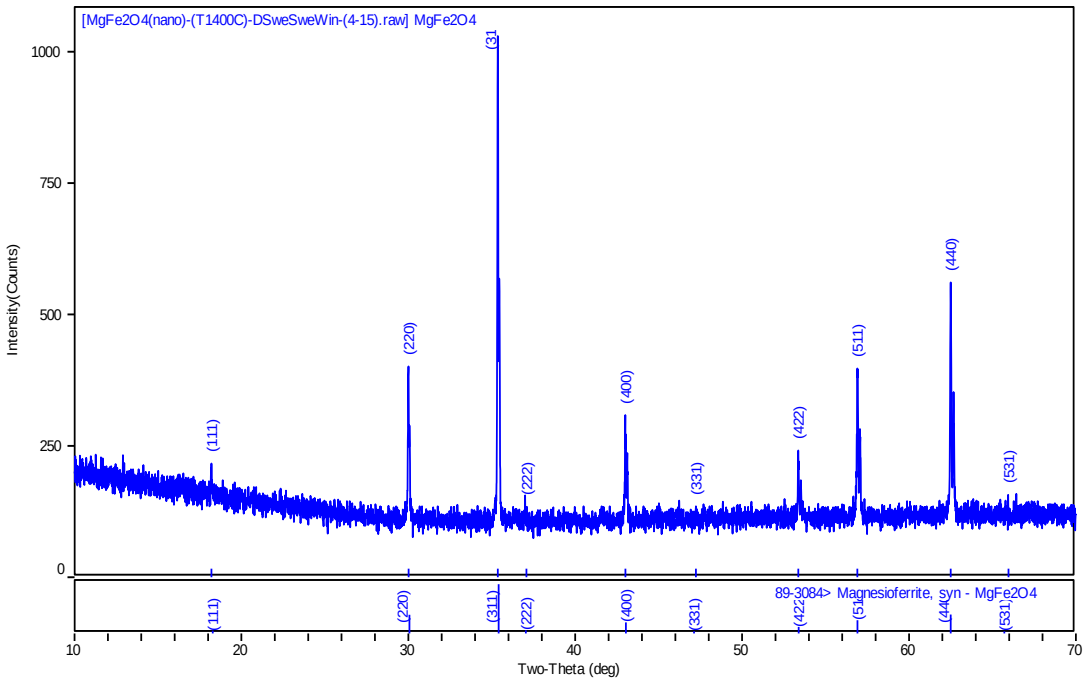

Figure 6 XRD spectrum of $MgFe_2O_4$ final-sintered at 1400ºC

Table 1 XRD data of $MgFe_2O_4$ pre-sintered at 900 ºC

No	2θ (deg)	Diffraction Peak (hkl)	FWHM (deg)	B (rad)	Lattice Constant a (Å)	Crystallite size (nm)
1	30.06	(220)	0.256	0.0045	8.4018	32.13
2	35.38	(311)	0.232	0.0041	8.4067	35.94
3	43.02	(400)	0.288	0.0050	8.4032	29.65
4	53.344	(422)	0.332	0.0058	8.4067	26.77
5	62.50	(440)	0.296	0.0052	8.3993	31.39
6	56.92	(511)	0.275	0.0048	8.3985	32.86
Average					8.4027	31.46

Table 2 Variation of average lattice constant and average crystallite size with final-sintering temperature

Final-sintering temperature (ºC)	Average lattice constant (Å)	Average crystallite size (n m)
1200	8.3808	57.60
1250	8.3847	48.10
1300	8.3637	37.14
1350	8.3816	67.35
1400	8.4051	72.10

Study on Microstructure and Surface Morphology of $MgFe_2O_4$

The microstructure and morphology play the important roles in determining magnetic and electrical transport properties. These studies for the materials are essential in order to understand the relationship between their processing parameters as well as the behavior when they are used in practical applications. Microstructures of the sintered $MgFe_2O_4$ have been analyzed by a high resolution scanning electron microscope (SEM). The SEM images of $MgFe_2O_4$ final-sintered at different temperatures are given in Figure 6 (a), (b), (c), (d) and (e). The variation of average grain size with the sintering temperature is shown in Table 2.From SEM images, it is confirmed that the microstructure is composed of regular grains. Moreover, the surface morphology represents a homogeneous structure. There is no impurity in the surface of $MgFe_2O_4$. With the increasing temperature, the grains become bigger and the average grain size increases from 0.95 μm to 1.60 μm. The pores are rarely visible between the grain boundaries and within the grain.

Table 3 Variation of average grain size with sintering temperature

Final-sintering temperature (ºC)	Average grain size (μ m)
1200	0.95
1250	1.03
1300	1.04
1350	1.38
1400	1.60

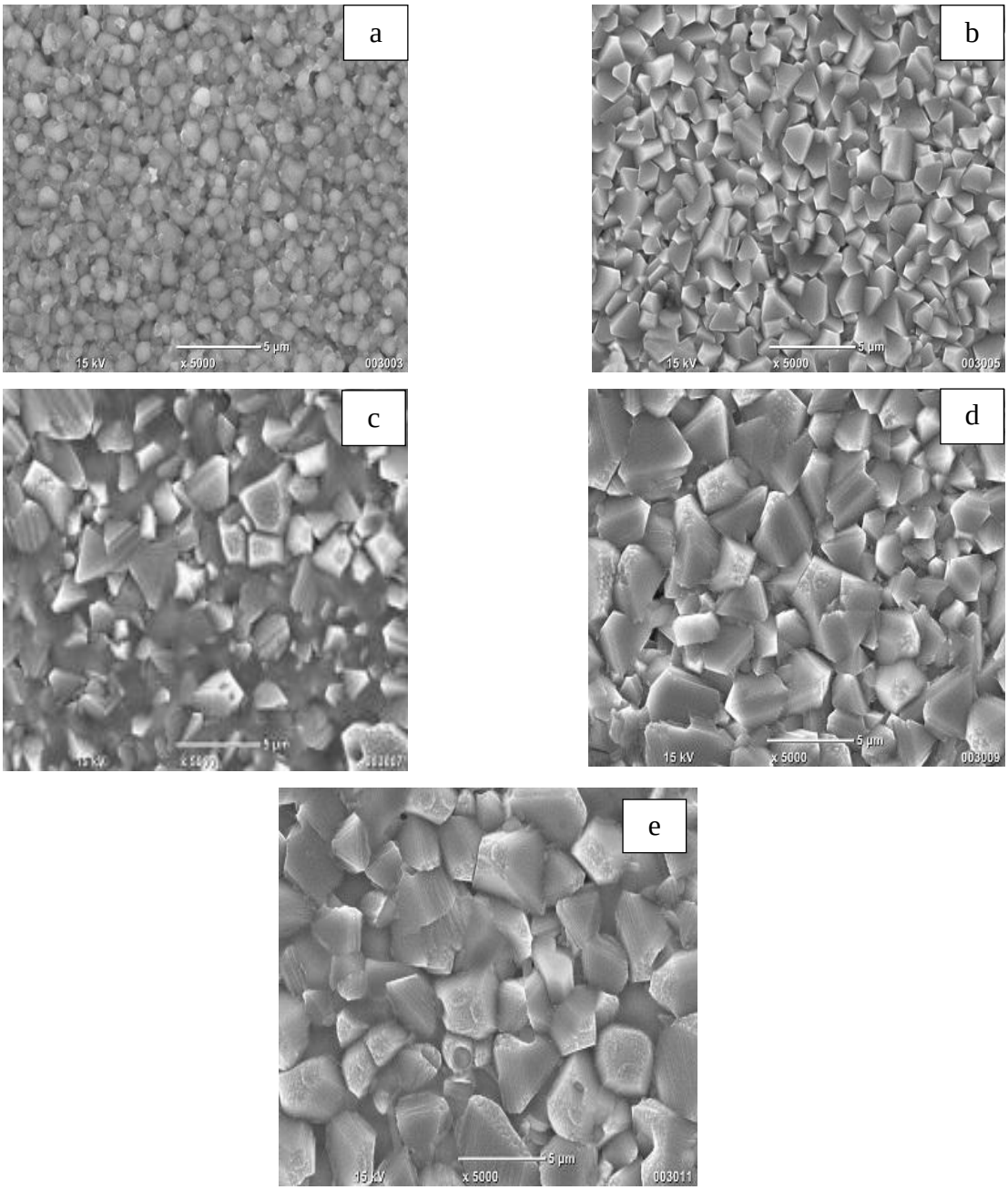

Figure 7 SEM micrographs of $MgFe_2O_4$ final-sintered at (a) 1200ºC (b) 1250ºC (c) 1300ºC (d) 1350ºC (e) 1400**ºC**

Study on the Molecular vibration

The molecular vibrations have been examined by Fourier Transform Infrared Spectroscopy (FTIR). The strength of IR peak is roughly dependent on the change in dipole moment during vibration. The FTIR spectra of $MgFe_2O_4$ powder which is final sintered at 1200ºC, 1250ºC, 1300ºC, 1350ºC and 1400ºC are presented in Figure 8 to Figure12. The vibrational frequencies of the chemical bonds in the $MgFe_2O_4$ nanoparticles can be assigned from FTIR spectra which were recorded in the region 400 cm^{-1} to 4000 cm^{-1}. This vibration is an indicative of formation of spinel ferrite structure. Therefore, it can be said that FTIR analysis strongly supports the XRD result.

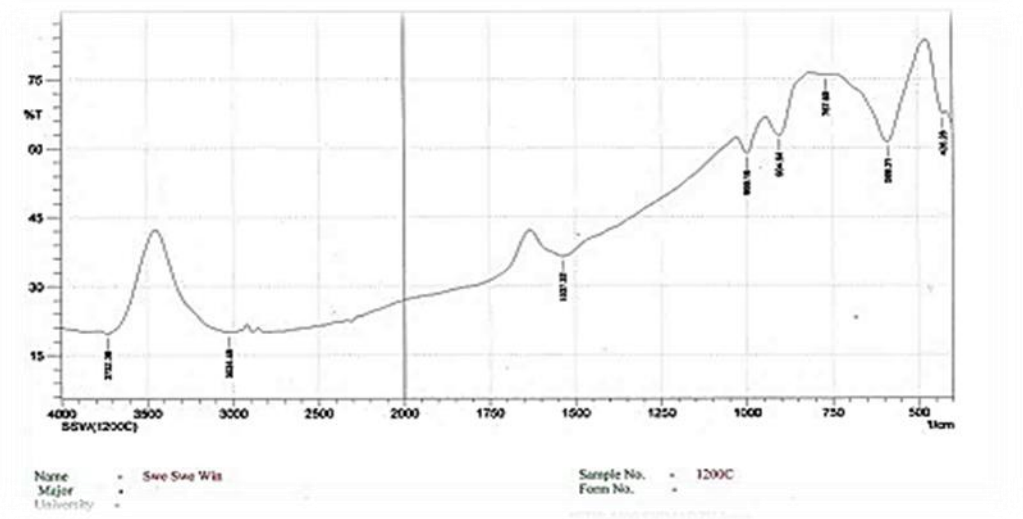

Figure 8 FTIR spectrum of $MgFe_2O_4$ final -sintered at 1200ºC

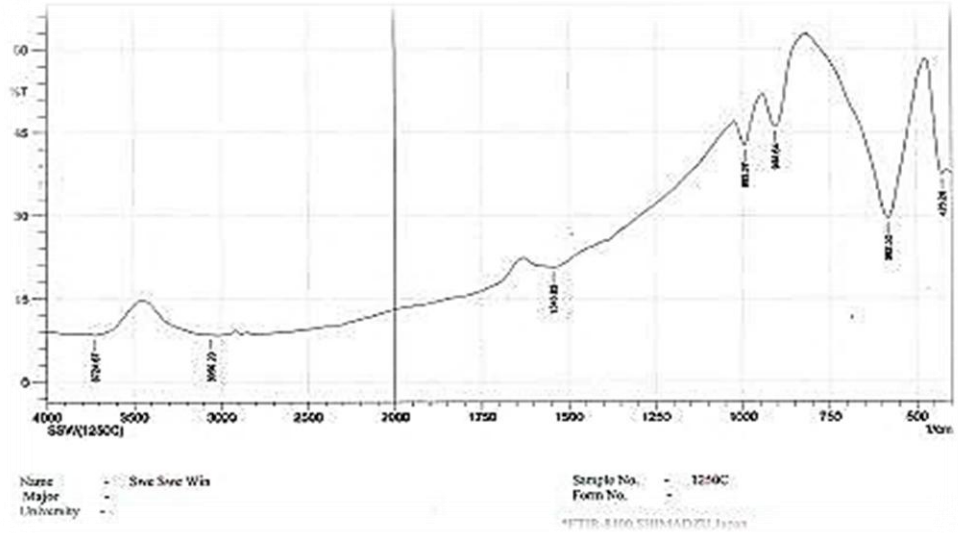

Figure 9 FTIR spectrum of $MgFe_2O_4$ final- sintered at 1250ºC

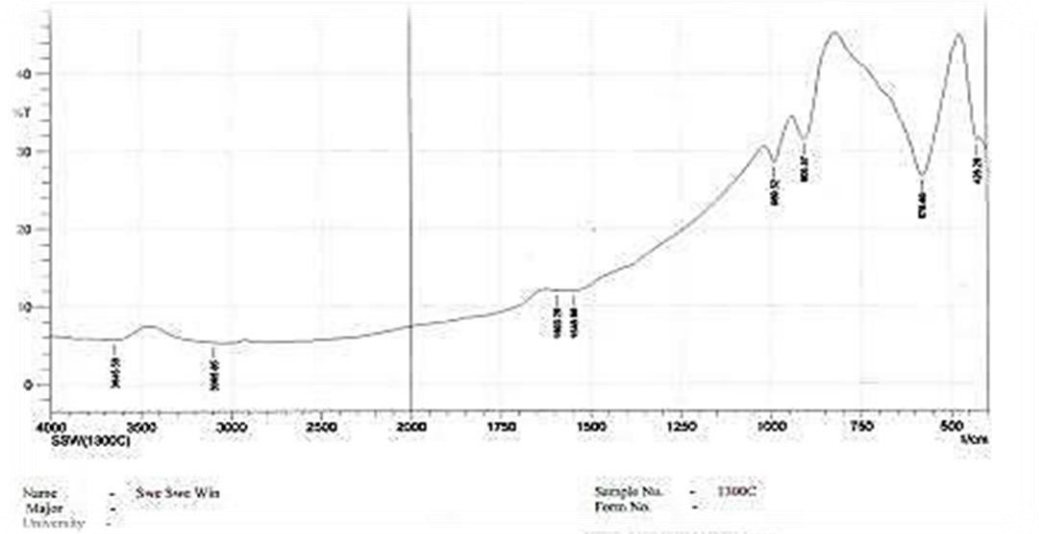

Figure 10 FTIR spectrum of $MgFe_2O_4$ final -sintered at 1300ºC

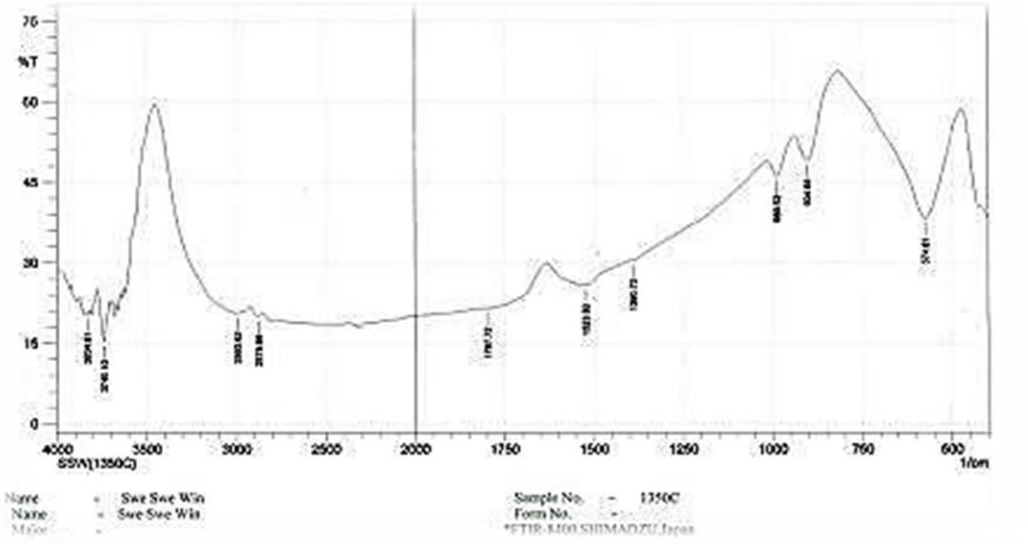

Figure 11 FTIR spectrum of $MgFe_2O_4$ final- sintered at 1350ºC

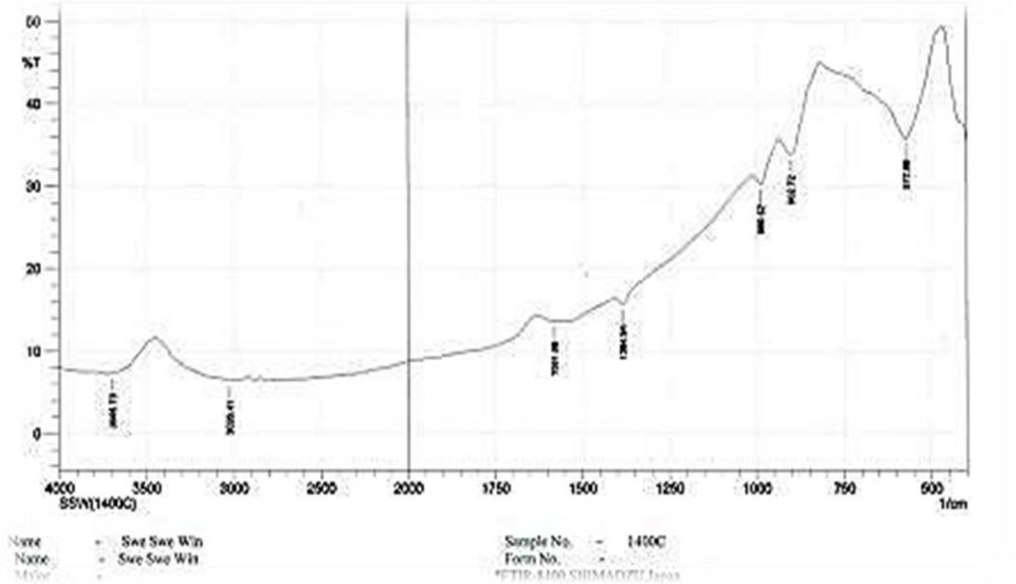

Figure 12 FTIR spectrum of $MgFe_2O_4$ final- sintered at 1400ºC

Study on Frequency Dependent Dielectric Constant

The electrical properties of samples have been measured using Fluke -189 LCR meter in the frequency range of 1kHz to 1 MHz. The dielectric constant (ε_r) was calculated by the relation

$$\varepsilon_r = \frac{Cd}{\varepsilon_0 A} \qquad (1)$$

where, d is the thickness, C is the capacitance, ε_0 is the permittivity of free space and A is the cross-sectional area of the pellet. The dielectric constant of samples has been determined from experimentally obtained capacitance values. The dielectric properties of ferrites are dependent upon several factors such as method of preparation, chemical composition, grain structure and grain size.

The variations of dielectric constant with frequency are shown in Figure 13. From these graphs, the values of dielectric constant of the samples show a variation in lower frequency

region. The variation of dielectric constant in lower frequency region is due to the grain boundary defects of ferrite and the dielectric constant becomes stable in higher frequency region. In the lower frequency region, the dipole polarization is hindered by the grain boundaries; however this effect can be overcome in the higher frequency region.

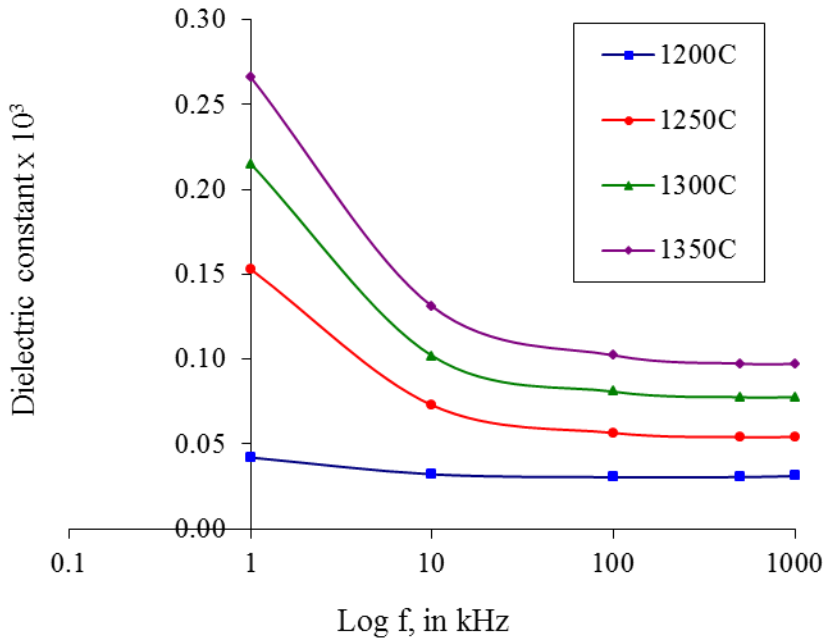

Figure 13 The variations of dielectric constant with frequency

Table 4 The variation of frequency dependent dielectric constant with temperature

Frequency (kHz)	Dielectric constant x10^3			
	1200℃	**1250℃**	**1300℃**	**1350℃**
1	0.0360	0.0600	0.1000	0.3900
10	0.0280	0.0130	0.0400	0.1600
100	0.0137	0.0070	0.0150	0.0450
500	0.0061	0.0010	0.0050	0.0160
1000	0.0140	0.0110	0.0120	0.0200

Conclusion

Magnesium ferrite, $MgFe_2O_4$, has been successfully prepared by co-precipitation method in this work. As the starting powder characteristics are strongly determined the product, the raw materials have been selected to ensure their purities. The XRD characterization of the sintered ferrite has been identified the formation of the typical cubic spinel structure. The lattice parameters and crystallite size have been calculated for further characterization. The sizes of crystallites in the sample have been evaluated by using the FWHM of the most intense peaks and the results confirm the formation of ferrite particles. The average crystallite size of $MgFe_2O_4$ decreased until nanometer range due to the synthesis by chemical co-precipitation technique. The average crystallite size is 31.6 nm when the sample is pre-sintered at 900℃ and becomes bigger at final-sintering temperatures. The typical lattice parameter values from 8.3637 Å to 8.4027 Å

have confirmed the consistency of synthesis route in this work. Based on the SEM micrographs, when the samples have been sintered at higher temperature, subsequent grain growth has taken place. At the final-sintering temperatures, the average grain size increases from 0.95 μ m to 1.60 μ m. The values of dielectric constant of the samples show a variation at relatively lower frequency range due to the grain boundary defects of ferrite and the dielectric constant becomes stable at relatively higher frequency range.

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