PREPARATION AND STRUCTURAL PROPERTIES OF COPPER FERRITE BY SOL-GEL METHOD

Su Su Tha[1] & Hsan Htoo[2]

Abstract

Copper ferrite materials were synthesized by sol-gel method. The X-ray diffraction (XRD) confirmed the phase formation and the structural characterization of these samples. The lattice parameter "a" of cubic structure was slightly decreased with increasing sintering temperature. The crystallite size of these samples was also evaluated. The grain size and grain boundary of ferrite samples were also analyzed by scanning electron microscope (SEM).

Keywords: *Copper Ferrite,* XRD and SEM

Introduction

Magnetic materials are widely used as components in various applications of industrial and medical equipments. Ferrites have emerged as novel materials with vast technological and scientific interest considering their brilliant physical properties such as reliable magnetization, high coercive force, large magnetocrystalline anisotropy as well as remarkable chemical stability and low cost. Since their discovery in the 1950 the degree of interest in them has grown enormously, and is still growing today. The ferrite materials may be classified into three different classes; spinel ferrites, garnet ferrites and hexagonal ferrites. The ferrites used for magnetic recording, data storage materials, radar absorbing materials due to their strong magnetic losses at the range of GHz frequency, magnetoelectric applications. These materials have a potential application at high frequency range due to their very low electrical conductivity, fairly large magneto-crystalline anisotropy, relatively large saturation magnetization, mechanical hardness, excellent chemical stability and low production costs [Gholamreza Nabiyouni et al./ JNS 3(2013)].

Various methods are used to synthesize ferrite nanoparticles, such as: combustion, mechano-chemical method, redox process, forced hydrolysis, co-precipitation, sol–gel, hydrothermal, polymer combustion method (PC), solid state method (SS), micro-emulsion, sonochemical, electrochemical and thermal decomposition method. Among the various methods, the sol-gel method is probably opted for homogeneity and improved characteristics.

The structure properties and the confirmation of cubic inverse spinel phase of copper ferrite nanoparticles are studied in the present work using the X-ray diffraction (XRD). The investigation of surface morphology of ferrite samples were carried out by using scanning electron microscope (SEM).

Materials and Method

Experimental Procedure

The nanoparticles composition of $CuFe_2O_4$ chemical were synthesized by the sol-gel method. The chemical precursors used in reaction were iron (III) nitrate nonahydrate [$Fe(NO_3)_3.9H_2O$], copper nitrate [$Cu(NO_3)_2$], and de-ionized water. In first the metal nitrates

[1] PhD Candidate, Lecturer, Department of Physics, Mawlamyine University
[2] Dr, Lecturer, Universities Research Centre, University of Yangon

dissolved in de-ionized water in required molar ratios. The obtained solution was heated using a magnetic stirrer on 70°C with stirring until forming the gel. Then the temperature was remained at 75°C until preparation a dry gel. Afterwards, the copper ferrite powder was heated in different temperatures such as 500°C, 600°C, 700°C, 800°C and 900°C for 1hr. Flow diagram of the sample preparation procedure of copper ferrite is given in Figure 1.

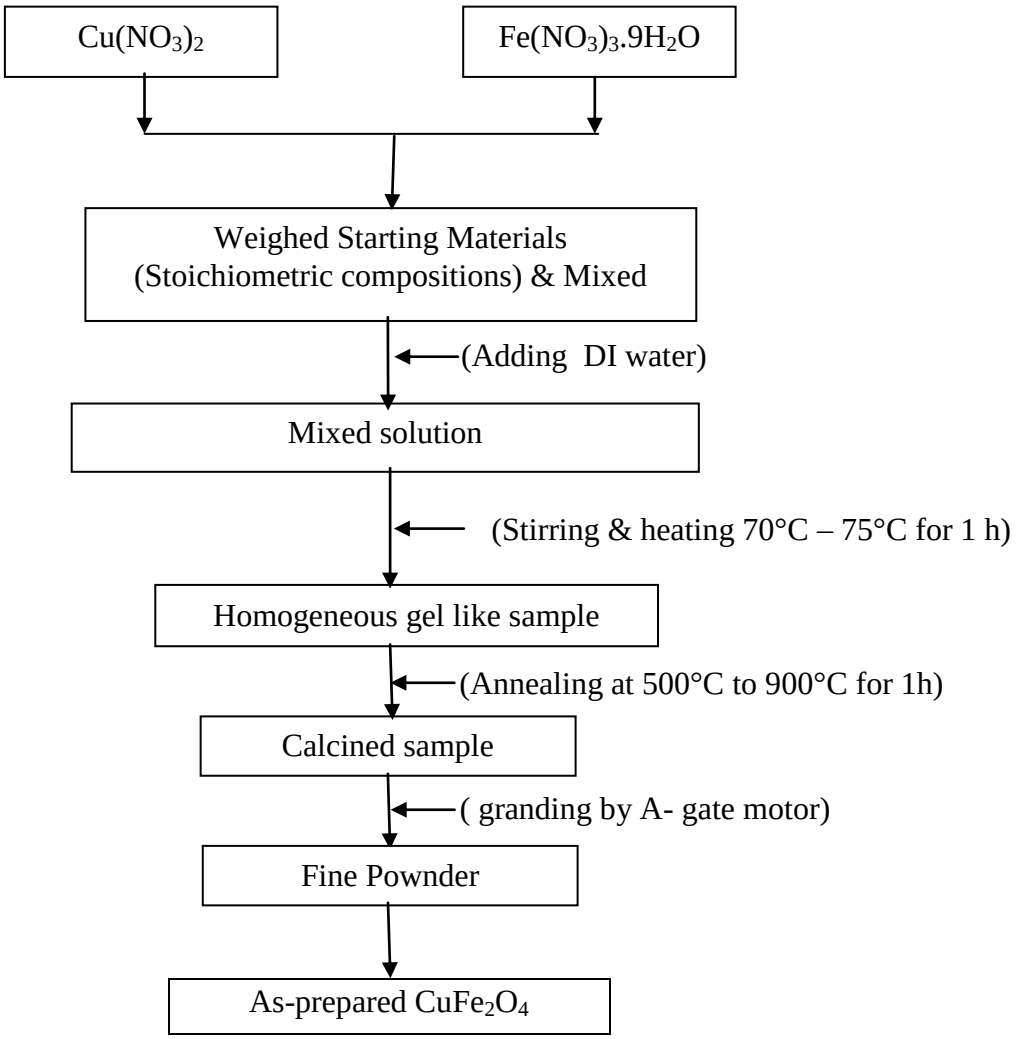

Figure 1 Flow diagram of the sample preparation procedure of $CuFe_2O_4$

Results and Discussion

Structure Analysis

XRD analysis was carried out to study the crystal structure and properties of $CuFe_2O_4$. It was performed using monochromatic Cu-K_α radiation (λ = 1.54056 Å) operated at 40 kV (tube voltage) and 40 mA (tube current). Specimen was scanned from 10º to 70º in diffraction angle, 2θ with step-size of 10º per minute. The observed XRD profiles of $CuFe_2O_4$ were shown in following figures. The reference profile was 25-0283 > $CuFe_2O_4$ of International Centre for Diffraction Data (ICDD) library file. The dominant peak are (111), (220), (311), (222), (400), (511) and (440) reflection. The crystal structure of $CuFe_2O_4$ is Cubic structure. Comparison of crystallite size of samples between various temperatures were investigated and presented in the following table. The crystallite size can be measured as following Debye-Scherrer formula.

$$D = \frac{k\,\lambda}{B\,\cos\,\theta}$$

Where, D = Crystallite size (nm). λ = The wavelength of X-ray use (1.5405 Å)

B = Full Width Half Maximum of dominant peak (radians)

θ = Angle of diffraction (radians), k = Scherrer constant

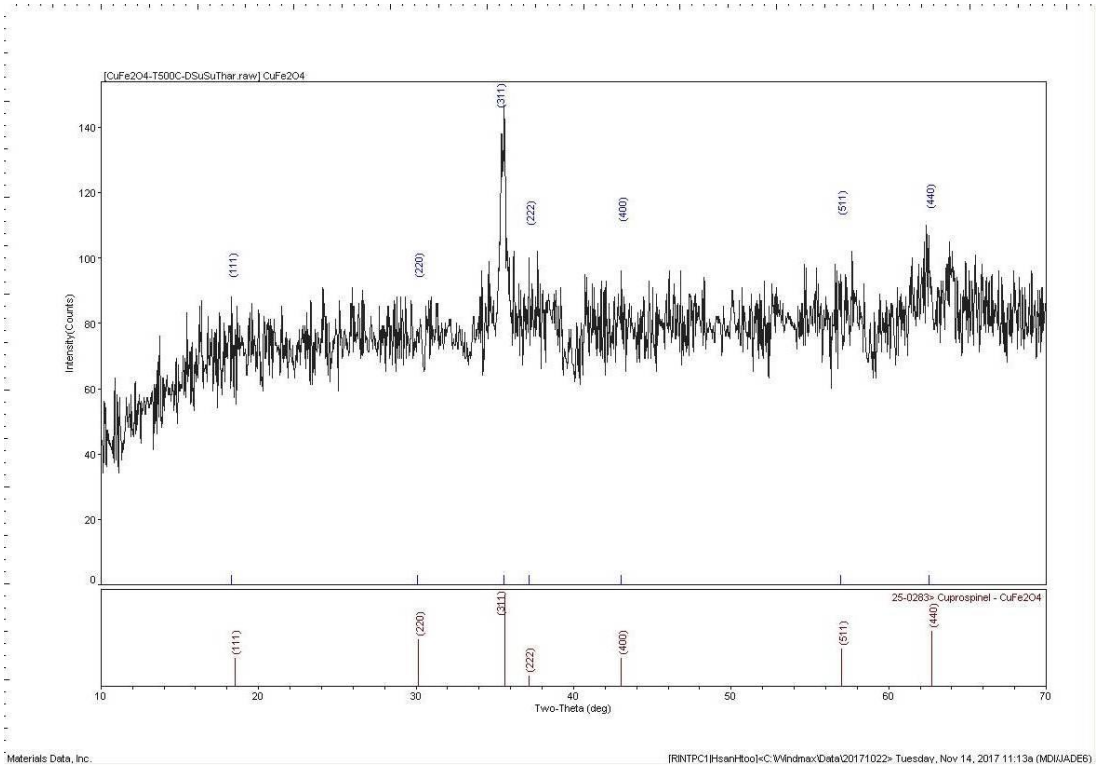

Figure 2 (a) XRD pattern for $CuFe_2O_4$ ferrite sample (500°C)

Peak Search Report (7 Peaks, Max P/N = 2.9) - [CuFe2O4-T500C-DSuSuTh

Close | Print | Save | Copy | Erase | Customize | Rescale | Help

#	2-Theta	d(Å)	(h k l)	BG	Height	I%	Area	I%	FWHM	XS(Å)	P/N
1	18.308	4.8419	(1 1 1)	62	30	42.9	233	13.2	0.132	933	1.6
2	30.098	2.9667	(2 2 0)	68	24	34.3	408	23.1	0.289	303	1.3
3	35.593	2.5203	(3 1 1)	77	70	100.0	1768	100.0	0.429	200	2.9
4	37.174	2.4166	(2 2 2)	79	29	41.4	108	6.1	0.063	>1000	1.4
5	43.021	2.1007	(4 0 0)	71	38	54.3	462	26.1	0.207	472	1.8
6	56.951	1.6156	(5 1 1)	76	35	50.0	421	23.8	0.204	507	1.7
7	62.558	1.4836	(4 4 0)	79	34	48.6	765	43.3	0.382	252	1.6

Figure 2 (b) XRD result data of Peak Search Report for $CuFe_2O_4$ ferrite sample (500°C)

Peak ID Extended Report (7 Peaks, Max P/N = 2.9) - [CuFe2O4-T500C-DSuSu

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#	2-Theta	d(Å)	Area	Area%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.308	4.8419	233	13.2	Cuprospinel	4.7951	30.0	(1 1 1)	18.488	0.180
2	30.098	2.9667	408	23.1	Cuprospinel	2.9619	50.0	(2 2 0)	30.147	0.050
3	35.593	2.5203	1768	100.0	Cuprospinel	2.5184	100.0	(3 1 1)	35.620	0.028
4	37.174	2.4166	108	6.1	Cuprospinel	2.4183	10.0	(2 2 2)	37.148	-0.026
5	43.021	2.1007	462	26.1	Cuprospinel	2.1009	30.0	(4 0 0)	43.017	-0.005
6	56.951	1.6156	421	23.8	Cuprospinel	1.6135	40.0	(5 1 1)	57.030	0.079
7	62.558	1.4836	765	43.3	Cuprospinel	1.4794	60.0	(4 4 0)	62.753	0.196

Figure 2 (c) XRD result data of Peak ID Report for $CuFe_2O_4$ ferrite sample (500°C)

Calculate Lattice Constants from Peak Locations and Miller Indices - [CuFe2O4-T50...

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18.308	4.8419	1	1	1	8.3864		
30.098	2.9667	2	2	0	8.3910		
35.593	2.5203	3	1	1	8.3588		
37.174	2.4166	2	2	2	8.3714		
43.021	2.1007	4	0	0	8.4029		
56.951	1.6156	5	1	1	8.3947		
					Average Lattice Constants =	8.3842	
					αβγ =	90.0	90.0	90.0

Higher-Order Peaks | Hide ESD Values | Help | Apply

Internal Standard d= 0.0000 2T(C)= 0.0 2T(O)= 1.0

Figure 2 (d) XRD result data of Structure Report for $CuFe_2O_4$ ferrite sample (500°C)

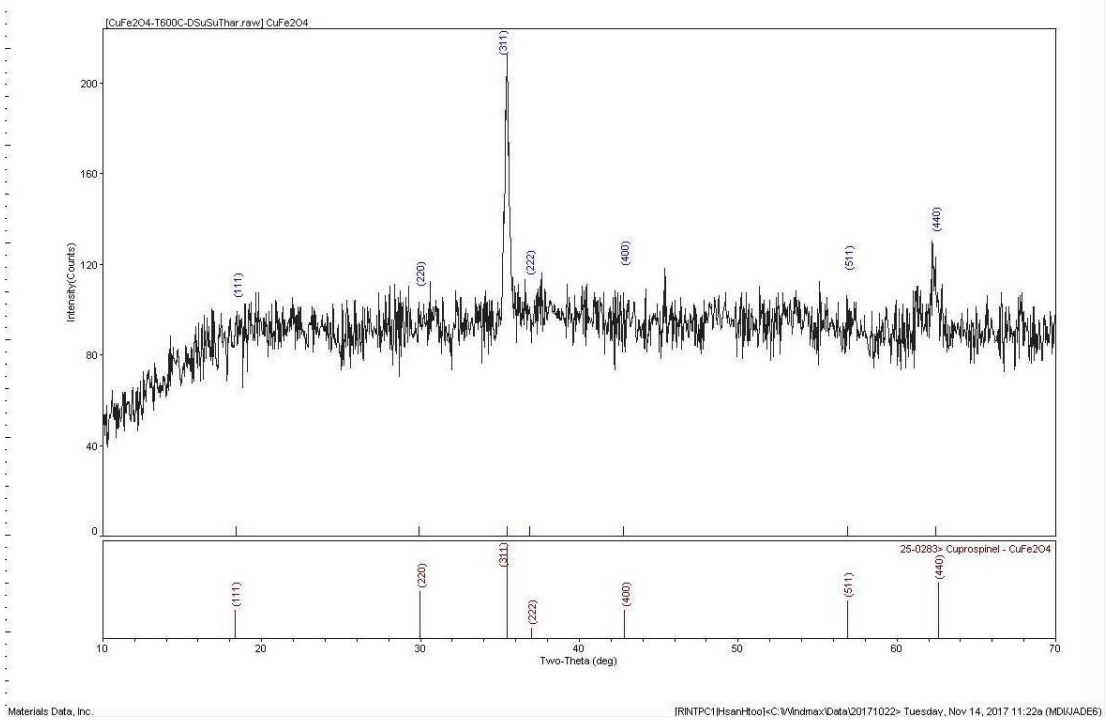

Figure 3 (a) XRD pattern for $CuFe_2O_4$ ferrite sample (600°C)

Peak Search Report (7 Peaks, Max P/N = 4.5) - [CuFe2O4-T600C-DSuSuT

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#	2-Theta	d(Å)	(h k l)	BG	Height	I%	Area	I%	FWHM	XS(Å)	P/N
1	18.406	4.8162	(1 1 1)	75	27	19.9	662	25.9	0.417	199	1.3
2	29.916	2.9843	(2 2 0)	89	18	13.2	108	4.2	0.102	>1000	0.9
3	35.453	2.5299	(3 1 1)	88	136	100.0	2554	100.0	0.319	275	4.5
4	36.849	2.4372	(2 2 2)	96	16	11.8	94	3.7	0.100	>1000	0.8
5	42.784	2.1118	(4 0 0)	83	33	24.3	808	31.6	0.416	211	1.5
6	56.884	1.6173	(5 1 1)	85	29	21.3	104	4.1	0.061	>1000	1.4
7	62.419	1.4865	(4 4 0)	92	39	28.7	526	20.6	0.229	450	1.7

Figure 3 (b) XRD result data of Peak Search Report for $CuFe_2O_4$ ferrite sample (600°C)

Peak ID Extended Report (7 Peaks, Max P/N = 4.5) - [CuFe2O4-T600C-DSuSu

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#	2-Theta	d(Å)	Area	Area%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.406	4.8162	662	25.9	Cuprospinel	4.8393	30.0	(1 1 1)	18.318	-0.088
2	29.916	2.9843	108	4.2	Cuprospinel	2.9783	50.0	(2 2 0)	29.977	0.062
3	35.453	2.5299	2554	100.0	Cuprospinel	2.5301	100.0	(3 1 1)	35.450	-0.003
4	36.849	2.4372	94	3.7	Cuprospinel	2.4290	10.0	(2 2 2)	36.978	0.129
5	42.784	2.1118	808	31.6	Cuprospinel	2.1089	30.0	(4 0 0)	42.847	0.063
6	56.884	1.6173	104	4.1	Cuprospinel	1.6179	40.0	(5 1 1)	56.860	-0.024
7	62.419	1.4865	526	20.6	Cuprospinel	1.4830	60.0	(4 4 0)	62.583	0.164

Figure 3 (c) XRD result data of Peak ID Report for $CuFe_2O_4$ ferrite sample (600°C)

Calculate Lattice Constants from Peak Locations and Miller Indices - [CuFe2O4-T60...

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18.406	4.8162	1	1	1	8.3419		
35.453	2.5299	3	1	1	8.3906		
42.784	2.1118	4	0	0	8.4473		
56.884	1.6173	5	1	1	8.4038		
62.419	1.4865	4	4	0	8.4091		

Higher-Order Peaks — Average Lattice Constants = 8.3986

Hide ESD Values — Help | Apply — αβγ = 90.0 | 90.0 | 90.0

Internal Standard — d= 0.0000 — 2T(C)= 0.0 — 2T(O)= — 1.0

Figure 3 (d) XRD result data of Structure Report for $CuFe_2O_4$ ferrite sample (600°C)

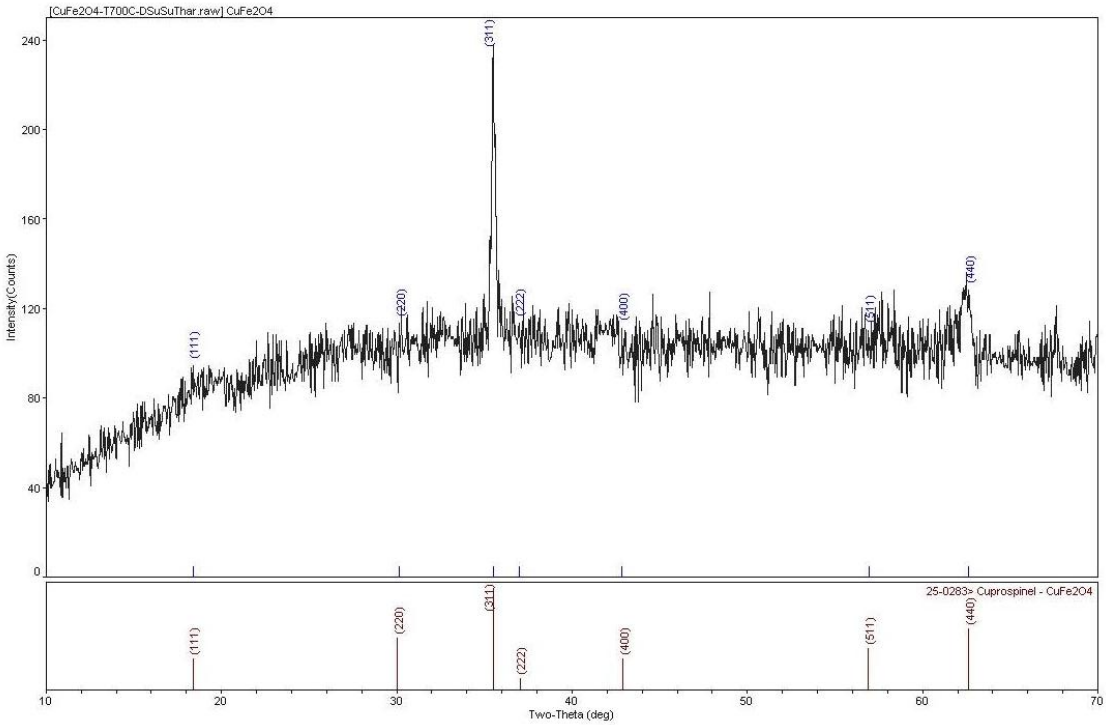

Figure 4 (a) XRD pattern for $CuFe_2O_4$ ferrite sample (700°C)

Peak Search Report (7 Peaks, Max P/N = 4.5) - [CuFe2O4-T700C-DSuSu

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#	2-Theta	d(Å)	(h k l)	BG	Height	I%	Area	I%	FWHM	XS(Å)	P/N
1	18.372	4.8252	(1 1 1)	77	17	12.1	430	15.6	0.430	192	0.9
2	30.140	2.9627	(2 2 0)	88	25	17.9	376	13.7	0.256	350	1.2
3	35.502	2.5265	(3 1 1)	98	140	100.0	2754	100.0	0.334	261	4.5
4	36.973	2.4293	(2 2 2)	99	14	10.0	289	10.5	0.351	249	0.7
5	42.850	2.1087	(4 0 0)	95	16	11.4	396	14.4	0.421	209	0.8
6	56.932	1.6161	(5 1 1)	95	15	10.7	859	31.2	0.974	93	0.7
7	62.643	1.4818	(4 4 0)	91	37	26.4	1417	51.5	0.651	145	1.6

Figure 4 (b) XRD result data of Peak Search Report for $CuFe_2O_4$ ferrite sample (700°C)

Peak ID Extended Report (7 Peaks, Max P/N = 4.5) - [CuFe2O4-T700C-DSuS

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#	2-Theta	d(Å)	Area	Area%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.372	4.8252	430	15.6	Cuprospinel	4.8262	30.0	(1 1 1)	18.368	-0.004
2	30.140	2.9627	376	13.7	Cuprospinel	2.9735	50.0	(2 2 0)	30.027	-0.112
3	35.502	2.5265	2754	100.0	Cuprospinel	2.5266	100.0	(3 1 1)	35.500	-0.002
4	36.973	2.4293	289	10.5	Cuprospinel	2.4258	10.0	(2 2 2)	37.028	0.054
5	42.850	2.1087	396	14.4	Cuprospinel	2.1065	30.0	(4 0 0)	42.897	0.047
6	56.932	1.6161	859	31.2	Cuprospinel	1.6166	40.0	(5 1 1)	56.910	-0.021
7	62.643	1.4818	1417	51.5	Cuprospinel	1.4820	60.0	(4 4 0)	62.633	-0.009

Figure 4 (c) XRD result data of Peak ID Report for $CuFe_2O_4$ ferrite sample (700°C)

Calculate Lattice Constants from Peak Locations and Miller Indices - [CuFe2O4-T70...

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30.140	2.9627	2	2	0	8.3796		
35.502	2.5265	3	1	1	8.3794		
62.643	1.4818	4	4	0	8.3821		

Higher-Order Peaks — Average Lattice Constants = 8.3804

Hide ESD Values — Help | Apply — αβγ = 90.0 | 90.0 | 90.0

Internal Standard — d= 0.0000 — 2T(C)= 0.0 — 2T(O)= — 1.0

Figure 4 (d) XRD result data of Structure Report for $CuFe_2O_4$ ferrite sample (700°C)

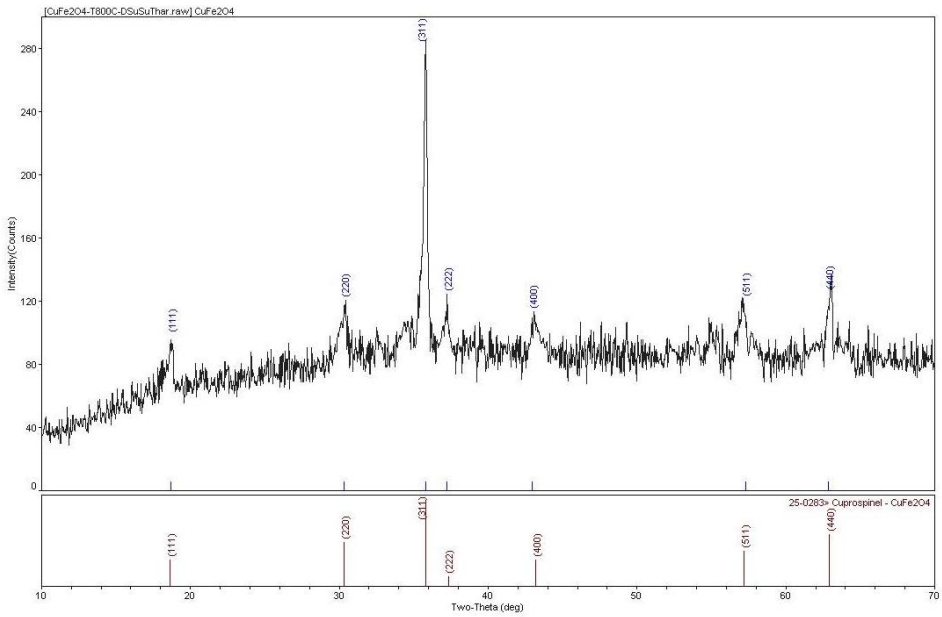

Figure 5 (a) XRD pattern for $CuFe_2O_4$ ferrite sample (800°C)

Peak Search Report (7 Peaks, Max P/N = 5.9) - [CuFe2O4-T800C-DSuSuT

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#	2-Theta	d(Å)	(h k l)	BG	Height	I%	Area	I%	FWHM	XS(Å)	P/N
1	18.715	4.7374	(1 1 1)	67	29	14.6	579	15.1	0.339	248	1.5
2	30.347	2.9429	(2 2 0)	82	36	18.1	894	23.4	0.422	201	1.7
3	35.807	2.5057	(3 1 1)	87	199	100.0	3824	100.0	0.327	268	5.9
4	37.255	2.4115	(2 2 2)	81	41	20.6	1016	26.6	0.421	205	1.9
5	42.956	2.1038	(4 0 0)	82	29	14.6	906	23.7	0.531	164	1.4
6	57.284	1.6070	(5 1 1)	85	34	17.1	1229	32.1	0.614	149	1.6
7	62.874	1.4769	(4 4 0)	86	37	18.6	768	20.1	0.353	275	1.7

Figure 5 (b) XRD result data of Peak Search Report for $CuFe_2O_4$ ferrite sample (800°C)

Peak ID Extended Report (7 Peaks, Max P/N = 5.9) - [CuFe2O4-T800C-DSuS

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#	2-Theta	d(Å)	Area	Area%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.715	4.7374	579	15.1	Cuprospinel	4.7518	30.0	(1 1 1)	18.658	-0.057
2	30.347	2.9429	894	23.4	Cuprospinel	2.9457	50.0	(2 2 0)	30.317	-0.029
3	35.807	2.5057	3824	100.0	Cuprospinel	2.5068	100.0	(3 1 1)	35.790	-0.016
4	37.255	2.4115	1016	26.6	Cuprospinel	2.4076	10.0	(2 2 2)	37.318	0.063
5	42.956	2.1038	906	23.7	Cuprospinel	2.0931	30.0	(4 0 0)	43.187	0.231
6	57.284	1.6070	1229	32.1	Cuprospinel	1.6091	40.0	(5 1 1)	57.200	-0.083
7	62.874	1.4769	768	20.1	Cuprospinel	1.4758	60.0	(4 4 0)	62.923	0.049

Figure 5 (c) XRD result data of Peak ID Report for $CuFe_2O_4$ ferrite sample (800°C)

Calculate Lattice Constants from Peak Locations and Miller Indices - [CuFe2O4-T80...

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18.715	4.7374	1	1	1	8.2055		
30.347	2.9429	2	2	0	8.3238		
35.807	2.5057	3	1	1	8.3105		
37.255	2.4115	2	2	2	8.3538		
42.956	2.1038	4	0	0	8.4150		
57.284	1.6070	5	1	1	8.3501		

Higher-Order Peaks — Average Lattice Constants = 8.3265

Hide ESD Values — Help | Apply — αβγ = 90.0 | 90.0 | 90.0

Internal Standard — d= 0.0000 — 2T(C)= 0.0 — 2T(O)= — 1.0

Figure 5 (d) XRD result data of Structure Report for $CuFe_2O_4$ ferrite sample (800°C)

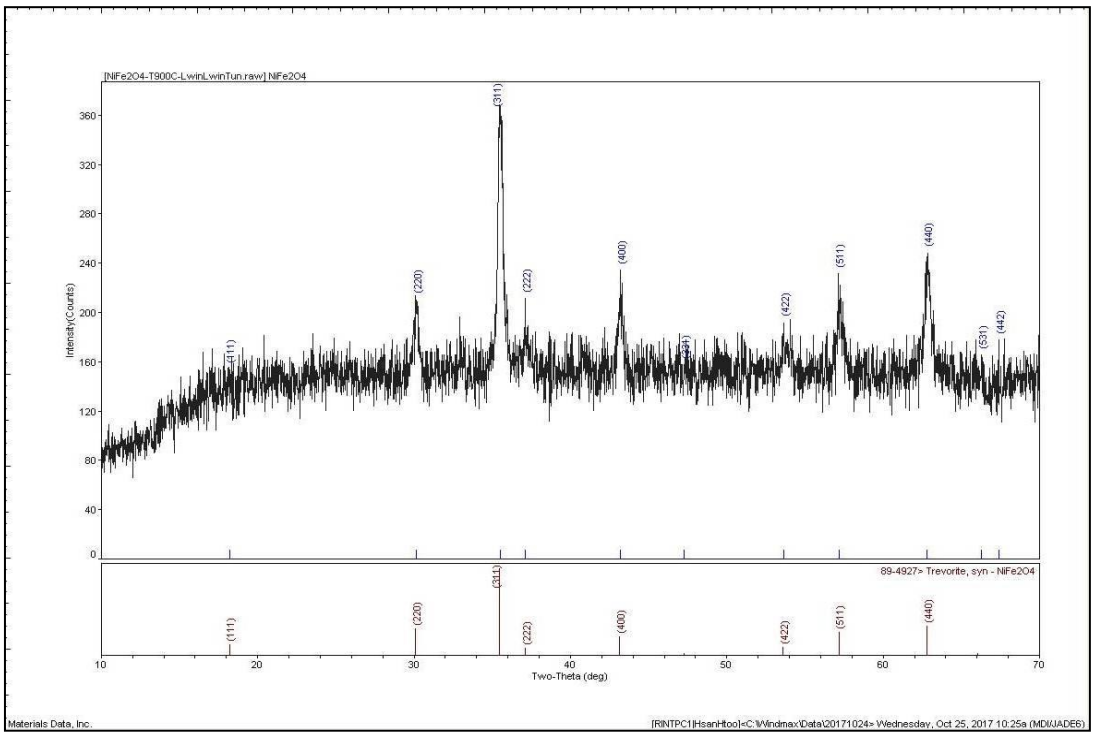

Figure 6 (a) XRD pattern for $CuFe_2O_4$ ferrite sample (900°C)

Peak Search Report (7 Peaks, Max P/N = 6.1) - [CuFe2O4-T900C-DSuSuTh

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#	2-Theta	d(Å)	(h k l)	BG	Height	I%	Area	I%	FWHM	XS(Å)	P/N
1	18.744	4.7301	(1 1 1)	80	21	10.1	427	10.6	0.346	243	1.0
2	30.325	2.9450	(2 2 0)	83	51	24.5	1985	49.2	0.662	126	2.2
3	35.844	2.5032	(3 1 1)	82	208	100.0	4033	100.0	0.330	266	6.1
4	37.333	2.4067	(2 2 2)	84	54	26.0	1450	36.0	0.456	188	2.3
5	43.194	2.0927	(4 0 0)	84	36	17.3	506	12.5	0.239	394	1.6
6	57.226	1.6085	(5 1 1)	76	49	23.6	1300	32.2	0.451	206	2.2
7	62.979	1.4747	(4 4 0)	92	61	29.3	1227	30.4	0.342	285	2.5

Figure 6 (b) XRD result data of Peak Search Report for $CuFe_2O_4$ ferrite sample (900°C)

Peak ID Extended Report (7 Peaks, Max P/N = 6.1) - [CuFe2O4-T900C-DSuSu

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#	2-Theta	d(Å)	Area	Area%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.744	4.7301	427	10.6	Cuprospinel	4.7518	30.0	(1 1 1)	18.658	-0.087
2	30.325	2.9450	1985	49.2	Cuprospinel	2.9457	50.0	(2 2 0)	30.317	-0.008
3	35.844	2.5032	4033	100.0	Cuprospinel	2.5068	100.0	(3 1 1)	35.790	-0.053
4	37.333	2.4067	1450	36.0	Cuprospinel	2.4076	10.0	(2 2 2)	37.318	-0.015
5	43.194	2.0927	506	12.5	Cuprospinel	2.0931	30.0	(4 0 0)	43.187	-0.007
6	57.226	1.6085	1300	32.2	Cuprospinel	1.6091	40.0	(5 1 1)	57.200	-0.026
7	62.979	1.4747	1227	30.4	Cuprospinel	1.4758	60.0	(4 4 0)	62.923	-0.055

Figure 6 (c) XRD result data of Peak ID Report for $CuFe_2O_4$ ferrite sample (900°C)

Calculate Lattice Constants from Peak Locations and Miller Indices - [CuFe2O4-T90...

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18.744	4.7301	1	1	1	8.1927		
30.325	2.9450	2	2	0	8.3296		
35.844	2.5032	3	1	1	8.3021		
37.333	2.4067	2	2	2	8.3371		
43.194	2.0927	4	0	0	8.3708		
57.226	1.6085	5	1	1	8.3579		

Higher-Order Peaks — Average Lattice Constants = 8.3150

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Internal Standard d= 0.0000 | 2T(C)= 0.0 | 2T(O)= | 1.0

Figure 6 (d) XRD result data of Structure Report for $CuFe_2O_4$ ferrite sample (900°C)

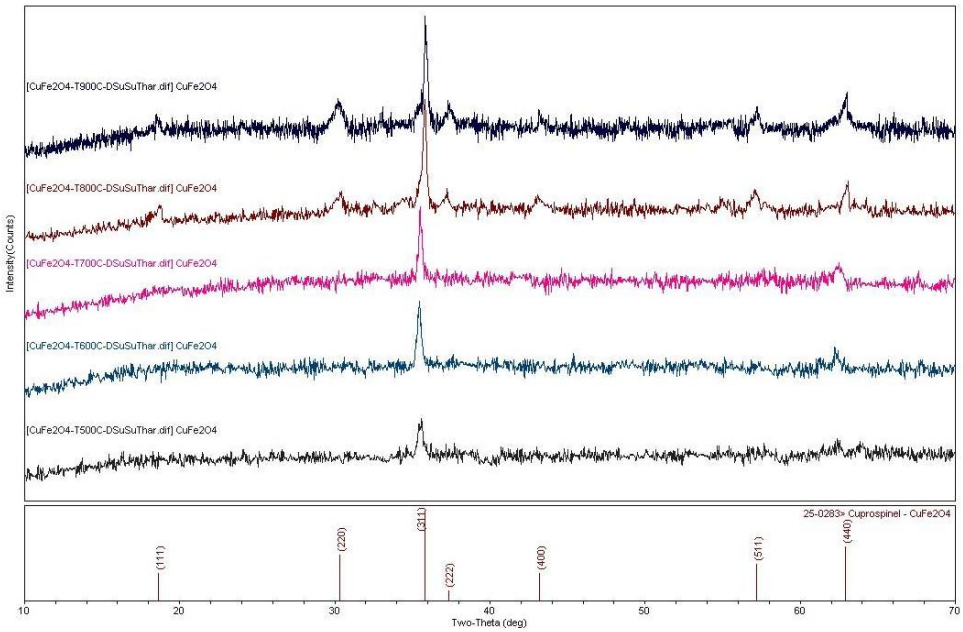

Figure 7 Peak comparison of XRD patterns for $CuFe_2O_4$ ferrite sample at various temperatures

Table 1 Comparison for Crystallite size of $CuFe_2O_4$ in various temperatures

Temperature	2θ (degree)	θ (radians)	FWHM (radians)	λ (Å)	cos θ	Crystallite size(nm)
500ºC	35.59	0.31	8.59×10^{-3}	1.5406	0.95	**16.9**
600ºC	35.45	0.31	5.57×10^{-3}	1.5406	0.95	26.1
700ºC	35.50	0.31	5.89×10^{-3}	1.5406	0.95	24.9
800ºC	35.81	0.31	5.71×10^{-3}	1.5406	0.95	25.5
900ºC	35.84	0.31	5.76×10^{-3}	1.5406	0.95	25.3

Table 2 Comparison for Lattice parameter of $CuFe_2O_4$ in various temperatures

Temperature	**d**	**h**	**k**	**l**	**Lattice Parameter(a)**
500ºC	2.5203	3	1	1	**8.3588**
600ºC	2.5299	3	1	1	8.3906
700ºC	2.5265	3	1	1	8.3794
800ºC	2.5057	3	1	1	8.3105
900ºC	2.5032	3	1	1	8.3021

Microstructure Observation

The microstructure and morphology play the important roles in the determining the magnetic and electric transport properties. These studied for the materials are essential in order to understand the relationship between their processing parameters as well as the behavior when used in practical applications. Microstructures of the sinter ferrite specimen were analyzed by a high resolution scanning electron microscope (SEM). The surface morphology and microstructural analysis of all the five samples of ferrites were carried out using scanning

electron microscope (SEM) JEOL-JSM 5610LV operated at 15kV in the secondary electron image (SEI) mode. The average values of the grain size for each sample were carried out comparison with SEM ruler. This method is based on the line intercept method.

The microstructures of the sintered ferrites with different temperature are shown in figure from 8(a) to (e). The measured grain size values are tabulated in table (3). It was found that the surface morphology of the samples is rough and non-uniform microstructure because the average particle size for every sample was different. Each grain usually contained a large number of atoms.

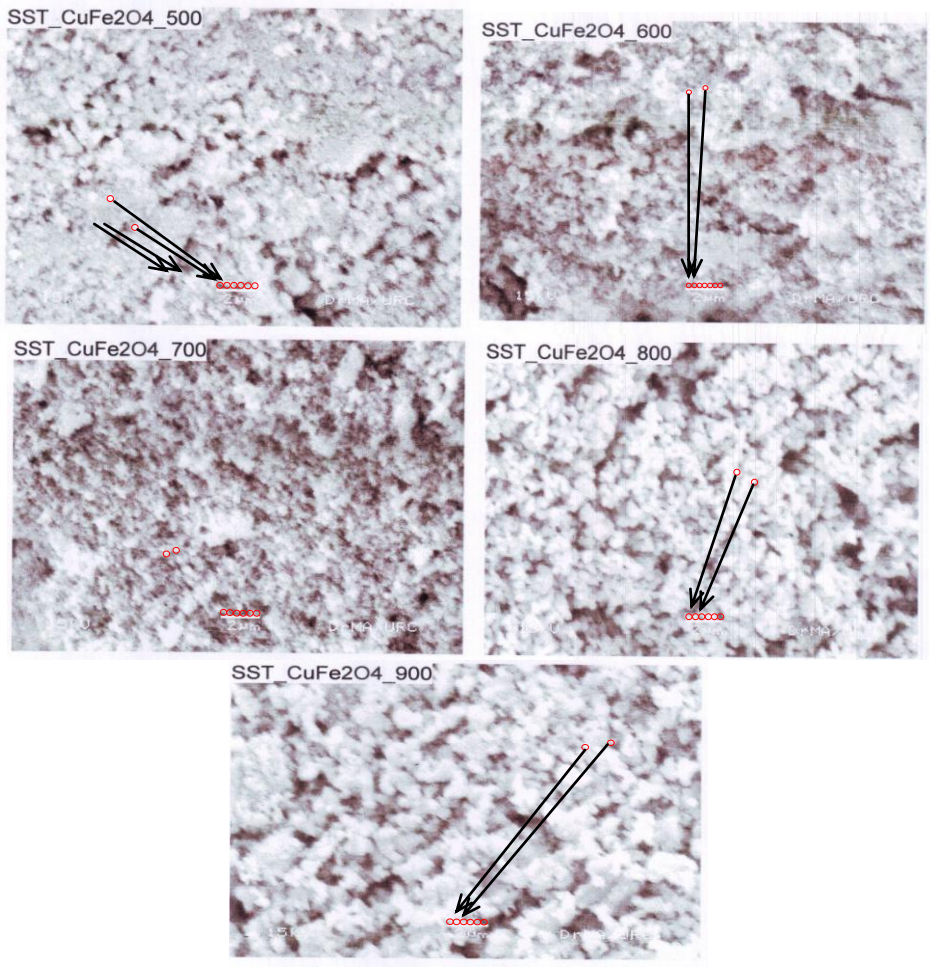

Figure 8 SEM image of copper ferrite (a) at 500˚C, (b) at 600˚C (c) at 700˚C, (d) at 800˚C and (e) at 900˚C

Table 3 Grain size for copper ferrite in various temperature

T(ºC)	QTY	SEM Ruler*(μm)*	Grain Size*(μm)*
500	5.5	2	0.3636
600	7	2	0.2857
700	6	2	0.3333
800	6	2	0.3333
900	6	2	0.3333

The scanning electron microscope (SEM) was used to analyze the microstructure and to determine the average grain size. The microstructure and morphology have an important role in determining the magnetic and electric transport properties and those were examined by a high resolution scanning electron microscope. These studied for the materials are essential in order to understand the relationship between their processing parameters as well as the behavior when used in practical applications. The microstructures of the prepared samples are shown in figures from 8(a) to (e). SEM images give information about the intergranular and intragranular pores as well as the sub-structural defects within the grains. Average grain size was determined by using SEM ruler.

Conclusion

Copper ferrites with the general formula, $CuFe_2O_4$ in various temperatures were successfully prepared by the sol-gel method. The broad peaks in the XRD patterns indicate a fine particle nature of the samples.

XRD results show that the as-prepared ferrite samples under investigation show the single phase (primitive) cubic structure. The lattice parameters were obtained in the range 8.3021 Å - 8.3906 Å. The lattice parameters agree with the literature value. The crystallite size of the samples also varies between 16.9 nm and 26.1 nm. According to experimental results, the sample at 500ºC is the smallest crystallize size of 16.9 nm. The surface morphology of the ferrite samples were determined by using the SEM photograph consists of grain varying from 0.2857 μm to 0.3636 μm. These grain sizes were approximately equal and mostly homogeneous.

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