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STUDY ON CHARACTERIZATION OF $Mg_{0.5}Cu_{0.5}Fe_2O_4$ FERRITE COMPOUND

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Abstract

Magnesium-Copper mixed spinel ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$, was prepared by usual ceramic method at 1100°C for 10 h in vacuum chamber (160 mmHg). Starting materials of Analar (AR) grade Magnesium Oxide (MgO), Copper Oxide (CuO), and Iron Oxide (Fe_2O_3) with desired stoichiometric composition were used to prepare the sample. The X-ray analysis was carried out to investigate the phase formation. The crystallite size was estimated by using collected XRD lines to examine the nanosized ferrite particles. SEM method was used to investigate the microstructural properties of the sample. D.C electrical conductivities of the sample were investigated in the temperature range 303 K – 1218 K to study the electrical conductivity.

Key word: $Mg_{0.5}Cu_{0.5}Fe_2O_4$ Ferrite Compound, Dc electrical conductivity, Activation energy.

Introduction

Ferrite, a ceramic-like material with magnetic properties and they are useful in many types of electronic devices, such as cell phones and wireless devices.

Ferrites are hard, brittle, iron-containing, and generally gray or black and are polycrystalline, *i.e.,* made up of a large number of small crystals. They are composed of iron oxide and one or more other metals in chemical combination. A ferrite is formed by the reaction of ferric oxide (iron oxide or rust) with any of a number of other metals, including magnesium, aluminum, barium, manganese, copper, nickel, cobalt, or even iron itself. A ferrite is usually described by the formula $M(Fe_xO_y)$, where M represents any metal that forms divalent bonds, such as manganese (Mn2+), nickel (Ni2+), cobalt (Co2+), zinc (Zn2+), copper (Cu2+), or magnesium (Mg2+). Nickel ferrite, for instance, is $NiFe_2O_4$, and Magnesium ferrite is $MgFe_2O_4$.

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The physical properties of ferrites are dependent on several factors, such as preparation method, sintering process and constituent elements. Several methods can be used to prepare ferrites, such as solid state reaction, mechanical milling, co-precipitation. The effect of various substituting cations on the structural, electrical, dielectric and magnetic properties of ferrites was the subject of an extensive research work which used techniques such as X-ray and neutron diffraction, thermal analysis, magnetization and electrical conductivity.

In the present work Magnesium-Copper mixed spinel ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ was prepared by usual ceramic method. Structural, microstructural, and its related temperature dependent electrical property of the sample were reported.

Experimental Procedures

2.1 Measurement

The present experimental work include the preparation of the $Mg_{0.5}Cu_{0.5}Fe_2O_4$ mixed ferrite, structural analysis by powder X-ray diffraction measurement, microstructural investigation by SEM measurement and D.C electrical conductivities of the sample were investigated in the temperature range 303 K – 1218 K.

2.2. Preparation of $Mg_{0.5}Cu_{0.5}Fe_2O_4$ Mixed Ferrite

Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$, was prepared by usual ceramic method. The starting materials of Analar (AR) grade Magnesium Oxide (MgO), Copper Oxide (CuO), and Iron Oxide (Fe_2O_3) were weighed with stoichiometric composition to prepare the sample.

The weighed powders were mixed and ground by an agate motor for 1 h to be homogeneous and to obtain fine grain powders. The powders were annealed at 1100°C for 10 h in vacuum chamber (160 mmHg) by using DELTA A Series Temperature Controller DTA4896. The K-type thermocouple was used as the temperature sensor.

Photographs of the experimental setup of sample preparation system, DELTA A Series Temperature Controller DTA-4896 and flow diagram of the Mg-Cu ferrite sample preparation are shown in Fig 1(a - c).

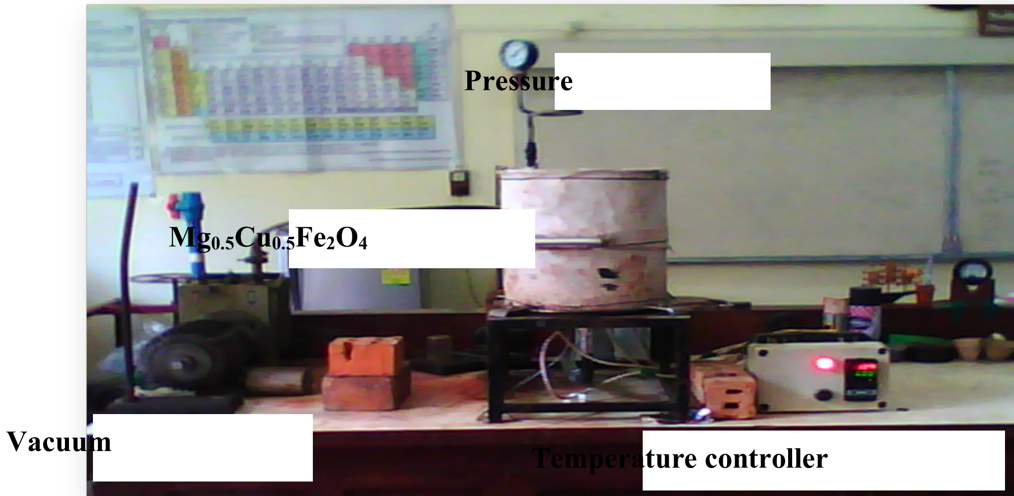

Figure 1(a) Photograph of the experimental setup of sample preparation

Figure1(b). Photograph of DELTAA SERIES DTA-4896 temperature controller

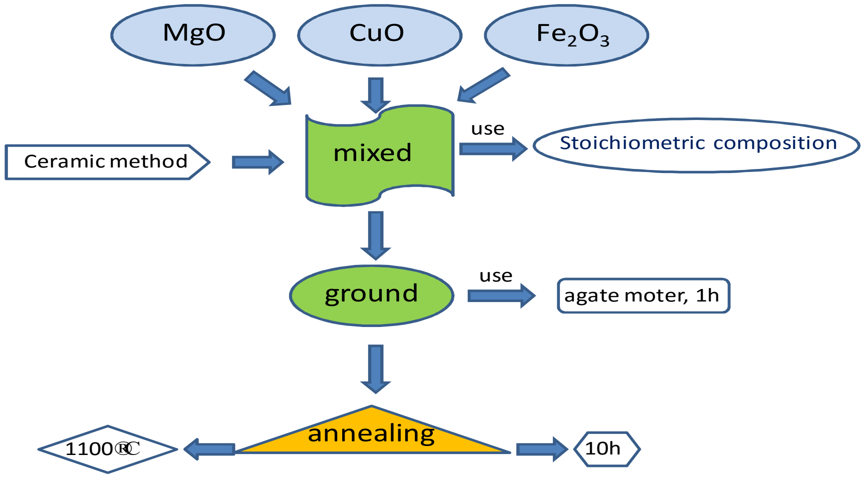

Figure 1(c). Flow diagram of the Mg-Cu ferrite sample preparation

2.3 X-ray Diffraction Measurement

Structural analysis and single phase examination of $Mg_{0.5}Cu_{0.5}Fe_2O_4$ were investigated by RIGAKU MULTIFLEX X-ray diffractometer using Ni-filter with CuK_a radiation, λ =1.54056 Å. The main reflections in the range $10° < 2\theta < 70°$ were observed, and the collected data were used to refine the unit cell parameters from the observed 2θ values with JCPDS (Joint Committee on Powder Diffraction Standards). Photograph showing the front-panel of the RIGAKU MULTIFLEX X-Ray Diffractometer is shown in Fig 2.

Figure 2. Photograph of the RIGAKU MULTIFLEX X-Ray Diffractometer

2.4 Characterization of Surface Morphology

The microstructure and morphology have an important role in determining the magnetic and electrical transport properties and those were examined by a high resolution scanning electron microscope. Morphological features of $Mg_{0.5}Cu_{0.5}Fe_2O_4$ mixed ferrite were investigated by using JEOL JSM-5610LV SEM. Photograph of the JEOL JSM-5610LV Scanning Electron Microscope is shown in Fig 3.

Figure 3. Photograph of the JEOL JSM-5610 LV Scanning Electron Microscope (SEM)

2.5 Conductivity Measurement

The dc electrical resistivity measurement on the ferrite was carried out by using digital multimeter. To measure the temperature variation of resistivity, a sample homemade apparatus was developed in the laboratory. The sample was placed in a sample holder that was immersed in a heating steel chamber surrounded by asbestos. Thermal conducting mica shield was used between the sample and the chamber to have a good thermal conductivity and to protect from electrical conduction.

The resistances were measured over a temperature range from 303 K to1218K. The variation of the temperature was sensed by k-type thermocouple. The dc conductivity σ of the sample is determined from the measured value and sample dimension using the relation: $\sigma = l/RA$, where l is the thickness of the sample, A is the area of the sample and R is the dc resistance. Photograph of the temperature dependent electrical conductivity measurement is shown in Fig 4(a) and the internal arrangement for the temperature dependent dc resistivity measurement is shown in Fig 4(b).

Figure 4(a) Photograph of the experimental setup of electrical resistance measurement

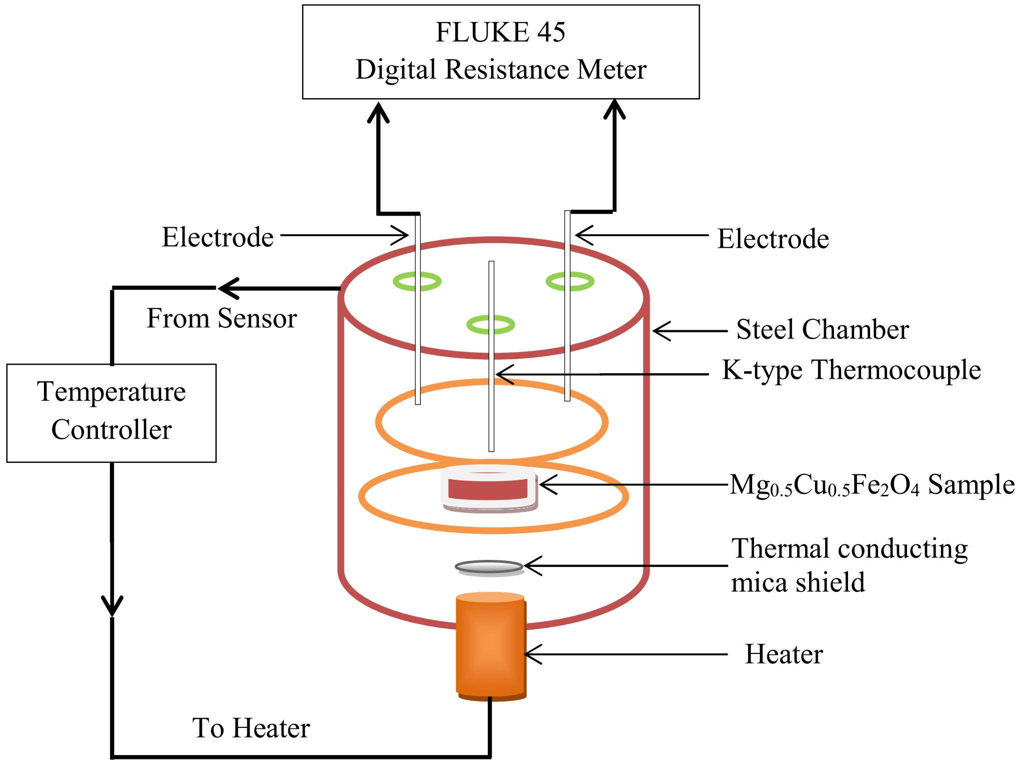

Figure 4(b). Sample chamber for temperature dependent electrical resistance measurement

Experimental Results

3.1 XRD Results

Structural analysis of the Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$, was investigated by powder X-ray diffraction (XRD) method. XRD pattern of the sample was collected on PC-controlled RIGAKU MULTIFLEX X-ray Diffractometer in the diffraction angle range 10° - 70° and as shown in Fig 5. The collected XRD lines were identified by JCPDS data library files of (1) Cat. No. 89-3084> Magnesioferrie, syn – $MgFe_2O_4$ and (2) Cat. No. 77-0010> $MgFe_2O_4$ – Copper Iron Oxide. XRD data are tabulated in Table 1.

The diffraction lines at 37.35° or (222) plane is represented by the Magnesium ferrite, $MgFe_2O_4$ and the line at 37.58° is represented by the

Copper ferrite, $CuFe_2O_4$. The diffraction line of small intensity at the diffraction angle of 38.46° is not assigned with JCPDS files. This may be attributed to small crystallite effects, crystal defects or chemical heterogeneity of the samples.

Table 1. XRD data of Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$

[Mg0.5Cu0.5Fe2O4-PPKhi.raw] MgCuFe2O4 - Full Chart — Peak ID Report

SCAN: 10.0/70.0/0.02/0.12(sec), Cu(40kV,40mA), I(max)=407, 09/03/10 17:55

PEAK: 25-pts/Quartic Filter, Threshold=3.0, Cutoff=0.1%, BG=1/0.7, Peak-Top=Summit

NOTE: Intensity = Counts, 2T(0)=0.0(deg), Wavelength to Compute d-Spacing = 1.54056Å (Cu/K-alpha1)

#	2-Theta	d(Å)	Height	Height%	Phase ID	d(Å)	I%	(h k l)	2-Theta	Delta
1	18.743	4.7305	44	11.2	Magnesioferrit...	4.7348	0.8	(1 1 1)	18.726	-0.017
2	30.596	2.9195	120	30.5	CuFe2O4	2.9177	23.7	(2 2 0)	30.615	0.019
3	35.939	2.4968	394	100.0	CuFe2O4	2.4938	100.0	(3 1 1)	35.983	0.045
4	37.346	2.4059	22	5.6	Magnesioferrit...	2.3951	2.4	(2 2 2)	37.520	0.174
5	37.578	2.3915	44	11.2	CuFe2O4	2.3890	9.3	(2 2 2)	37.620	0.042
6	43.579	2.0751	80	20.3	Magnesioferrit...	2.0776	23.2	(4 0 0)	43.524	-0.055
7	53.950	1.6981	51	12.9	Magnesioferrit...	1.6997	14.3	(4 2 2)	53.896	-0.055
8	57.478	1.6020	148	37.6	Magnesioferrit...	1.6033	37.4	(5 1 1)	57.426	-0.053
9	63.073	1.4727	181	45.9	Magnesioferrit...	1.4738	61.3	(4 4 0)	63.019	-0.054

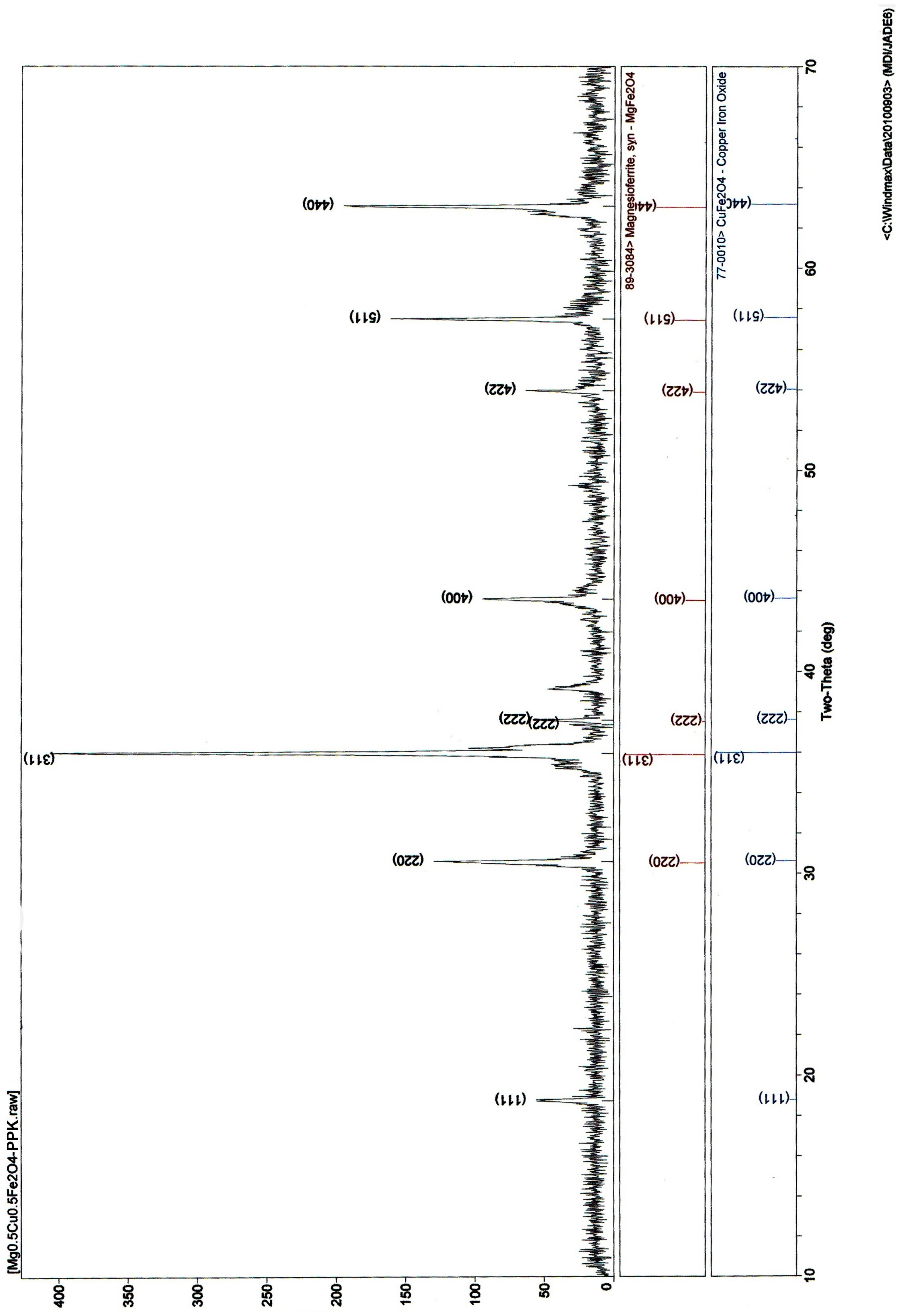

Fig 5 XRD pattern of Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$

3.2 Microstructural Analysis

Scanning Electron Microscopy (SEM) was employed for morphological features of grain shape, grain size and sample homogeneity of Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ powders. SEM micrograph of Magnesium-Copper mixed spinel ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ is shown in Fig 6. As shown in figure, the grain shape of the sample is block. The grain sizes of the sample are about 0.20 μm – 1.80 μm. Some pores are found. Most of the samples are found to be well-crystallized grains.

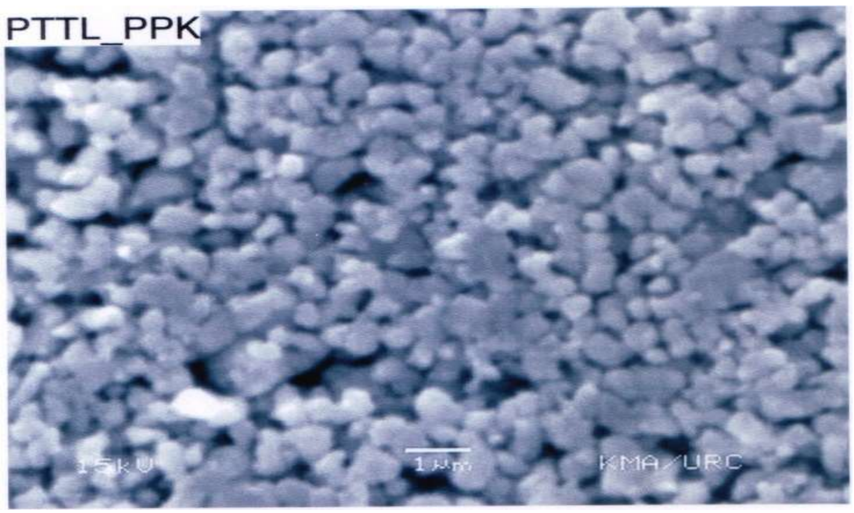

Figure 6. SEM image of Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$

3.3 Temperature Dependent Electrical Conductivity Study

The electrical nature of a material is characterized by its conductivity (or, inversely, its resistivity) .

Electrical conductivity of a ferrite obeys the following Arrhenius expression.

$$\sigma = \sigma_0 \exp(-E_a / kT)$$

where σ is the conductivity, σ_0 is the pre-exponential factor, E_a is the activation energy for electrical conduction, k is the Boltzmann constant and T is the absolute temperature.

Arrhenius plot of Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ in the temperature range of 303 K - 1218 K is shown in Fig 7(a-b). The experimental data are tabulated in Table 2. The electrical conductivity of the sample was found to be clearly increased at 1068 K and it indicated with the ellipse shape ring.

The electrical conductivity (σ) of the sample can be written as the form:

$$\sigma_{dc} = \sigma_0 \exp(-E_a / kT)$$

$$\ln \sigma_{dc} = -E_a / kT + \ln \sigma_0$$

$$= (-E_a / k)(1/T) + \ln \sigma_0$$

Comparing the above equation with the experimental linear equation, y = mx + c, then the value of slope will give the value of ($-E_a/k$). From Fig 7(b), the activation energy E_a can be obtained by using the slope of the ln (σ) versus 1000/T graph.

$$E_a/k = 7.5578 \times 1000$$

$$E_a = 7.5578 \times 1000 \times k$$

$$E_a = 7.5578 \times 1000 \times 1.38\text{E-}23$$

$$E_a = 1.0430 \times 10^{-19}\ \text{J}$$

$$E_a = 0.6519\ \text{eV}$$

Electrical conductivities of the sample are obtained as 1.9232×10^{-6} S m^{-1} at 303 K (start temperature) and 8.3840×10^{-6} S m^{-1} at 1218 K (end temperature) respectively. Temperature dependent electrical conductivity results show that the $Mg_{0.5}Cu_{0.5}Fe_2O_4$ sample exhibited as a normal ionic conductor due to its electrical conductivity is found as $\sigma < 10^{-3}$ S m^{-1}.

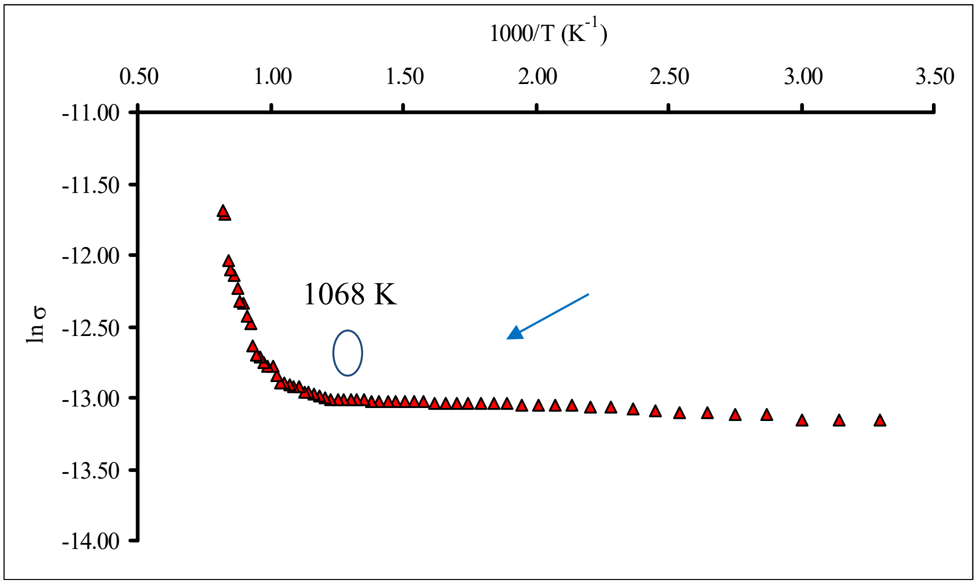

Figure 7(a) Arrhenius's plots of the conductivity with reciprocal temperature of the Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ (303 K – 1218 K)

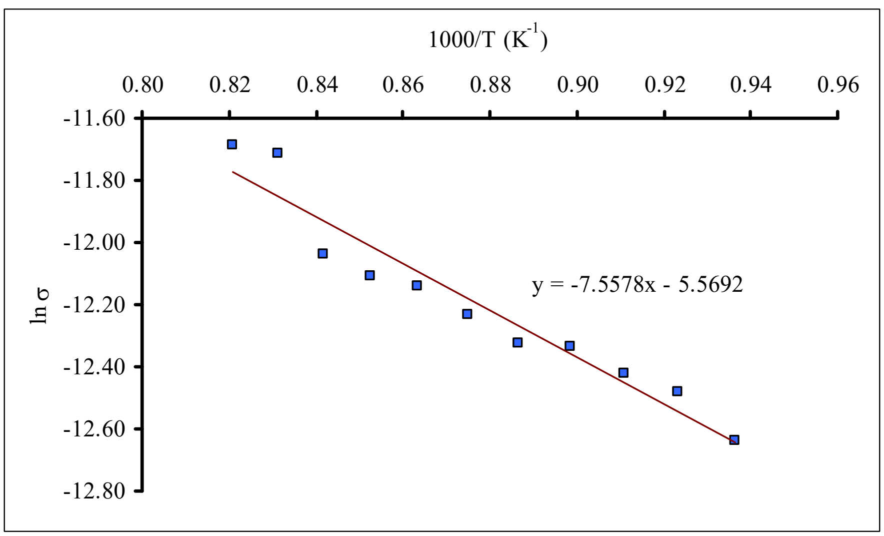

Figure 7(b) Arrhenius's plots of the conductivity with reciprocal temperature of the Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$ (1068 K – 1218 K)

Table 2. The experimental data of the Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$

T(K)	1000/T (K^{-1})	R (Ω)	σ (S m^{-1})	ln σ
303	3.3003	2.2460E+07	1.9232E-06	-13.1615
318	3.1447	2.2449E+07	1.9242E-06	-13.1610
333	3.0030	2.2242E+07	1.9421E-06	-13.1518
348	2.8736	2.1485E+07	2.0105E-06	-13.1171
363	2.7548	2.1396E+07	2.0189E-06	-13.1130
378	2.6455	2.1201E+07	2.0374E-06	-13.1038
393	2.5445	2.1192E+07	2.0383E-06	-13.1034
408	2.4510	2.1000E+07	2.0569E-06	-13.0943
423	2.3641	2.0633E+07	2.0935E-06	-13.0767
438	2.2831	2.0400E+07	2.1174E-06	-13.0653
453	2.2075	2.0364E+07	2.1212E-06	-13.0635
468	2.1368	2.0243E+07	2.1339E-06	-13.0576
483	2.0704	2.0154E+07	2.1433E-06	-13.0532
498	2.0080	2.0146E+07	2.1441E-06	-13.0528
513	1.9493	2.0068E+07	2.1525E-06	-13.0489
528	1.8939	1.9986E+07	2.1613E-06	-13.0448
543	1.8416	1.9972E+07	2.1628E-06	-13.0441
558	1.7921	1.9935E+07	2.1668E-06	-13.0422
573	1.7452	1.9895E+07	2.1712E-06	-13.0402
588	1.7007	1.9878E+07	2.1730E-06	-13.0394
603	1.6584	1.9840E+07	2.1772E-06	-13.0375
618	1.6181	1.9817E+07	2.1797E-06	-13.0363
633	1.5798	1.9736E+07	2.1887E-06	-13.0322
648	1.5432	1.9723E+07	2.1901E-06	-13.0316
663	1.5083	1.9720E+07	2.1905E-06	-13.0314
678	1.4749	1.9681E+07	2.1948E-06	-13.0294
693	1.4430	1.9592E+07	2.2048E-06	-13.0249
708	1.4124	1.9540E+07	2.2106E-06	-13.0222
723	1.3831	1.9493E+07	2.2160E-06	-13.0198
738	1.3550	1.9473E+07	2.2182E-06	-13.0188

T(K)	1000/T (K^{-1})	R (Ω)	σ (S m^{-1})	ln σ
753	1.3280	1.9458E+07	2.2199E-06	-13.0180
768	1.3021	1.9427E+07	2.2235E-06	-13.0164
783	1.2771	1.9406E+07	2.2259E-06	-13.0153
798	1.2531	1.9290E+07	2.2393E-06	-13.0094
813	1.2300	1.9284E+07	2.2400E-06	-13.0090
828	1.2077	1.9147E+07	2.2560E-06	-13.0019
843	1.1862	1.8785E+07	2.2995E-06	-12.9828
858	1.1655	1.8542E+07	2.3296E-06	-12.9698
873	1.1455	1.8485E+07	2.3368E-06	-12.9667
888	1.1261	1.8420E+07	2.3450E-06	-12.9632
903	1.1074	1.7674E+07	2.4440E-06	-12.9219
918	1.0893	1.7644E+07	2.4482E-06	-12.9202
933	1.0718	1.7427E+07	2.4787E-06	-12.9078
948	1.0549	1.7307E+07	2.4959E-06	-12.9009
963	1.0384	1.7130E+07	2.5216E-06	-12.8906
978	1.0225	1.6419E+07	2.6308E-06	-12.8482
993	1.0070	1.5400E+07	2.8049E-06	-12.7841
1008	0.9921	1.5370E+07	2.8104E-06	-12.7822
1023	0.9775	1.4873E+07	2.9043E-06	-12.7493
1038	0.9634	1.4398E+07	3.0001E-06	-12.7169
1053	0.9497	1.4252E+07	3.0309E-06	-12.7067
1068	0.9363	1.3325E+07	3.2417E-06	-12.6394
1083	0.9234	1.1390E+07	3.7924E-06	-12.4825
1098	0.9107	1.0733E+07	4.0246E-06	-12.4231
1113	0.8985	9.8317E+06	4.3935E-06	-12.3354
1128	0.8865	9.7073E+06	4.4498E-06	-12.3226
1143	0.8749	8.8663E+06	4.8719E-06	-12.2320
1158	0.8636	8.1033E+06	5.3306E-06	-12.1420
1173	0.8525	7.8235E+06	5.5213E-06	-12.1069
1188	0.8418	7.2950E+06	5.9213E-06	-12.0370
1203	0.8313	5.2669E+06	8.2014E-06	-11.7112
1218	0.8210	5.1522E+06	8.3840E-06	-11.6892

Conclusion

4.1 Conclusion

Magnesium-Copper mixed ferrite, $Mg_{0.5}Cu_{0.5}Fe_2O_4$, was prepared by usual ceramic method at 1100°C for 10 h in vacuum chamber (160 mmHg). Structural analysis and lattice parameters determination of the sample were investigated by XRD method. XRD pattern showed that $Mg_{0.5}Cu_{0.5}Fe_2O_4$ mixed ferrite belongs to cubic structure at room temperature. The lattice parameters are obtained as $a = b = c = 8.28$ Å. The crystallite size was also obtained as 37.95 nm. It is consistent with the ceramically prepared ferrite.

SEM micrograph shows that sample exhibited the fine grained microstructure with small grain size of block shape. Most of the samples were found to uniform and obtained as 0.20 μm – 1.80 μm. Some pores are found and most of the samples are found to be well-crystallized grains.

Electrical conductivities of the sample were increased with increasing temperatures. The sample exhibited as a normal ionic conductor due to its electrical conductivity is found as $\sigma < 10^{-3}$ S m^{-1}.

Magnesium-Copper ferrite device can be used for many application such as chlorine gas sensors, color imaging, cores of audio frequency, microwave absorber, high frequency transformers coils and medical diagnosis.

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