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Preparation and study structure properties of Zinc-Copper ferrite (ZnO_X $CuO_{1-X}Fe_2O_3$)nanoparticles

Tahseen H. Mubarak [1], Karim H. Hassan [2], Zena Mohammed Ali Abbas[3],WafaaAbdulsattarShatti[4]
[1,3,4] *Iraq, University of Diyala, College of Science, Department of physics*
[2] *Iraq, University of Diyala, College of Science, Department of Chemistry*

Abstract: *Nanomaterial's play a pivotal role in physical, spinel ferrites are useful magnetic materials because of their low cost and high electromagnetic performance over a wide frequency range as compared to the pure metals. In this research we have Preparedzinc-copper ferrite (ZnO_X CuO_{1-X} Fe_2O_3) nanoparticles by using powder metallurgy route,where x = (0.2).A mold is designed in diameter (1cm) with a hight of (3cm), and the weight of the sample is (1.3 g), and ferrite parts are formed by pressingwith a pressure of (700) psi,the sintering temperature of (1000 °C) for four hours .X-ray Diffraction (XRD) to find the lattice parameters and crystallite size of the sintered ferrite specimensconfirmed the formation of tetragonal single phase zinc-copper ferrite nanoparticles. The particle size calculations were done using XRD Scherer's formula (41.867nm) depending on the sentring temperature and time. The study of Scanning electron microscope (SEM) micrographs reveals a less number of pores with smaller lump size, and a homogeneous system with agglomerates of submicronic particles,the crystallites are in order of micrometer with relatively smooth surface.Density measurements are performed using Archimedes' principle,density of the sintered sample is measured by Archimedes method using distilled water as fluid medium(4.892 g /cm3).*

Keywords: *ZnO_X CuO_{1-X} Fe_2O_3, nanoparticles, powder metallurgy,SEM, ferrite*

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I. Introduction

Nanosized ferrite materials have attracted great attention in recent years. They exhibit unusual physical and chemical properties significantly different from those of the bulk materials due to their extremely small size and large specific surface area [1,2]. Ferrites are usually non-conductive ferrimagnetic ceramic compounds derived from iron oxides such as hematite (Fe_2O_3) or magnetite (Fe_3O_4) as well as oxides of other metals, and are like most other ceramics, hard and brittle. In terms of their magnetic properties, the different ferrites are often classified as "soft" or "hard", which refers to their low or high magnetic coercivity [3]. Zinc oxide is an inorganic compound with the formula ZnO. It usually appears as a white powder, nearly insoluble in water. The powder is widely used as an additive into numerous materials and products including plastics, ceramics, glass, cement ,rubber and lubricants [4].Zinc oxide is an important basic material due to its low cost, large band gap (3.37 eV), large excitonbinding energy (60 MeV),and luminescent properties [5]. It is widely used in many applications,such as catalyst, gas sensor, filtering materials for ultraviolet light, microbe resistant defenceclothing [6] and also as antimicrobial and retanning agent [7]. Cupric oxide (CuO) is a black material which melts above 1200°C with some loss of oxygen ,and is used as a pigment in clay glazes. Several colors, including red, blue, and green, can be derived from it. As a mineral, it is known as tenorite. It is red in color and does not dissolve in water or any organic solvents.Copper (II) oxide has application as a p-type semiconductor. It is used an abrasive to polish optical equipment, and to produce dry cell batteries in addition to its use in wet cell batteries as cathode [8].Cupric oxide (CuO) is an important transition metal oxide with a narrow band gap (Eg 1.2 eV) andforms the basis of several interesting high temperature superconductors and giant magneto resistance materials [9]. Iron (III) oxide or ferric oxide is inorganic compound with the formula Fe_2O_3. It is one of the three main oxides of iron, the other two being iron (II) oxide (FeO), which is rare, and iron (II,III) oxide (Fe_3O_4), which also occurs naturally as the mineral magnetite. As the mineral known as hematite, Fe_2O_3 is the main source of iron for steel industry. Fe_2O_3 is ferromagnetic, dark red, and readily attacked by acids. Rust is often called iron (III) oxide, and to some extent this label is useful, because rust shares several properties and has a similar composition. To a chemist, rust is considered an ill-defined material, described as hydrated ferric oxide [10].

II. Experimental

Ferrite sample preparation

Raw materials of high purity are used, because the presence of impurities affects the properties of prepared materials. All the chemical materials used are commercial one and are of analytical grade.

Stoichiometric calculations

The appropriate weight percentage of each oxide to be mixed for different composition is calculated by the following formula;

Weight % of oxide = (molecular weight of oxide X required weight of the sample)/ (sum of the molecular weight of the given composition).The weights of oxides powder needed to prepare the required formula are given in table (1).

Table (1) Symbols of prepared

Sample	Chemical formula
zinc-copper ferrites	ZnO_X CuO_{1-X} Fe_2O_3

Mixing

The powders (nano-ZnO,nano-CuO and nanoFe_2O_3) are mixed to obtain a uniform distribution of the components. These are mixed using a variable speed electric mixer for two hours for the purpose of obtaining a homogeneous mixture and the non-agglomerated mixtures are then dried in an oven at 80 °C for 3 hours.

Pellet formation

A mold is designed for the manufacture of samples in the form of pellet in diameter (9mm) and thickness (5mm) and the weight of the sample is (1.3 g). It uses hydraulic press with a pressure of (500-700 psi), and the diameter of the mold used (1cm) with a hight of (3cm).

Sintering

The green Pellets are loaded on refractory plates (pure alumina container) and sintered at temperature of (1000 °C) for four hours, and then cooled in the furnace to room temperature.

The X-ray diffraction pattern were recorded using XRD-6000 with CuKα (λ=1.5406A°) that have an accelerating voltage of (220/50)HZ which is produced by SHIMADZU company, and the scanning electron microscope used in imaging the nanoparticles was a VEGA//EasyProbe which is a favorable combination of a scanning electron microscope and a fully integrated energy dispersive X- ray microanalyser produced by TESCAN, s.r.o., Libušinatrída.

III. Result And Discussion

1.XRD diffraction test

X-ray diffractometer is used to identify the types of ferrites from their crystalline phases. The lattice parameters and crystallite size of the sintered ferrite specimens are evaluated from XRD analysis. The broad peaks in the XRD patterns indicate a fine particle nature of the particles. Table (2) shows the XRD data of ferrites sintered at 1000°C.

Table (2) XRD data of ferrite sintered at 1000 °C (Strongest 3 peaks)

1000 °C				
Sample	2 Theta (deg)	d(Å)	FWHM (deg)	Intensity (counts)
zinc-copper ferrite	35.7064	2.51256	0.20370	453
	57.2534	1.60780	0.20160	227
	30.3765	2.94017	0.17560	150

Fig. 1 shows the XRD pattern of $(ZnO)_x(CuO)_{1-x}$(Fe_2O_3) where the value of x =(0.2). The diffraction peaks of Fe_2O_3disappear completely but a small amount of CuO remains as second phase beside the tetragonal copper ferrite,Fig. 1 illustrates ferrites (zinc-copper ferrites) single phase of tetragonal .

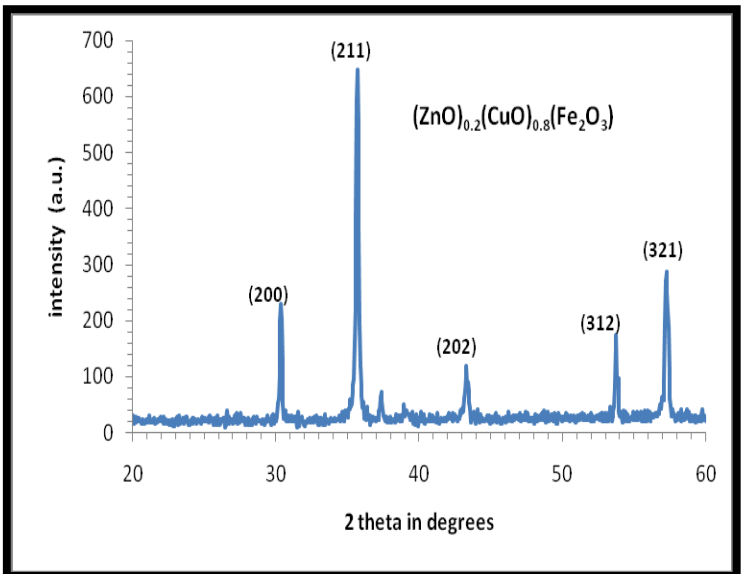


Fig. (1) XRD pattern of (zinc-copper ferrite) sintered at 1000 °C for 4 hours

In ($ZnO_XCuO_{1-X}Fe_2O_3$) ferrites, the solid interaction between ZnO,CuO and Fe_2O_3, leading to the formation of zinc copper ferrite.The sample show all the characteristic peaks of ferrite material with most intense peak (211),which confirms the formation of tetragonal structure ferrite. This result is identical to the result obtained by KenfackFlaurance in the study of Cu-Ni-Fe-O system (2004)[11].
The particle size of ferrite sample is calculated using Scherrer's[12,13] formula is shown in table (3) together with lattice parameters .
$D = 0.9\lambda / \beta \cos \theta$
where λ is the wavelength of X-ray (0.15406 nm).β (FWHM)

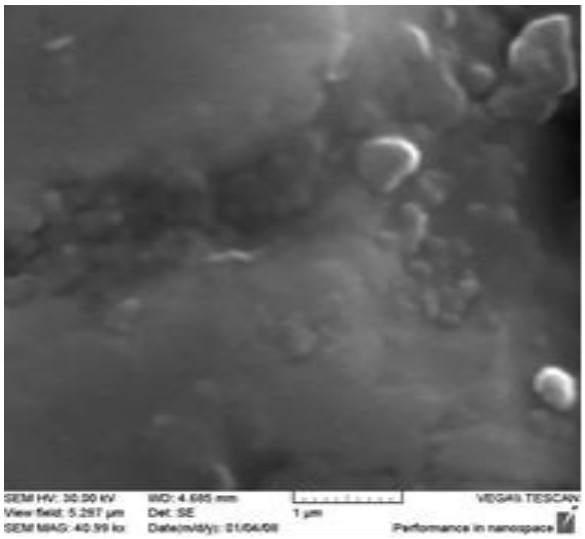
Table (3) particle size of ferrites and lattice parameters

Sample	2 Theta (deg)	d(Å)	FWHM (deg)	particle size (nm)	Tetragonal a,c(Å)
zinc-copper ferrite	35.7064	2.51256	0.2037	41.867	a=5.880, c=0.955

In tetragonal structureferrite, the lattice parameters (a,c) are calculated using the relation[14].

$$d_{hkl}=\frac{1}{\left(\frac{h^2+k^2}{a^2}+\frac{l^2}{c^2}\right)^{1/2}}$$

2- Scanning electron microscope (SEM) test



Scanning electron microscope (SEM) applied to samples sintered at 1000°C is used to determine the lump size. From the micrograph, we can observe the formation of soft agglomerates with irregular morphology constituting the quite fine particles. The study of SEM micrographs reveals a less number of pores with smaller lump size, and a homogeneous system with agglomerates of submicronic particles. Since the ceramic method involves sintering of the stoichiometric mixtures at high temperatures, the crystallites are in order of micrometer with relatively smooth surface. The aggregate of crystallites of various sizes indicates a size distribution in the micrographs. The variation in size of the particles among the different samples is due to the difference in processing temperatures. When the ferrite powders with more crystalline content are used for samples sintered at high temperatures, the local shrinkage speed is much higher than the densification speed of the ceramics.

3-Density test

Experimentally,density measurements are performed using Archimedes' principle. The errors associated with this method derive mainly from thermal, surface effects pore presence and specimen size. Density of the sintered sample is measured by Archimedes method using distilled water as fluid medium. The apparent density is shown in table (4).

Table (4) apparent density of ferrites

Sample	Density g /cm^3 at 1000°C
zinc-copper ferrite	4.892

There is an increase in the density and shrinkage of the sintered pellets with an increase of, time and temperature, but the porosity shows otherwise.

IV. Conclusions

Zinc-copper ferrite nanoparticles under investigation show tetragonal copper ferrite spinel structure, The particle size of ferrite sample is calculated using Scherrer's formula about 41.867 nm, the lattice parameters (a,c) : a=5.880 nm,c=0.955nm SEM micrographs reveals a less number of pores with smaller lump size, and a homogeneous system with agglomerates of submicronic particles

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