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Fabracation and study of Structural properties for the compounds ($Mg_{1-x}Zn_xFe_2O_4$) prepared by chemical precipitation method

Saif Amer Mahdi
Physics department , College of Science , Tikrit University , Tikrit , Iraq
Saifamer76@gmil.com

Abstract
In this paper were prepared Magnesium – Zinc ferrites having the chemical formula ($Mg_{1-x}Zn_xFe_2O_4$) ,(X=0.0, 0.4 and 0.6) were prepared by using chemical precipitation method and these samples were annealing at 700ºC for (3hr). Then the structural and physical properties have been measured for the samples prepared. XRD analysis showed that all the samples having face-centered cubic (FCC) structure and is miller indices (220),(331),(222) (400),(422),(511),(440) The Lattice constant (a) increased with the Zinc concentration (8.36 – 8.67 ºA) increase and also increasing of the homogeneous surface structure caused best in the physical properties, while the Microscope examinations showed that the average roughness surface and Grain size (Dnm) were decreasing with the increase in Zn-concentration .

Keywords: Magnesium – Zinc ferrites, chemical precipitation method , XRD analysis

1-Introduction
The continuous search for new materials with the best properties led to discover materials that were not known previously, with the availability of these materials appeared many uses which based in its work on the unique properties owned. Magnetic ceramic such as (Ferrites) one of these materials, which took its way to develop to use in the construction of electrical circuits which operates at low and high-frequency [1]. The Ferrites cubic with spinel-structure ($MeFe_2O_4$) or ($MeOFe_2O_3$) [where the symbol Me refer to any bivalent metal belongs to the transition elements in the periodic table] which take a crystalline cubic structure achieves a balance between negative and positive charges and is characterized by tightly compacting the oxygen ions with positive ions bivalent (Me^{+2}) or a trivalent (Fe^{+3}) and distributed in binary crystalline structure, and from here came the naming of this type of Ferrite as "spinel Ferrites," and spinel Ferrites materials have commercial significance due to the unique electrical and magnetic properties which is owned and this type of material have been the subject of comprehensive studies by some physicists and chemists[2] The researchers (Muhammad Ajmai) and (Asghari Maqsood 2007) studied the effect of adding ZnO on the synthetic and electrical properties for compounds ($Ni_{1-x}Zn_xFe_2O_4$), the results indicate that the particle size reach highest value (6.16 nm) and the lattice constant increases from (8.337A°) to (8.490A°) and the porosity reduced to (10.2%), and the increase in the lattice constant has been interpreted due to the fact that the density of zinc ions is greater than the density of nickel ions [3]. J. Jacob and his group in 2008 have been prepared nanoparticles for nickel compound ($NiFe_2O_4$) by chemical precipitation at temperatures (275,450,600) °C for (2hr) and used the (XRD) technique in order to characterize the structural and synthetic for this sample and shown cubic image, also found that the particle size increases (10nm) to (23nm) with gradual increase in the temperatures. [4]. The researchers (Mahamuni and BK Chougule 2010) have studied the structural characteristics of the compounds $Ni_xZn_{1-x}Fe_2O_4$ and proportions (x= 0.2,0.4,0.6,0.8) and observed through X-ray diffraction analysis that all the samples have the cubic crystalline structure, also found the lattice constant decreased with increasing the values of (X) while the particle size had been within the range (30 ~ 50) nm. [5]. In 2014 KV. Kumar and his group studied synthetic properties of Fritat Ni_{1-X} Cu $_X$ Fe_2O_4 (X = 0, 0.2, 0.4,0.5, 0.6, 0.8 and 1.0) and prepared by using the chemical precipitation at (700°C) for (5 hrs), and observed from samples of X-ray diffraction, all these samples have a single-phase and cubic crystalline structure, they found that the particle size within the range (58.91-367) nm The density (ρ_{x-ray}) and the porosity increases with copper concentration [6], it is worth noting the importance of this research through the preparation of Ferrite Zinc- Magnesium using the method of chemical precipitation and understand the experimental variables during the samples preparation such as mixing, pressing and bonding materials and select the raw materials and the degree of purity, as well as, select suitable homogeneous samples and the conditions of annealing , also understand the nature and characteristic of the crystal structure by using X-ray diffraction technique and atomic force microscope, to show homogeneity and free of gaps and defects of synthetics. Topology surface is also studied in this paper by using an AFM to show the homogeneity of samples and it is free from gaps and synthetics defects as well as to determine the particle size.

2-Experimental details
This section includes the materials used in this research as well as the scientific method which follow to prepare the compound of ($Mg_{1-x}Zn_xFe_2O_4$) in different proportions of zinc (x=0.0,0.4 and 0.6) and the basic stages of the method of samples preparation by chemical precipitation technique, as well as other operations for synthetics testing and other tests which is used to evaluate the prepared samples also, the following raw materials has been used Iron nitrate

$Fe_2O_3(NO_3)_{2.6}H_2O$ (% 98) and magnesium nitrate Mg $(NO_3)_{2.}6H_2O$ (%98) and Zinc nitrate $Zn(NO_3)_{2.}6H_2O$) (99%), the creation of Ferrites requires high accuracy at all steps to prevent pollution caused by impurities.
The preparation steps can be provided as follows:
Firstly, Prepared the samples in different molar ratios (x = 0.0,0.4 and 0.6) labelled A1, A2 and A3 respectively, and have been calculated the weight ratios of samples accurately by sensitive Scale of type (Denver) made in German and accuracy up to (10^{-4} gram), then these compounds have been dissolved by distilled water for each compound separately, and mixing the Solutions with each other by using Magnetic stirrer and temperature (60˚C) during two hours to get a complete homogeneity of the compounds, then were added the sodium hydroxide solution to the compound and doing titration process to reach a PH equal to 7, and these compounds are filtered and then dried at temperature 100ºC, and mix all the compounds by using (agate mortar) to triturating well, and to obtain a homogeneous mixture and accurate size of particle with a homogeneous sizes, the importance of this process is obtaining a homogeneous powders affect on the interaction rate of these materials, also in the physical properties of the final outcome, then have been added the material bond (polyvinyl alcohol PVA) approximately (2%) from the total weight of the sample in order to consolidate and cohesion the mix during the output process from the molding and not to get distortions or defects for the sample surface, and this proportion will not have effect on the main components of Ferrites and also it will evaporate during the final heating. Thus, in this experiment, the samples were compressed at 5 Ton during 4 min, to suitable with the requirements of the devices used to study, in order to get the required tests, that is by hydraulic mechanic compressor up to the extent of pressure (15 ton) by annealing process, the samples (compressed samples) were heated to temperature (700°C) for (3hr.) by using an electric oven, which can reach temperature up to (1100°C), then cooled samples gradually until it reaches room temperature. In this process, the internal spread occurs for neighbouring particles. Thus, it will be allowed to spread and centred in its place and cohesion with each other. The porosity are considerably less because of the spread in the gaps and vacuums for samples surface. Finally, we get solid ceramic samples and less brittle, also observes that diameter of samples is least for little bit as a result of the heating process, where increase in the degree of annealing temperature leads to reduce the poresity, thus increasing particles size (grains size)) [7]. The tests carried out on the prepared Ferrites samples were included the synthetics tests by examining the X-ray diffraction (XRD), and atomic force microscope (AFM), and calculate the density and porosity. As, the properties of prepared Ferrites are affected by increasing of the concentration of zinc, as well as the density of the prepared samples and extent of particles size (grains size) for the sample, which are depend on the degree of sintering temperature.

3-Results and Discussion

These tests included microscopic examination of atomic force microscope (AFM) to a statement fine structure, and the calculation of particle size of the fine structure and the degree of homogeneity of the constituent chemical elements of the samples that are meant studied, it has also been conducting tests X-ray diffraction to note phase Ferrites other interfaces and phase generated, fixed lattice constant measure, as measuring the density of sintered within examination conducted bulk density measurements within this type of examination were measured for Bulk density samples, and by measuring the mass of each sample is completely dry. It is possible to measure the size of each sample mathematically as they are of cylindrical shape of a homogeneous mathematical relation [8]:

$$V = \pi r^2 h \text{ ----- (1)}$$

Where r is the radius of the samples , h is the thickness of the samples
It has been calculated Bulk density (ρ_d) of the following mathematical relation[8]:

$$\rho_d = \frac{m}{V} \text{-------- (2)}$$

where m is mass sample and V is the size of the sample that was previously calculated. we have adopted a examination of X-ray diffraction (XRD) in order to get the same material Ferrites structure spinl. X-ray diffraction device (XRD-6000Shimadzu) product from a company (SHIMAZU Japanese origin) and the target used in the tube X is (Cuk_α) having been diagnosed with X-ray diffraction pattern by identifying on the upper peaks sites that appear when you highlight a package of X-rays to the extent of 2 Θ = (20° -80°) on the surface of the material. As can be some of the compositional variables of lattice expense (a) through the following mathematical relationships[9]:

$$a = d (h^2 + k^2 + l^2)^{1/2} \text{ ----- (3)}$$

where a is lattice constant. d: is the value of the distance between the parallel levels, and (hkl) Miller corresponding coefficients for each level in this sample. The use of an atomic force microscope AFM (AA 3000 SPM Scanning probe Microscope) fitted by a company (Angstrom Advanced Inc). This gives microscope two-dimensional analysis (2 D) and three-dimensional (3 D) surfaces studied samples, an atomic force microscope two uses to identify the exact details of the topography and structures granular crystalline on the surface of the prepared forms and gives a high resolution image topography shows the crystal structure of the material surface details, and can also be give us some calculations on the form and distribution of the grains size and rate of roughness and the rate of surface elevation a contagious by computer program by the company to obtain thermal interactions and turning oxides used in

practice to Ferrites required of a compound $Mg_{1-x}Zn_xFe_2O_4$ values (x = 0.0, 0.4 and 0.6) it has been confirmed through X-ray diffraction technique. It was observed structural spinl for all samples and well and with crystalline structures cubic form of type (FCC) and shown in Fig. 1 (a–c).

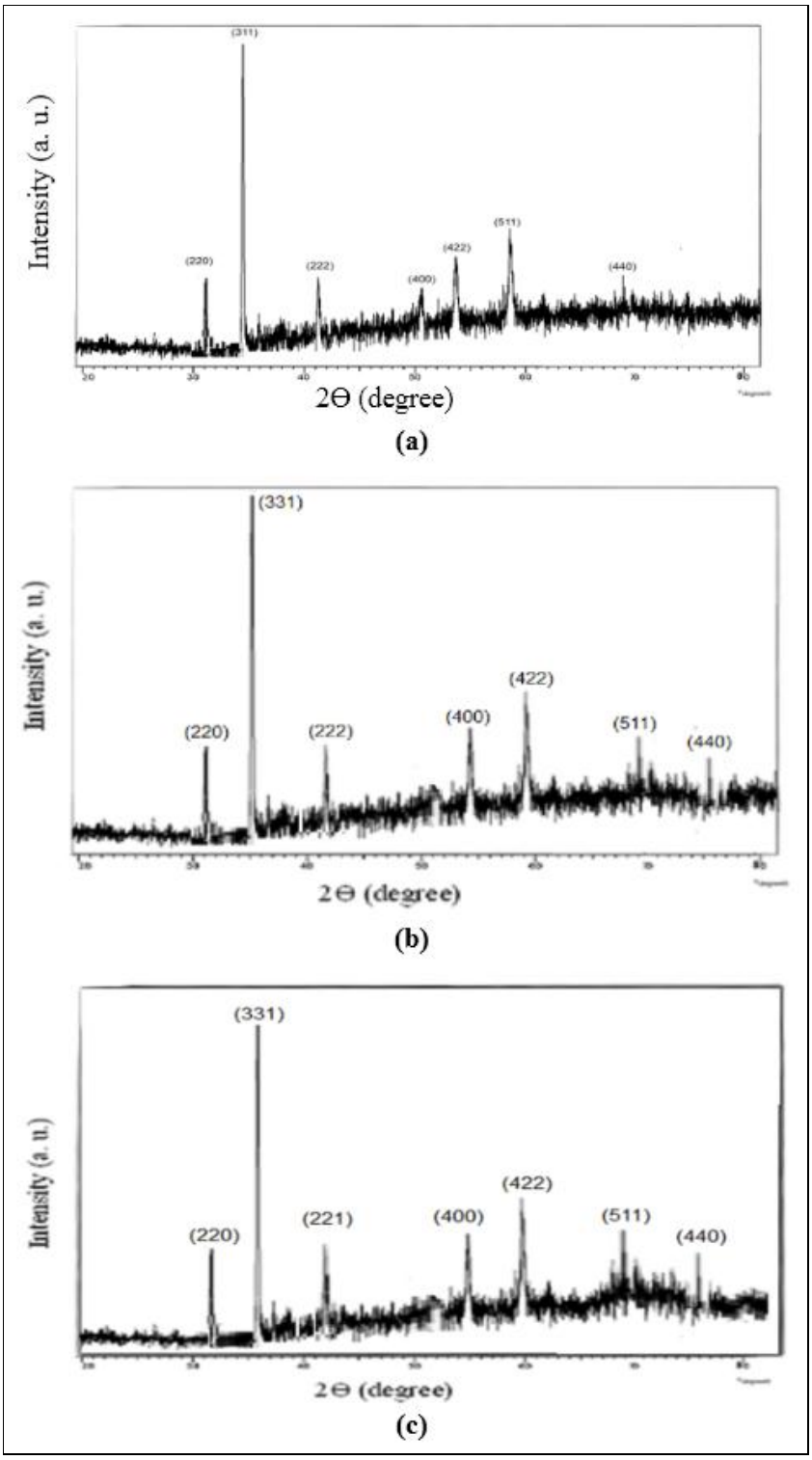

Fig. 1 (a – c). X-ray diffraction pattern of A1, A2 and A3, respectively

As well as notes of shape low intensity of some diffraction peaks with increasing zinc concentration due to the increased prevalence of positive ions at different locations (A-site) and (B-site) within the lattice, as shown in Table (1).

Table 1 represents the lattice constant and bulk density of compounds A1, A2 and A3, respectively

Samples	Lattice constant (°A)	Bulk density (gram/cm^3)
A1	8.36	10.028
A2	8.41	7.222
A3	8.67	4.417

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It was also study change in the bulk density of the samples prepared with different concentrations by using the relation (2). As shown in Fig. 2(a-c) the bulk density increases with the value of (x). The increase in density increase of zinc concentrate due to the atomic weight of zinc (amu) (7.14 gm/cm^3) greater than the atomic weight of manganese (amu) (1.738 gm/cm^3).

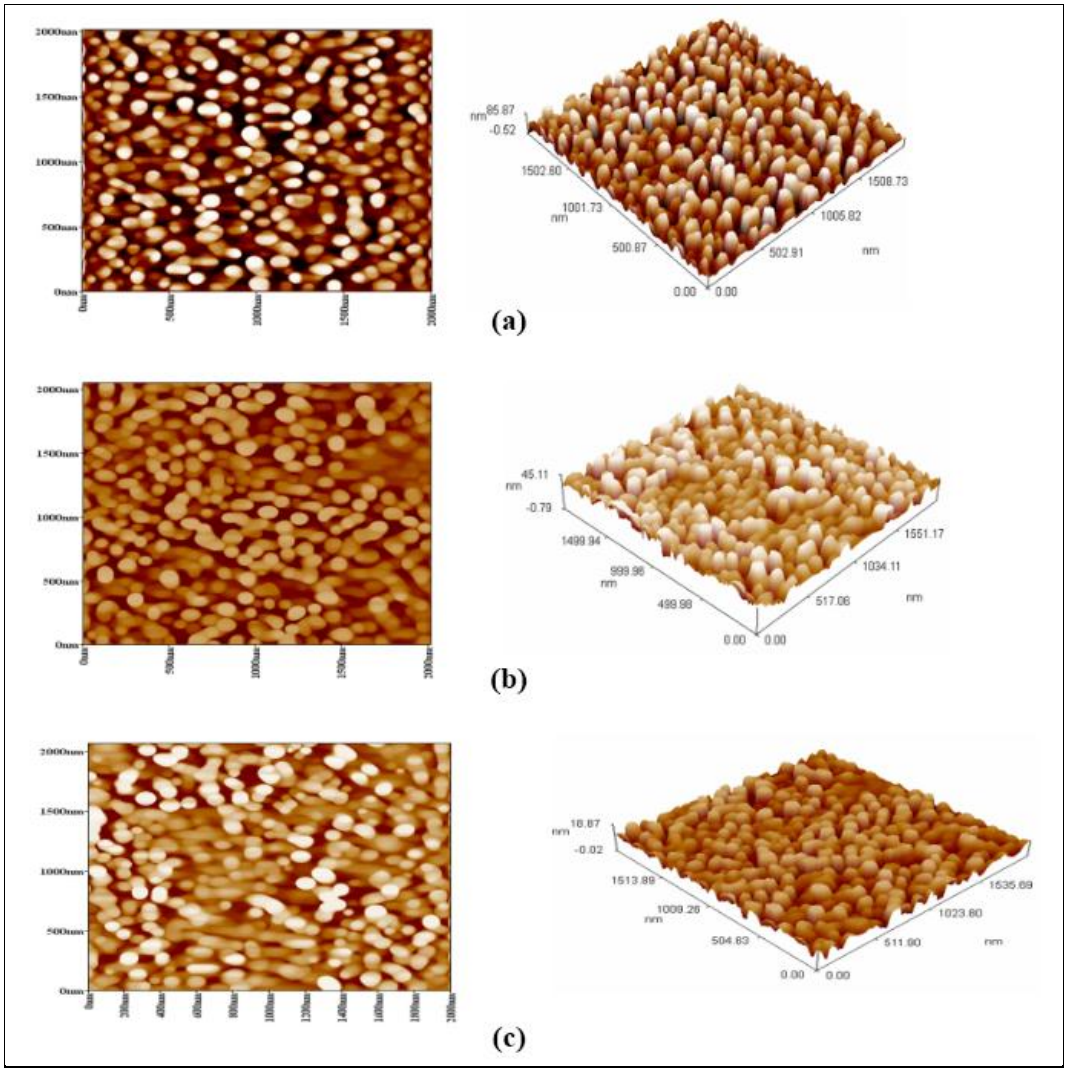

Fig. 2 (a–c) AFM images 2D & 3D compounds A1, A2 and A3, respectively

Increasing the concentration of zinc it causes increase the weight of the samples and thus lead to increased Bulk density. AFM has been conducting tests of compounds $Mg_{1-x}Zn_xFe_2O_4$ and values (x = 0.0, 0.4 and 0.6) as in Figure 2 which shows images of topographic analytical two-dimensional (2D) and three dimensions (3D), respectively. It has been found that the rate of the square root of the average value of the surface roughness decreases with increasing zinc concentration as shown in Table 2 It was found that the square root of the rate, the value of surface roughness and grains size rate decreases with increasing zinc concentration as shown in Table (2).

Table 2 shows the results obtained from the AFM examination of for compounds A1, A2 and A3.

samples	Roughness Average (nm)	Root Mean Square (nm)	Grain size (nm)
A1	21.6	24.9	91.25
A2	6.48	7.78	82.56
A3	2.48	2.93	67.65

The decreases in surface roughness rate with increasing zinc concentration explain the growth crystalline occurring horizontally on the surface and get well homogeneity between the grains this reduces of surface defects of the samples .

In Fig. 3(a–c) the distribution of grains groupware of samples are shown

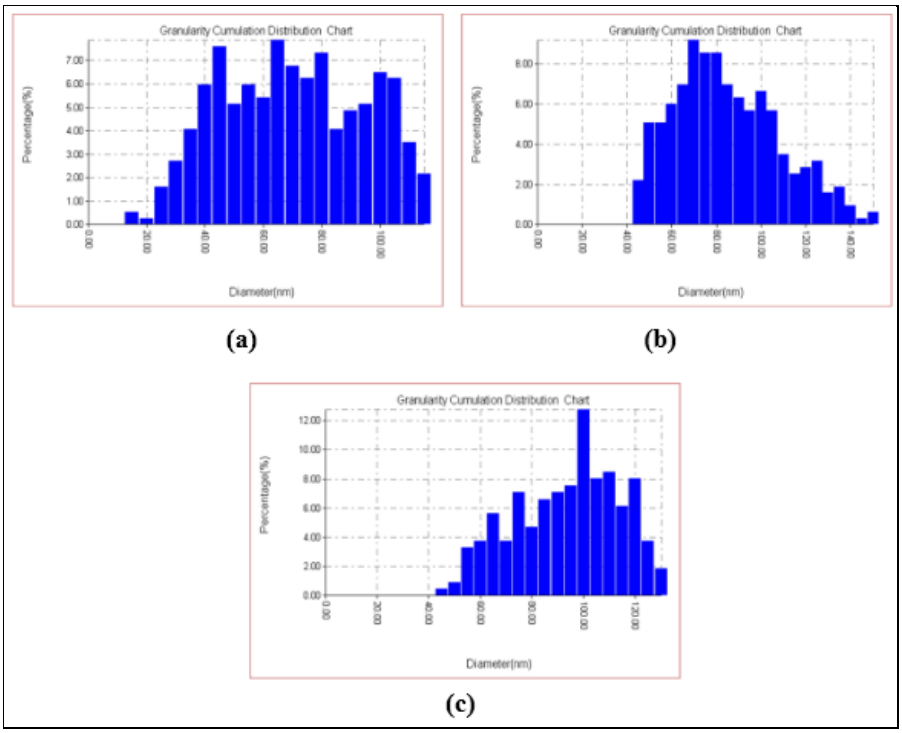

Fig. 3(a – c) the distribution of grains groupware of compounds A1, A2 and A3, respectively

It has been found that the average particle size increases with increasing values (X) This is consistent with the results of X-ray diffraction in that the grains size increases with increasing zinc concentration, but the rate of particle size measured by AFM is larger than the particle size measured by XRD and the reason for this is that AFM measures the size of the grains on the surface of the sample while the XRD measures the particle size within the sample and the grains are larger at the surface than it is in the interior, and this is in line with the findings of the researcher [10].

4- Conclusions

1- The crystal structure of all samples under study is cubic Spinel structure

2- The increase in zinc concentration it causes increase the weight of the samples and thus lead to increased Bulk density

3- Examinations an atomic force microscope showed that there are homogeneous in compositional structure and decrease the surface roughness of the Ferrites samples rate with increasing zinc concentration.

5- References

1- W.D. Kingery, D.R. Uhmann & H.K. Bowen (1976) " Introduction to ceramics" 2nd Edition, John Wiley and Sons, New York.

2- A . J. B. Fuller (1987) "Ferrites at microwave frequencies " Peter peregrines Ltd. London,.

3- M. Ajmai, A. Maqsood, (2007)" Materials Science and Engineering ", 139,pp (164–170),.

4- P.U. Mahamuni and B.K. Chougule (2010) "Structural, Dielectric and Magnetic Properties of Nanocrystalline Ni-Zn Ferrites", Scholar Research Library,Vol.1, No.1, p(112-118),.

5- J. Jacob, M. Abdulkhadar, A. Lonappan and K.T. Mathew (2008) "Microwave dielectric properties of nanostructured nickel ferrite", Bull. Mater. Sci., Vol. 31, No. 6, pp(847–851).

6- K. V. Kumar, R. Sridhar, D. Ravinder, and K. R. Krishna (2014) "Structural Properties and Electrical Conductivity of Copper Substituted Nickel Nano Ferrites", International Journal of Applied Physics and Mathematics, Vol. 4, No. 2, P (113-115).

7- B.D. Cullity "Introduction to Magnetic Materials (1972) ", Wesley, Mas achusetts , U.S.A .

8- Annual Book of ASTM Standards (1979), Part 13 , PP (180-182).

9- M. J. Iqbal, M. N. Ashiq, P. Hernandez-Gomezb and J. M. Munoz, (2007) "Synthesis, Physical, Magnetic and Electrical Properties of Al-Ga Substituted Co-precipitated Nanocrys - talline Strontium Hexaferrite, " Journal of Magnetism and Magnetic Materials, Vol. 320, No. 6, pp (881-886).

10-A. Ashok, T. Somaiah, D. Ravinder, C. Venkateshwarlu, C. S. Reddy, K. N. Rao, M. Prasad (2012) "Electrical Properties of Cadmium Substitution in Nickel Ferrites", World Journal of Condensed Matter Physics, Vol. 2 ,p (257-266).

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تصنيع ودراسة الخواص التركيبية لمركبات $Mg_{1-x}Zn_xFe_2O_4$ المحضرة بطريقة الترسيب الكيميائي

سيف عامر مهدي

قسم الفيزياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

Saifamer76@gmil.com

الملخص

تم في هذا البحث تحضير مركبات المغنيسيوم – الزنك ذات الصيغة الكيميائية ($Mg_{1-x}Zn_xFe_2O_4$) حيث أنّ (X=0.0, 0.4 and 0.6) وذلك باستخدام طريقة الترسيب الكيميائي , تم تلدين النماذج المحضرة عند درجة حرارة (700˚C) ولمدة (3hr). أجريت الفحوصات التركيبية والفيزياوية للنماذج المحضرة ,بين تحليل حيود الأشعة السينية أنّ جميع النماذج كانت ذات تركيب مكعب متمركز الأوجه (FCC) ذات معاملات ميل (400),(222),(311),(220) (511),(422) وإن زيادة تركيز الزنك يؤدي إلى زيادة ثابت الشبيكة (a) (8.36 ـ 8.67) وكذلك زيادة تجانس البنية التركيبية مسبباً تحسن في الخواص الفيزيائية, بينما الفحوصات المجهرية فقد أظهرت نقصان معدل خشونة السطح والحجم الحبيبي (Dnm) مع زيادة تركيز الزنك.

الكلمات المفتاحية: المغنيسيوم – الزنك فرايت , طريقة الترسيب الكيميائي , تحليل حيود الاشعة السينية