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Structural properties of $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ solid solution**

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Abstract: Crystallography information files (CIF) were designed formed $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ solid solution with the substitution factor $x=0$ to 1 with an increment of 0.1 depending on Vegard's law by using crystallography software. The effect of the substitution factor has been studied on some parameters and properties of the Co-Cu ferrite system, such as the effect of substitution factor on the lattice parameter, the volume of unit cell, and the density of the unit cell. Also, XRD patterns were estimated by crystallography software depending on the mathematical models of XRD. The XRD results showed a slight shift in the peak position varying with the substitution factor, these are due to the change in lattice parameter caused by the substitution of ions with different ionic radii. XRD also showed an increment in peak intensity varying with the substitution factor, that's due to an increase in the concentration of Cu which led to an increase in the density of electrons.

Keywords: cobalt copper ferrite, spinel solid solution, electron density map

1 Introduction

Ferrites are ferromagnetic oxide have a wide range of applications in material science due to their magnetic properties

[1, 2]. ferrite is classified as hard and soft ferrite depending on its permanent magnetization and the value of the coercive field [2, 3]. The hard ferrite usually has a hexagonal crystal structure (space group $P6_3/mmc$) [4, 5, 6], Hexa-ferrites have various technological and industrial applications in magnetic recording media, magnets, frequency devices, microwave devices due to their properties such as high Curie temperature and coercively, huge magnetization [5]. Strontium hex ferrite ($\text{SrFe}_{12}\text{O}_{19}$) and barium hexa-ferrite are the most famous material belong to this type [4, 7]. The soft ferrite is a material with a spinel structure with the formula AB_2X_4 where A and B are cations while X is an anion like oxygen, sulphur, selenium, and Tellurium [8, 9]. for the ferrite Spinel X is an oxygen O^{2-} and the trivalent anion is Iron Fe^{+3} . The A-site is located inside the tetrahedral void while B-site is located inside the octahedral void. The spinel ferrite has a cubic crystal structure (space group) [10, 11]. the soft ferrite has low coercively and high magnetization, it has 4 octahedral voids and 8 tetrahedral voids. Type of spinel ferrite and the net magnetization are determined according to the orientation of the voids, the most popular spinel ferrite is ferrimagnetic and antiferromagnetic which has the 180-degree domain [12, 13]. Also, spinel ferrite is classified as normal ferrite with the formula AFe_2O_4 and the inverse spinel ferrite with the formula $[\text{Fe}^{+3}]_{\text{Tetrahedral void}}[\text{Fe}^{+3}\text{A}^{+2}]_{\text{Octahedral void}}\text{O}_4$ according to the Crystal field stabilization energy (CFSE)[14–16] and the spin state in the d orbital of the A and B sites of the spinel [15, 17]. In this study, it will focus on CoFe_2O_4 which has an inverse spinel and substitute of Cu with various concentration and its effect on the crystal structure, XRD, and density.

2 Method

In this work, crystallography information file(CIF) number COD#1533163 from crystallography open database was used to build $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ solid solution by substitution of Cu^{+2} with Co^{+2} by factor x using crystallography software [18, 19] i.e. the occupancy of the Cobalt is changed by factor x and adding new Cu^{+2} at the same position of Cobalt by a

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Figure 1: Crystal structure of $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$.

factor $(1-x)$. the lattice parameter was calculated according to Vegard's Law as shown in the following equation [20].

$$a(A_xB_{1-x}) = (x) \cdot a_A + (1-x) \cdot a_B \quad (1)$$

where $a(A_xB_{1-x})$ is the lattice parameter for the solid solution, a_A is the lattice parameter for the A site atom, and a_B is the lattice parameter for the B-site atom, and x is the substitution factor (for this study $x=0, 0.1, 0.2 \dots 1$). The solid solution for this study is $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ of based on two compounds, the first is CoFe_2O_4 with lattice $a=8.3806 \text{ \AA}$ [21] and the second was CuFe_2O_4 with lattice $a=8.37 \text{ \AA}$ [22]. Also, both Cobalt ferrite and copper ferrite have the same crystal structure and the same space group (Fd-3m).

2.1 Estimation of X-ray diffraction pattern (XRD)

The XRD pattern was estimated using VESTA software [18] and Mercury software [23, 24, 25], the intensity of the XRD pattern was calculated using the following equation [24]

Figure 2: a) Variation of lattice parameter with substitution factor(x); b) Variation of lattice volume with substitution factor(x).

$$I_{hkl} = S \cdot L_p(2\theta) \cdot A \cdot P_{hkl} \cdot F_{hkl}^2 \quad (2)$$

Where I_{hkl} is the intensity of peak diffracted from hkl lattice plane, hkl are the Miller indices, S is the scaling factor, $L_p(2\theta)$ is the Lorentzian polarization factor, A is the X-ray absorption factor, P_{hkl} is the preferred orientation factor, and F_{hkl} is the structural factor. The XRD pattern intensity depends on many factors as described in Eq. (2), it depends on the nature X-ray source and the technique of generation of X-ray and many factors but the intensity of the peak mainly depend on the structural factor as shown in Eq. (3) [24]

$$I_{hkl} \approx F_{hkl}^2 \quad (3)$$

The structural factor depends on many factors; these are shown in Eq. (4) [24]

$$F_{hkl} = \sum_n f_n \cdot O_n \cdot e^{(2\pi i \{hx+ky+lz\})} \cdot e^{-W_n} \quad (4)$$

Table 1: Substitution of Cu^{+2} with Co^{+2} in AB_2X_4 system to build $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$.

Site	Atom	Composition	Occupancy	Wyckoff position	Coordinate		
					x	y	z
A-Site	Fe;Co;Cu		1				
	Fe^{+3}	$0.627\text{Fe}^{+3} + (0.373x)\text{Co}^{+2}$	0.627	16d	0.5	0.5	0.5
	Co^{+2}	$+(0.373[1-x])\text{Cu}^{+2}$	$0.373 \cdot x$	16d	0.5	0.5	0.5
	Cu^{+2}		$0.373 \cdot (1-x)$	16d	0.5	0.5	0.5
B-site	Fe;Co;Cu		1				
	Fe^{+3}	$0.745\text{Fe}^{+3} + (0.255x)\text{Co}^{+2}$	0.745	8a	0.125	0.125	0.125
	Co^{+2}	$+(0.255[1-x])\text{Cu}^{+2}$	$0.255 \cdot x$	8a	0.125	0.125	0.125
	Cu^{+2}		$0.255 \cdot (1-x)$	8a	0.125	0.125	0.125
X-site	O^{2-}	O^{2-}	1	32e	0.2566	0.2566	0.2566

Where the summation is over all atoms (n), f_n is the X-ray form factor which is a function of 2θ and the atomic number Z , O_n is site occupation or the factor of the coordinate of atoms, $\text{Exp}(2\pi i(hx+ky+lz))$ is the interface term which is a vector product between the position of atoms in the unit cell and a scatter vector of the hkl plane, and W_n is Debye-Waller factor or the atomic displacement factor which is caused by thermal vibration. All the previous terms affect the intensity of the XRD peak, so special crystal structure software were used to calculate the structural factor and the peak intensity and finally plotting the XRD pattern.

The relationship between the structural factor and the electron density in any plane is shown in Eq. (5). [18]

$$F_{hkl} = \int_V e^{2\pi i(hx+ky+lz)} \cdot \rho_{xyz} dV \quad (5)$$

Where ρ_{xyz} is the density of electron in the plane causing the diffraction.

2.2 Estimation of density

The density of the built CIFs is calculated using crystal maker software according to Eq. (5) [26, 27]

$$\rho = \frac{\text{Mass of unit cell (gm)}}{\text{Volume of Unit cell (cm}^3\text{)}} = \frac{n \times M}{V \times N_a} \quad (6)$$

Where ρ is the density in g/cm^3 , n is the number of atom per unit cell, M is the Molar Mass of the atoms, V is the volume of unit cell and N_a is Avogadro's number ($N_a = 6.022 \times 10^{23} \text{ mol}^{-1}$).

Figure 3: XRD patterns for $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$.

3 Result and discussion

The properties of solid solution $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ depend on the value of the substitution factor, the CIF file for $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ is shown in Figure 1.

The substitution of copper ion was performed depending on Vegard's Law, the occupancy of both A and B sites of the composition (i.e. Co and Cu in AB_2O_4) because the cobalt ferrite is an inverse spinel of Co^{+2} occupied in both A and B sites, Table 1 shows the process of substitution of Cu^{+2} ion with Co^{+2} with their Wyckoff position and coordinates of atoms.

Both Co^{+2} and Cu^{+2} have the same position in the crystal because both of them have the same oxidation state (i.e. +2) with different ionic radii, For Co^{+2} the ionic radii are $0.58 \text{ \AA}$ for the A-site (for Co^{+2} inside the tetrahedral void with coordination number IV) while for the Co^{+2} in B-site

Figure 4: Intensity of for $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ peaks (a): 111 peak; (b): 022 peak; (c): 113 peak; (d): 222 peak; (e): 004 peak; (f): 224 peak; (g): 115 peak; (h): 044 peak; (i): 135 peak; (j): 026 peak; (k): 335 peak; (l): 444 peak; (m): 155 peak; (n): 246 peak; (o): 137 peak; (p): 008 peak; (q): 337 peak

is $0.745 \text{ \AA}$ (for Co^{+2} inside the octahedral void with coordination number VI) [28, 29]. The ionic radii for Cu^{+2} is $0.57 \text{ \AA}$ for the A-site while for Cu^{+2} in B-site is $0.73 \text{ \AA}$ [28, 30]. In general, Co^{+2} radius is slightly greater than Cu^{+2} in about

$0.01\text{--}0.012 \text{ \AA}$, this is the reason for variation in the lattice parameter of the solid solution, the lattice parameter of CoFe_2O_4 is $a=8.3806 \text{ \AA}$ which is slightly greater than for CuFe_2O_4 $a=8.37 \text{ \AA}$ (i.e. the difference is about $0.01 \text{ \AA}$). Figure

Figure 5: Electron density of different planes for $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ peaks (a):111 plane; (b): 022 plane; (c): 113 plane; (d):222 plane; (e):004 plane; (f): 224 plane; (g): 115 plane; (h):044 plane; (i):135 plane; (j): 026 plane; (k): 335 plane; (l):444 plane; (m): 155 plane; (n):246 plane; (o): 137 plane; (p):008 plane; (q):337 plane.

2. Shows the variation of the lattice parameter and lattice volume with the substitution factor.

The lattice parameter and volume of unit cell increase slightly with the substitution factor x due to the difference in ionic radii. The volume of unit cell varying with x is estimated in Eq. (7).

$$V \left(\text{\AA}^3 \right) = 2.23063x + 586.3758 \quad (7)$$

3.1 X-ray diffraction pattern (XRD)

The calculated XRD patterns showed that there is a small shift in the peak position for all peaks. The shifting in peak position increase slightly with decreasing the substitution factor x , that's because the increasing of lattice parameter leads to an increase in the d spacing, and the increase in d space leads to a decrease in the 2θ position according to Bragg's law [31]. Figure 3: Shows the XRD patterns calculated using Mercury software.

The second result of the XRD is a very slight shift in the value of the intensity varying with the substitution factor. Figures 4a-q shows the variation of peak intensity with the substitution factor on the y-axis. It's known that the X-ray is scattered or diffracted from electronic clouds, which means for any peak the more intensity, the more electron density in that plane caused the diffraction. By decreasing the substitution factor the peak intensity increase i.e. increasing Cu^{+2} concentration lead to increase peak intensity, which means more density of electrons or the probability of finding electrons in this plane increase [11, 30, 32]. By comparing the last result with the atomic number of Co which has an atomic number equal to 27 while for Cu is equal to 29, that means 27 electrons for Co and 29 electrons for Cu [33]. The variation of intensity is affected by the ratio of electron density of both Co/Cu i.e. $27/29=0.931$ multiplied by the substitution factor the last result is also agreed with Vegard's Law [30, 34].

The XRD peak intensity is directly proportion with the square of the structure factor ($I_{hkl} \sim F_{hkl}^2$), also the structural factor is direct proportion to the density of electron of the plane caused the diffraction ($F_{hkl} \sim \rho_{xyz}$) so the intensity of the peak is proportional to the density of electrons for any plane caused the diffraction ($I_{hkl} \sim (\rho_{xyz})^2$) [18]. The distribution of the density of electrons is shown in Figure 5a-q for the lattice planes caused the diffraction. The distribution of electrons inside the lattice depends on many factors such as the type of bonds between atoms, the position of atoms in the plane of lattice, the atomic number of the basis, and the type of the lattice. In Figure 5 the density distribution occurs in the red region with a probability of 100%. The scale bars in Figure 5. Is between 0-1 from blue to red which refers to the probability of finding electrons from 0% at blue to 100% at the red region, i.e. XRD occurs from the red region only, and the intensity of the XRD peak indicates that the diffraction occurs from the red region of the crystal planes of the lattice rather than from that completely plane.

Figure 6: Density of $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ solid solution.

3.2 Density estimation

The density of the CIF files was calculated using crystal maker software, it is known that the density of the unit cell is in direct proportion to the mass and inversely proportional to the volume of unit cell. The density of the solid solution depends on the reduced mass of solid solution (dependent on Vegard's law). The volume of unit cell varied with the substitution factor because $V=a^3$ for the cubic lattice and the lattice also varied with the substitution factor according to dependence on Vegard's law. The results for the density of the $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ is shown in Figure 6. The results showed that the density decrease the substitution factor, this behavior of the density of the solid solution is due to the increment in volume of unit cell with the substitution factor ($\rho \sim 1/V$, $V \sim x$, so increase x lead to decrease density). Also, the density of solid solution depends on the variation of molar masses with the substitution factor ($\rho \sim M$, $M \sim 1/x$, so increase x leads to a decrease in the density). The equation for the density of the $\text{Co}_x\text{Cu}_{1-x}\text{Fe}_2\text{O}_4$ solid solution is shown in Eq. (8).

$$\rho \left(\frac{\text{g}}{\text{cm}^3} \right) = -0.12475x + 5.41839 \quad (8)$$

4 Conclusion

The XRD showed a slight shift in peak position due to the change in the lattice size, and a slight change in the peak intensity due to the change in concentration of Cu that leads to an increase in the density of electrons. Also, the density of the solid solution decreases linearly with the substitution factor. These results must be taken into consideration in

studying the solid solution and in the experimental design of the Co-Cu ferrite solid solution.

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