

The effect of calcination temperature on the structural properties of Nickel -Lanthanum ferrite nano powders

Mahmood A. Mohammed¹, Mohammed Z. Hasan², Bushra k. Sulaiman³

^{1,3}Northern Technical University, Technical Institute, Mechanics Department, Kirkuk, Iraq

² Northern Technical University, Technical College, Fuel and Energy Engineering Dep. Kirkuk , Iraq,

Abstract

Nickle-Lanthanum ferrite ($\text{Ni La}_x\text{Fe}_{2-x}\text{O}_4$) with $x=0, 0.04, 0.08, 0.12, 0.16$ and 0.2 , were prepared by sol-gel method. Nickel-Lanthanum ferrite nano powders are burnt at temperature of (200°C) . The obtained ferrite powders were calcined at temperatures of 400 and 600°C for three hours and the powders characterized by XRD to study the structural properties and the crystallinity of the ferrite powders . XRD patterns show a nano sized particles in the range of $(20-39\text{ nm})$ with different (x) of La ions and different calcined temperatures. Also the lattice constant and theoretical powder density were calculated and found to be in the ranges of $(8.32-8.36\text{ \AA})$ and $(5.35 - 5.75\text{ g/cm}^3)$ respectively.

1. Introduction

The magnetic ceramic structures with ions of (Fe^{+3}) as the main component are called Ferrite [1]. Many author's worked on ferrite research field, in the recent years has been created several models which completes the obtained results by researchers on metal oxides from research on metallic compounds [2]. Ferrimagnetic are oxide magnetic materials with dark brown or black cubic structures own the ceramic property which being hard and brittle. The magnetic properties of the ferrite materials are changed by controlling the preparation method, type of materials used and thermal treatments, [3]. Electric resistivity of ferrites can be varied from 10^{-4} to $10^9\ \Omega\cdot\text{m}$, as a function of their chemical composition [4]. Ferrites Considered a magnetic materials with high magnetic performance at the same time it has an insulating material behaviors, therefore it has been chosen in the product industry which operates at medium and high frequencies. They show dielectric characteristics, so they are not electrically conductive. This gives them preference over transition metals that have magnetic properties in many applications. Ferrites also have a spontaneous magnetization below Curie temperature (T_c), [5, 6]. Due to rapid progress in the fabrication of nanostructures, ferrite materials can be synthesized with nano sized particles. The chemical and physical properties of nano materials differ from those of bulk materials with the same chemical composition [7]. Many research groups are contributed to study the nano particle ferrites due to their many applications in magnetic devices, radar absorbing materials, Magnetic Suspension and computer hard drive magnets, etc. [8].

2. Experimental technique

Sol-gel auto-combustion method was used to synthesis Ni-La ferrite nano powder. $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ with purity $>99\%$, $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ with purity $>99.9\%$, $[\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}]$ with purity $>99\%$, $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ with purity $>99.9\%$ and $[\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}]$ were used to prepare present ferrite.

Ni-La ferrite powders were prepared in three stages as shown below:

- 1- Dissolve the each of nitrates and citric acid separately in 10 ml of distilled water and then mix the nitrate and citric acid all in one baker according to the percentage standard stoichiometric weight, dissolving and mixing are done by using a magnetic stirrer. After the mixing is completed, the ammonia solution is added in droplets to the solution until to obtain the pH equals to 7 with continuous stirring to get a dark brown solution.
- 2- After obtaining a clear brown solution with a pH equal to 7, the solution is stirred with heating at temperature of 90°C for three hours until to get a gel and then dry the gel inside an oven at 120°C for 24 hours
- 3- Increase the temperature of dry gel until the product gets burned at 200°C and thus we get the dark brown ferrite powders.

The burnt fine powders were calcined grinded to get nano particle ferrites.

3. X-ray characteristics measurements

Phase formation and powder size of prepared ferrites can be studied by using the X-ray diffraction. While, d-spacing are calculated by equation;

$$n\lambda = 2d \sin \theta \quad \dots\dots\dots(1)$$

,where ($\lambda = 0.15406$ nm) is the wave length of x-ray with copper source , and (θ) is the Bragg's angle .For the crystallite size of the powders less than 100 nm can be determined by equation below;

$$D = 0.9 \lambda / \beta \cos \theta \quad \dots\dots\dots(2)$$

where, D is the crystallite size, λ is the X-ray wavelength, θ is the Bragg's angle and β is the full width at half maximum(FWHM) of the main peaks in the XRD spectrum,[9].

Lattice constant (a) is defined as the unit cell of the crystal and calculated using equation as follows;

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

where,(a) is lattice constant, (λ) is the X-ray wavelength, and ($h k l$) is Miller index.

(d_x) is represents the x-ray density. The spinal lattice contains eight formulas per unit cell, as the equation:

$$d_x = 8 M_w / N.a^3 \dots\dots\dots(4)$$

where, (N) is Avogadro's number, M_w is the molecular weight

4.Results and discussions

XRD patterns for prepared ferrite powders at 200^oC denoted to Ni-La ferrite system showing the presence of (NiO) phase in XRD spectrum, as shown in figures (1),(2) and (3). The calcined powders at temperatures 200,400 and 600^oC tends to the presence of metal oxide phase which denote by (*) in the ferrite powders with composition (x=0,0.04,0.08,0.12,0.16,0.2) It means that some of calcined temperatures are not enough to transfer it to single phase structure. .These figures also shows XRD spectrum with planes (220), (311), (222), (400), (422), and (511).

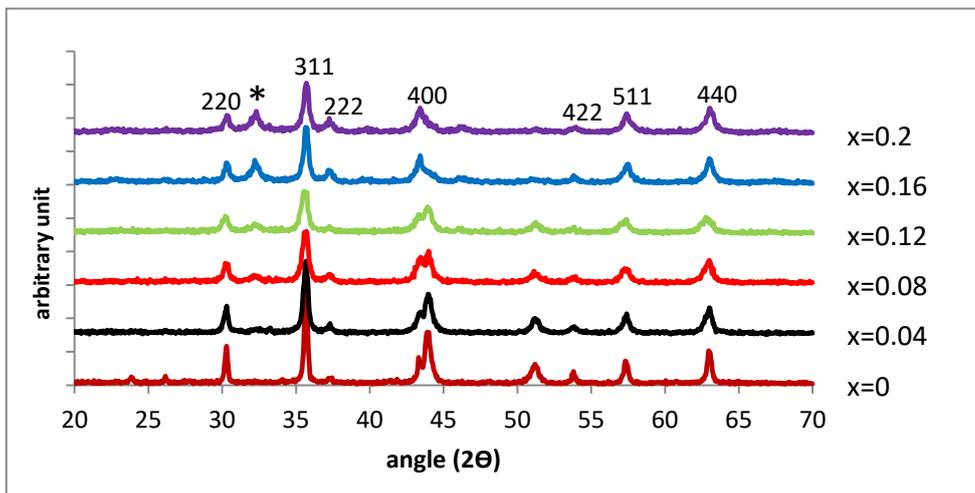


Figure (1): shows XRD pattern of the ferrite powders with different composition at temperature of 200 °C .

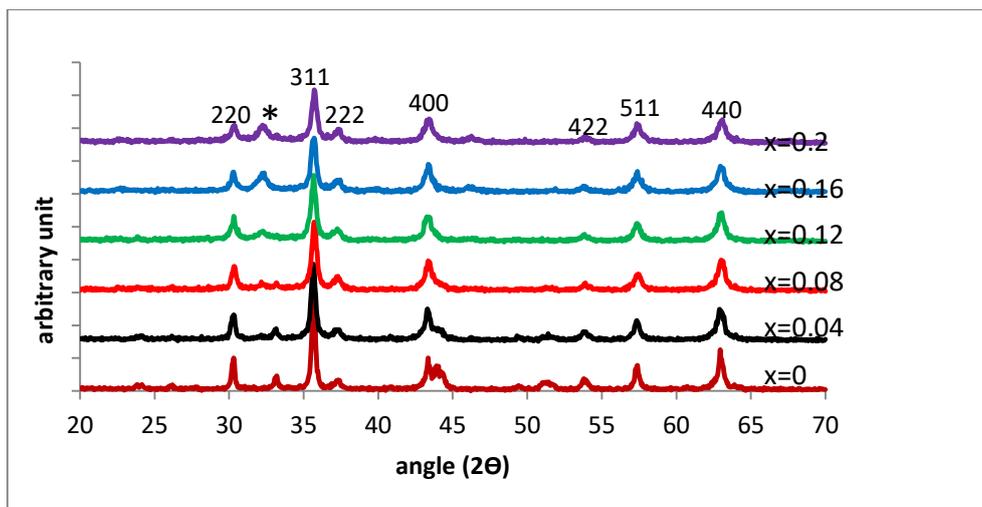


Figure (2): shows XRD pattern of the ferrite powders with different composition at temperature of 400 °C

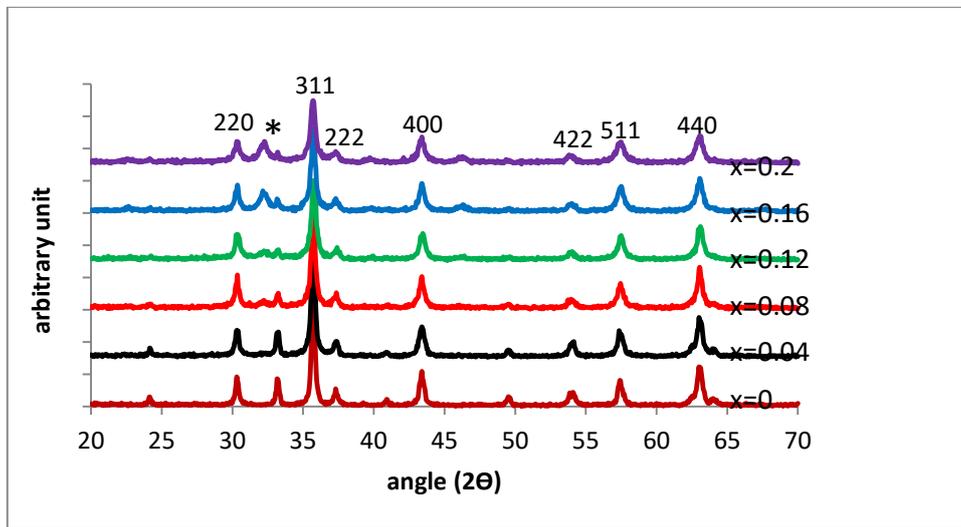


Figure (3): shows XRD pattern of the ferrite powders with different composition at temperature of 600 °C .

The crystallite size of the ferrite powders were calculated by Scherer formula (2) with FWHM of the main peak [311] and found it in the range of 20-38 nm. And this shows the ferrite powder is synthesized immediately by sol-gel method. Figures (4) shows that temperatures 200 and 400 °C, While the average crystallite size values are approximated with an increasing of substitution values (a) at calcined temperature of 600 °C, these variations in crystallite size with calcination temperatures of the ferrite powders are due to the recrystallization of the prepared ferrite structure.

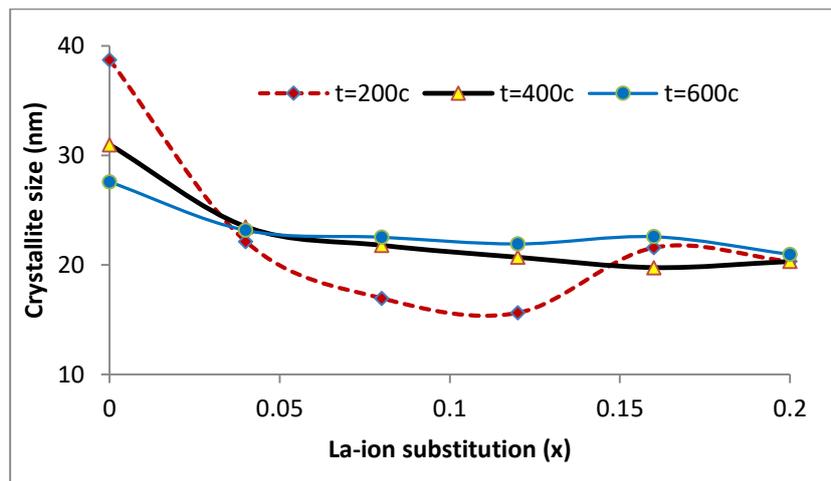


Figure (4): shows the crystallite size as a function of (x)

The lattice constant (a) for main peak [311] of the Ni-La ferrite powders was calculated by using equation (3). The lattice constant values (a) varied in the range of (8.3388 - 8.36114 Å) with increasing the La-ion substitution (x) from 0.0 to 0.2 for all ferrite powders calcined at temperatures 200, 400, 600 °C. These variations are due to the ionic radius of La⁺³ ion (1.15 Å) is greater than that of Fe⁺³ ion (0.64 Å) as shown in figure (5).

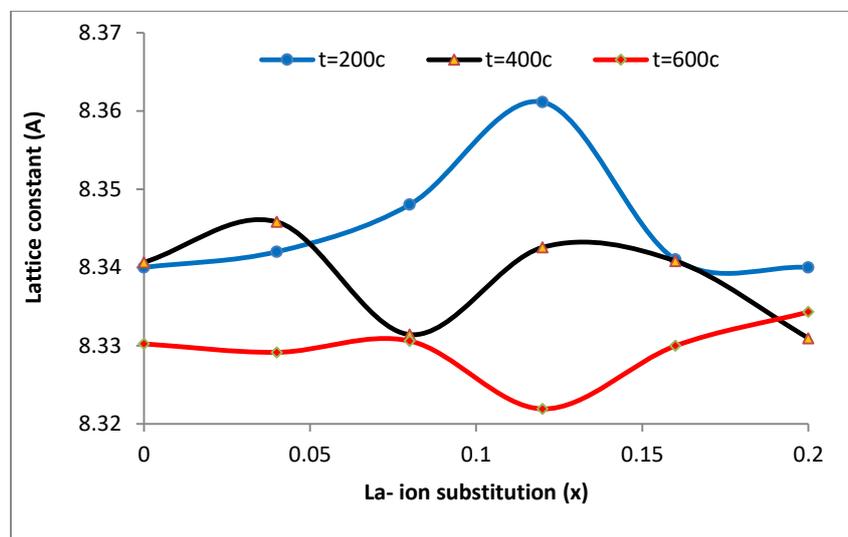


Figure (5): shows La ion concentration (x) with lattice constant (a) calcined at temperatures of 200, 400, 600 °C.

X-ray density (ρ_x) of Ni-La ferrite nano powders was calculated using equation (4). It observed increases from 5.37 to 5.8 gm/cm³ for all prepared ferrite compositions at different calcinations temperatures. It concludes that the lattice parameter was increased with increasing La⁺³-ion substitution so the density is increased with increasing of La⁺³. And these are due to substitution of Fe⁺³ ions (0.64 Å³) by larger La⁺³ ions (1.15 Å³) in the Ni-La ferrite system, and figure (6) shows these indications.

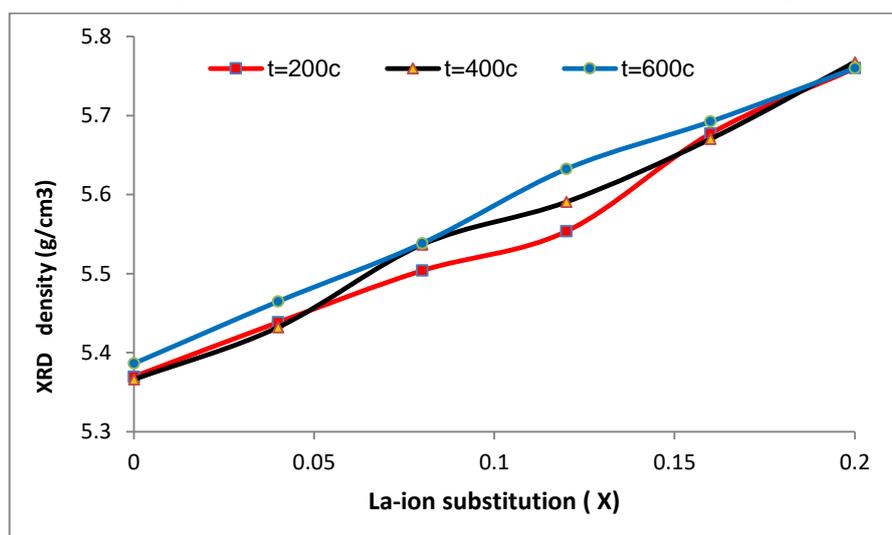


Figure (6): shows an effect of La ion concentration (x) on the XRD or theoretical density calcined at temperatures of 200, 400, 600 °C.

5. Conclusion

In the present work, the research is study the action of La-substitution and calcinations temperatures on the characteristic of the La- substituted Ni-ferrite system, this study is to find the optimum La-ion concentration. From this work, the ferrite system conclusions are:

- Sol-gel method is a suitable to prepare the Ni La_xFe_{2-x}O₄ ferrite with nano sized particles in the range of (20-39 nm), reactive specific surface area 30-70 m²/gm and polycrystalline ferrites.
- Ni-La ferrite nano powder was successfully prepared by using sol-gel method at 200°C.
- Crystallite size is inversely proportional to the calcined temperatures and it decreases with increase in the La⁺³ substitution for Ni-La ferrite system.
- Theoretical density increased with increasing of calcined temperatures and it decreases with increasing in the La⁺³ ion concentration for Ni-La ferrite system.

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