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Physical and Mechanical Properties of Synthesized Doped Nanoferrite

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Abstract

Nanoferrite materials have been synthesized by sol-gel auto combustion method. The effect of doping different percentages of Y_2O_3 (0.34 µm) on the physical and mechanical properties of selected mixed ferrite [$(Li_{2.5}Fe_{0.5})$ $0.9(Co_4Fe_2O_4)$ $_{0.1}$] by adding 10% Cobalt ferrite was studied. Physical properties (i.e. .density, porosity and water absorption) were affected by the doping, where the density increased about 32% at 6 wt% Y_2O_3, while porosity has a drastically decreased about 80% at 6% Y_2O_3 and has a correlation effect on the mechanical properties(Splitting tensile strength and Vickers microhardnss). The fracture strength at 1 % wt. of Y_2O_3 has doubled value of the undoped sample and then decreased. The same behavior shows with the testing of Vickers micro hardness.SEM (Scanning electron microscopy) micrographs revealed that the microstructure of the fracture surface of the samples consist of detached approximately closely packed particles and also showed the formation of micro agglomerated particles with some voids . By doping with Y_2O_3 the pores decreased and a dense material obtained

Keywords: *Nanoferrite, Y_2O_3, density, splitting strength, Vickers micro hardness.*

1. Introduction

Nano crystalline lithium ferrite has been investigated with last years due to its potential use in the microwaves field as a replacement for garnets or as a memory core [1, 2]. Due to its important in construction and engineering of many electromagnetic and microwave devices, lithium ferrite has been widely investigated material [2,3]. This material crystallizes in the spinal structure AB_2O_4, where A and B denote lattice site tetrahedrally and octahedrally coordinated by oxygen ions respectively[4] .Lithium ferrite is an unusual and in the same respect, a remarkable material. Where, several research programs have been undertaken to study its fundamental properties and to develop high-power microwave materials from it. The distinctive properties of lithium ferrite are the following:

1- The lithium ion is monovalent; i.e., in order to preserve charge balance, Li^+enters the lattice in combination with Fe^{3+} ion. The compound may be thought of as $(Li_{2.5}Fe_{0.5})Fe_2O_4$. Lithium ferrite can be prepared with low value of ΔH.

2- An ionic ordering can be established in lithium ferrite. Lithium enters the spinel lattice on the octahedral sites (i.e., the spinel is inverted).

The rare earth ions have unpaired 4f electrons that have a role to originate magnetic anisotropy due to their orbital shape, where the magneto-crystalline anisotropy in ferrite in related to 4f-3d coupling between the transition metal and rare earth ions, thereby doping rare-earth into spinal Li-Fe ferrite can improve their electrical and magnetic properties[5]. Wende and Langbein (2006), investigated the thermal decomposition of freeze - dried Li-Mn (II)-Fe (III) formatted precursor by differential thermal analysis (DTA), *thermal gravimetric analysis* (TGA) and mass spectroscopy. It was found that the thermal decomposition of a homogeneous freeze - dried lithium manganese Tiron formats, followed by an annealing, is a suitable method for preparing a single phase solid solution ferrite ($Li_XMn_{1-X}Fe_{2-2X}$ O4, with $0 \leq X \leq 1$), at relatively low temperature [6]. Altavilla et al. (2009), have been studied the

synthesis of monodispersed Fe_2O_4 (M=Fe, Co, Ni) ferrite nanoparticles: effect of reaction temperature on the particle size. The possibility of preparing monodispersed transition metal-oxides nano- particles covered by functionalized long chain organic molecules, in the sub size range of 20 nm, has recently opened an entire field of research [7].

De Fazio et al. (2011), showed the electromagnetic properties of manganese-zinc ferrite with lithium substitution. Polycrystalline manganese-zinc ferrite with lithium substitution of composition $Li_{0.5x}Mn_{0.4}Zn_{0.6-x}Fe^{2+}{}_{0.5x}O_4$ ($0.0 \leq x \leq 0.4$) were prepared by the usual ceramic method [8]. Arana et al. (2012) studied the Li-substituted Mn-Zn ferrite structural and magnetic properties after different thermal treatments. Lithium of composition $Zn_{0.6}Mn_{0.4}Fe_2O_4$ and $Li_{0.2}Zn_{0.2}Mn_{0.4}Fe_{2.5}O_4$ were prepared by the self-combustion sol-gel method. Incorporating Li to the crystalline lattice increased the saturation magnetization and promoted a decrease in the secondary phase's segregation [9]. Rosaiah and Hussien (2013) studied the preparation of the ferrite by hydrothermal synthesis. XRD spectrum exhibited predominate (200) orientation peak at $2\theta = 43.63$ corresponding to cubic structure. Electric and dielectric properties were studied over a frequency range of 1Hz-1MHz [10]. Agami et. al. (2014), investigated the structural, IR, and magnetic studies of annealed Li-ferrite nanoparticles nano-particles of spinel Li-ferrite, $Li_{0.5}Fe_{2.5}O_4$, were prepared by sol-gel auto combustion technique and annealed at different temperatures (T_a = 673, 873, and 1073 K)[11].The aim of this research is studding the physical and mechanical properties of synthesized nnoferrites doped by Y_2O_3.

2. Experimental Work

2.1. Sample Preparation

The numbers of samples and the percentage of Y_2O_3 are listed in Table (1).

Table 1,
Shows the numbers of samples and Percentage of Y_2O_3

No.	Number of samples	Percentage of Y_2O_3
1	3	0.5
2	3	1
3	3	2
4	3	4
5	3	6

2.1. Preparation of Nano-Ferrite by Co-Precipitation

1. Hydrated cobalt nitrate is dissolved in 50/50 % distilled water – ethanol ratio with 0.5(Molarity) M.
2. Hydrated lithium nitrate is dissolved in 50/50 % distilled water – ethanol ratio with 0.5 M.
3. Hydrated iron nitrate is dissolved in 50/50 % distilled water – ethanol ratio with 0.5 M.
4. The cobalt solution is mixed with iron solution, where the ratio of cobalt solution to iron solution was selected according to a definite chemical stoichiometric ratio as (Fe: Co=2:1) by using magnetic stirrer.
5. The lithium solution is mixed with iron solution, where the ratio of lithium solution to iron solution was selected according to a definite chemical stoichiometric ratio as (Fe: Li =5:1) by using magnetic stirrer.
6. The two solutions are mixed by using magnetic stirrer.
7. 7. Addition of the resulting cobalt ferrite with different weight percentage (2, 5, 10, 15 and 20 wt. %) to lithium ferrite.
8. Addition of surfactant material (glucose).
9. Addition of ammonium hydroxide drops to the mixed solution until the gel bed was formed.
10. PH of the solution was measured, where the gel formation begins at PH 6.5.
11. Addition of citric acid that leads to hear notifying the combustion which helps in reducing the particle size of produced gel.
12. Filtrate the solution with filter papers to get out the gel.

2.2. Drying and Calcination

The filtered gel was then dried at temperature 80°C for 6 hours in a programmed electrical oven. The gel was then crushed and calcined at temperature 800°C at heating rate 10°C/min for 1 hour. The powders were finally cooled by switching off the furnace to room temperature.

2.3. Powder Compaction

Poly vinyl alcohol (PVA) with 2 wt. % was mixed with the powder for (30) min. Then, the powder was pressed unixally in Stave stainless steel at (374) MPa pressure to have a compacted specimen with diameter (10mm) and thickness (4 mm).

2.4. Sintering Process

The sintering processes of the compact samples were carried out in air atmosphere. The sintering temperature used was 1200 °C for two hours, with a heating rate and cooling rate of 10°C/min as shown in the Figure (1).Then, the dimensions and masses of sintered samples were measured to determine apparent density , porosity and water absorption using Archimedes method while splitting tensile strength measured by Brazilian test and the micro hardness measured by Vickers test .

3. Results and Discussion

Physical Properties of Doped Nano-Ferrite

Apparent Density and Porosity

Figure (2) presents the apparent density versus different weight percentages of Y_2O_3. It has been shown that the apparent density of prepared nano-ferrite samples is increased with increasing Y_2O_3 content. This may be due to the removal of micro pores in their microstructure during the sintering process. During the sintering, the time of sintering and the temperature are very important parameters to satisfy the diffusion of particles and then increasing mass flow rate through the pores which leads to increase the apparent density and decrease the porosity, as shown in Figure (2). Sintering at high temperature causes high diffusion rates and higher densification.

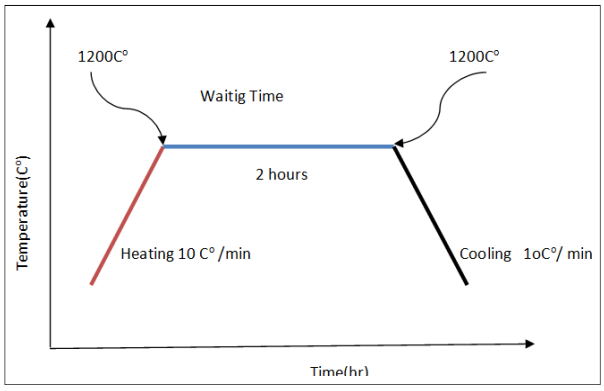

Fig. 1. Single heating cycle of the applied sintering process.

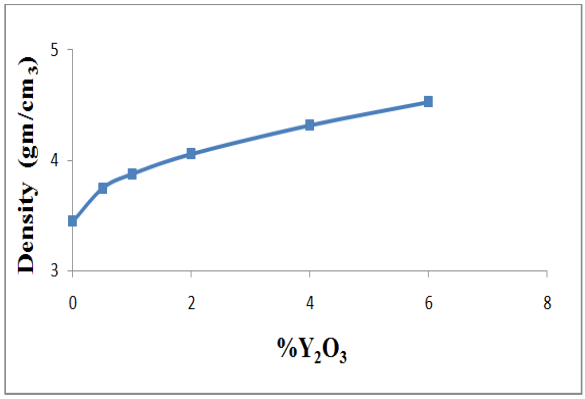

Fig. 2. Density for ferrites at different percentages of Y_2O_3.

Figure (3) shows the effect of Y_2O_3 addition on the porosity of ferrite system sintered at 1200°C for two hours. As seen the porosity decreased rapidly with increasing the Y_2O_3 content which may be due to the filling of the pores, which gives higher densification as shown in the density results.

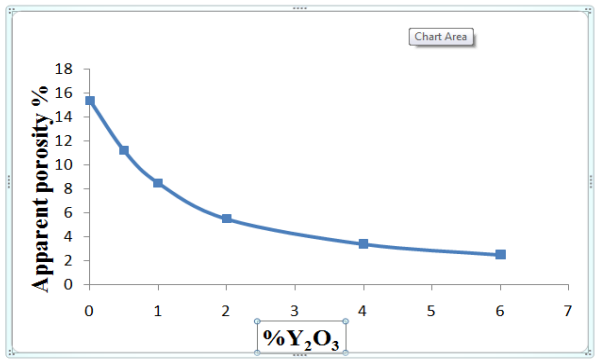

Fig. 3. Apparent porosity for different percentages of Y2O3.

Water Absorption

Water absorption is shown in Figure (4) for different weight percentages of Y_2O_3. The water absorption decareases with increasing the precentage of Y_2O_3. It is well known that the water absorption is the physical property upon that dopends on the appearent porosity, where the water enters the open pore channel.

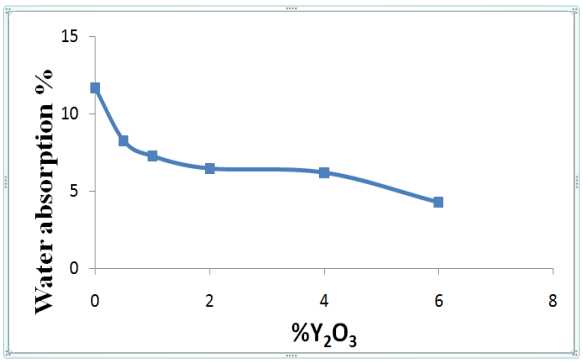

Fig. 4. Water absorption for different weight percentages of Y_2O_3.

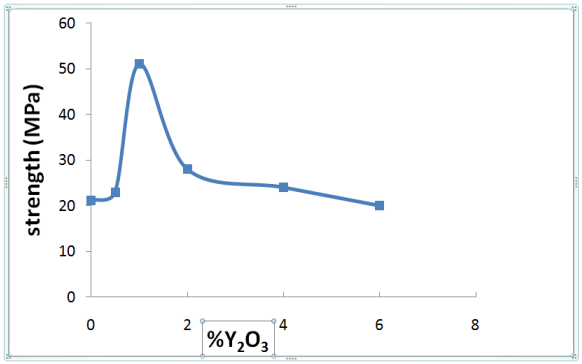

Fig. 5. The splitting tensile strength of nano-ferrite at different percentages ofY_2O_3 sintered at 1200°C for 2 hrs.

4. Mechanical Properties

Splitting Tensile Strength (Brazilian Test)

Figure (5) shows the effect of Y_2O_3 percentages on the fracture strength of nano-ferrite which is sintered at 1200℃ for two hours. It is clear a bell shape formed from the relation between Y_2O_3 percentage and fracture strength of the nano-ferrite which increased with increasing the Y_2O_3 content having the highest value (51 MPa) at 1wt. % Y_2O_3 and then decreased. As shown in the figure that the addition of Y_2O_3, especially at 1wt. % has highly improved the splitting tensile strength of the ferrite system, its value is more than twice that of the ferrite system. The increasing in the splitting tensile strength of the doped ferrite is due to density improvement with less porosity which leads to increasing the particles bonding. After 1wt. % Y_2O_3, the fracture strength decreases with further addition of Y_2O_3, and this may be due to the presence of residual porosity in the ferrite system. Residual stresses in the samples are another factor that may have contributed to the reduction in splitting tensile strength [12]. There is a difference in the thermal expansion coefficient between ferrites and Y_2O_3, and this mismatch in the thermal expansion coefficient could produce residual stresses near ferrites-Y_2O_3 grain boundaries. And, that could also result in micro cracking which lowers the strength values [13].

Vickers Micro Hardness

The micro hardness of material is an important mechanical property because it relates how much the material will inelastic deformed when a surface load is applied. The indentation diameters of micro Vickers tester for sintered samples are very small and do not appear in Vickers tester instrument. The light optical microscope with a computer program was used to analyze the image and calculate the micro hardness. The Vickers micro hardness value of the ferrite system doped with different percentages of Y_2O_3 that sintered at 1200° C for 2 hrs is shown in Figure (6). It is clear that the hardness of samples increased with increasing Y_2O_3 content until 1wt. % and then decreased while has the same behavior of splitting tensile strength. Because the hardness value are highly correlated with the relative density and porosity, so reducing the number of defects in a sample is a common way of decreasing its micro hardness[14]. The hardness has a maximum value at 1 wt.% Y_2O_3 which will result in a material being more resistance to the indentation at a given load, which will signify that the material will be able to plastically deform more so than the ferrite ceramic. Sometimes, the mechanical properties, such as hardness are decreased when the grain size is decreased in the nano range, as reported by Andrievski and Glezer [15]. Furthermore, in this work, Vickers micro hardness is decreased slightly after 1% Y_2O_3.

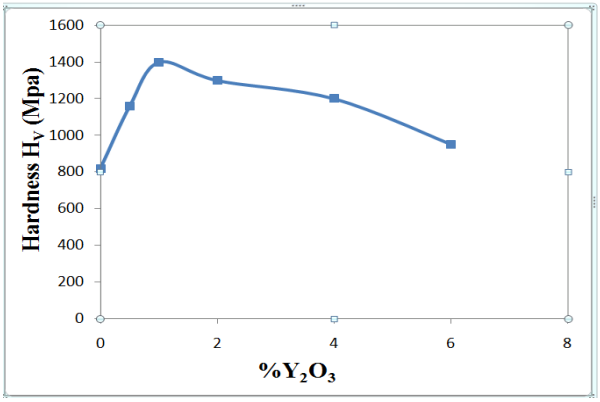

Fig. 6. Vickers micro hardness for the different percentages of Y_2O_3.

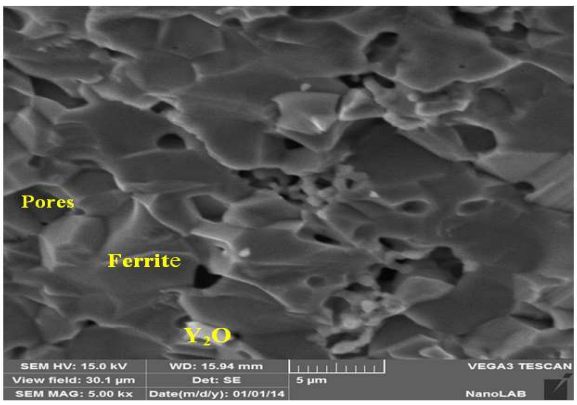

Fig. 7. SEM image of fracture surface for 0.5% Y_2O_3 additives.

5. SEM of Doped Nano-Ferrites

SEM imaging was conducting to observe the shape and morphology of samples. The SEM micrographs were taken from the fresh fracture surface of a ferrite body compacted and sintered at 1200° C from the composition $[(Li_{0.5}Fe_{2.5}O_4)_{1-x}(CoFe_2O_4)_X]_{1-y}(Y_2O_3)_y$ obtained with the classical ceramic technology presented. These micrographs are shown in Figure (7) to Figure (11) . It is clear that the fracture surface is an intergranular fracture (equiaxal), and the microstructure displays an irregular (non- equiaxal) fine grain microstructure with average grain size that are slightly larger than the ferrite powders particle size.

It is evident from the micrographs that the microstructure of the surface consists of detached, approximately closely-packed particles. Also, these images show the formation of micro agglomeration particles and some voids, where pores are located at the junctions of agglomerates.

The black and dark regions correspond to the ferrites particles and pores respectively, while the lighter areas are for Y_2O_3 phase. Furthermore, it is clear that by increasing Y_2O_3, the porosity decreased and denser materials obtained. The crack propagation occurs near the pores in the microstructure, where the pores act as stress density resulting in easy crack propagation path. It is also clear that some of the grains have grown when the sintering at 1200 ℃ in the preferred orientation. The fracture nature of the prepared nano-ferrite from lithium ferrite-10% cobalt ferrite – Y_2O_3 seems to be brittle which is clear from the fracture samples after the indirect tensile test (Brazilian test).

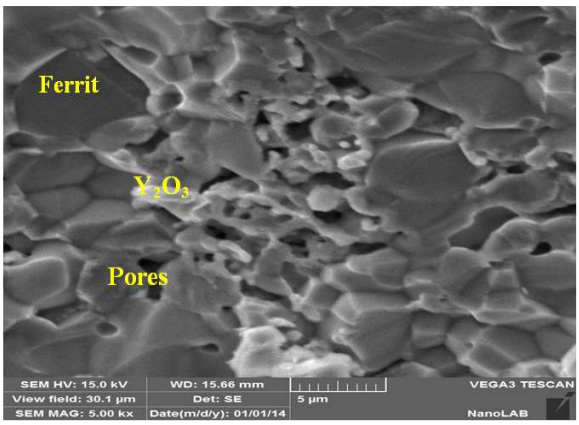

Fig. 8. SEM image of fracture surface for 1% Y_2O_3 additives.

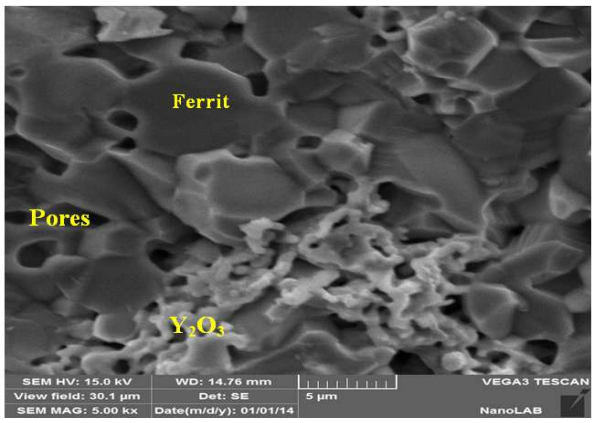

Fig. 9. SEM image of fracture surface for 2% Y_2O_3 additives.

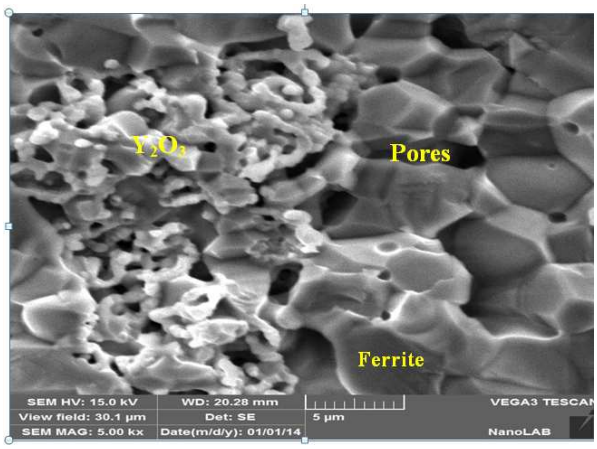

Fig. 10. SEM image of fracture surface for 4% Y_2O_3 additives.

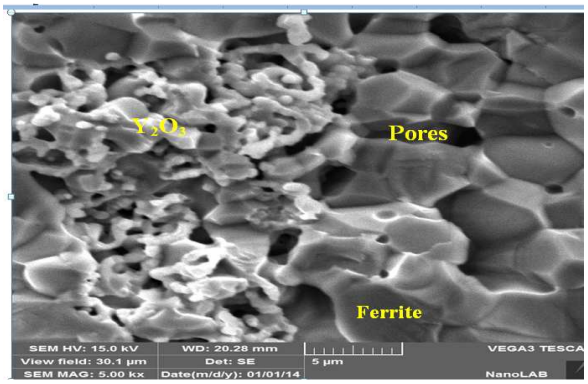

Fig. 11. SEM image of fracture surface for 6% Y_2O_3 additives.

6. Conclusions

To summarize the main ideas obtained, the following conclusions can be concluded from this work:

1- Mixed ferrites (lithium ferrites-cobalt ferrite) were successfully prepared by sol-gel technique.
2- SEM micrographs are established that the use of nanopowder produced by sol-gel technology leads to uniform and dense ferrite bodies, where the structure is compact with fewer amounts of pores.
3- Physical properties, such as density have incresed with Y_2O_3 content.
4- Porosity and water absorption decrease with the increasing content of Y_2O_3.
5- Fracture strength, micro hardness and the other properties are improved by the addition of Y2O3, especially at 1 wt. %.

7. Referances

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دراسة الخواص الفيزيائية والميكانيكية للفرايت النانوي المطعم

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الخلاصة

حضرت مواد الفرايت النانوية بطريقة (السول – جيل) ذاتية الاحتراق وتم دراسة تأثير اضافة نسب مختلفة من اليتيريا ذو حجم حبيبي (٠.٣٤) مايكرون على الخواص الفيزيائية والميكانيكية للفرايت المحضر $[(Li_{2.5}Fe_{0.5})_{0.9}(Co_4Fe_2O_4)_{0.1}]$. ان الخواص الفيزيائية مثل (الكثافة والمسامية وامتصاصية الماء) تأثرت باضافة اليتيريا حيث ازدادت الكثافة بزيادة نسب الاضافة اما المسامية فقد تقلصت بصورة كبيرة والتي لها تأثير على الخواص الميكانيكية مثل (متانة الشد وصلادة فيكرز) . تضاعفت متانة الكسر عند بنسبة (١%) من اليتيريا ومن ثم تناقصت . نفس التصرف عند حساب صلادة فيكرز. اظهرت صور الالكتروني الماسح بأن التركيب المجهري لسطح الكسر للنماذج يحتوي تقريبا على حبيبات متجاورة متراصة وكذلك تكوين دقائق مايكروية متجمعة مع وجود فجوات . وبعد باضافة اليتيريا قلت المسامات وتم الحصول على مادة اكثر تراصاً.