

Synthesis of nanocrystalline NiCoMn ferrite and investigation their physical properties

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Abstract: Nickel Ferrites nanoparticles doped with cobalt and manganese ($\text{Ni}_{0.99}\text{Co}_{0.01}\text{Mn}_{0.01}\text{Fe}_{1.99}\text{O}_4$) were synthesized by sol–gel combustion method. The effect of calcination temperature on the properties of the ferrite was also studied. The structural properties of all samples investigated using X-ray diffraction technique. The XRD analysis indicated that the all samples have cubic spinel structure. Their $M(H)$ loops have been traced using a Vibrating Sample Magnetometer (VSM). The saturation magnetization (M_s) was increased as the calcination temperature increased.

Keywords: Ferrites; nickel ferrite; dopants, sol–gel combustion method.

Introduction

In the recent years, ferrite nanoparticles have been the subject of much interest because of their applications in permanent magnets, magnetic drug delivery, microwave devices, high density information strong technology and gas or humidity sensors [1,2]. Ni ferrites doped with small amounts of Co and Mn has indicated to be a material with high sensivity for oxidizing gases[2,3]. In this work, ferrite nanoparticles were synthesized by sol-gel combustion method. This method is a unique combination of the chemical sol–gel process and the combustion process. In this method calcination temperatures are relatively low and produces homogeneous powder[5].

Experimental

In this work, the ferrite compositions ($\text{Ni}_{0.99}\text{Co}_{0.01}\text{Mn}_{0.01}\text{Fe}_{1.99}\text{O}_4$) synthesized from nickel nitrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], ferric nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$], cobalt nitrate [$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], manganese nitrate [$\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$] and citric acid [$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$]. The stoichiometric molar amounts of metal nitrate and citric acid was first dissolved in to deionized water to form the sol. The molar ratio of nitrates to citric acid was 1:1. Ammonia was also slowly added to the sol to adjust the pH value at about 7 For all samples preparation processes. During this procedure, the sol was continuously stirred by a magnetic agitator. Then, the sol was heated at 75 °C and stirred continuously till to obtain a xerogel. then the xerogel heated in oven, at 200 °C for 2 h and then at 300 °C for 0.5 h. At a proper temperature ignition started and the dried gel burnt in a self-propagating combustion manner until all the gel was burnt out completely to form a fluffy loose powder. Finally, the as-burnt powders were calcined in the furnace at 500 °C, 700 °C and 900 °C for 2 h with a heating rate of 3 °C/min to obtain the single phase ferrite.

The composition and crystal structure of the product were determined by x-ray diffraction (x-ray source is of Cu $K\alpha$ with $\lambda = 1.54056 \text{ \AA}$). Magnetization properties were studied by a

vibrating sample magnetometer (VSM Lake Shore 7400), through the heystersis loops measurments.

Results and Discussion

Fig. 1 shows the XRD patterns at room temperature for the $\text{Ni}_{0.99}\text{Co}_{0.01}\text{Mn}_{0.01}\text{Fe}_{1.99}\text{O}_4$ samples. (a) the pattern for sample that was calcinated at 500 °C, (b) is that of for sample that calcinated at 700 °C and (c) is that of for the sample calcinated at 900 °C.

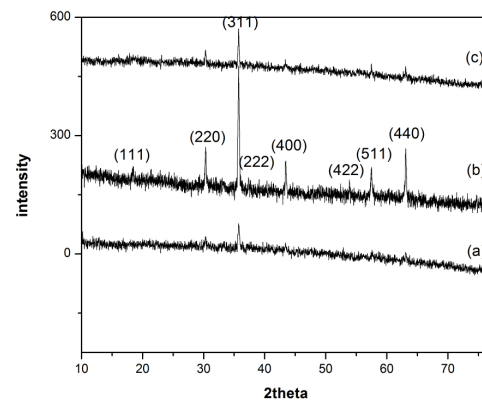


Fig. 1: XRD patterns at room temperature for ferrite $\text{Ni}_{0.99}\text{Co}_{0.01}\text{Mn}_{0.01}\text{Fe}_{1.99}\text{O}_4$ which calcinated at 500 °C, 700 °C and 900 °C.

According to Fig.1: Choosing 500 °C for calcination is not enough for appearing the spinel structure. It seems that 700 °C is the suitable temperature for producing the cubic spinel structure. Although by increasing the calcination temperature to 900 °C, the peaks become narrower and intensities decrease, resulting to reduce the crystallinity. Therefore it is observed that at 700 °C all diffraction peaks correspond to cubic spinel structure and thus the phase completed at this temperature.

The average crystallite size has been evaluated from the full width at half maximum of (311) peak in the XRD pattern, using the Debye–Scherrer formula:

$$D = 0.9 \lambda / b \cos\theta$$

Where $\lambda = 0.154056$ nm, is the wavelength of the Cu $K\alpha$ line, θ is the Bragg angle and b is the full width of the diffraction line at half of the maximum intensity measured in radians (FWHM). With increasing of temperature, nanoparticle sizes increase. The values of lattice constant are a little larger than that of for NiFe₂O₄ (0.8320 nm), which indicates the incorporation of Co (ionic radius 0.088 nm) and Mn (ionic radius 0.097 nm) dopants in the spinel lattice of Ni ferrite.

Temperature (°C)	500(°C)	700(°C)	900(°C)
Crystallite (nm)	28.3	43	57.5
Lattice constant (nm)	0.8333	0.8324	0.8327

Table. 1: crystallite size for the powders calcined at 500 °C, 700 °C and 900 °C for 2 h

The magnetic properties of samples have been characterized using a vibrating sample magnetometer. Figure. 2 presents the results of VSM measurement on the Ni_{0.99}Co_{0.01}Mn_{0.01}Fe_{1.99}O₄ at 500 °C, 700 °C and 900 °C, at room temperature.

Increasing in saturation magnetization (M_s) was observed with increasing the temperature from 500 °C to 900 °C. The observed increase in M_s values by calcination is believed to be due to growing of grains.

The value of coercivity field (H_c) of samples decrease with increasing of calcination temperature. When the size arrived to the critical diameter of a single domain, the coercivity decrease because of the change from single domain to a multi-domain structure[4].

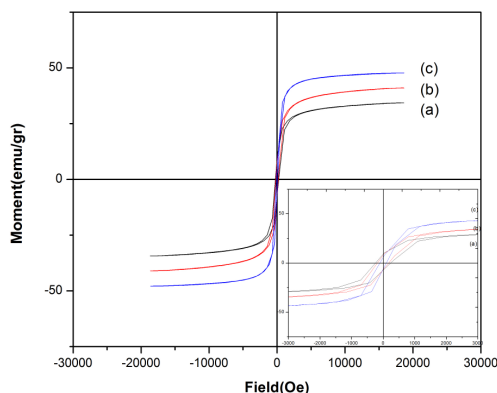


Fig. 2: Magnetization curves for powders calcined at 500 °C and 700 °C and 900 °C for 2 h.

The inset of Fig. 2 shows hysteresis loops are started in the field range of $\sim \pm 3000$ Oe for the sample that was

calcinated at 500 °C (a) and is $\sim \pm 2000$ Oe for that calcinated at 700 °C (b) and is $\sim \pm 1500$ Oe for that calcinated at 900 °C (c).

Conclusions

From this research, one can concluded that nanocrystalline NiCoMn ferrite can be synthesized by sol-gel auto combustion technique. With this method it is possible preparation of the ferrites at relatively lower temperatures and much shorter duration than that required in the conventional solid-state route. Up to this step of our research, 700 °C is the best set temperature for calcination process.

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