Preparation of Zinc/Nickel Ferrite Nanostructures by DC Reactive Magnetron Sputtering

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Abstract
In this work, zinc/nickel ferrite ($ZnNiFe_2O_4$) nanostructures were synthesized by co-sputtering of Zn, Ni and Fe targets in presence of oxygen. A dc sputtering system employing closed-field unbalanced magnetron was used for the preparation of these films. The structural and optical properties of the prepared films were introduced by XRD, EDX, SEM, AFM, FTIR and UV-visible spectroscopy. Results showed that these films are polycrystalline, highly pure, with average particle size of 20-25nm and average surface roughness of 0.465nm. Also, they showed high transmittance in the ultraviolet region and their transmittance decreased in the visible region and then increased in the infrared region. These nickel ferrite nanostructures were prepared at low production cost, high reliability and reasonable structural purity.

Keywords: Magnetron co-sputtering; Reactive sputtering; Nickel ferrite; Nanostructures
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1. Introduction

Due to its increasing uses in many new applications, especially magnetic, magneto-optic and gas sensors, synthesis and preparation of nickel ferrite were the goals of many research works and studies during the last two decades. Moreover, its capability to host another metal within its molecular structure, such as Zn, Cu, Co, Mn and Cu, has encouraged researchers to find many other fields not easily introduced with the original structure [1-3]. As well, capping the surface of nickel ferrite structures with organic or organometallic agents made them good candidates for ferrofluids, magneto-optics, spintronics, biomedical applications and anodes for batteries [4-8].

These compounds were prepared as thin films or powders by several different methods and techniques. The most common ones are sol-gel methods [9], the ball-milling technique [10], co-precipitation [11], electrospinning method [12], the hydrothermal method [13], the reverse micelles process [14], and the micro-emulsion method [15].

Ferrite nanostructures exhibit featured magnetic properties compared to the larger scale structures [16-18]. Therefore, the interest in synthesizing these nanostructures has increased drastically within the last decade to overcome some technological problems in many modern industries [19-21]. The importance of magnetic measurement and testing applications will continue to rise corresponding to technological trends such as electric mobility, robotics, miniaturization and automation technologies, as well as promising magnetic materials among which nickel ferrite is one of the best [22,23].

In this work, a dc reactive magnetron sputtering technique was used to prepare zinc/nickel ferrite ($ZnNiFe_2O_4$) nanostructures at low pressures with employment of Zn, Ni and Fe targets. The structural and optical properties of the prepared nanostructures are studied.

2. Experimental Part

The dc plasma sputtering system used in this work contains of a vacuum chamber, two discharge electrodes, vacuum pumps and accessories, and cooling and heating facilities. The chamber could be evacuated down to 10^{-3} mbar by a rotary pump and to 10^{-5} mbar by a diffusion pump. The base vacuum was determined by the purpose of the discharge process. Argon at maximum pressure of 0.8mbar was used as the discharge gas and its pressure was finely controlled by needle valve. More details on the dc plasma sputtering system can be found in references [24-26].

The inter-electrode distance could be easily varied from 0 to 10cm as the system was operated. The cathode was cooled down to about 10°C to prevent the secondary electron emission while the anode could be heated by an underneath heater or kept at room temperature. The two targets of highly pure nickel (Ni, 0.9999) and iron (Fe, 0.999) were mounted on the cathode with some geometrical arrangement suitable for the work, as shown in Fig. (1), while the glass substrate on which the films were deposited was placed on the anode. All results presented in this work were obtained by using the concentric arrangement of the Ni and Fe targets.

Highly-pure oxygen gas was flowing to the chamber throughout needle valve to represent the reactive gas required to form the compound of nickel ferrite. The mixture of argon and oxygen gases could be controlled by a gas mixing unit before entering the chamber.

The film thickness was measured to be about 178nm for the sample prepared at 4 cm inter-electrode distance, 0.58 mbar gas pressure, 40:60 Ar:O_2 mixing ratio, 1.8kV discharge voltage, 25mA discharge current and deposition time of 4 hours.

The characterization and measurements included x-ray diffraction (XRD), scanning electron microscopy (SEM), electron-dispersive x-ray diffraction (EDX), atomic force microscopy (AFM), Fourier-transform infrared (FTIR) spectroscopy and UV-visible spectroscopy.

3. Results and Discussion

In the FTIR transmission spectrum of the prepared nanostructured $NiFe_2O_4$ thin film in Fig. (1), distinct peaks are observed around 430, 590 and 780 cm^{-1}. They are attributed to the octahedral metal stretching vibration (Zn-O) and (Ni-O) and the tetrahedral metal intrinsic stretching vibration (Fe-O), respectively.

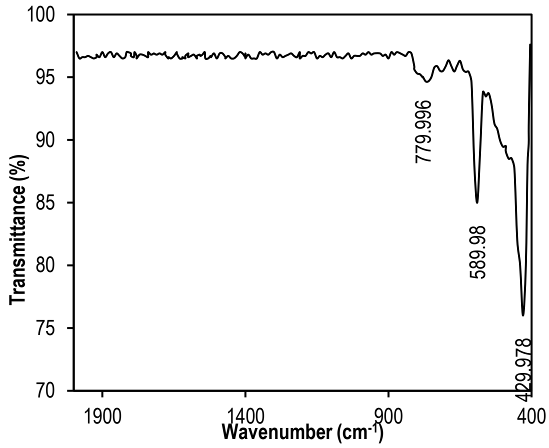

Fig. (1) The transmission FTIR spectrum of the prepared sample

Figure (2) shows the transmission spectrum in the spectral range of 200-1000nm. It appears totally transparent in the range of 200-343nm while this transmittance gradually decreases to reach its minimum at 406nm before increasing again to keep approximately constant value (88-89%) beyond 600m. In general, this film can be considered as optically transparent.

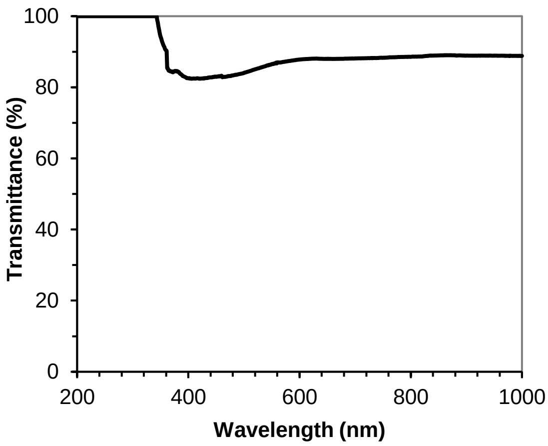

Fig. (2) The UV-visible spectrum of the prepared sample

Figure (3) shows the XRD patterns of the prepared sample and six peaks can be apparently observed at 30.17, 35.53, 43.22, 53.70, 57.16 and 62.60°, which correspond to crystal planes of (220), (311), (400), (422), (511) and (440), respectively. These planes are characteristic for $ZnNiFe_2O_4$ structure and since no other peaks are observed, this may highlight that the prepared sample is highly pure. The XRD pattern confirms the formation of spinel cubic structure of $ZnNiFe_2O_4$.

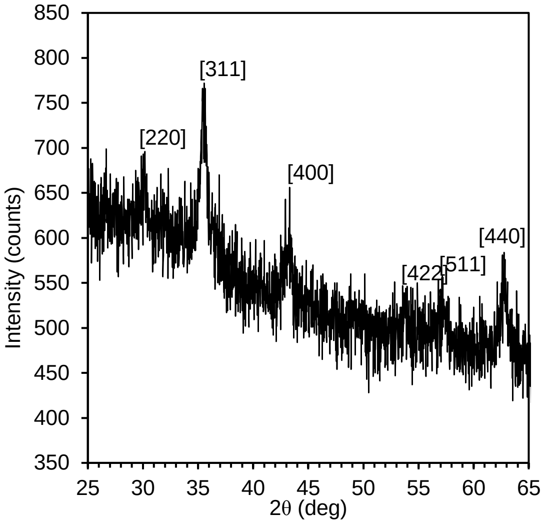

Fig. (3) The XRD pattern of the prepared sample

Figure (4) shows the SEM image of the prepared sample and the minimum particle size is 20-25nm and the homogeneity over the tested area is reasonably accepted. Figure (5) shows the morphology of the prepared sample determined by the AFM. The average roughness of the surface is about 0.465nm. The homogeneity of the prepared surfaces is clearly observed, which encourages using these nanostructures in some applications requiring high and homogeneous surfaces at the nanoscale, such as magnetic, magneto-optic and gas sensing.

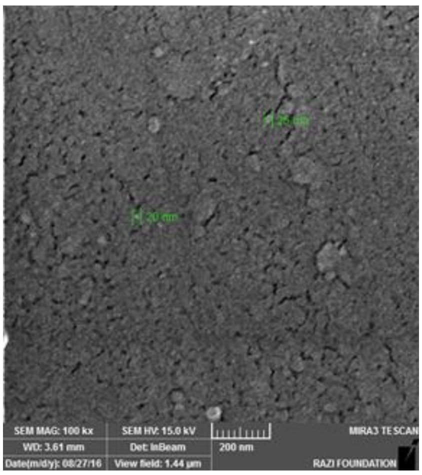

Fig. (4) The SEM image of the prepared sample

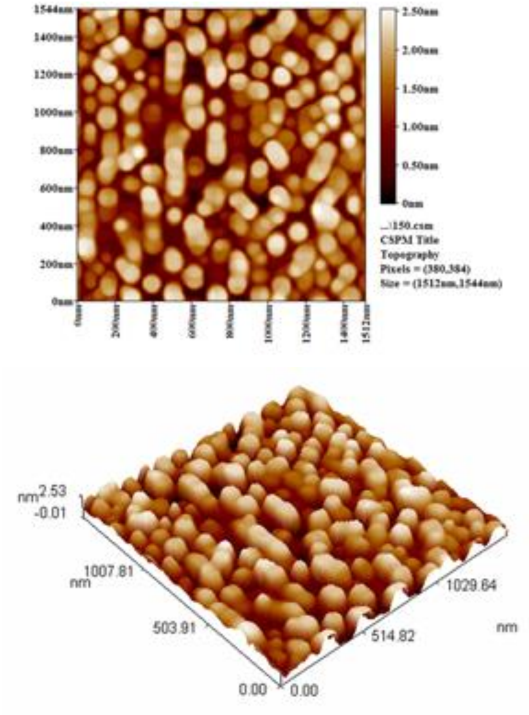

Fig. (5) The AFM image of the prepared sample

4. Conclusions

According to the obtained results, polycrystalline zinc/nickel ferrite ($ZnNiFe_2O_4$) nanostructures can be prepared by a low-pressure dc magnetron sputtering technique. The structural and optical characterizations of the prepared nanostructures showed that they have high structural purity, minimum particle size of 20-25nm, average surface roughness of 0.465nm, high transmittance (>80%) in the spectral range of 200-1000nm. The preparation of these nanostructures by the employed technique is reasonably described as low cost and high reliability process.

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