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Structural Characteristics of Nickel Ferrite Nanoparticles Synthesized by New Arrangement of Concentric Targets in DC Reactive Magnetron Sputtering

Nickel ferrite (NiFe₂O₄) nanostructures were synthesized by co-sputtering of Ni and Fe targets in presence of oxygen. A dc plasma sputtering system employing closed-field unbalanced magnetron at the anode was used for the preparation of these films. The structural characteristics of the prepared films were determined and the results showed that these films are polycrystalline, highly pure, with average particle size of 20-25nm and average surface roughness of 0.465nm. These nickel ferrite nanostructures were prepared at low production cost, high reliability and reasonable structural purity.

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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].

This compound was 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].

Nickel 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 nickel ferrite (NiFe₂O₄) nanostructures at low pressures with employment of concentric Ni and Fe targets. The structural characteristics of the prepared nanostructures are studied.

2. Experiment

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.

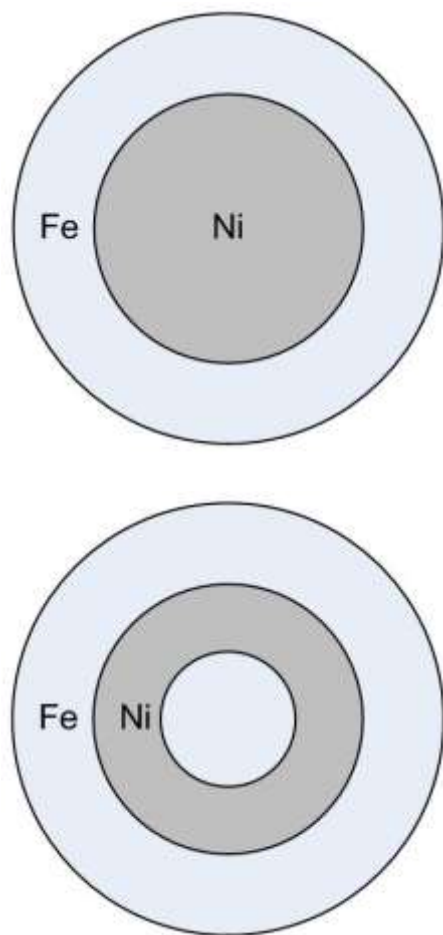


Fig. (1) The two geometrical arrangements of the two targets used in this work

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:O₂ 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), atomic force microscopy (AFM) and Fourier-transform infrared (FTIR) spectroscopy.

3. Results and Discussion

In the FTIR transmission spectrum of the prepared nanostructured NiFe₂O₄ thin film in Fig. (2a), two distinct peaks are observed around 430 and 590 cm⁻¹. They are attributed to the octahedral metal stretching vibration (Ni-O) and the tetrahedral metal

intrinsic stretching vibration (Fe-O), respectively. Other peaks observed in the range 2800-3600 cm⁻¹ are assigned to the stretching modes of the free or adsorbed water. Figure (2b) 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 600nm. In general, this film can be considered as optically transparent.

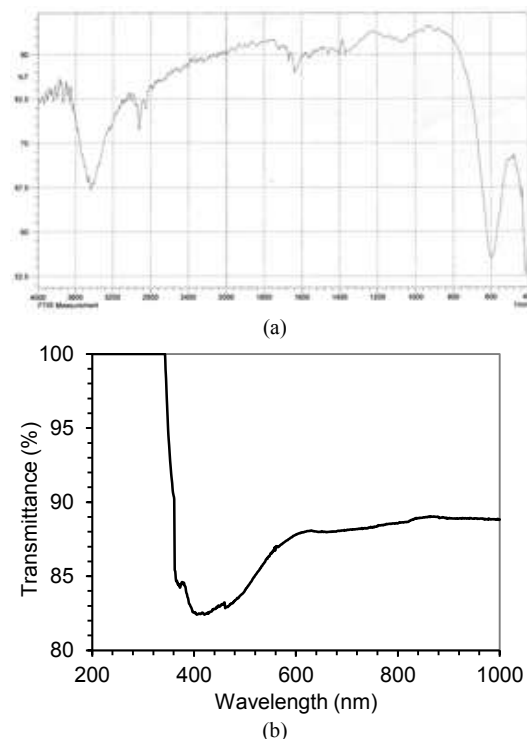


Fig. (2) The transmission FTIR (a) and UV-visible (b) spectra of the prepared sample

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 NiFe₂O₄ 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 NiFe₂O₄.

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.

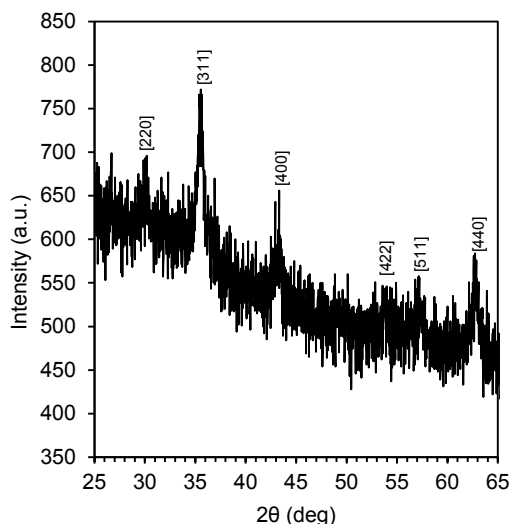


Fig. (3) The XRD pattern of the prepared sample

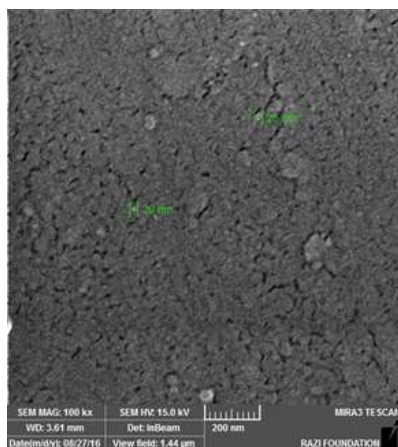


Fig. (4) The SEM image of the prepared sample

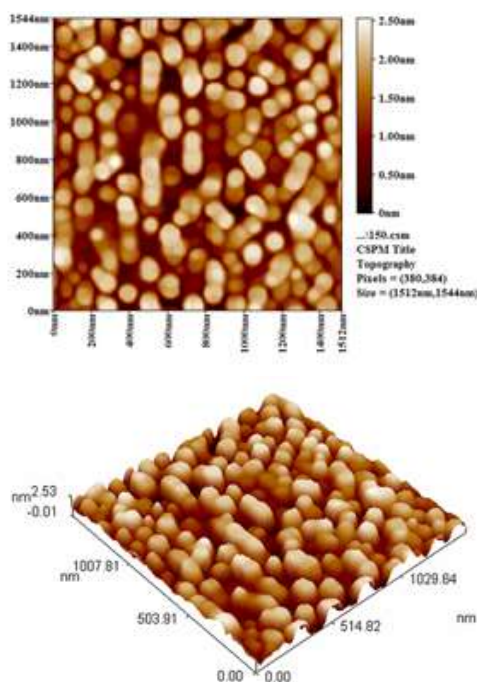


Fig. (5) The AFM image of the prepared sample

4. Conclusion

According to the obtained results, polycrystalline nickel ferrite (NiFe_2O_4) nanostructures can be prepared by a low-pressure dc magnetron sputtering technique using a special arrangement of concentric targets to be sputtered. The structural characterizations of the prepared nanostructures showed that they have high structural purity, minimum particle size of 20-25nm, and average surface roughness of 0.465nm. The preparation of these nanostructures by the employed technique is reasonably described as low cost and high reliability process.

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