Magnetic Properties of Nickel Oxide Nanoparticles Synthesized By Sol-Gel Method

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Abstract

Nickel oxide nanoparticles were synthesized by a sol-gel process. The effect of various synthetic reaction and heat treatment conditions on the structure and magnetic properties of NiO nanoparticles was also studied. As-synthesized NiO nanoparticles have been characterized by X-ray diffraction, transmission electron microscopy, UV–VIS and FTIR spectroscopy. XRD revealed the NiO nanoparticles have single cubic phase structure with average particle size of 20 nm. NiO nanoparticles exhibited nanocrystalline grains with a randomly oriented morphology. The increase in the band gap can be related to the quantum confinement effects of the NiO nanoparticles. Electrical conductivity of synthesized NiO nanoparticles was found to be increased with the temperature. Magnetic behavior of the synthesized NiO nanoparticles was studied by using Vibration Sample Magnetometer (VSM). The squareness ratio (SQR) for the NiO nanoparticles was found to be 82.68 x 10^{-3} indicating that the NiO nanoparticles do not have the properties of a recording medium.

Keywords: Nickel oxide, nanoparticles, sol-gel, magnetic properties, squareness ratio

Introduction

Nanometer-scale materials with the size of 1 to 100 nm have attracted considerable interest in recent years due to the departure of properties from bulk phases arising from quantum size effects. Nickel oxide (NiO) is a technologically important material with applications in catalysts, electrochromic film, gas sensors, fuel cell, anode of organic light-emitting diodes, magnetic materials and thermoelectric materials, owing to its p-type conductivity, wide band gap ranging from 3.6 eV to 4.0 eV, excellent chemical stability, electrical and optical properties [1-5]. In general, the resistivity of NiO nanoparticles can be decreased by increasing the concentration of Ni^{2+} vacancies [6] during annealing in air or doping with monovalent atoms, such as Li [7] or Al [8] atoms. Jang et al [7] reported the deposition of low resistivity NiO doped with Li atoms by radio frequency magnetron sputtering. Nandy et al. [8] reported that the doping of NiO films with Al atoms by magnetron sputtering can enhance the conductivity of NiO. Several approaches have been used to prepare NiO nanoparticles such as pulsed laser deposition [9], sputtering [10], e-beam evaporation [11], electrochemical method [12] and sol–gel method [13–16]. Among these, the sol–gel method is the most cost-effective for producing large-area films, and provides excellent control of the composition and homogeneity. On the other hand, it is still challenging to develop simple, fast and versatile methods for the synthesis of highly conducting NiO nanoparticles.

Experimental

Synthesis of NiO nanoparticles: Nickel oxide (NiO) nanoparticles were prepared by sol–gel process [17] in which, 0.1M nickel carbonate was added in 100ml starch solution and the mixture was stirred for half an hour. Then ammonia was added drop wise in the solution under constant stirring. After complete addition of ammonia, the solution was allowed to settle for overnight and then filtered using membrane filtration assembly, washed using deionized water and ethanol to remove the impurities and then dried at 80°C in hot air oven. Dried sample was treated at different temperatures in order to maintain the stability of compound. The colour of the sample was changed from green to faint gray at 100°C to 750°C.

Instruments for characterizations

X-ray diffraction (XRD) analysis was conducted on Philips PW1710 automatic X-ray diffractometer with Cu-Kα radiation (λ=1.5404Å), with a scanning speed of 10°min−1. FTIR spectra was performed on Shimatzu FTIR-8101A Spectrophotometer in the wavelength range of 400–4000 cm^{-1}. TEM analysis of NiO was carried out on Phillips model-CM200 with resolution 2.4Å. The electrical conductivity of the compressed pellets of NiO nanoparticle was determined by two probe method. The pellets were prepared with the help of hydraulic press (Kimaya Engineers, India) by applying a pressure of 5000 kg/cm^2. The magnetization measurements of synthesized sample was carried out using vibrating sample magnetometer (VSM) Lakeshore-3107 model at room temperature in the magnetic field range - 8000Oe to +8000Oe .

Results and discussion

The nickel oxide nanoparticles were analyzed by X-ray diffraction technique to study structural identification and changes in the crystallinity. The XRD pattern of nickel oxide is shown in Fig. 1. It reveals cubic phase comprising a strong reflection along (1 1 1) plane and a weak reflection along (2 2 2) plane. The crystalline peaks at 2θ = 37·53°, 43·60°, 63·09°, 75·67°, 79·58° which have been identified as peaks of single phase cubic structure of NiO with diffracting planes (111), (200), (220), (311), (222) respectively matched with JCPDS file 73-1523 [18]. The grain size of the crystallites (mean crystallite diameter) was found to be 20 nm as calculated using the well-known Scherrer's formula.

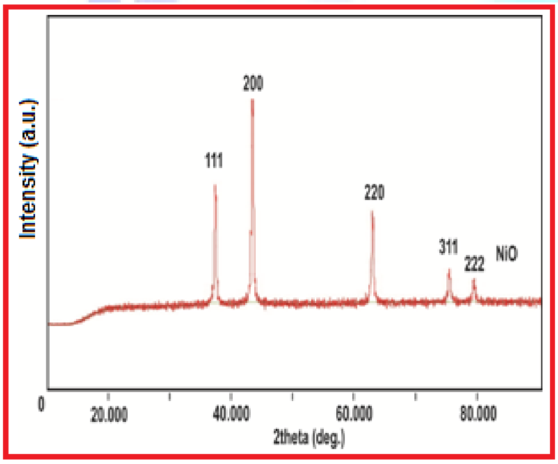

Fig. 1: XRD pattern of NiO

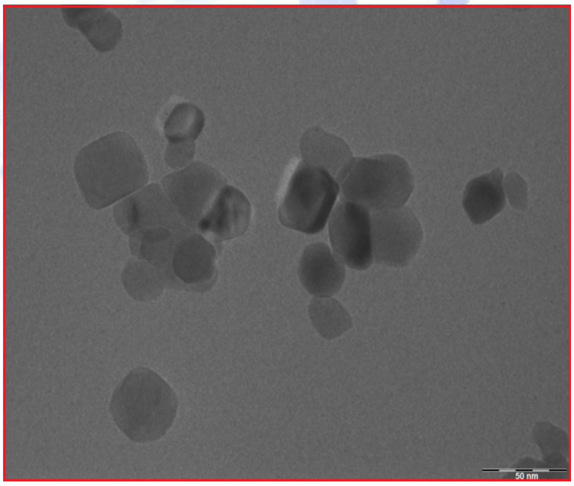

Fig. 2: TEM image of NiO

Transmission electron micrograph of NiO nanoparticles is shown in Fig. 2. It shows the particles are mostly irregular spherical shape with average particle size of 20 nm which is consistent with that estimated from the XRD data. From the TEM image of NiO, it could be concluded that this preparation method is appropriate to obtain the NiO nanoparticles with very small size.

FT-IR spectrum of NiO nanoparticles is shown in Fig. 3. The bands 874 cm^{-1}, 662 cm^{-1} are assigned to Ni–O vibration. The other bands clearly indicate that the sample consists of hydroxide ions and their presence in the IR spectrum may be due to the absorption of water. The band at 1476 cm^{-1} is attributed to the bending vibration of water molecule due to the absorbed moisture. The broad absorption band centered at 3720 cm^{-1} is attributable to the band O–H stretching vibrations and the weak band near 1476 cm^{-1} is assigned to H–O–H bending vibrations mode. These observations provided the evidence to the effect of hydration in the structure.

UV-visible spectrum of NiO nanoparticles is shown in Fig. 4. Nickel oxide is a high band-gap semiconductor with the absorption edge in the UV region and no absorption in the visible region. The presence of Ni^{3+} ions in the oxide lattice show charge transfer transition with the consequent absorption in the visible region [19]. The optical absorption band gap of the NiO nanoparticles was estimated to be 3.14eV.

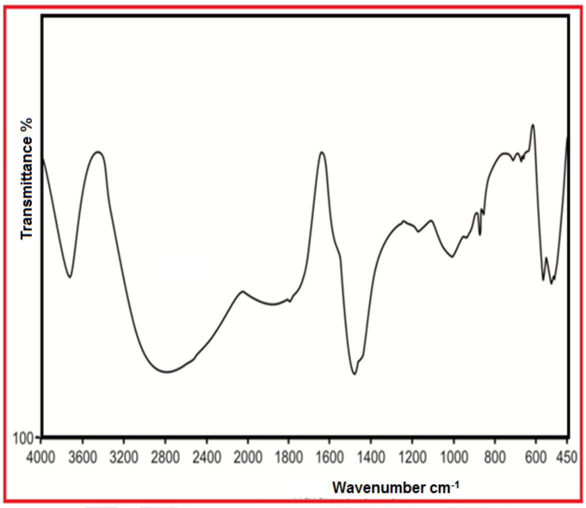

Fig. 3: FTIR spectrum of NiO

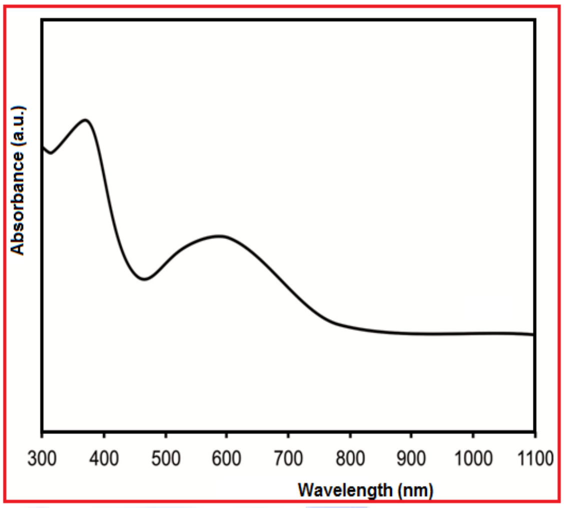

Fig. 4: UV-VIS spectrum of NiO

Fig. 5 shows the electrical conductivity of NiO nanoparticles at different temperature determined by using two probe method. The conduction mechanism of the NiO is believed to be related to the concentration of the electrical carrier, which is the oxygen vacancy existed in the structure. It is observed from the graph, increase in the conductivity with temperature must mainly regarded as due to the thermally activated mobility of the carriers (electrons or holes) rather than to a thermally activated generation of these. The room temperature electrical conductivity of NiO was found to be 0.01 $x10^{-6}$ S/cm. Increase in conductivity with increase in temperature proves semiconducting behavior of NiO nanoparticles [20].

The specific magnetization curve for NiO nanoparticles obtained from room temperature VSM measurement is shown in Fig. 6. The saturation magnetization of NiO was found to be 1.19 emu/g with coercive field 452.66 Oe and retentivity 98.4 x 10^{-3} emu/g shows its ferromagnetic nature [21]. The squareness ratio (SQR = *Mr/Ms*) is an important assessment of the quality of the magnetic materials which decides the recording medium properties of the magnetic materials. The SQR for NiO nanoparticles was found to be 82.68 x 10^{-3} indicating that the pure NiO do not have the properties of a recording medium

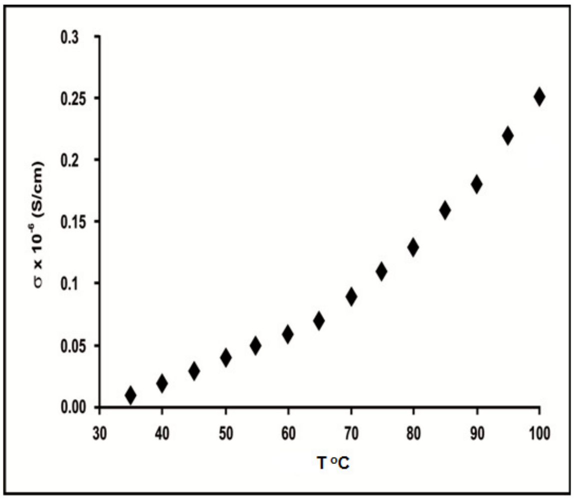

Fig. 5: Electrical conductivity of NiO

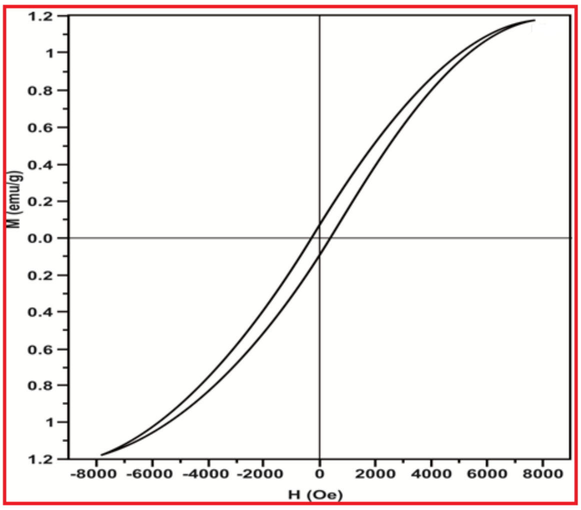

Fig. 6: Magnetic Behavior of NiO

Conclusions

Nickel oxide nanoparticles were successfully synthesized by a sol-gel. XRD revealed the NiO nanoparticles have single cubic phase structure with average particle size of 20 nm. NiO nanoparticles exhibited nanocrystalline grains with a randomly oriented morphology. The increase in the band gap can be related to the quantum confinement effects of the NiO nanoparticles. Electrical conductivity of synthesized NiO nanoparticles was found to be increased with the temperature. From SQR value, NiO nanoparticles can not show the properties of a recording medium.

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