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Short communication

Facile synthesis and characterization of nanostructured chromium oxide

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ABSTRACT

Chromium oxide nanoparticles have been synthesized using chromium nitrite and sodium oleate. The synthesized material was structurally characterized using X-ray powder diffraction and scanning electron microscopy. The X-ray diffraction study reveals that synthesized powder is phase pure chromium oxide. The scanning electron microscopy confirms the nanostructure of the particles. The morphology of Cr_2O_3 was like rice grain with an average grain size of 100 nm. The magnetic property of the chromium nanoparticles was studied. The magnetic properties were studied as a function of temperature and applied magnetic field. In the zero-field-cooled (ZFC) and FC measurements, irreversibility was observed at 250 K. It was observed that chromium oxide nanoparticles show ferromagnetic behavior below the blocking temperature.

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1. Introduction

Transition metal oxides have attracted a great deal of attention in recent years, due to their unique physical, chemical, and magnetic properties [1–3]. Among various transition metal oxides, Cr_2O_3 is one of the widely studied compound due to its wide band gap (~3.4 eV) and p-type semiconducting nature [4–8]. Cr_2O_3 has extensive applications in optical and electronic devices, catalysis, wear resistance materials, and advanced colorants [9–11]. Bulk Cr_2O_3 is an antiferromagnetic material with a Neel temperature of 307 K, whereas, nanostructured Cr_2O_3 shows magnetic properties which is due to the presence of uncompensated surface spins [12].

Makhlouf has studied the magnetic properties of nanostructured chromium oxide synthesized using chromium hydroxide $Cr(OH)_3$ [13]. Nanocrystalline Cr_2O_3 particles were synthesized in a microwave plasma using chromium hexacarbonyl as precursor [12]. The magnetic behavior of the nanoparticles was described by a modified Langevin function in the temperature range from 10 to 300 K. Pei et al. [6] have synthesized Cr_2O_3 nanoparticles using a hydrothermal technique. It was observed that the higher calcination temperature and higher total concentration lead to the formation of larger average particle size.

Nanophase Cr_2O_3 with an initial average grain diameter of 10 nm was synthesized by the gas-condensation method followed by in-situ consolidation [14]. The nanophase powder showed superparamagnetic behavior, in both its powder and consolidated forms. Cr and Cr_2O_3 nanoparticles were synthesized by arc-discharge subsequent annealing the as-prepared Cr nanoparticles [15]. Cr nanoparticles showed antiferromagnetic properties, in addition to a weak-ferromagnetic component at lower fields. The weak-ferromagnetic component could be due to uncompensated surface spins. The Cr_2O_3 nanoparticles exhibited an exchange bias in the hysteresis loops. This could be taken as the exchange coupling between the uncompensated spins at the surface and the spins in the core of the Cr_2O_3 nanoparticles. Enhancement of magnetization below the antiferromagnetic ordering temperature Neel temperature in Cr_2O_3 nanoparticles was reported [16]. The enhancement of magnetization below Neel temperature was systematic and larger for sample with smaller particle size.

In the present manuscript, we report detailed structural and magnetic characterization of nanostructured Cr_2O_3 synthesized using a novel technique. This novel technique allows us to synthesize phase controlled magnetic nanoparticles of Cr_2O_3.

2. Experimental details

Chromium oxide was synthesized using chromium nitrate and sodium oleate. Both the compounds of high purity were purchased from Alfa Assar and used without further purification. The intermediate complex chromium oleate was synthesized by dissolving 10 mmol of $Cr(NO_3)_2$ and 10 mmol of sodium oleate in 300 ml of DI water under vigorous stirring conditions. The precipitate was filtered and washed several times with DI water. After drying at room temperature, the precipitate was transferred into Pyrex tube and sealed. The tube was heated with a rate of 3 °C/min from room temperature to 400 °C and annealed at same temperature for 4 h. The sample was brought at room temperature naturally.

The crystal structure, phase purity and particle size of the synthesized powder were characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The XRD spectrum of the sample was recorded with Bruker AXS X-ray diffractometer using

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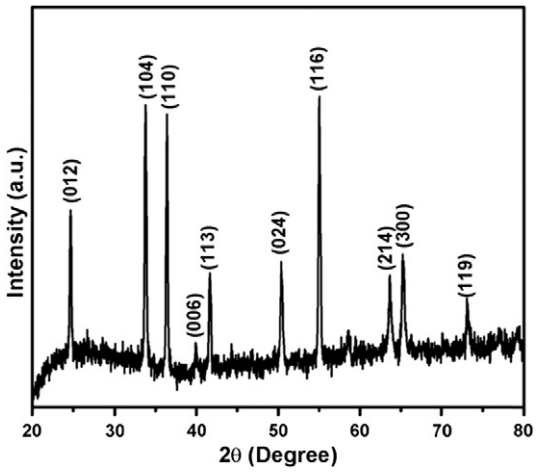

Fig. 1. XRD patterns of synthesized Cr_2O_3 nanoparticles.

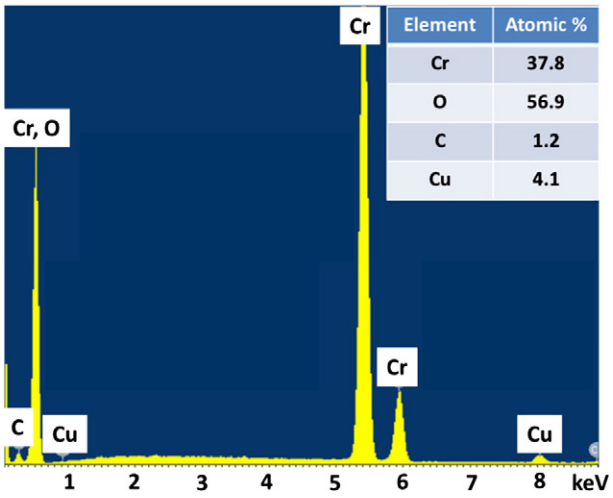

Fig. 3. EDS patterns of synthesized Cr_2O_3 nanoparticles along with atomic percent of elements.

2θ-θ scan with CuK_α (λ = 1.5405 Å) radiation which operated at 40 kV and 40 mA. The particle size and morphology of the powder sample were studied using a JEOL JSM-840A scanning electron microscope and an FEI Quanta 200 field emission scanning electron microscopy (FESEM) equipped with an Oxford INCA 250 silicon drift X-ray energy dispersive spectrometer (EDS). Quantum Design MPMS XL-T superconducting quantum interference device (SQUID) magnetometer was used to study the magnetic properties of the material.

3. Results and discussion

The crystal structure and phase purity of the synthesized powder were studied using X-ray diffraction method. The X-ray diffraction pattern of the powder sample is shown in Fig. 1. The observed XRD patterns are in good agreement with the literature value for Cr_2O_3 (JCPDS card no. 85–0869) which corresponds to the rhombohedral $R\left(\bar{3}\right)c$ Cr_2O_3 phase. As seen in the XRD patterns, we do not observe any additional peaks which indicate high phase purity of the synthesized Cr_2O_3. The lattice parameters of the hexagonal phase of Cr_2O_3 were calculated using the following expression [17]

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\frac{\left(h^2 + hk + k^2\right)}{a^2} + \frac{l^2}{c^2} \quad (1)$$

where, d is the inter-planar spacing, h, k, & l are the Miller indices, and a & c are the lattice parameters of the hexagon. The lattice constants of a = 4.934 Å and c = 13.54 Å were estimated for nanostructured Cr_2O_3, which are comparable to the lattice constant for bulk Cr_2O_3 (a = 4.958 Å and c = 13.58 Å).

The particle size and morphology of the Cr_2O_3 were examined using scanning electron microscopy (SEM). Fig. 2 shows the SEM images of the synthesized Cr_2O_3. As seen in the figure, the synthesized Cr_2O_3 is of nanometer size. The morphology of the Cr_2O_3 is like rice grain with an approximate length of 100 nm. Elemental analysis and their ratio were investigated using energy-dispersive X-ray spectrometer (EDS) analysis. The EDS pattern of the sample is shown in Fig. 3. The EDS pattern shows the presence of Cr, O, Cu, and small amount of C. The relative concentration ratio of Cr to O is approximately 2:3. The presence of Cu in the EDS pattern is due to the sample holder which is made of Cu, whereas the small amount of C may be from the organic part of sodium oleate.

The magnetic properties of the nano structured Cr_2O_3 were studied as a function of temperature and applied magnetic field. The temperature dependence magnetization (M *vs.* T) was studied in zero-field-cooled (ZFC) and field-cooled (FC) process under different applied magnetic field (Fig. 4). In ZFC measurements, the Cr_2O_3 nano powder was cooled from high temperature to 5 K without applying any external field. After reaching 5 K, a magnetic field was applied and the

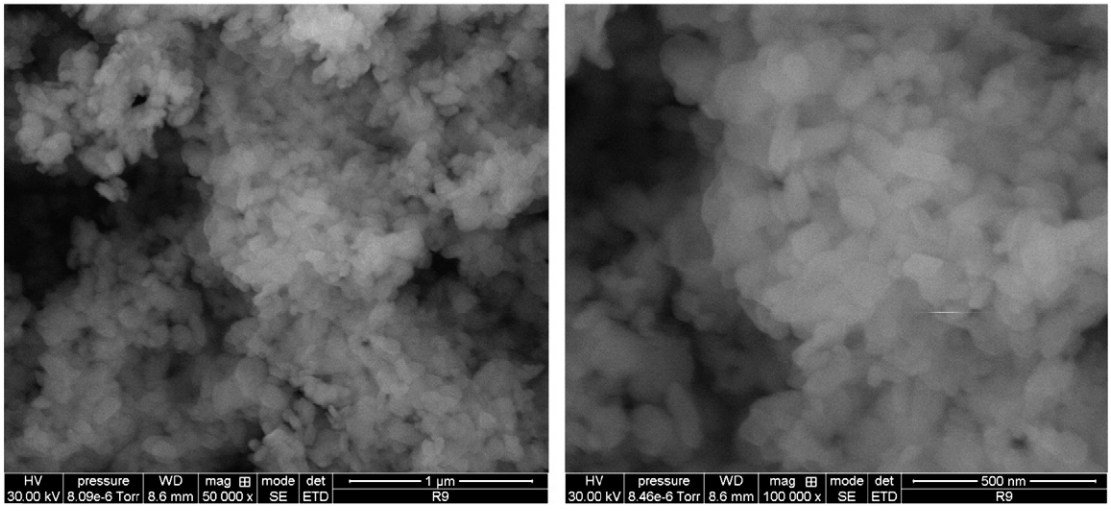

Fig. 2. SEM images of synthesized Cr_2O_3 nanoparticles at different magnifications.

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