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Phase transition of magnetite by mechanical alloying

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Abstract : Nanocomposite of Fe_3O_4 (magnetite) and α-Fe_2O_3 have been prepared by mechanical grinding Fe_3O_4 powder at ambient with 20 : 1 ball to powder mass ratio (BPMR) for different durations. Crystalline phases and several microstructure parameters such as volume fraction of the individual phase, particle size, and strain have been determined by X-ray diffraction technique. A very interesting phase transformation kinetics has been noticed. With progress of milling volume fraction of Fe_3O_4 reduces and that of α-Fe_2O_3 increases. After 10 hr of milling, content of Fe_3O_4 phase reduces to as low as 0.05 volume fraction. After 6 hr of milling particle size of Fe_3O_4 phase reduces from 54 nm to 5 nm. Then the size of the particles increases to 10 nm and remains almost unchanged upto 10 hr milling. The growth of α-Fe_2O_3 phase has been initiated from the Fe_3O_4 phase with very small particle size (4 nm) and in course of milling duration particle size grows upto 11 nm after 6 hrs of milling in an unusual manner. DC electrical conductivity measurements were carried out in the temperature range 110–500 K. Resistivity of the nanocomposites exhibits semiconducting nature comprised of three different activation energies in an unusual manner. Conduction mechanism can be explained on the basis of microstructure induced in composites during milling.

Keywords : Magnetite, phase transition, mechanical alloying, XRD, TEM

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1. Introduction

Nanophase materials with average grain sizes ranging from 5 to 20 nm, exhibit properties that are often different and novel relative to those of conventional materials [1]. The electronic, magnetic, optical and chemical properties of the materials have been found to vary differently from those of the bulk form and to depend sensitively on size, shape and composition [2]. A number of techniques have been developed to synthesize nanoparticles. Inert gas condensation [3], laser ablation [4], supersonic expansion method [5], sputtering technique [6] are few among the physical methods. Various chemical methods such as sol-gel technique [7], reduction of melt quenched glasses [8], (AOT)/heptane inverse micelled method [9], and electrodeposition method have been employed [10].

However material processing by these methods has the following shortcomings. In some cases methods are not cost effective and yield of materials is very small in some cases. Mechanical alloying is a powerful method for the production of a variety of fine particles [11]. Apart from quantum size effects and surface and interface effects, a decrease in the particle size causes definite and systematic changes in the crystal symmetry and lattice parameters, particularly in oxide nanoparticles [12,13]. In this paper we discuss the effect of size induced structural distortions on the properties of nanoconposite of Fe_3O_4 and α-Fe_2O_3 prepared by mechanical grinding of Fe_3O_4.

2. Material preparation

Fe_3O_4 (99%) procured from Aldrich was employed for milling in Fritsh pulverisette 5 planetary ball mill. Hardened steel vials of 80 cm^3 volume and hardened steel vials of 10 mm diameter were used for milling. Magnetite powder (Fe_3O_4) was crushed and disintegrated in the grinding bowl, keeping the ball to powder mass ratio 20 : 1. The rotational speed of the mill was set at 300 rpm. The powder was milled in ordinary atmosphere without any additive under 'closed' milling condition. Small quantities of sample were taken from the bowl after 1, 2, 4, 6, and 10 hrs of milling in order to check the phase formation during milling time by X-Ray diffraction technique. The X-Ray diffraction patterns of the samples were recorded using Ni-filtered Cu-K$_\alpha$ radiation

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temperature region by the conductivity of the nanostructured α-Fe_2O_3. We believe that defect states induced in the α-Fe_2O_3 nano particles give rise to finite conductivity of α-Fe_2O_3. Intimate mixing of the nanosized Fe_3O_4 particles in the α-Fe_2O_3 matrix of nanodimension gives rise to a large number of grain boundaries. The atoms, which reside on the grain boundaries, control the temperature dependent resistivity of the magnetite. TEM microstructure of the magnetic nanocomposite reveals such intimate mixing of Fe_3O_4 and α-Fe_2O_3 as shown in the Figure 6.

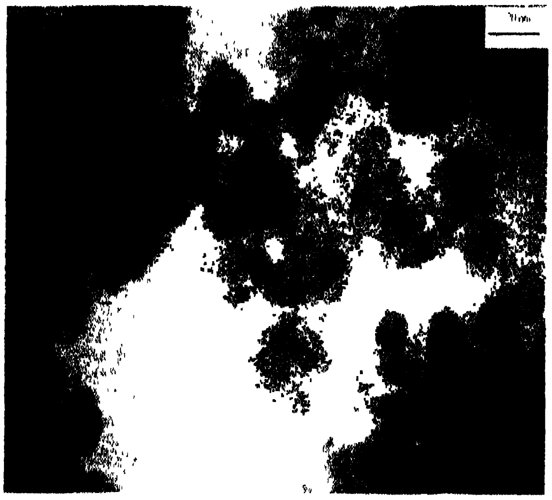

Figure 6. TEM microstructure of the sample S10

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