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Research Paper

Synthesis and Characterization of *α*-Co Nanoparticles Dispersed in Silica Matrix

C. Adaikalameri and E. Thirumal[1]
[1]Department of Nuclear Physics,
Faculty of Arts & Science,
Bharath Institute of Higher Education and Research,
Chennai-600073, India

Address for Correspondence

C. Adaikalameri and E. Thirumal[1]
[1]Department of Nuclear Physics,
Faculty of Arts & Science,
Bharath Institute of Higher Education and Research,
Chennai-600073, India

Abstract

Ultrafine particles of pure amorphous cobalt were chemically synthesized by borohydride reduction of a cobalt salt in aqueous condition. This material when subjected to a controlled annealing in aqueous medium at 80 °C for about an hour yielded phase pure nanocrystalline particles of cobalt with a hexagonal close packed (hcp) structure as revealed by an X-ray diffraction (XRD) and trnasmission electron microscopy measurements (TEM) analysis. Silica coating was carried out using Stöber process. Pure cobalt and Co/SiO_2 samples were annealed at various temperatures. The influence of thermal treatment on the crystal structure of Co particles was examined. The estimated crystalline size of as-prepared cobalt was 23 nm and increases with annealing temperature. The magnetization reach a maximum 96 emu/g. The dielectric constant of the sample is almost independent of frequency up to 1 MHz. Our method provides a promising route to fabricate magnetic materials in the high-frequency range.

INTRODUCTION

Cobalt nanocrystals display a wealth of size-dependent structural, magnetic, electronic, and catalytic properties. There has been a considerable amount of research involving the

preparation, structure, and properties of magnetic cobalt nanoparticles in the past decade [1-4]. The high magneto crystalline anisotropy of hcp Co has spurred intensive studies of Co-based nanostructures for magnetic storage purposes [5]. Cobalt nanoparticles coated with insulators have been prepared and studied for the applications in AC electrical and electronic devices [6]. With the growing interest in building advanced materials using nanoscale building blocks, there is a need to control the sizes, shapes, and structures. Different crystalline structures provide some practical benefits. In practice, cobalt nanoparticles often possesses mixed structures in which low energy stacking faults introduce a combination of fcc and hcp character, making the synthesis of single structured cobalt a challenging task. Puntes *et al* achieved size and shape-controlled cobalt nanorods as well as spherically shaped nanocrystals [7]. In this work, we reported the successful preparation of structure-controlled hcp cobalt nanoparticles and further coating with silica.

EXPERIMENTAL PROCEDURE

Fine particles of Co were prepared by reducing the cobalt salt $CoCl_2.6H_2O$ with sodium borohydride ($NaBH_4$) as reducing agent. The reaction was carried out in aqueous medium at room temperature. 100 ml of 1 M solution of $NaBH_4$ was slowly added with vigorous stirring to 250 ml of a 0.1 M $CoCl_2.6H_2O$ solution in a 500 ml round bottom flask. After the reduction of Co^{2+} into metallic Co, the sample was filtered by magnetic decantation and washed thoroughly with distilled water to remove all residual ions from the reaction mixture. Repeated washing is essential to remove unwanted impurities, such as elemental boron and its compounds with Co. The recovered powder was redispersed in 100 ml fresh distilled water. Post hydrothermal annealing was carried out in constant temperature water bath at 80 °C for an hour in necked 100 ml round bottom flask with an air exposure. The solution was mechanically stirred during heating. After the solution was allowed to cool to room temperature (RT), the Co powders were collected and washed with ethanol. The samples were dried at RT in air atmosphere for further studies.

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In order to prepare the Co/SiO_2 nanocomposite, Co nanoparticles were redispersed in 25 ml of 4.2 vol% of ammonia (28% NH_3 in H_2O) in ethanol and immediately 25 ml of 10 vol% TEOS in ethanol was added slowly under vigorous stirring for 24 h and the product was then aged for 48 h. The resulting Co/SiO_2 nanocomposite powder was washed with acetone and dried by flash heating at 60 ºC. Portions of this nanocomposite were also annealed in N2 atmosphere at temperatures such as 300 and 500 ºC, for 1 h. The samples were characterized by X-ray diffraction (XRD) using Cu-Kα_1 radiation in the 2θ range of 20º – 90º, vibrating sample magnetometry (VSM) over an applied field of -7 kOe to + 7 kOe and transmission electron microscopy (TEM).

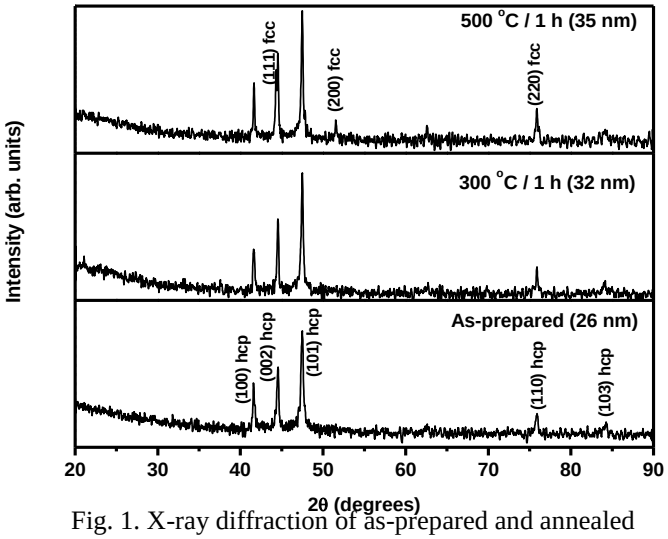

Fig. 1. X-ray diffraction of as-prepared and annealed Co/SiO_2 powder.

RESULTS AND DISCUSSIONS

The borohydride reduction method was widely used to synthesis cobalt nanoparticles, however there is no report on the synthesis of pure hcp cobalt nanoparticles. Here we first time synthesized the hcp Co nanoparticles with modified annealing process. The X-ray diffraction profile of the silica encapsulated Co nanoparticles is shown in figure 1, indicating that the as-synthesized and annealed at 300 ºC nanopowders correspond to hcp structure. Further, annealed at 500 ºC shows the fcc phase appears and then the Co particles were a mixture of fcc and hcp phases.

The average grain size of the synthesized cobalt nanoparticles in silica matrix was estimated by the Scherrer formula from the (101) diffraction peak of hcp phase. The estimated crystalline size of as-prepared cobalt was 26 nm and increases to 32 nm and 35 nm for with annealing temperature of 300 ºC and 500 ºC respectively. Figure 2 shows a TEM image of silica particles encapsulated nanocrystalline hcp cobalt, it can be seen that aggregation 30 to 50 nm particles due to large attractive van der Waals and magnetic forces between the particles. The increased particle size was attributed to deposition of silica layer on the cobalt crystalline.

Figure 3 shows the hysteresis curve recorded at room temperature. The shape of the magnetization curve and the small hysteresis are typical of ferromagnetic behaviour, and magnetization nearly reached saturation at a field of 10kOe. The measured saturation magnetization, obtained at 15 kOe was 96 emu/g of Co/SiO_2 composite. The coercive force of the composite was 336 Oe. The frequency variation of the dielectric constant (DC) of the annealed samples has been studied at room temperature. As observed in figure 4 the DC is quite low and is in the range of 30-100. It is observed that for each sample the dielectric constant nearly constant from 100 Hz to 1 MHz, and slight increase in dielectric constant was observed with annealing temperature. The dielectric constant of any material, in general is due to dipolar, electronic, ionic, and interfacial polarizations. At low frequencies dipolar and interfacial polarizations are known to play the most important role. At high frequencies electronic and ionic polarizations are the main contributiors. A constant and low value of the DC at all frequencies is might be attributed to electronic polarization of cobalt and electronic as well as ionic polarization from silica matrix.

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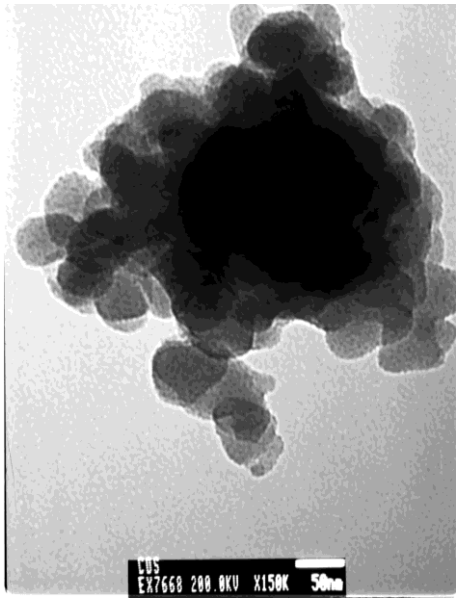

Fig. 2. TEM micrograph of the as-prepared Co/SiO_2 composite powder

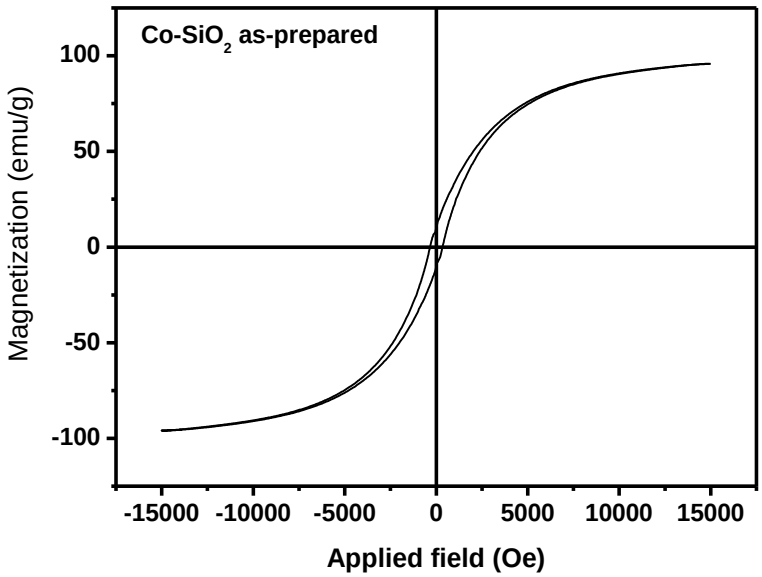

Fig. 3. Magnetization versus magnetic field plot for Co/SiO_2 composite powder at room temperature.

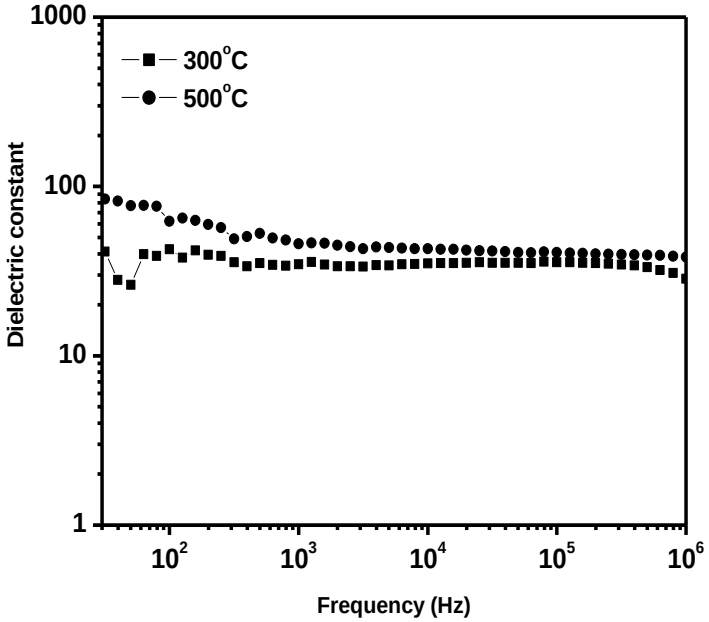

Fig.4. Variation of the dielectric constant with frequency of Co/SiO_2 nanocomposite measured at room temperature for annealed at indicated temperature.

CONCLUSION

Thermodynamically stable nanocrystalline (26 nm) hcp-Co powders were successfully prepared by chemical method and its coating with silica was achieved by modified Stöber method. Co nanoparticles dispersed in silica matrix was found in TEM microstructure. The stable Co/SiO_2 nanocomposite with relatively high saturation magnetization 96 (emu/g) and dielectric behavior of this materials are favorable for high frequency applications.

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