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Studies on the effect of sintering conditions on the magnetostriction characteristics of cobalt ferrite derived from nanocrystalline powders

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Abstract

The effect of sintering conditions on the magnetostriction characteristics of sintered cobalt ferrite derived from nanocrystalline powders is studied. It is observed that the magnetostriction coefficient as well as the slope of the magnetostriction as a function of field depends on the microstructure of the sintered product. There is a direct correlation between the magnetostriction characteristics and density as well as porosity of the sintered products. Intragranular pores are found to be strongly influencing the magnetostriction characteristics. Similarly, correlation is found between the magnetostriction parameters and the Curie temperature, indicating the role of cation distribution which affects the magnetic anisotropy. Sintering the compacts made from nanocrystalline powders at 1450 °C for a very short duration is found to give high values of magnetostriction coefficient. However, higher values of magnetostriction at low magnetic fields are obtained for samples sintered at lower temperatures for longer duration and having relatively lower density.

Keywords: Sintering; Magnetostriction; Cobalt ferrite; Nanocrystalline; Microstructure

1. Introduction

Sintering is one of the crucial steps in the processing of ceramic magnetic materials aimed at various applications. Sintering studies on different ferrites and the corresponding changes in the performance parameters such as permeability, dielectric constant, loss factor, etc. are well documented in the literature.[1,2] The performance parameters, in general, depend on the microstructure, density, porosity, grain size, etc. of the sintered products. Magnetostriction is the change in dimensions of a magnetic material in the presence of an applied magnetic field and magnetostrictive materials are used as sensors and actuators for various applications.[3,4] The magnetostriction coefficient, $\lambda = \Delta L/L$, and the strain derivative or slope of the magnetostriction at low magnetic fields, $d\lambda/dH$, are the important magnetostriction parameters considered from the application point of view. High magnetostriction coefficient and strain derivative are required for different applications. Among the different ferrites, cobalt ferrite in its single crystalline form shows high magnetostrictive strain of ~600 ppm, depending on the composition and crystallographic direction.[5,6] Cobalt ferrite is an ideal material for future magnetostrictive applications instead of the currently used Tb-Fe-Dy-based alloys[7] because of its low cost, high electrical resistance, easy processability, good mechanical properties, etc. There have been many attempts to make sintered polycrystalline cobalt ferrite with high magnetostriction at low magnetic fields by changing the processing conditions.[8–16] Sintered metal bonded cobalt ferrite composite showing a magnetostrictive strain up to 230 ppm at low applied magnetic fields has been proposed to be useful for magnetomechanical sensor applications.[8,9]

Most of the previous reports on the studies on the magnetostriction of cobalt ferrite are on the materials synthesized by the ceramic process involving high-temperature solid state reactions. Magnetostriction coefficient up to 225 ppm is reported for sintered cobalt ferrite derived from bulk powders with low strain derivative ($\leq 1.5 \times 10^{-9}$ A^{-1} m).[8–10] However, a large value for the strain derivative, 4.34×10^{-9} A^{-1} m, is reported for sintered cobalt ferrite derived from bulk powders, even though the magnetostriction coefficient obtained is ~150 ppm.[14] It has been reported that the magnetostriction coefficient of cobalt ferrite sintered from bulk powders depends on grain size.[11,12] Recently,

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it has been reported that higher magnetostriction coefficient (>300 ppm) can be attained for cobalt ferrite by starting from nanocrystalline powders.[17,18] So far, there have been no attempts to study the effect of sintering of cobalt ferrite compacts derived from nanocrystalline materials at lower temperatures (<1300 °C) on the magnetostriction characteristics. It is widely known that sintered products derived from nanocrystalline powders exhibit improved magnetic permeability and permittivity compared to the compacts derived from bulk powders.[19,20] The advantage of the nanocrystalline powders is that they are more sinterable due to the fine particle nature as well as the high surface area.[21–24] In this manuscript, we report the effect of sintering conditions on the magnetostriction characteristics of the compacts made from nanosized particles of cobalt ferrite. The objective of the present study is to investigate the influence of sintering processes on the densification, microstructure, and magnetostriction characteristics of sintered cobalt ferrite derived from nanocrystalline materials.

2. Experimental

Nanocrystalline cobalt ferrite was synthesized by an auto-combustion method using the corresponding metal nitrates and glycine, as reported previously.[17] Stoichiometric amounts of cobalt nitrate, $Co(NO_3)_2\cdot 6H_2O$ (Aldrich), and ferric nitrate, $Fe(NO_3)_3\cdot 9H_2O$ (Aldrich), taken in the 1:2 M ratio, were dissolved separately, in minimum amount of distilled water. Glycine (Merck), corresponding to the glycine to nitrate ratio of 0.09 was also dissolved separately in minimum amount of distilled water. All the three solutions (cobalt nitrate, ferric nitrate, glycine) were mixed thoroughly by ultrasonicating for 2 min. The mixed solution was then transferred to a crystallizing dish and the dish with its contents was subjected to heating on a laboratory hot plate at a temperature of about 200 °C. After complete evaporation of water, the resulting viscous liquid ignited automatically giving rise to a fluffy mass of cobalt ferrite. The particle size of the as-synthesized powders was obtained as 4 nm.[17] Bulk cobalt ferrite powder was prepared by the ceramic method, as reported previously.[11]

The as-synthesized powder was mixed with 2% solution of poly vinyl alcohol as a binder and uniaxially pressed into cylindrical shaped specimens (∼10 mm diameter ×∼15 mm length) under a pressure of 8 MPa. For sintering studies, the compacts were heated from room temperature to a high temperature and held at this temperature for different durations and then cooled back to room temperature slowly. In this work, samples were sintered in the temperature range 1000–1500 °C with a heating rate of 5 °C/min, cooling rate of 20 °C/min and different holding times. Initially samples were sintered at different temperatures for a fixed sintering time of 10 min to optimize the temperature, and then sintered for different durations at a fixed temperature to optimize the sintering time. Samples were also sintered at 1000 and 1100 °C for 20 h each to study the effect of longer duration of sintering at lower temperatures on the values of magnetostriction.

The sintering characteristics of the samples were studied by thermo-mechanical analysis (TMA). A Perkin Elmer Pyris Diamond thermo-mechanical analyzer was used to study the sintering behavior in terms of linear shrinkage as a function of temperature. Density of the sintered cylindrical compacts were calculated from their volume and weight and the relative density was calculated from the ratio of the measured density to the theoretical density of cobalt ferrite calculated from the crystal structure parameters. The green density of the compacts is obtained as 45% of the theoretical density of cobalt ferrite (5.275 g/cm^3). Magnetic measurements were made on a PAR EG&G vibrating sample magnetometer. Magnetization was measured as a function of field at room temperature, and Curie temperature was determined from measurement of magnetization as a function of temperature above 300 K. The magnetostriction measurement was carried out on the sintered cylindrical specimens with dimensions ∼7 mm diameter ×∼10 mm length. Magnetostriction was measured at room temperature parallel to the direction of the applied magnetic field by using 350 Ω resistive strain gages (Micro-Measurements, USA) bonded on to the sintered samples. Details of the magnetostriction measurements are reported elsewhere.[17]

Magnetic field annealing was carried out on the samples sintered at 1200 and 1450 °C for 10 min as well as on the samples sintered at 1000 and 1100 °C for 20 h. Magnetic field annealing was carried out at 300 °C in a magnetic field of 400 kA/m for 30 min. The annealing field was applied perpendicular to the cylindrical axis of the sintered pellet. After cooling the sample to room temperature in the annealing field, magnetostriction was measured along the cylindrical axis by applying the measuring field perpendicular to the direction of the annealing field.

3. Results and discussion

Fig. 1 shows a comparison of the sintering curves of cobalt ferrite made from nanocrystalline and bulk powders. The sintering behavior of the nanocrystalline powder sample differs from that of the powder sample synthesized by the ceramic method. In the case of the ceramic sample, effective sintering starts only

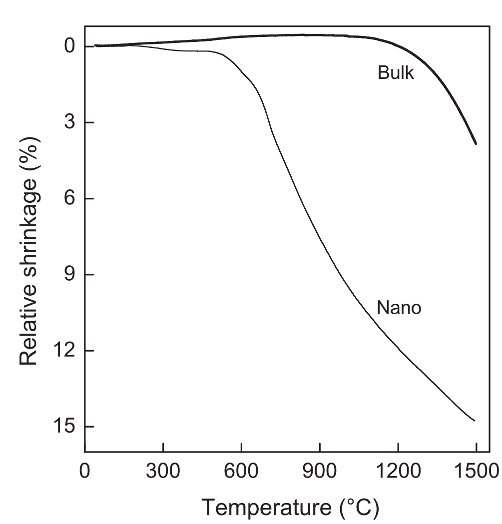

Fig. 1. Comparison of the sintering curves of the compacts made from nanocrystalline and bulk powders.