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# Dependence of the magnetic and magnetoelastic properties of cobalt ferrite on processing parameters

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#### ARTICLE INFO

### ABSTRACT

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### 1. Introduction

The need for advanced automated systems in industries is increasing due to the requirement to scale up production. Most automated systems depend heavily on high sensitivity sensor or energy efficient actuator devices. Stress sensors and actuators can be developed by exploiting the magnetomechanical coupling capability in magnetostrictive materials. It is important to optimize the properties of these materials for such applications. This has sparked considerable research interest in magnetostrictive materials in developing devices for different applications, especially where noncontact operation is crucial. Non-contact operation capability is very useful for devices because accuracy, repeatability and linearity are degraded over time due to wear and tear in devices operating in contact mode. Suitable magnetostrictive materials for such devices should possess sufficient amplitude of magnetostriction for the intended applications and high sensitivity of magnetostriction to applied magnetic field (strain derivative). Materials based on cobalt ferrite are candidates for such device development because of their desirable magnetostrictive properties [1].

Cobalt ferrite (CoFe<sub>2</sub>O<sub>4</sub>) and its derivatives CoM<sub>X</sub>Fe<sub>2-X</sub>O<sub>4</sub> (where M stands for a metal ion) have high strain derivative, good chemical stability and are suitable for developing robust magnetomechanical devices. Moreover, since it has been shown that magnetostriction amplitude of as low as 30 ppm is sufficient

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The dependence of the magnetic and magnetoelastic properties of highly magnetostrictive cobalt ferrite on processing parameters has been investigated. The cobalt ferrite samples used in this study were prepared via conventional ceramic processing methods. The processing parameters of interest were sintering temperature, holding time at the sintering temperature and powder compaction pressure. It was observed that the crystal structure, composition and saturation magnetization of the samples studied did not vary with changes in processing parameters but coercive field decreased with increasing sintering temperature. The amplitude of peak to peak magnetostriction was dependent on the holding time and powder compaction pressure. The strain derivative on the other hand was found to depend on powder compaction pressure at any given sintering temperature or holding time. The results show how the magnetoelastic properties of cobalt ferrite can be varied by changing the processing parameters.

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for several applications [2], the typical amplitudes of magnetostriction, 100–225 ppm, reported for cobalt ferrite in various studies [3–5] are high enough for many applications. As a result, sensor and actuator designs based on cobalt ferrite are expected to be durable, chemically stable and exhibit good response to stimulus in application.

Cobalt ferrite has a partially inverse spinel crystal structure. Fig. 1 [6] shows the cation and oxygen distribution around the tetrahedral and octahedral sites of a spinel crystal structure. For cobalt ferrite, it is generally accepted that a large fraction of Co<sup>2+</sup> are on the octahedral sites (B-sites) and the rest are on the tetrahedral sites (A-sites) [7]. In comparison with other ferrite materials, the high amplitude of magnetostriction in cobalt ferrite is thought to be due to the  $Co^{2+}$  on the B-sites of the spinel structure. This suggests that changes in site occupancy of Co<sup>2+</sup> and deviation of targeted composition from stoichiometry (CoFe<sub>2</sub>O<sub>4</sub>) affect the amplitude of magnetostriction and strain derivative of cobalt ferrite. With two different cation sites, Coferrite offers the potential for tailoring its magnetostrictive properties by influencing the site occupancy of the cations. Substitution of cations such as Mn<sup>3+</sup>, Ga<sup>3+</sup>, Cr<sup>3+</sup>, Ge<sup>4+</sup>/Co<sup>2+</sup> and Al<sup>3+</sup> have been shown to affect the magnetostrictive properties of Co-ferrite in different ways depending on the site into which they substitute [3]. Since magnetostrictive properties also depend on heat treatment and other processing parameters, the capability to optimize magnetostrictive properties without cation substitution is desirable.

The effect of quenching heat treatments and the influence of vacuum sintering on the magnetostrictive properties of Co-ferrite have been studied [8,9]. Both studies showed degradation of magnetostriction and strain derivative due to cation redistribution

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Fig. 1. (A) Tetrahedral site surrounded by four oxygen ions. (B) Octahedral site bounded by six oxygen ions. (C) Spinel crystal structure. Shaded and non-shaded parts represent octants of similar cation occupancy. (D) Two octants showing cation and oxygen distribution. After Cullity [6].

and development of additional phase, respectively. Since it is possible to alter magnetostrictive properties by altering the processing parameters, this study aims at investigating in detail the dependence of the magnetic and magnetostrictive properties of Co-ferrite prepared via conventional ceramic methods on compaction pressure, sintering temperature and holding time during the preparation of Co-ferrite.

#### 2. Experimental details

 $Co_3O_4$  and  $Fe_3O_4$  were mixed in their appropriate proportions and calcined twice for 24 hrs at 1000 °C in air following the chemical equation:

$$Co_3O_4 + 2Fe_3O_4 = 3CoFe_2O_4$$

Calcining the samples twice was necessary to ensure complete solid state reaction of the oxides. After calcining, the powder samples were pressed at 87 or 127 MPa, sintered in air at 1250, 1350 or 1450 °C and held at those temperatures for 24 or 36 h. To confirm a complete formation of the spinel phase, X-ray diffractometry (XRD) was used to study the crystal structure of the samples. The spectra were measured at increaments of  $0.02^{\circ}$ on a Philips PW1710 automated powder diffractometer with copper (CuK $\alpha$ ) radiation at 35 kV and 40 mA. The microstructures and final compositions of the samples were studied using an FEI XL30 FEG environmental scanning electron microscope (ESEM) equipped with an Oxford Instrument Inca X-ray analyzer for energy dispersive X-ray spectroscopy (EDX).

Magnetic properties were studied with a SQUID magnetometer up to a maximum applied field of 50 kOe. Room temperature magnetostriction ( $\lambda$ ) was measured parallel to the applied field using strain gauges attached to the samples. The sensitivity of magnetostriction to applied magnetic field ( $d\lambda/dH$ ) was determined by differentiating the magnetostriction curve with respect to the magnetic field.

#### 3. Results and discussion

The X-ray diffraction patterns shown in Fig. 2 are representative of all samples investigated. Five of the twelve patterns were



**Fig. 2.** X-ray diffraction patterns for a selected subset of representative samples showing the effect of varying sintering temperature (A–C), holding time (B and D) and powder compaction pressure (B and E).

selected to show different effects of the processing parameters studied on the crystal structure of cobalt ferrite. Samples A, B and C have different sintering temperatures, samples B and D have different holding times at a sintering temperature while samples B and E have different powder compaction pressures. All XRD spectra showed only the peaks of the single spinel phase crystal structure. Furthermore, no shifts in the peak positions were observed indicating that no observable changes in lattice constants took place due to the variation of the processing parameters. SEM and EDX analyses were also carried out to further confirm the presence of a single phase by investigating the microstructure and determining the final compositions of the samples studied.

Fig. 3 shows backscattered electron micrographs of the samples. Backscattered electron imaging is used because unlike the secondary electron imaging, the image contrast is a function of the elemental composition of the sample studied.

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