Chapter 3

Effect of Particle Size on the Magnetostriction of Sintered Cobalt Ferrite

3.1 Introduction

Ferrites are the most widely used magnetic materials for many technological applications [1-4]. For many of the technological applications, ferrite powders need to be compacted into specific shapes and sintered under suitable conditions to tune the performance parameters. The properties of the sintered products are generally affected by the composition, stoichiometry, processing conditions, microstructure, density, grain size, etc, and these are very important factors determining the final performance of the ferrites [3]. In the recent years, research work is focused on nanocrystalline ferrites due to their importance in understanding the fundamentals in nanomagnetism as well as for their potential applications, including biomedical applications [5-10]. There are several reports in the literature on the magnetic properties of spinel ferrites derived from nanoparticles with different crystallite sizes [11, 12]. Sintered ferrite products derived from nanocrystalline powders have been shown to exhibit improved performance parameters, such as magnetic permeability and dielectric constant, compared to their bulk counterparts [13-18].

Among the different spinel type ferrites, cobalt ferrite, CoFe$_2$O$_4$, is known for its high magnetocrystalline anisotropy and high coercivity due to the presence of highly
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anisotropic Co$^{2+}$ ions [19, 20]. In addition, the material has good mechanical hardness, chemical stability, and high saturation magnetization. Cobalt ferrite, CoFe$_2$O$_4$, has the mixed spinel structure, represented as $(Co_{1-x}Fe_x)^T[Co_xFe_{2-x}]^O$O$_4$, where $x$ is the degree of inversion, which depends on the heat treatment conditions, and the superscripts T and O represent the tetrahedral and octahedral sites in the spinel structure, respectively [21]. There are several studies reported in the recent literature on the magnetostrictive properties of sintered, un-substituted [22-31] and substituted [32-41] polycrystalline cobalt ferrite, as discussed in detail in Chapter 1. In brief, the magnetostriction coefficient and strain derivative of sintered cobalt ferrite depend on the processing parameters such as the method of synthesis, pressure applied while making the compacts for sintering, sintering atmosphere, temperature, and duration of sintering [26, 27, 31, 42]. Since cobalt ferrite is an ideal ceramic material for future magnetostrictive applications, it is necessary to achieve relatively large magnetostrictive strain at room temperature by changing the synthesis and processing conditions. The highest value of magnetostriction coefficient so far reported for sintered polycrystalline cobalt ferrite is 230 ppm, synthesized by the ceramic method and processed under simple conditions [23].

Preliminary studies have shown that relatively larger magnetostriction coefficient (~200 ppm) can be obtained for sintered cobalt ferrite derived from nanocrystalline powders compared to the material derived from bulk powders [25]. Out of three different low temperature methods used for the synthesis of nanoparticles, particles of size of 30 nm synthesized by an auto-combustion method gave higher values of magnetostriction when compared to the nanoparticles synthesized by other low-temperature methods such as co-precipitation and citrate-gel methods and having smaller particle sizes (10-15 nm). However, so far there have been no detailed and systematic studies reported in the literature to explore the effect of initial particle size of the starting powders used for compaction and the sintering conditions on the magnetostriction characteristics of the sintered compacts derived from nanocrystalline materials.
Nanocrystalline cobalt ferrite can be synthesized by various wet chemical methods such as co-precipitation [43, 44], sol-gel [45-47], hydrothermal [48, 49], autocombustion [25, 50, 51], microemulsion [52, 53], etc. Out of this, autocombustion method is a very simple process to obtain nanocrystalline cobalt ferrite. Kikukawa et al have reported that the particle size of cobalt ferrite can be varied in the glycine-nitrate process by changing the fuel (glycine) to oxidizer (nitrate) ratio in the combustion synthesis [51]. Nanosized particles could be obtained by using smaller glycine to nitrate (G/N) ratios. The objective of the present work is to study the effect of the initial particle size of the powders on the ultimate value of magnetostriction of cobalt ferrite after sintering. The nanocrystalline cobalt ferrite powders of varying particle sizes below 100 nm are synthesized by the autocombustion method by changing the glycine to nitrate ratio, as reported in the literature [51].

3.2 Synthesis

Cobalt ferrite nanoparticles were synthesized by the autocombustion method, using glycine (G) as fuel and the corresponding metal (M) nitrates (N) as the oxidizer, following the previous reports [25, 50, 51]. The main controllable processing variable is the G/N or M/G ratio. G/N ratio corresponds to the complete combustion is called the stoichiometric ratio. For the synthesis of cobalt ferrite from the corresponding metal nitrates, the stoichiometric G/N ratio is 0.55:1 or M/G ratio is 1:1.47 according to the reaction,

\[
9 \text{Co(NO}_3\text{)}_2 + 18 \text{Fe(NO}_3\text{)}_3 + 40 \text{NH}_2\text{CH}_2\text{COOH} \\
\rightarrow 9 \text{CoFe}_2\text{O}_4 + 56 \text{N}_2 + 80 \text{CO}_2 + 100 \text{H}_2\text{O}
\]

(or)

\[
\text{Co(NO}_3\text{)}_2 + 2 \text{Fe(NO}_3\text{)}_3 + 4.44 \text{NH}_2\text{CH}_2\text{COOH} \rightarrow \text{CoFe}_2\text{O}_4
\]

where, G/N = 4.44/(2 x 3 + 1 x 2) = 4.44/8 = 0.555.
For the synthesis of cobalt ferrite, stoichiometric amounts of cobalt nitrate, \(\text{Co(NO}_3\text{)}_2\cdot6\text{H}_2\text{O}\) (Aldrich), and ferric nitrate, \(\text{Fe(NO}_3\text{)}_3\cdot9\text{H}_2\text{O}\) (Aldrich), taken in the 1:2 molar ratio, were dissolved separately, in minimum amount of distilled water. Glycine (Merck), corresponding to different glycine to nitrate ratios, ranging from 0.05 to 0.75, 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 minutes. 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. This procedure was repeated for different G/N ratios. The sample codes and the corresponding G/N ratios are indicated in Table 3.1.

To compare the properties of the nanocrystalline cobalt ferrite as well as the sintered products derived from the nanocrystalline powders, bulk cobalt ferrite was also synthesized by the ceramic method, starting from the oxides of cobalt and iron taken in the stoichiometric ratio. For this, stoichiometric amounts of \(\text{CoCO}_3\) (Aldrich) and \(\text{Fe}_2\text{O}_3\) (Aldrich) were weighed and mixed together in an agate mortar. The mixture was first heated at 1000 °C for 12 h and again for 24 h at the same temperature after an intermediate grinding. The resulting powder was further heated at 1100 °C for 72 h with two intermediate grindings.

To study the effect of particle size on magnetostriction of sintered cobalt ferrite, nanocrystalline cobalt ferrite powders were also synthesized by co-precipitation and citrate methods. For the co-precipitation method, cobalt and iron nitrates were taken in the 1:2 molar ratio and dissolved in 100 ml distilled water. By using a burette, the mixed solution was added to 20% of KOH (SD Fine Chemicals) solution taken in a round bottom flask. The precipitate formed was filtered and washed several times with distilled water until the pH is around 7. The precipitate was dried overnight in an oven at 100 °C which was eventually converted to a black powder of cobalt ferrite. For the citrate gel method, stoichiometric amounts of the corresponding metal nitrates were dissolved in
distilled water. Water solution of citric acid (SD Fine Chemicals), corresponding to two moles of citric acid per mole of metal, was added to the metal ion solution. The resulting solution was evaporated on a water bath and finally a thick gel was formed. This precursor gel was dried in an oven at 100 °C and the dried precursor was calcined at 500 °C for 4 h to get the cobalt ferrite powder.

### 3.3 Studies on As-synthesized Powder Samples

#### 3.3.1 Phase Purity and Structural Characterization

Figure 3.1 shows the powder XRD patterns of the different cobalt ferrite samples synthesized by varying the G/N ratio from 0.05 to 0.75 (sample codes G1 to G13). All the reflections in the experimental patterns are very broad, and the width of the reflections decreases with increasing G/N ratio, indicating increasing crystallite size. Only the most intense reflection of cobalt ferrite is observed for G1 as a very broad peak. The next most intense reflection started appearing with increased intensity for G2 and G3 and all the reflections of cobalt ferrite are observed for sample G4 and others. For the combustion synthesis of cobalt ferrite using glycine, the complete combustion reaction corresponds to the G/N ratio of 0.55, according to the balanced reaction as explained in section 3.2. It is found that single phase ferrite is formed up to the G/N ratio of 0.47 (sample G10). FeO is found to be formed as an impurity phase at the stoichiometric G/N ratio of 0.55 (sample G11). At higher G/N ratios, due to the presence of excess glycine, Fe metal impurity is formed, as indicated in Figure 3.1. Kikukawa *et al* previously reported that single phase spinel type ferrites can be obtained from precursors with a G/N ratio less than about 0.5 [51].
Table 3.1: Details of the cobalt ferrite samples synthesized by the autocombustion method using metal nitrates and glycine.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>G/N ratio</th>
<th>M/G ratio</th>
<th>Flame temperature (±1°C)</th>
<th>D_{XRD} (±1nm)</th>
<th>Lattice parameter (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>8.382</td>
</tr>
<tr>
<td>G1</td>
<td>0.05</td>
<td>0.125</td>
<td>190</td>
<td>3</td>
<td>8.340</td>
</tr>
<tr>
<td>G2</td>
<td>0.09</td>
<td>0.25</td>
<td>215</td>
<td>4</td>
<td>8.340</td>
</tr>
<tr>
<td>G3</td>
<td>0.14</td>
<td>0.375</td>
<td>230</td>
<td>5</td>
<td>8.381</td>
</tr>
<tr>
<td>G4</td>
<td>0.19</td>
<td>0.5</td>
<td>385</td>
<td>7</td>
<td>8.385</td>
</tr>
<tr>
<td>G5</td>
<td>0.23</td>
<td>0.675</td>
<td>442</td>
<td>14</td>
<td>8.384</td>
</tr>
<tr>
<td>G6</td>
<td>0.28</td>
<td>0.75</td>
<td>640</td>
<td>22</td>
<td>8.390</td>
</tr>
<tr>
<td>G7</td>
<td>0.33</td>
<td>0.875</td>
<td>720</td>
<td>34</td>
<td>8.385</td>
</tr>
<tr>
<td>G8</td>
<td>0.38</td>
<td>1.0</td>
<td>770</td>
<td>40</td>
<td>8.393</td>
</tr>
<tr>
<td>G9</td>
<td>0.42</td>
<td>1.125</td>
<td>811</td>
<td>61</td>
<td>8.387</td>
</tr>
<tr>
<td>G10</td>
<td>0.47</td>
<td>1.25</td>
<td>934</td>
<td>78</td>
<td>8.389</td>
</tr>
<tr>
<td>G11</td>
<td>0.55</td>
<td>1.47</td>
<td>867</td>
<td>55</td>
<td>8.389</td>
</tr>
<tr>
<td>G12</td>
<td>0.66</td>
<td>1.67</td>
<td>856</td>
<td>57</td>
<td>8.380</td>
</tr>
<tr>
<td>G13</td>
<td>0.75</td>
<td>2.0</td>
<td>573</td>
<td>60</td>
<td>8.350</td>
</tr>
</tbody>
</table>
Figure 3.1: Powder XRD patterns of as-synthesized cobalt ferrite powders using different glycine/nitrate ratios (G1 to G13) as well as by the ceramic method (G0). Impurity peaks are marked as *FeO and #Fe.
It is known that the temperature of the combustion reaction has an important effect on the crystallite size of the resulting powder [51]. By adjusting the G/N ratio, one can control the reaction temperature and thereby control the crystallite size of the products. The temperature of the combustion reaction was measured by a Laser thermometer (METRAVI MT-14). As shown in Figure 3.2, flame temperature initially increased with increasing the G/N ratio, highest flame temperature is obtained for G/N = 0.47 and the flame temperature is decreased further with increasing glycine content. It was found that from G1 to G10, flame temperature increases and correspondingly, the crystallite size also increases. This is due to the increasing exothermicity of the combustion reaction with increasing amount of the fuel [54]. A low flame temperature of 190 °C was observed in the case of G1. The highest flame temperature of 934 °C was obtained for G10 for which the G/N ratio is close to stoichiometric ratio (0.47). At this G/N ratio, complete combustion reaction takes place. When the G/N ratio is further increased beyond 0.47, the flame temperature was found to be decreased due to the lesser amount of the oxidizer available for the combustion reaction.
The average crystallite size is calculated using the Scherrer formula, $D_{\text{XRD}} = \frac{0.9\lambda}{\beta \cos \theta}$, where $D_{\text{XRD}}$ is the crystallite size. The calculated average crystallite sizes are shown in Table 3.1. The crystallite size increases with increasing G/N ratio and reaches a maximum for G/N = 0.47 at which the flame temperature also is maximum. There is a direct correlation between the flame temperature and the crystallite size, similar to that reported by Chick et al [54], in the case of synthesis of perovskite oxides using the glycine-nitrate process. Relatively smaller crystallite sizes are obtained when impurity phases are also formed along with the ferrite phase for G/N $\geq 0.5$. The cubic lattice parameter is calculated for all the samples by least squares method of refinement of the XRD patterns and the values are given in Table 3.1. For G3 to G12, the calculated lattice parameter is comparable to the value reported for cobalt ferrite (8.391 Å, JCPDS # 22-1086) [55]. For G1 and G2, the values are slightly smaller, probably because only few peaks are observed and the peaks are very broad, so that there could be errors in the calculation of the lattice parameter. The lattice parameter is slightly smaller for G12 and G13, possibly due to the formation of a different composition because Fe impurity in large amounts is observed in these samples.

The transmission electron micrographs of some of the as-synthesized powder samples (G2, G4, G6 and G8) are shown in Figure 3.3. The average particle sizes are obtained as 6, 8, 25 and 45 nm, respectively for G2, G4, G6 and G8 and these values are found to be comparable to the average crystallite sizes calculated from XRD line broadening. This clearly shows that nanocrystalline cobalt ferrite has been formed by the present method of synthesis. Since the average crystallite sizes calculated for G2, G4, G6 and G8 are comparable to the particle sizes obtained from TEM measurements, the average crystallite size is considered as the particle size for all samples. Therefore, the crystallite sizes are used as particle sizes in the rest part of this thesis.
3.3.2 Magnetic Measurements

Room temperature magnetic measurements on the as-synthesized powder samples are shown in Figure 3.4. Since formation of impurity phases is observed in samples G11, G12 and G13, magnetic studies have been performed on samples G1 to G10 only. Magnetic hysteresis loop is not observed for G1 and G2 (Figure 3.5) having very small particle sizes of 3 nm and 4 nm, respectively, indicating superparamagnetic nature of the particles. This is further confirmed from field cooled (FC) and zero field cooled (ZFC) magnetization measurements for the samples G1 and G2 as shown in Figure 3.6. Both FC and ZFC magnetization are measured in a field of 8 kA/m (100 Oe). Superparamagnetic particles are characterized by a maximum at the superparamagnetic blocking temperature in the zero field cooled magnetization measurements. The blocking temperatures for G1 and G2 are obtained as 90 K and 150 K, respectively. These values are comparable to that reported in the literature for cobalt ferrite nanoparticles of similar sizes [56].
As shown in Figure 3.4 and Table 3.2, magnetization of the as-synthesized samples is not saturated at the highest field of measurement and the magnetization decreases with decreasing particle size. This is due to the increasing surface contribution for smaller particles where the surface layer is considered to be magnetically dead [57, 58]. Similarly, the coercivity initially increases with particle size, reaches a maximum value for G7 of particle size 34 nm, and then decreases. This behavior corresponds to the change over from single domain to multi-domain particles above a critical size. The critical size of 34 nm obtained for the present cobalt ferrite is comparable to the critical size reported for other spinel ferrites [59-62].

Table 3.2: Magnetic properties of the as-synthesized powder samples. M is the magnetization at 1200 kA/m, the highest field used for the measurement.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>M (Am²/kg)</th>
<th>Hc (kA/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>0.7</td>
<td>0</td>
</tr>
<tr>
<td>G2</td>
<td>1.9</td>
<td>0</td>
</tr>
<tr>
<td>G3</td>
<td>13.2</td>
<td>15.0</td>
</tr>
<tr>
<td>G4</td>
<td>26.3</td>
<td>32.5</td>
</tr>
<tr>
<td>G5</td>
<td>43.2</td>
<td>60.1</td>
</tr>
<tr>
<td>G6</td>
<td>62.0</td>
<td>72.6</td>
</tr>
<tr>
<td>G7</td>
<td>67.5</td>
<td>74.0</td>
</tr>
<tr>
<td>G8</td>
<td>69.8</td>
<td>67.3</td>
</tr>
<tr>
<td>G9</td>
<td>75.6</td>
<td>62.0</td>
</tr>
<tr>
<td>G10</td>
<td>67.5</td>
<td>81.5</td>
</tr>
<tr>
<td>G11</td>
<td>41.2</td>
<td>89.2</td>
</tr>
<tr>
<td>G12</td>
<td>85.8</td>
<td>68.3</td>
</tr>
<tr>
<td>G13</td>
<td>91.2</td>
<td>65.9</td>
</tr>
</tbody>
</table>
Figure 3.4: Room temperature $M$ vs $H$ curves of the as-synthesized powder samples.

Figure 3.5: Enlarged $M$-$H$ curves of G1 and G2.
Figure 3.6: Zero field cooled (ZFC) and field cooled (FC) magnetization curves of G1 and G2, measured in a field of $H=8$ kA/m (100 Oe).
Temperature variation of magnetization for all the as-synthesized samples has been measured above room temperature, in a field of 8 kA/m, as shown in Figure 3.7. Strikingly, only the samples G1, G2 and G3 showed a sharp magnetic transition at the expected Curie temperature of cobalt ferrite. The measured Curie temperature is close to 793 K and is comparable with that of the bulk powder as well as the value reported in the literature [35, 63]. The large increase in the magnetization above 600 K for G1, G2 and G3 is likely to be due to the growth of the particles at higher temperatures, as reported previously for other ferrites [58]. Whereas all the rest of the samples showed either more than one magnetic transition or a lower transition temperature in the temperature range 700-800 K. This is likely to be due to the compositional inhomogeneity such that different particles in a sample can have different compositions in the series Co_{1+x}Fe_{2-x}O_4 close to $x = 0$ and/or the formation of CoFe_2O_4 with different degree of distribution in
different particles represented as $(Co_{1-x}Fe_x)^7[Co_xFe_{2-x}]O_4$ during the formation of the product in the combustion reaction.

Figure 3.8: Sintering behavior of the cobalt ferrite samples synthesized from different glycine/nitrate ratios compared with ceramic sample.

3.3.3 TMA Analysis

For magnetostriction studies, the samples need to be sintered into specific shapes. The sintering characteristics of the samples are, therefore, studied by thermo-mechanical analysis (TMA). Sintering curves of different samples synthesized by the autocombustion method are compared with that obtained by the ceramic method in Figure 3.8. The sintering behavior of the powder samples synthesized by autocombustion method differs from that of the powder samples synthesized by the ceramic method. In the case of the ceramic sample (G0), effective sintering starts only above 1200 °C. However, for the
nanocrystalline samples G1 and G2, the onset of the sintering process is about 500 °C and this temperature slowly increases up to 800 °C for G8 with increasing crystallite size. These results show that the nanocrystalline cobalt ferrite can be sintered at relatively lower temperatures compared to the ceramic sample, as reported previously [25]. However, TMA analysis shows that the shrinkage is continued with increasing temperature and that the samples are not completely sintered up to a maximum temperature of 1500 °C.

3.4 Studies on Sintered Samples

Most of the studies reported in the literature on sintered cobalt ferrite are made on compacts sintered at 1450 °C [22, 23, 25]. Also, TMA studies indicate that the sintering is not completed up to the temperature of 1500 °C. Hence, all the samples were sintered under identical conditions for comparison of the results. Pressed compacts were heated in a programmable furnace to a temperature of 1450 °C at the heating rate of 5 °C/min and held at this temperature for 10 minutes. The samples were then cooled back to room temperature at the rate of 20 °C/min.

3.4.1 XRD Studies

Figure 3.9 shows the X-ray diffraction patterns of the sintered samples. The sample codes SG0 and SG1 to SG10 correspond to the sintered samples derived from the corresponding powders G0 and G1 to G10. In the XRD patterns, all the observed peaks correspond to spinel ferrite and no other impurity phases are formed after sintering. XRD patterns of all the sintered samples are identical and matching well with that of the pattern shown and indexed for the bulk sample (SG0). The lattice parameter for the sintered samples SG1 to SG9 is found to be comparable to that obtained for the bulk sample and the literature value for cobalt ferrite (8.391 Å). Unusually low value for the lattice parameter is obtained for SG10. This could be due to a slight difference in the composition for this sample. A very low intensity impurity peak due to FeO was observed in the XRD pattern of the corresponding powder sample G10 (see Figure 3.1).
Figure 3.9: Powder XRD patterns of the sintered samples.
3.4.2 Density Measurements

Figure 3.10 shows the variation of the density of the sintered pellets as a function of G/N ratio. The densities of the sintered pellets were calculated from the volume and the weight of the pellets, as well as by the Archimedes method. The relative density was calculated from the ratio of the measured density to the theoretical density of cobalt ferrite (5.276 g/cm³) calculated from the crystal structure parameters. Density is almost comparable for SG1 and SG2 and then increased to the highest density of 86% for SG4. Density is further decreased down to ~79% with increasing G/N ratio. The variation in the sintered density for particles with larger sizes could be due to the characteristics of the powders obtained under different G/N ratios. The relatively larger density of SG10 compared to SG9 could be due to the presence of minor amounts of impurity, as discussed in the previous section. Large increase in the flame temperature in the combustion reaction was observed for larger G/N ratios (Figure 3.2) which could affect the surface characteristics of the powders and control the sintering behavior and hence the density of the sintered compacts.

![Figure 3.10: Density of the sintered products as a function of G/N ratio.](image)
3.4.3 Magnetic Measurements

Magnetic measurements were carried out on crushed small pieces of sintered pellets. It has been shown previously that magnetic characteristics of the crushed pieces of sintered cobalt ferrite with arbitrary sizes and shapes affect the magnetic characteristics due to the random orientation of the grains with respect to the direction of the applied magnetic fields as well as due to the shape anisotropy [64]. Since the sintered sample SG10 showed a difference in the lattice parameter, probably due to a different composition, further studies are made on sintered samples from SG1 to SG9 only. In the case of the sintered samples, the magnetization is found to be saturated and the saturation magnetization varies between 77.9 to 83.4 Am²/kg, for the samples SG1 to SG9, as shown in Figure 3.11 and Table 3.3. There is a corresponding variation of the coercivity also. The small variation in the saturation magnetization and coercivity could be due to the cation distribution as well as the microstructure and orientation of the grains in the sintered products.

Figure 3.11: Room temperature magnetization curves for the different sintered cobalt ferrite samples.
Temperature variation of magnetization for all sintered samples (SG1 to SG9) measured above room temperature is shown in Figure 3.12. It is found that all sintered samples show comparable Tc,s, close to the reported value for the bulk material (~793 K). The large increase in the magnetization above 600 K and the flat region between 700 K and Tc are due to the changes in the magnetocrystalline anisotropy of cobalt ferrite [64]. Curie temperature is a measure of the degree of distribution of the cations in the tetrahedral and octahedral sites of the spinel lattice. The comparable Tc,s of the sintered products indicate almost similar distribution of the cations in all the samples. The cation distribution in the tetrahedral and octahedral sites of the spinel lattice is expected to affect the magnetization, magnetic anisotropy and Curie temperature of the material. The magnetic properties of different sintered samples are compared in Table 3.3.

![M-T curves of the sintered cobalt ferrite samples, measured in a magnetic field of 8 kA/m (100 Oe).](image)
3.4.4 Magnetostriction Studies

Some of the previous studies reported on the magnetostriction measurements on sintered cobalt ferrite are made on samples sintered at 1450 °C [23, 25]. Hence, to compare the magnetostriction characteristics of sintered samples derived from the nanocrystalline powders with that reported in the literature, all the studies are made on pressed pellets sintered at 1450 °C for 10 minutes. Figure 3.13 shows the magnetostriction curves recorded up to a maximum magnetic field strength of 800 kA/m, in the parallel ($\lambda_{\text{par}}$) and perpendicular ($\lambda_{\text{per}}$) directions, respectively, with respect to the direction of the applied magnetic field. The magnetostriction is negative along the parallel direction, as reported [26, 29, 32, 34]. Higher magnetostriction is obtained along the parallel direction for all the samples sintered from nanocrystalline materials ($\geq$ 200 ppm) compared to the value of 150 ppm for the sample prepared by the ceramic method with a starting particle size > 1 μm and processed under identical conditions. Maximum value of magnetostriction above 300 ppm is achieved for the samples sintered from the smallest particles (G1 and G2), with the highest value of 315 ppm for SG2. SG1 and SG2 show some peculiarities in the magnetostriction along the perpendicular direction also. These two samples show negative magnetostriction along the perpendicular direction and all other samples show positive values along this direction. This is an indication for the favorable alignment of the domains and easy axis of magnetization in the sintered pellets derived from very small particles. Moreover, compared to SG1, SG2 shows higher magnetostriction at lower magnetic fields and maximum value of magnetostriction for SG2 is obtained at a lower field. However, higher magnetostriction at low fields (below 200 kA/m) is obtained for sample SG8. These differences are much clear in Figure 3.14 which shows the strain derivative ($d\lambda/dH$) as a function of magnetic field measured along the parallel direction.

The anisotropic magnetostriction, $\lambda_a = \lambda_{\text{par}} - \lambda_{\text{per}}$ as well as the volume magnetostriction $\omega = \lambda_{\text{par}} + 2\lambda_{\text{per}}$ are also of interest for these two samples. Figure 3.15 shows the variation of maximum value of magnetostriction ($\lambda_{\text{max}}$) along the parallel direction, maximum value of the strain derivative, anisotropic and volume
magnetostrictions as a function of the particle size of the starting powders. The magnitude of $\lambda_a$ is comparable for all samples, whereas the signs are different for SG1 and SG2. Similarly, these two samples show very high volume magnetostriction of around 400 ppm and for all other samples, the value is less than 200 ppm. Thus, the samples sintered from very small particles, G1 and G2, show extremely large values for the magnetostriction coefficient. Apart from the changes in the value of magnetostriction, the slope of the magnetostriction ($d\lambda/dH$) is also an important factor for stress sensing applications. Higher values of the strain derivative are desirable for better magnetomechanical coupling [32]. The highest value of strain derivative of $1.97 \times 10^{-9}$ A$^{-1}$m is obtained in the case of SG2 which is larger than the previously reported strain derivative value of $1.3 \times 10^{-9}$ A$^{-1}$m for bulk material [23].

Table 3.3: Magnetic and magnetostrictive properties of sintered pellets.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Density (%)</th>
<th>$M_s$ (Am$^2$/kg)</th>
<th>$H_c$ (kA/m)</th>
<th>$T_c$ (°C)</th>
<th>$\lambda_{\text{max}}$ (ppm)</th>
<th>$d\lambda/dH$ (10$^{-9}$A$^{-1}$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SG0</td>
<td>89</td>
<td>78.5</td>
<td>19.1</td>
<td>521</td>
<td>-151</td>
<td>-0.56</td>
</tr>
<tr>
<td>SG1</td>
<td>80</td>
<td>79.6</td>
<td>14.1</td>
<td>515</td>
<td>-307</td>
<td>-1.14</td>
</tr>
<tr>
<td>SG2</td>
<td>80</td>
<td>81.6</td>
<td>12.4</td>
<td>520</td>
<td>-315</td>
<td>-1.97</td>
</tr>
<tr>
<td>SG3</td>
<td>84</td>
<td>82.8</td>
<td>7.4</td>
<td>525</td>
<td>-205</td>
<td>-1.03</td>
</tr>
<tr>
<td>SG4</td>
<td>86</td>
<td>83.3</td>
<td>5.5</td>
<td>523</td>
<td>-200</td>
<td>-1.12</td>
</tr>
<tr>
<td>SG5</td>
<td>85</td>
<td>83.4</td>
<td>6.7</td>
<td>524</td>
<td>-218</td>
<td>-1.00</td>
</tr>
<tr>
<td>SG6</td>
<td>84</td>
<td>82.8</td>
<td>6.0</td>
<td>521</td>
<td>-232</td>
<td>-1.06</td>
</tr>
<tr>
<td>SG7</td>
<td>82</td>
<td>82.8</td>
<td>8.1</td>
<td>524</td>
<td>-252</td>
<td>-1.49</td>
</tr>
<tr>
<td>SG8</td>
<td>80</td>
<td>81.3</td>
<td>11.1</td>
<td>516</td>
<td>-254</td>
<td>-1.35</td>
</tr>
<tr>
<td>SG9</td>
<td>79</td>
<td>77.9</td>
<td>33.6</td>
<td>526</td>
<td>-235</td>
<td>-1.01</td>
</tr>
</tbody>
</table>
Figure 3.13: Magnetostriction curves of different sintered samples as a function of magnetic field measured along the parallel ($\lambda_{\text{par}}$) and perpendicular ($\lambda_{\text{per}}$) directions. The magnetostriction of the ceramic sample (SG0) is shown for comparison.
Figure 3.14: Strain derivative of different sintered samples as a function of magnetic field measured along the parallel direction.

Figure 3.15: Variation of (a) maximum value of magnetostriction along the parallel direction, (b) strain derivative, (c) anisotropic magnetostriction and (d) volume magnetostriction as a function of the particle size of the starting powders.
3.4.5 Microstructure

A comparison of the coercivity and density of the sintered products and magnetostriction parameters, as shown in Table 3.3, indicate that in general, there are no correlations between the magnetic characteristics or the density with the maximum value of magnetostriction. However, there are some minor correlations observed with the microstructure of the sintered compacts as shown in Figure 3.16. SG1 and SG2 with the highest magnetostriction ($\lambda > 300$ ppm) show microstructures with clear and small grains. For SG3 to SG6, larger grains with intra-grain pores and defects are observed, even though these samples show lower porosity and higher sintered densities in the range 84–86% compared to 80% density for SG1 and SG2. For SG7 to SG9, continuous and fused grains without any shape are observed. Thus, it appears that the microstructure, which depends on the initial particle size, could be one of the decisive factors for the higher and lower values of magnetostriction and strain derivative of the sintered samples.

3.4.6 Effect of Particle Size

The results from the magnetostriction studies on different sintered samples derived from starting powders of different particle sizes show that the maximum value of the magnetostriction coefficient is not exactly correlated with the initial particle size, even though compacts derived from the smallest particles show higher magnetostriction coefficient. However, the different particle sizes are obtained by changing the glycine to nitrate ratio during the synthesis. It is possible that the powder characteristics are different because of the different amounts of fuel used and this affects the sintering characteristics and microstructure and thereby the ultimate value of magnetostriction. To study the effect of particle size on the maximum value of magnetostriction, the particle size of G2, which shows the highest value of magnetostriction after sintering (SG2) is changed by pre-calcination at different temperatures. It is known that particle growth takes place if nanocrystalline materials synthesized at low temperatures are further calcined at higher temperatures [65, 66]. Therefore, the as-synthesized G2 powder was calcined at two different temperatures to vary the particle size.
Figure 3.16: SEM images of the sintered samples. All micrographs shown are under the same magnification.
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XRD patterns of the calcined powders are shown in Figure 3.17. The average crystallite sizes of the calcined powders are calculated using the Scherrer equation and the values are shown in Table 3.4. The calculated average crystallite size is assumed as the average particle size. There is substantial increase in the particle size after calcining at the two different temperatures for different durations. Similarly, there is an increase in the magnetization as well as coercivity of the powders with increasing particle size, as expected. The magnetization curves of the calcined powders are compared in Figure 3.18 and the magnetic parameters are compared in Table 3.5.

Table 3.4: Details of the calcined G2 powders.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Calcination conditions</th>
<th>$D_{\text{XRD}}$ (±1nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G2</td>
<td>As-synthesized</td>
<td>4</td>
</tr>
<tr>
<td>G2a</td>
<td>180 °C /30min</td>
<td>7</td>
</tr>
<tr>
<td>G2b</td>
<td>400 °C/5hr</td>
<td>21</td>
</tr>
</tbody>
</table>

Figure 3.17: Powder XRD patterns of as-synthesized and calcined G2 powders.
Figure 3.18: Room temperature magnetization curves of the as-synthesized and calcined G2 powders.

Figure 3.19: Magnetostriction curves of different sintered samples derived from G2 as a function of magnetic field measured along the parallel ($\lambda_{par}$) direction.
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Figure 3.20: SEM images of the sintered samples derived from G2. All micrographs are under the same magnification.

Table 3.5: Magnetic and magnetostrictive characteristics of sintered pellets derived from as-synthesized and calcined G2 powders.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>D (%)</th>
<th>$M_s$ (Am$^2$/kg)</th>
<th>$H_c$ (kA/m)</th>
<th>$\lambda_{\text{max}}$ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SG2</td>
<td>80</td>
<td>81.6</td>
<td>12.4</td>
<td>-315</td>
</tr>
<tr>
<td>SG2a</td>
<td>80</td>
<td>82.0</td>
<td>15.8</td>
<td>-301</td>
</tr>
<tr>
<td>SG2b</td>
<td>78</td>
<td>84.1</td>
<td>15.1</td>
<td>-204</td>
</tr>
</tbody>
</table>

Magnetostriction of the pellets sintered from the calcined powders derived from G2 is measured as a function of magnetic field. The results are shown in Figure 3.19. The maximum value of magnetostriction is decreased after increasing the particle size of the starting powder, as expected. The SEM figures shown in Figure 3.20 indicate that there is not much difference in the microstructures of the sintered compacts derived from the as-synthesized and pre-calcined G2 powders, suggesting that there is not much role for the microstructure also in deciding the value of magnetostriction. An important observation is that the decrease in the maximum value of magnetostriction of the sintered samples
derived from the pre-calcined powders with different particle sizes (G2a and G2b) is much more than that observed for sintered compacts derived from the as-synthesized starting powders of comparable particle sizes (G4 and G6). This shows that apart from the particle sizes of the starting powders, there are other factors such as the characteristics of the starting powders contributing to the maximum value of magnetostriction of the sintered compacts. The characteristics of the starting powders can be varied by the synthesis conditions.

Figure 3.21: Powder XRD patterns of CoFe₂O₄ samples synthesized by the citrate-gel and co-precipitation methods.

To verify the above assumptions, nanocrystalline powders of cobalt ferrite were synthesized by co-precipitation and citrate-gel methods. Figure 3.21 shows the XRD patterns of single phase cobalt ferrite obtained by co-precipitation and citrate precursor methods. Only the most intense peak of cobalt ferrite is observed in the XRD pattern of the sample prepared by the co-precipitation method and the peak is very broad than that
of the peaks observed in the XRD pattern of the powder synthesized by the citrate precursor method, indicating smaller crystallite size in the former case. The average crystallite size is calculated using the Scherrer formula and the calculated values are shown in Table 3.6.

Table 3.6: Different properties of as-synthesized powder samples synthesized by the co-precipitation and citrate precursor methods.

<table>
<thead>
<tr>
<th>Synthesis method</th>
<th>$D_{XRD}$ (±1nm)</th>
<th>$M_s$ (Am$^2$/kg)</th>
<th>$H_c$ (kA/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co-precipitation</td>
<td>5</td>
<td>6.4</td>
<td>0</td>
</tr>
<tr>
<td>Citrate</td>
<td>22</td>
<td>65.0</td>
<td>82.6</td>
</tr>
</tbody>
</table>

Figure 3.22: Room temperature magnetization curves of the as-synthesized powders synthesized by the co-precipitation and citrate methods.
Room temperature magnetic measurements on the as-synthesized powder samples obtained by co-precipitation and citrate-gel methods are shown in Figure 3.22. The as-synthesized powder obtained by the co-precipitation method of particle size 5 nm show superparamagnetic behavior as expected for such small particles. High magnetization with large coercivity is obtained for the sample synthesized by the citrate-gel method of particle size of 22 nm.

Figure 3.23 shows the magnetostriction curves of sintered samples obtained by the co-precipitation and citrate methods. Maximum value of magnetostriction as 160 ppm is obtained for sintered sample obtained by the co-precipitation method and 216 ppm in the case of sintered sample obtained by the citrate method. The microstructures of both sintered samples are almost comparable, as shown in Figure 3.24. Even though the particle size of the powders obtained from co-precipitation method is comparable with that of the sample G2 synthesized by the combustion method, the maximum value of magnetostriction of the former is nearly one half of the value of the latter.

![Figure 3.23: Magnetostriction as a function of magnetic field recorded in the parallel direction to the applied magnetic field.](image)
Figure 3.24: SEM images of the sintered samples synthesized by (a) co-precipitation and (b) citrate-gel methods.

Table 3.7: Magnetic and magnetostrictive properties of sintered pellets obtained by co-precipitation and citrate methods.

<table>
<thead>
<tr>
<th>Synthesis method</th>
<th>D (%)</th>
<th>$M_s$ (Am$^2$/kg)</th>
<th>$H_c$ (kA/m)</th>
<th>$\lambda_{\text{max}}$ (ppm)</th>
<th>$d\lambda/dH$ ($10^{-9}$A$^{-1}$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co-precipitation</td>
<td>78</td>
<td>78.5</td>
<td>12.4</td>
<td>-160</td>
<td>-0.84</td>
</tr>
<tr>
<td>Citrate</td>
<td>87</td>
<td>82.3</td>
<td>10.9</td>
<td>-215</td>
<td>-1.05</td>
</tr>
</tbody>
</table>

The magnetostriction characteristics of the sintered samples derived from starting powders of comparable particle sizes obtained by the three different methods are compared in Figure 3.25 and Table 3.8. The results show that for particles of comparable sizes, sintered compacts derived from the powders obtained by the auto-combustion method give higher values of magnetostriction and strain derivative. Also, there is not much correlation between the sintered density and magnetostriction characteristics. From these results it can be concluded that the magnetostriction of sintered ferrite depends on and can be controlled by the synthesis and processing conditions and by adjusting the particle size of the initial starting powders used for sintering the pellets.
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Table 3.8: Comparison of the magnetostrictive properties of sintered pellets derived from powders of comparable sizes obtained by different synthesis methods.

<table>
<thead>
<tr>
<th>Synthesis Method</th>
<th>Sample Code</th>
<th>$D_{XRD}$ (±1nm)</th>
<th>D (%)</th>
<th>$\lambda_{max}$ (ppm)</th>
<th>$d\lambda/dH$ ($10^{-9}$ A$^{-1}$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Autocombustion</td>
<td>G2</td>
<td>4</td>
<td>80</td>
<td>-315</td>
<td>-1.97</td>
</tr>
<tr>
<td>Co-precipitation</td>
<td>Cop</td>
<td>5</td>
<td>78</td>
<td>-160</td>
<td>-0.84</td>
</tr>
<tr>
<td>Autocombustion</td>
<td>G3</td>
<td>5</td>
<td>84</td>
<td>-205</td>
<td>-1.03</td>
</tr>
<tr>
<td>&quot;</td>
<td>G2a</td>
<td>7</td>
<td>80</td>
<td>-301</td>
<td>-1.40</td>
</tr>
<tr>
<td>&quot;</td>
<td>G4</td>
<td>7</td>
<td>86</td>
<td>-200</td>
<td>-1.12</td>
</tr>
<tr>
<td>&quot;</td>
<td>G2b</td>
<td>21</td>
<td>78</td>
<td>-204</td>
<td>-1.01</td>
</tr>
<tr>
<td>&quot;</td>
<td>G6</td>
<td>22</td>
<td>84</td>
<td>-232</td>
<td>-1.06</td>
</tr>
<tr>
<td>Citrate-gel</td>
<td>Cit</td>
<td>22</td>
<td>87</td>
<td>-215</td>
<td>-1.05</td>
</tr>
</tbody>
</table>

Figure 3.25: Magnetostriction as a function of magnetic field recorded in the parallel direction to the applied magnetic field, for sintered pellets derived from powders of comparable sizes obtained by different synthesis methods. The sample codes are defined in Table 3.8.
3.4.7 Effect of Magnetic Field Annealing

Magnetic field annealing has been reported to be very effective in enhancing the magnetostriction coefficient and strain derivative of cobalt ferrite due to the induced easy axis direction parallel to the annealing field, which affects magnetization processes and domain configuration [24, 30]. Higher magnetostriction coefficient perpendicular to the direction of the annealing field has been achieved by magnetic field annealing because of the reorientation of the domains along the measurement direction. In a very recent study, Muhammed et al investigated the effect of different powder compaction pressures in the range of 87 to 278 MPa and magnetic field annealing in an external magnetic field of 8000 kA/m (10 T), at 300 °C for 3 hours [31]. The field annealing process causes an induced uniaxial anisotropy, which results in a reduction of coercivity as well as increase in the magnetostrictive strain up to 400 ppm along the hard axis for the compacts made under a very high pressure of 150 MPa.

In the present work, we have compared the effect of magnetic field annealing on the samples SG1, SG2 where the starting particle sizes are very small and comparable for G1 and G2. These two samples gave comparable values for maximum magnetostriction but SG2 showed relatively larger magnetostriction at lower magnetic fields as well as high strain derivative of $1.97 \times 10^{-9}$ A$^{-1}$m along the parallel direction among all the other samples. Moreover, the compacts were made under a very low pressure of 8 MPa compared to the high pressures used in the reported work where high magnetostriction is observed [31]. For comparison of the results, sintered cobalt ferrite sample SG8 which gave higher magnetostriction at lower magnetic fields when compared to SG1 and SG2, derived from G8 with intermediate particle size of 40 nm, as well as the sample obtained by ceramic method (SG0) also are studied after magnetic field annealing. Magnetic field annealing of the sintered pellets was carried out at 300 °C in a magnetic field of 400 kA/m for 30 min. The sample was cooled back to room temperature in the presence of the same field after annealing. The annealing field was applied perpendicular to the
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The cylindrical axis of the sintered pellet which is perpendicular to the measurement direction as well as the direction of the measuring field in the case of $\lambda_{\text{par}}$.

Figures 3.26 shows the magnetostriction curves of the sintered samples SG1, SG2, SG8 and SG0, measured at room temperature, as a function of magnetic field applied along the parallel ($\lambda_{\text{par}}$) and perpendicular ($\lambda_{\text{per}}$) directions, before and after magnetic field annealing. The maximum values of magnetostriction and strain derivative of the different sintered samples, before and after field annealing, are compared in Table 3.9. The relative change in the maximum value of magnetostriction after and before field annealing, $\Delta \lambda_{\text{par}} = (\lambda_H - \lambda_0)/\lambda_0$, measured along the parallel direction, where $\lambda_0$ and $\lambda_H$ are the maximum value of magnetostriction before and after field annealing, respectively, is compared in Table 3.9. It is observed that there is not much change in the maximum value of magnetostriction after field annealing for sample SG1, whereas 10%, 39% and 83% increases are observed for the samples SG2, SG8 and SG0, respectively. Even though SG8 showed relatively lower maximum value of magnetostriction and strain derivative before field annealing compared to SG2, comparable values for maximum value of magnetostriction and strain derivative are obtained after magnetic field annealing for both samples.

Table 3.9: Magnetostrictive properties of different samples, before and after magnetic field annealing.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Before magnetic annealing</th>
<th>After magnetic annealing</th>
<th>$\Delta \lambda_{\text{par}}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\lambda_{\text{par}}$ (ppm)</td>
<td>$d\lambda_{\text{par}}/dH$ (10^{-9}A^{-1}m)</td>
<td>$\lambda_{\text{par}}$ (ppm)</td>
</tr>
<tr>
<td>SG1</td>
<td>-307</td>
<td>-1.14</td>
<td>-304</td>
</tr>
<tr>
<td>SG2</td>
<td>-315</td>
<td>-1.97</td>
<td>-345</td>
</tr>
<tr>
<td>SG8</td>
<td>-254</td>
<td>-1.35</td>
<td>-353</td>
</tr>
<tr>
<td>SG0</td>
<td>-151</td>
<td>-0.56</td>
<td>-276</td>
</tr>
</tbody>
</table>
Another important observation is that, after annealing the sample in a magnetic field, higher magnetostriction is observed at lower magnetic fields for all samples and the field at which maximum magnetostriction is observed is reduced to 300 kA/m. This is clearly evident from the strain derivative curves of all samples compared in Figure 3.27, before and after magnetic field annealing. There is not much change in the strain derivative of SG2 after magnetic annealing, except that the maximum value is shifted to
lower fields. On the other hand, about 50% increase in the strain derivative at low fields is observed after magnetic field annealing for samples SG1 and SG8 whereas 150% increase is observed for the bulk sample (SG0), as shown in the Figure 3.27. These observations show that higher magnetostriction can be achieved without magnetic field annealing if the sintered compacts are made from smaller particles obtained by the autocombustion method of synthesis and magnetic field annealing is very effective in enhancing the magnetostriction and strain derivative for samples sintered from larger particles.

Figure 3.27: Field derivative of the magnetostriction for SG0, SG1, SG2, and SG8, parallel to the applied magnetic field. The open and closed symbols represent measurements before and after annealing in a field of 400 kA/m, respectively.
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Figure 3.28: Anisotropic magnetostriction of SG0, SG1, SG2 and SG8 along the parallel direction. The open and closed symbols represent the measurements before and after annealing in a field of 400 kA/m, respectively.

The maximum value of magnetostriction for the bulk sample SG0 is obtained as 151 ppm, less than the reported values, probably due to the difference in the processing parameters compared to the reports in the literature [23]. As shown in Figure 3.26 and Table 3.9, after annealing the sample in a magnetic field, the maximum value of magnetostriction of SG0 is increased to 275 ppm. Similarly, the strain derivative is increased from $0.56 \times 10^9$ A$^{-1}$m to $1.38 \times 10^9$ A$^{-1}$m, after magnetic annealing.

It is interesting to note that the magnetostriction coefficients $\lambda_{\text{par}}$ and $\lambda_{\text{per}}$ have opposite signs for the sintered samples derived from the larger particles (SG8 and SG0)
before magnetic annealing, whereas both values have the same sign after magnetic annealing. In the case of SG2, both $\lambda_{\text{par}}$ and $\lambda_{\text{per}}$ are negative before and after magnetic field annealing. However, $\lambda_{\text{par}}$ increases and the transverse value decreases (less negative) after the field annealing. Similarly, the sign of $\lambda_{\text{per}}$ is reversed for the sample derived from the smaller particles (SG1 and SG2) and after magnetic annealing, $\lambda_{\text{par}}$ and $\lambda_{\text{per}}$ have opposite signs. Similar features have been reported previously [31], indicating changes in the different single crystal contributions to the polycrystalline behavior after magnetic field annealing and that isotropic case is not valid.

It is found that the magnitude of anisotropic magnetostriction, $\lambda_a = \lambda_{\text{par}} - \lambda_{\text{per}}$, is comparable before and after magnetic field annealing, for SG0, SG1, SG2 and SG8, as shown in Figure 3.28. Comparable values for the anisotropic magnetostriction are obtained for the samples derived from the nanocrystalline particles derived from the autocombustion method. This shows that higher magnetostriction characteristics can be obtained for sintered compacts derived from nanocrystalline powders synthesized by the autocombustion method using glycine as the fuel.

### 3.5 Conclusions

The magnetic and magnetostrictive properties of sintered cobalt ferrite derived from nanocrystalline powders synthesized by the autocombustion method using glycine have been investigated. The results show that the particle size of the as-synthesized powders is one of the factors deciding the magnetostriction parameters of the compacts made from powders synthesized by this method. High values of the magnetostriction coefficient and strain derivative are obtained for compacts sintered from particles of very small sizes obtained from the autocombustion method. However, comparison of the effect of the initial particle size derived from different methods of synthesis indicated that particle size alone is not the deciding factor for the high values of magnetostriction. For the powders derived from the autocombustion method, there are no correlations between the magnetic and magnetostrictive parameters of the sintered products. However, some correlation between the magnetostriction and microstructure of the sintered products is observed.
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The present results indicate that sintering nanocrystalline powders of very small particle sizes obtained by the autocombustion method is effective for obtaining high values of magnetostriction for sintered cobalt ferrite. Pre-sintering of the particles during synthesis, and pores in the grain and orientation of the grains in the sintered products are the likely parameters controlling magnetostriction.

Magnetostriction values of 315 ppm without magnetic annealing and 345 ppm after magnetic annealing, at room temperature, are the highest values reported so far for sintered polycrystalline cobalt ferrite obtained under simple processing conditions. The present results show that it is possible to get very high magnetostriction and coupling coefficient for sintered cobalt ferrite derived from nanocrystalline powders of very small particle size synthesized by the autocombustion method using glycine.
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