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Structural Magnetic properties of polycrystalline Dy0.4 Sm0.6 FeO3 (DSFO) compounds synthesized By Sol-gel Dy0.4 Sm0.6 FeO3 Sintered at different temperatures

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Abstract:

This paper explores the structural and magnetic properties of polycrystalline DSFO compounds synthesized via sol-gel route and sintered at 800°C, 1000°C and 1200°C. X-ray diffraction (XRD) analysis with Rietveld refinement confirms that, the samples sintered at 800°C and 1000°Cex hibit a single phase with out any detectable impurity,where as the samples intered at 1200°C contains a small amount of $Dy_3Fe_5O_{12}$cubic garnet impurity. The M-H hysteresis analysis shows an increase in magnetisation (i,e M_m& $M_r$and coercive filed H_C) with increasing sintering temperature, the enhancement is more significant from 800^{o}C to 1000^{o}C compared to 1200^{o}C. Mössbauer spectra reveals a slight increase in the hyperfine field with rising sintering temperature indicates improved magnetic ordering. The observed smaller isomer shift suggests the presence of Fe^{3+}state. Overall,the samples intered at1000°Cexhibits relatively superior structural, and magnetic properties than the other samples.

Keywords: Orthoferrite, Sol-gel, XRD, magnetisation, Mossbauer spectra

Introduction:

Multiferroic Magneto-Electric coupling has drawn considerable attention from the researchers due to intriguing underlying physics involved and their promising technological applications.Phenomen asuch as Electricfield induced magnetizations witching and vice-versa,temperature driven magnetization reversal, and the interplay between spin and lattice have create interest in the scientific community, high lighting the potential of these materials for advanced functional devices [1,2]. Magneto-Electric coupling enables to manipulate magnetisation through application of electricfield and electric polarization can be altered with externalmagneticfield. This unique bidirectional control makes such materials highly efficient candidates for use in sensors,spintronic systems and memory storage devices with ultra-lowpower consumption[3- 6].

Nevertheless, identifying material that exhibit pronounced Magneto-Electric coupling remains challenging, as the ferromagnetism typically relies on the presence of electrons in d-orbital,whereasferroelectricitytendstoprefertheirpresence.Amongthematerialsexhibiting Magneto-Electric coupling, the $RFeO_3$family (where, R= Rare earth element) has garnered substantial attention from the researchers for its remarkable Magnetic, magneto-optic and multiferroic characteristics.

$RFeO_3$crystallizes in the Orthorhombic perovskite structure with Pbnm space group, where R^{3+} ion occupy eight corners of the unit cell and Fe^{3+} ions octa hedrally coordinated with six oxygen ions forming FeO_6octahedra [7,8]. The magneto-electric behaviour of $RFeO_3$family majorly governed by the super-exchange interactions among Fe^{3+}-Fe^{3+}, Fe^{3+}-$R^{3+}$and R^{3+}-R^{3+}ions.DuetothethreetypesofmagneticinteractionsandthearrangementofFe^{3+}spins,$RFeO_3$compounds exhibit three distinct magnetic configurations, each of which characterised with a specific crystallographic easy axis of magnetisation namely Γ_1, $\Gamma_2$and Γ_4. All these configurations are temperature dependant,$\Gamma_4$typically found at high temperature while any of the three may occur at low temperature [9,10]. In these compounds there exist a characteristic temperature known as Spin-Reorientation Transition temperature (SRT) at which easy axis of magnetisation shifts from one crystallographic axis to another, indicating a change in the magnetic configuration [11-13].

Among $RFeO_3$compounds $SmFeO_3$has demonstrated outstanding device-relevant properties includingrapidmagneticswitchingandhightemperaturerotationofeasyaxisofmagnetisation around 480K. Notably, this represents the highest SRT temperature reported within the entire $RFeO_3$family. Similarly, $DyFeO_3$exhibit SRT around 35K which is the lowest SRT reported among $RFeO_3$ family. In the context of SRT, doping with Dy at Sm site in $SmFeO_3$ is expected to induce a variation in spin-reorientation transition temperature, spanning a wide range from approximately35Kto480K.Onesuch$Dy_{0.4}Sm_{0.6}FeO_3$has been reported to exhibit SRT around 240-270K.this compound has been selected for further exploration in this study [10-12].

Extensive investigation has been devoted to modify the properties of $RFeO_3$compounds through doping at both the R-site and Fe-site, as the structural and multiferroic properties of ABO_3 perovskites can be effectively tailored by substitution at either theA-site or B-site [13- 16]. It has been reported that the synthesis methods significantly influence the properties of materials.Varioustechniqueshavebeenemployedforthepreparationof$RFeO_3$,including Sol- gel, hydrothermal and solid-state reaction method etc [16-20].

The Sol-gel synthesis route is widely recognised as a versatile and effective synthesis technique, offering advantages such as improved chemical homogeneity, precise stoichiometric control and high purity compared to other approaches [1]. In this method critical synthesis parameters such as chelating agent, calcinations and sintering temperatures significantly impact the structural and functional properties of the compound. Recent studies have emphasized the critical role of selecting suitable chelating agent and optimizing sintering temperature in synthesizing $YFeO_3$ nanomaterials [21,22]. It has been reported that 800^{o}C sintering temperature yields better results in case of $SmFeO_3$[23], while 1000^{o}C is more effective for $ErFeO_3$[24], similar findings have been made for $DyFeO_3$ and $LaFeO_3$[25,26].

The present study focuses on exploring how sintering temperature effects the structural and magnetic properties of $Dy_{0.4}Sm_{0.6}FeO_3$, hereafter referred to as DSFO.

Experimental Method

Polycrystalline $Dy_{0.4}Sm_{0.6}FeO_3$(DSFO) samples were synthesized via the Sol-gel route and subsequently sintered at 800°C, 1000°C and 1200°C. Stoichiometric amounts of $Dy(NO_3)_3{\cdot}5H_2O$,$Sm(NO_3)_3.6H_2O$and$Fe(NO_3)_3{\cdot}9H_2O$ were dissolved in deionised water with stirring to achieve a homogenous solution. Specific amount of citric acid was added as a chelating agent, and the pH of the solution was adjusted to 6-7 by adding adequate amount of ammonium hydroxide. The solution was stirred consistently and heated at 80°C until a dense and viscous gel formed, after which ethylene glycol is added. The gel was then subjected to continuous heating at 150°C yielding a black powder through auto-combustion. This powder was calcinated at 600°C for 4 hours, further ground and pressed into 10mm diameter circular pellets.Thesepelletsweresinteredinairatmosphereat800°C,1000°C and 1200°C for 4hours to study the effects of sintering temperature on the material properties. X-ray diffraction (XRD) measurements were carried out using a Brucker D8 Advanced diffractometer with Cu-Kαradiation. Room temperature magnetization measurements were performed using a commercial Vibrating Sample Magnetometer (VSM). Room temperature Mössbauer spectroscopy was conducted in transmission geometry using ^{57}Co(Rh) radioactive source with standard PC based Mössbauer spectrometer.

Results and Discussion:

Structuralcharacterisationofpreparedsampleswasperformedwithusing Rietveldrefinement of XRD with FullProf software, assuming an orthorhombic crystal structure with Pbnmspace group. X-ray diffraction analysis is a versatile tool for extracting the detailed structural information and also the structural phase purity of the prepared samples intertwined the magnetic phase transitions, and the crystallinity can significantly influence the saturation magnetization and the coercivity of the samples.

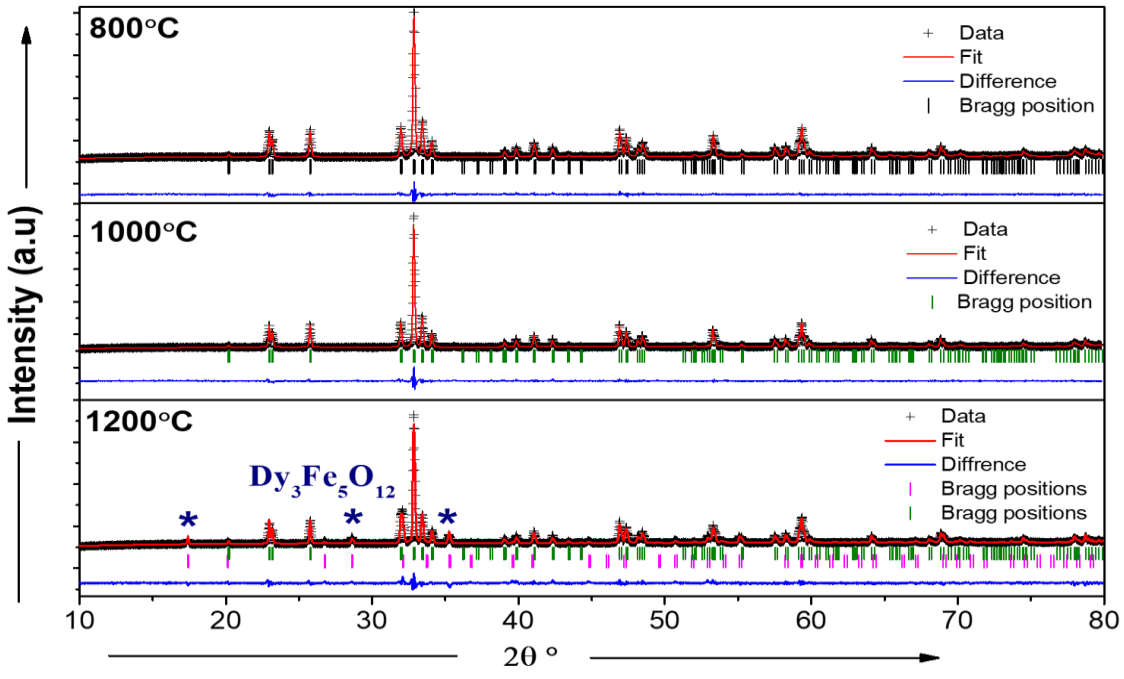

Fig.1. Rietveld refined X-ray diffraction pattern of DSFO orthoferrite samples sintered at 800°C, 1000°C and 1200°C

XRD patterns of the DSFO samples sintered at800°C,1000°C and1200°CpresentedinFig.1. All the samples exhibit orthorhombic perovskite structure in consistent with literature [1]. However, a minor secondary phase $Dy_3Fe_5O_{12}$garnet impurity was observed in samples intered at 1200°C. This garnet phase formation attributed to partial transformation of orthorhombic perovskite into a cubic garnet structure under high temperature conditions as reported earlier [1,11]. The variation in unit cell volume, with sintering temperature shown in Fig. 2a.Aslight linear increment observed in lattice parameters and unit cell volume with increasing sintering temperature due to thermal expansion with increased sintering temperature. However, a reduction in unit cell volume noticed for the samples interedat1200°C may be associated with the formation of garnet impurity phase. A similar trend was observed in the intensity and FWHM of Bragg peaks. The variation in intensity of most intense peak (112) along with its corresponding peak width (FWHM, □=2□2-2□1) as a function of sintering temperature is graphically illustrated in Fig.2b.The increase in the peak intensity accompanied by a decrease in FWHM from 800°C to 1000°C indicates reduced lattice strain and enhanced crystallinity. However, the subsequent decrease in intensity and broadening of FWHM at 1200°C suggests adeterioration in phase purity and crystallinity likely associated with formation of $Dy_3Fe_5O_{12}$secondary phase at elevated sintering temperature.

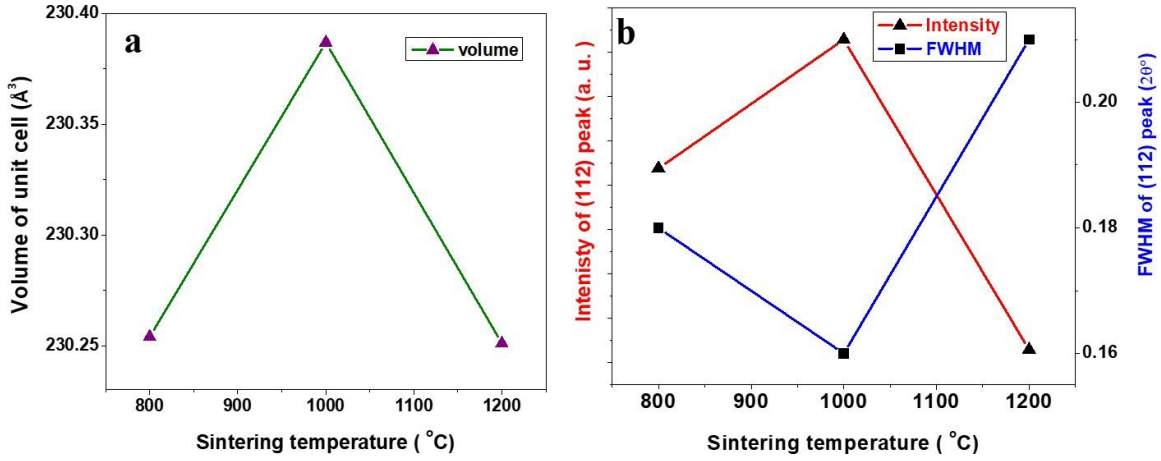

Fig. 2. a). The variation in unit cell volume, b). intensity of (112) most intense peak and corresponding peak FWHM, as a function of sintering temperature of DSFO orthoferrite samples sintered at 800°C, 1000°C and 1200°C Similar trend was noticed in the measurements of bond lengths and bond angles [1].As light increment in Fe-O bond lengths was observed from 800°C to 1000°C followed by minor reduction at1200°C.These results suggests that elevated thermal energy not ably influence the Fe-O coordination environment.Form the experimental observations, it is evident that, increasing the sintering temperature from 800°C to 1000°C enhances the crystallinity and reduces the lattice strain in DSFO.However, further increase to 1200°C induces phase transformation and emergence of impurityphases.Therefore,1000°Cisidentifiedasoptimalsinteringtemperatureforachievingsuperior structural quality in DSFO sample.The magnetic behaviour of $RFeO_3$compounds is primarily governed by the Fe^{3+} spin arrangement and underlying magnetic symmetry. The arrangement of FeO_6 octahedra in which Fe^{3+}ion is coordinated by sixO^{2-}ions and each oxygen ion is shared between two neigh bouring octahedra, facilitating Fe^{3+}-O^{2-}-Fe^{3+} super-exchange interactions. According to the Dzyaloshinskii-Moriya (DM) anti-symmetric exchange mechanism, the spins of adjacent Fe^{3+} ions are slightly can ted

rather than perfectly antiparallel, leading to acanted antiferromagnetic ordering with a week ferromagnetic component [10,16].The variation of magnetization with the applied magnetic field of DSFO samples sintered at different temperatures was investigated and these were illustrated in Fig. 3. The absence of saturation magnetisation in these M-H loops indicates that, all the samples exhibit antiferromagnetic behavior with weak ferromagnetic component [10,13].

The corresponding magnetisation parameters, including Remanent magnetisation (M_r), maximum magnetisation (M_m- magnetisation measured at an applied field of 20000 Oe) and coercive magnetic field (H_c) values were summarised in table 2.

Table 2. Room temperature of DSFO orthoferrite samples sintered at 800°C, 1000°C and 1200°C

Sintering temperature	M – H at 300 K		
	M_r (emu / g)	H_c (Oe)	M_m(emu/g)
800°C	0.31	3343	1.32
1000°C	0.57	3562	2.51
1200°C	0.62	3274	2.79

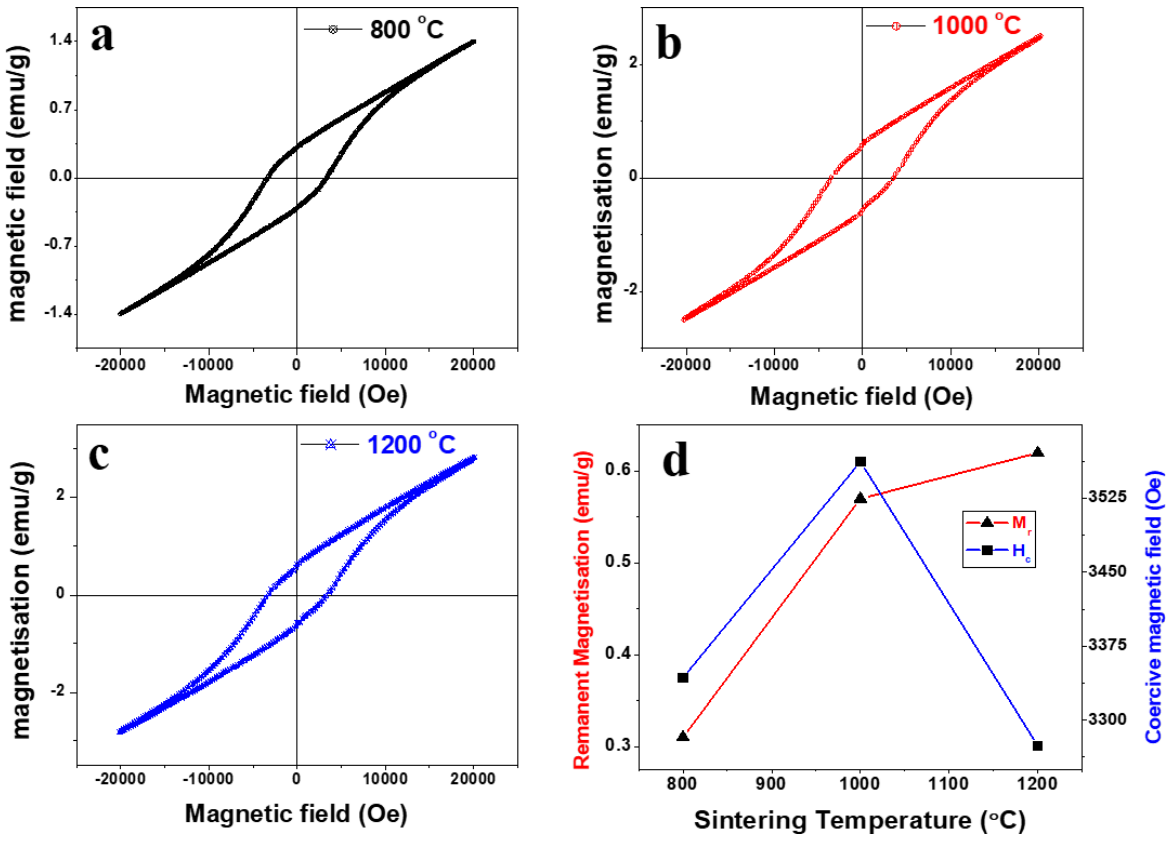

Fig. 3. Room temperature M-H hysteresis loops of DSFO orthoferrite samples sintered at 800°C Fig. (a),

1000°C Fig.(b) and 1200°C Fig. (C). The variation of $M_r$and $H_c$values as a function of sintering temperature represented in Fig. (d).It is noticed that,an increasing intering temperature results in noticeable enhancement in both the $M_r$and M_m, with the more significant changes observed from 800°C to 1000°C compared tochanges between 1000°Cto1200°C(seeFig.3(d)).The magnetization is strongly influenced by the localionic distribution with in the FeO_6 octahedra and their super-exchange interactions.

The observed increase in lattice parameters and Bragg peak intensities from the XRD analysis indicates the improved crystallinity and reduced octahedral distortion in FeO_6, correlates the enhanced magnetic properties. However, the observed non-linear trend in the variation of Mr and Mm from1000°Cto1200°Cisattributed to impurity phase. Further more, the variations in Fe-O bond lengths corroborates the changes observed in magnetisation.

A noticeable increase in coercive field was observed as the sintering temperature rise from 800°C to 1000°C (See Fig. 3(d)), Coercivity is primarily influenced by lattice strain (defects) and domain wall interactions. The enhanced do main wall interactions make it harder to reverse the magnetisation, there by increasing the coercivity of the sample. In present study, the observed narrowing of Bragg peak widths in the XRD patterns indicates a reduction in lattice strain and improved crystallinity, which in turn promotes stronger domain wall interactions- accounting for increased coercivefield from 800°Cto1000°C.However, subsequent reduction observed in coercive field for the sample sintered at 1200°C is attributed to impurity phases and decline in crystallinity, which likely diminish domain wall interactions.

The M-H analysis reveals that, increasing the sintering temperature from 800°C to 1000°C leads to a notable enhancement in magnetic properties of DSFO samples, however decline in these parameters is observed for 1200°C, suggesting the sample sintered at 1000°C exhibit higher values of M_r, $M_m$and $H_c$compared to other samples.

Mössbauer spectroscopy offers critical insights into the electronic environment of iron atoms in the sample, particularly about their oxidation states and coordination geometry. Variations in peak width and intensity of Mössbauer peaks are closely associated with the changes in the structural and magnetic ordering, which are influenced by external factors such as sintering temperature and elemental doping. Isomer shift in Mössbauer spectroscopy is related to the electron density at the nucleus and provides direct information about the oxidation state and bonding environment of the iron atoms. Hyperfinefiled reveals the details about them agnetic orderings within the sample.Additionally, broadening of Mössbauer peaks is signature of the lattice stain and phonon interactions at elevated temperatures [27]. The room temperature Mössbauer spectra of DSFO orthoferrite samples sintered at 800°C, 1000°C and 1200°C are presented in Fig. 4.

The spectra were analysed using the NORMOS-SITE software, assuming asinglesextet configuration consistent with the polycrystalline nature of the samples [15,27]. The intensity ratio of the magnetically ordered components is fixed at 3:2:1: 1:2:3. The extracted Mössbauer parameters are summarised in table 3.

The relatively low isomer shift values observed across all DSFO samples confirm the presence of Fe^{3+}state.Aslightincrease noticed in the hyperfine field with rising sintering temperature, with more

pronounced from 800°C to 1000°C than 1000°C to 1200°C, indicating the enhanced magnetic ordering, in agreement with the magnetisation parameters obtained from M-H hysteresis loops. A minor decrease in spectral line width from 800°C to 1000°C suggests reduce in lattice strain and distortion within the FeO_6 octahedra. Conversely, the peak broadening at 1200°C is attributed to reduced crystallinity and the presence of impurity phase, the same is corroborate with the XRD data. The Mössbauer study demonstrates that the sample sintered at 1000°C exhibits superior magnetic characteristics and structural quality compared to other samples.

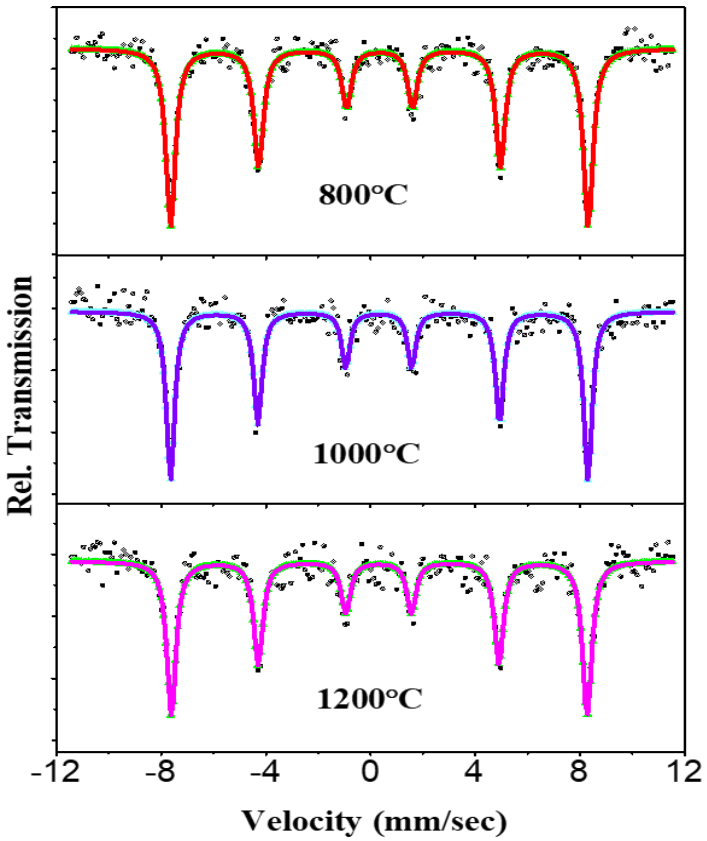

Fig.4.Room temperature Mössbauer spectrum of DSFO ortho ferrite samples sintered at 800°C, 1000°C and 1200°C.

Table 3. Room temperature Mössbauer parameters of DSFO orthoferrite samples sintered at 800°C, 1000°C and 1200°C

Parameters	800°C	1000°C	1200°C
Isomer shift (mm/s)	0.34 (0.002)	0.32 (0.003)	0.32 (0.001)
Quadrupole splitting (mm/s)	0.01 (0.010)	0.02 (0.010)	0.02 (0.030)
Hyperfine field (Tesla)	49.20 (0.020)	49.40 (0.030)	49.42 (0.020)
Line width	0.44 (0.030)	0.38 (0.020)	0.42 (0.010)

Conclusions:

The structural and magnetic properties of polycrystalline DSFO synthesized via the sol-gel method and sintered at 800°C, 1000°C and 1200°C have been systematically investigated. X- ray diffraction (XRD) analysis confirms that samples sintered at 800°C and 1000°C exhibit a single-phase orthorhombic, where as thesamplesinteredat1200°Ccontains a small amount of $Dy_3Fe_5O_{12}$cubic impurity. As light linear increase in lattice parameters, unit cell volume and Fe-Obondlengthswiththeincreasingsinteringtemperatureobserved,where as slight reduction was noted at 1200°C due to impurity. The increase in the intensity of (112) peak along with decrease in its FWHM with rising sintering temperature, suggests improved structural quality and reduced lattice strain leading to lower distortion in the FeO_6 octahedra. Crystalline size calculationfurtherrevealsimprovedcrystallinityincreasesfrom800°Cto1000°C,however,a slight decline is observed 1200°Clikely due to impurity phase. The magnetization data(M-H) demonstrates a clear increase in magnetisation with rising sintering temperature. The enhancement in magnetisation (Mr and Mm) is more significant from 800^{o}C to 1000^{o}C comparedto1200^{o}C.However,as light reduction in coercive field observed in1200^{o}Csample probably due to presence of impurity phases and decrease in crystallinity, which may weaken the domain wall interactions and thus reduce coercivity. Mössbauer spectra shows a gradual increase in the hyperfine field with rising sintering temperature, indicating enhanced magnetic ordering, The observed lower isomer shift across all samples suggest the presence of Fe^{3+}state.

As light narrowing of spectral line width from800^{o}Cto1000^{o}C indicates reduced lattice strain and lower octahedral distortion. However, the peak broadening at 1200^{o}C is attributed to reduced crystallinity and the presence of impurity phase. Over all observations collectively demonstrates that the sintering DSFO sample at1000°Cyields optimal structural, and magnetic properties compared to the samples sintered at 800^{o}C and 1200^{o}C.

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Conflict of Interest:

The author has no conflict of interest regarding the research.

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