Research Article

Dielectric and optical properties of Ni-doped $LaFeO_3$ nanoparticles

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Abstract
Un-doped and Ni-doped lanthanum ferrite nanoparticles were synthesized by solid-state method. Ni concentration-dependent structural, dielectric and optical properties of synthesized nanoparticles were investigated. X-ray diffraction patterns confirmed predominant single-phase orthorhombic crystal structure with space group Pbnm in all samples. Average crystallite size was found to vary from 19 to 21 nm with Ni concentration. The field emission scanning electron microscopy revealed nanocrystalline structure with homogenous distribution of particles. UV–Vis–NIR diffuse spectra captured at room temperature indicate that $La_{1-x}Ni_xFeO_3$ is an indirect band gap material. The band gap varies from 1.70 to 1.85 eV with changing Ni concentration. Frequency-dependent dielectric constant, dielectric loss and ac conductivity were studied at room temperature. The dielectric constant was found to increase with increasing Ni content at high frequency. The loss factor resulted from domain wall resonance exhibited an identical dispersion behavior of dielectric constant. At high frequencies, the dielectric losses of orthoferrites were found to be low. This is attributed to the restricted motion of domain wall pointing its plausible practical applications in magnetically tunable filters and oscillators.

Keywords Solid-state method · Nanocrystalline $LaFeO_3$ · Orthoferrites · Dielectric properties · Optical properties · Band gap

1 Introduction

Ferrites are a class of ceramic materials consisting of mixtures of various metal oxides. It finds many potential applications in the modern world [1–4]. Nowadays, a significant attention has been given to lanthanum ferrites ($LaFeO_3$) due to its exchange bias, opto-magnetic and multiferroic properties. These ferrites are candidates for memory devices, low power-consuming spintronic devices, oxygen permeation membranes, opto-magnetic sensors and electrode material in solid oxide fuel cells (SOFCs) [5, 6]. Manikandan et al. [7–10] also reported pure and doped ferrite nanomaterials for different applications such as photocatalyst for the degradation of dyes, catalytic oxidation of alcohols, antibacterial activity and sensor materials.

The rare-earth-based orthoferrites, $RFeO_3$ (R denotes rare-earth element), are orthorhombically distorted perovskites with four formula units per unit cell. In this structure, Fe^{3+} occupies the orthorhombic site and R^{3+} situates on the twofold axis [11–13, 13–19].

The orthorhombic structure of $LaFeO_3$ belongs to Pbnm space group displaying p-type semiconductor. It is an attractive electroceramics due to mixed ionic/electronic conductivity and chemical stability at high temperature [20]. It exhibits antiferromagnetic phenomena with Neel temperature (T_N) at 750 K [21]. Neutron diffraction studies on model $LaFeO_3$ proved that each Fe^{3+} ion is confined to six(6) Fe^{3+} ions with spins pair antiparallel [22]. Recently, ferromagnetism has also been reported for LFO nanoparticles. The effects of length scale, shape and anisotropy prevailed

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at surface which shows the magnetization increased with decreasing particle size [23–25].

Significant research efforts are being made to search for new class of oxide materials with smaller band gaps to enhance visible light absorption. The doping at La and/or Fe site of $LaFeO_3$ conducted by researchers is shown to display significant effects on many properties of $LaFeO_3$. Some investigations have been reported for LFO bulk ceramics [26–36]. Improved dielectric and optical properties of doped LFO are important for its practical applications. In this context, for the first time we thoroughly investigated the effect of Ni doping on the structural, dielectric and optical properties of $LaFeO_3$ nanoparticles. $La_{1-x}Ni_xFeO_3$ (x: 0.0, 0.10 and 0.20) nanoparticles were synthesized with the variation in Ni doping level by solid-state reaction technique.

2 Experimental techniques

$La_{1-x}Ni_xFeO_3$ (x = 0.0, 0.10 and 0.20) nanoparticles were synthesized by a solid-state reaction technique. The analytical-grade base materials, namely La_2O_3, NiO and γ-Fe_2O_3, were taken in stoichiometric proportion. Then, it was milled using in zirconia balls for 24 h to obtain homogeneous mixture. Next, calcination was carried out at 700 °C for 2 h followed by remilling in alcoholic media for 10 h. Next, the suspensions were dried at 120 °C. Then, obtained powders were pressed into disk-shaped samples of 15 mm in diameter under a compressive force of 40 kN. The pressed pellets were sintered at 750 °C for 2 h embedded in corresponding powders to avoid volatilization. The phase structure of the sintered powders was determined by X-ray diffractometry (XRD; PANalytical Empyrean). The microstructure of the polished samples was investigated by scanning electron microscopy (JSM-7600F, JEOL). For dielectric measurement, the sintered samples were properly flattened to obtain parallel and smooth surfaces. Then, a silver paste (SPI Flash-Dry Silver Paint) was applied on both polished surfaces and dried at 250 °C for 4 h to provide connection of electrodes. Dielectric property measurement was conducted by a precision impedance analyzer (6500B, Wayne Kerr Electronics, UK) at ambient condition at frequency ranging from 10 Hz to 100 MHz. Optical properties were measured by UV–VIS–NIR spectrometry (Lambda-1050, PerkinElmer, USA).

3 Results and discussion

3.1 X-ray diffraction

X-ray diffraction (XRD) patterns were captured at Bragg angle (2θ) ranging from 20° to 80°. Figure 1 shows room temperature XRD patterns of $La_{1-x}Ni_xFeO_3$ (x = 0.0, 0.10 and 0.20) samples. The peak intensity was shown to modulate with Ni content compared to pure $LaFeO_3$. However, all samples were shown to confirm single-phase orthorhombic crystal structure with Pbnm space group.

Evidently, a small trace of secondary phase γ-Fe_2O_3 was found to present in XRD patterns of both pure and Ni-doped samples. However, the apparently unavoidable formation of secondary phases during the synthesis of $LaFeO_3$-based materials has been reported in several articles [37, 38].

Obtained patterns were also further analyzed by the Rietveld refinement using Xpert HighScore plus with ICDD database. The crystallite size and strain are presented in Table 1. Average crystallite size was found to vary from 19 to 21 nm with the variation in Ni dopant level.

The ionic radius (0.69 A) of Ni^{2+} is much less than the ionic radius (1.03 A) of La^{3+} [39]. Thus, a significant strain is generated in parent LFO, which is found to be increased with Ni^{2+} content concomitant with the Rietveld refinement strain data as shown in Table 1. Crystallite size was found to decrease with increasing Ni^{2+} doping concentration. Similar result was reported for Ni-doped $BiFeO_3$ [40]. In contrast, with increasing Ni^{2+} doping concentration crystallite size was shown to increase in $ZnFe_2O_4$ [10, 41–44]. In this context, we postulate that the initial crystal structure of parent material might play an important role. However, it is established through density functional theory (DFT) calculations that Mn^{2+}, Co^{2+}, Ni^{2+} and Cu^{2+} are both the A- and B-site dopants [45]. Ca^{2+}, Sr^{2+}, Ba^{2+} and Fe^{2+} are found to be A-site and

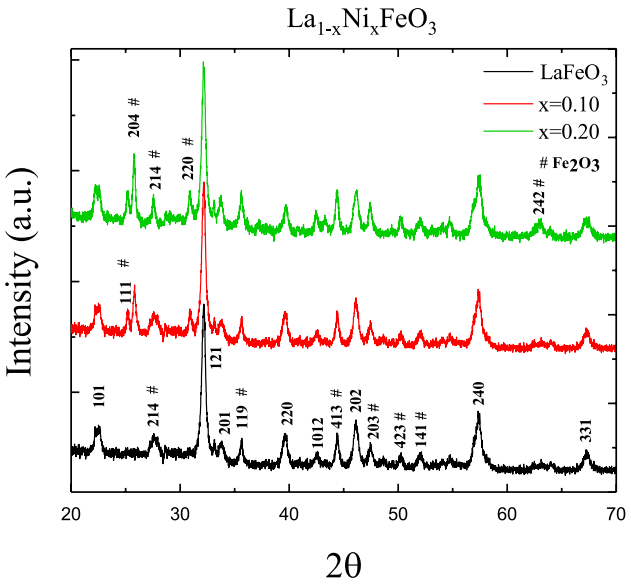

Fig. 1 X-ray diffraction (XRD) patterns of $La_{1-x}Ni_xFeO_3$ (x = 0, 0.1, 0.2) samples

Table 1 Crystal parameters, crystallite size and lattice strain of LFO and Ni-doped LFO

Samples ($La_{1-x}Ni_xFeO_3$)	Crystal parameters (Å)	Volume (Å^3)	D (nm)	ϵ (%)	GOF
$x = 0.0$	$a = 5.58803$, $b = 7.84259$, $c = 5.54317$	242.9274	20.7	0.607	1.90
$x = 0.1$	$a = 5.6177$, $b = 7.85131$, $c = 5.54495$	244.5712	19.6	0.641	2.30
$x = 0.2$	$a = 5.61863$, $b = 7.85281$, $c = 5.55731$	245.2309	18.9	0.663	2.63

D, crystallite size, ϵ, lattice strain; GOF, goodness of fitting

Mg^{2+} is considered to be B-site dopants. We doped Ni^{2+} to substitute A-site La^{3+} ion, but the increase in cell parameters and the volume of unit cell obtained from Rietveld analysis contradicts with our initial prediction. Thus, we postulate that that Ni^{2+} substitutes B-site Fe^{3+} ion of radius 0.55 A which demonstrates a good agreement with observed cell expansion and may rule out the possibility of the A-site substitution by Ni^{2+} dopant.

3.2 Scanning electron microscopy

Shown in Fig. 2 are the scanning electron micrographs of the polished samples of $La_{1-x}Ni_xFeO_3$ ($x = 0, 0.1, 0.2$) nanoparticles which were calcined at 700 °C for 2 h. Evidently, all micrographs exhibit irregular particle shapes with a wide range of particle size distribution. The mean particle size of $La_{1-x}Ni_xFeO_3$ ($x = 0, 0.1, 0.2$) ceramics decreases with increasing Ni content. That means Ni doping was found to suppress grain growth. Inhibition of grain growth was also

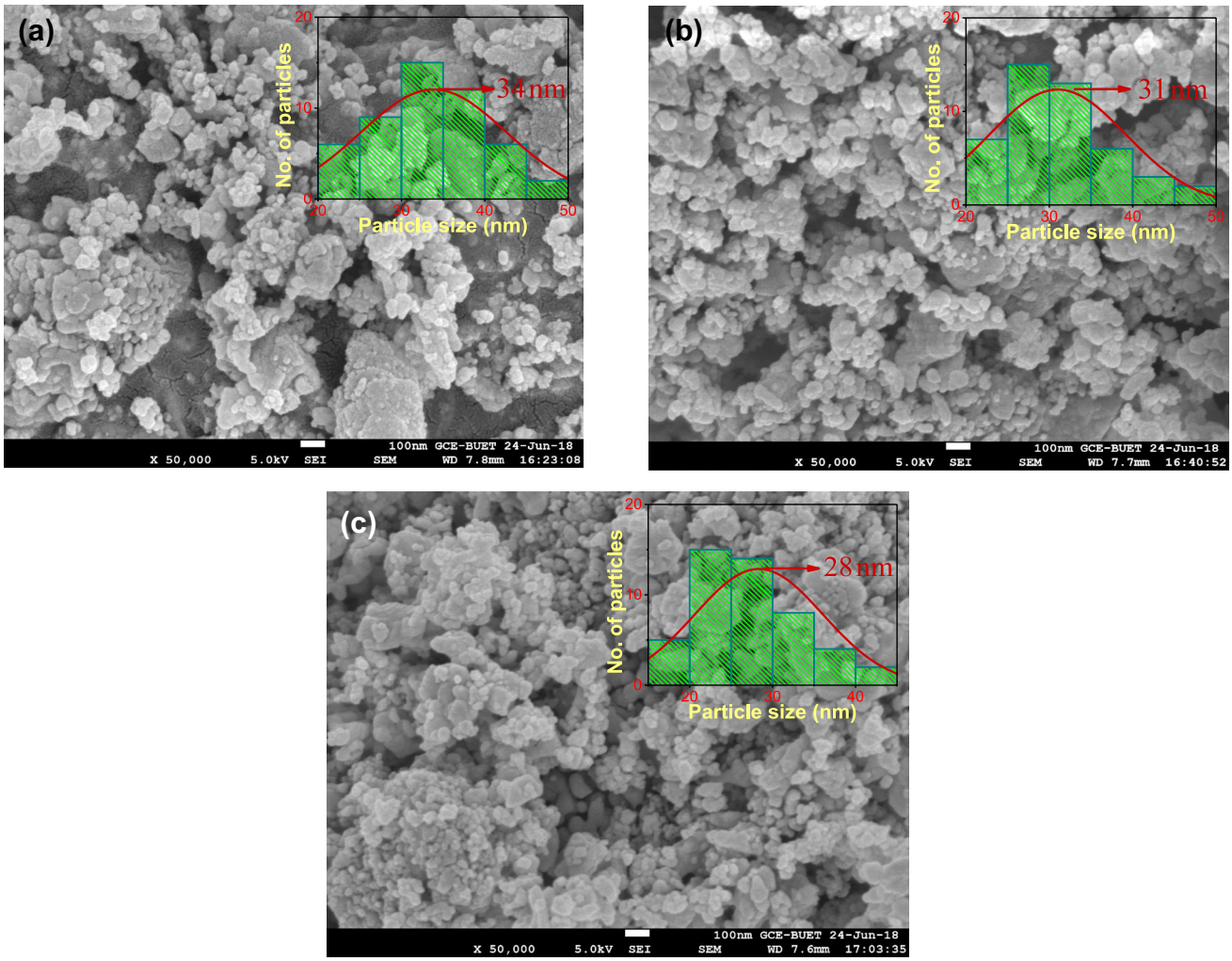

Fig. 2 FESEM micrographs of $La_{1-x}Ni_xFeO_3$: **a** $x = 0.0$, **b** $x = 0.10$ and **c** $x = 0.20$

reported for Ni-doped $BiFeO_3$ [40]. The decreasing grain size of Ni-doped LFO can be resulted from the suppression of O_2 vacancy concentration (the slower the O_2 ion movement, the smaller the grain growth rate) and due to the difference in ionic radius of La^{3+} and Ni^{3+} [46–48]. In addition, oxygen vacancies are presumably generated due to Ni doping in LFO resulting structural distortion with concurrent reduction in crystallinity hence reducing the grain size.

However, during calcining the compositions at 700 °C high surface energy leads to neck formation by diffusion in solid state and nanoparticles agglomerate by the process of evaporation–condensation. In addition, both the particle size and crystal size of Ni-doped LFO nanoparticles reduce with increasing Ni content demonstrating the retardation of grain growth.

3.3 Dielectric properties

The room temperature measurement of dielectric behavior is presented in Fig. 3. Figure 3a shows how the dielectric constant varies with frequency (10^2–10^7 Hz). The increase in dielectric constant at low frequency is remarkable. The frequency regions may be subdivided into three regimes: (I) the first region (up to 103 Hz) in which the dielectric constant sharply decreases with frequency, (II) the region in which the dielectric constant decreases comparatively

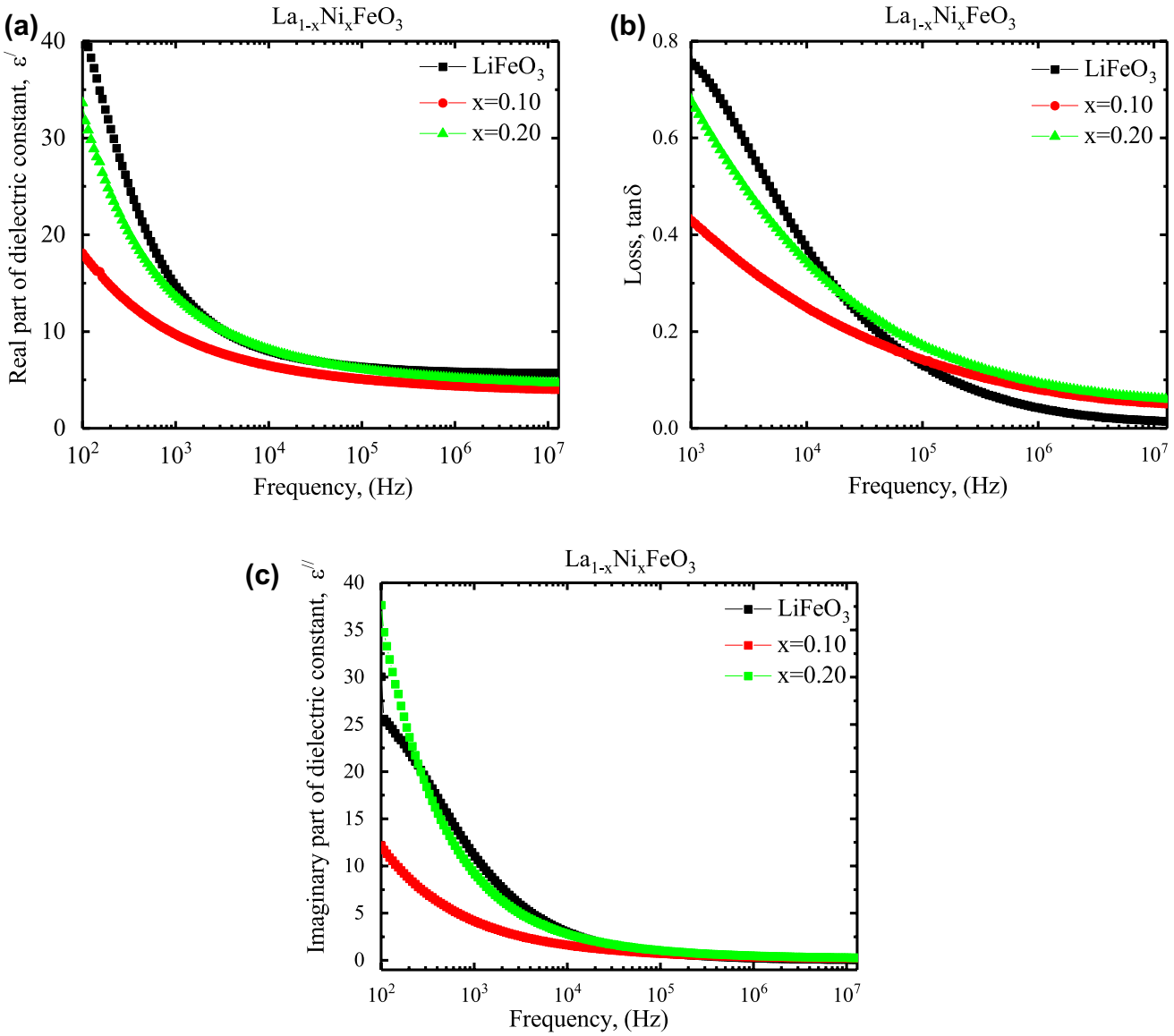

Fig. 3 Frequency dependency of $La_{1-x}Ni_xFeO_3$ (x = 0.1, 0.2) recorded at RT in the wide frequency range from 100 Hz to 120 MHz: **a** real part of the dielectric constant (ϵ'), **b** imaginary part of the dielectric constant (ϵ'') and **c** tangent losses (tan δ)

slowly with frequency and (III) the frequency region in which the dielectric constant remains almost constant.

Similar type of dielectric behavior has also been observed in our investigated other dielectric materials [49, 50] and may be exemplified by polarization mechanism. At low frequencies, the dielectric constant is attributed to all types of polarization mechanisms and the contribution from various types of polarizations start to decrease with the increase in frequency. The dipolar including interfacial polarizations play role to dielectric constant at low frequencies. Basically, Fe^{3+}/Fe^{2+} ions bring about dipolarity in ferrites. The electron exchange between these ions results the dipolar polarization, and the dipole alignment takes place by AC field. After a certain frequency of AC field, the dipolar polarization does not contribute to the dielectric constant. This is because the electron exchange between Fe^{2+} and Fe^{3+} cannot follow alternating field. The dielectric constant is attributed only to the electronic polarization at very high-frequency region [51]. The electronic polarization is independent on frequency, resulting in a constant value of dielectric constant at high frequency. The effect of trace secondary phase may also be noted. The dielectric dispersion of γ-Fe_2O_3 was absent after annealing above 550 °C [52]. Thus, the observed dielectric behavior of LFO was not expected to alter remarkably.

The lagging in the polarization with respect to the AC field can be represented by the dielectric loss (tanδ). Figure 3b shows the frequency dependence of tanδ for $La_{1-x}Ni_xFeO_3$ system where no loss peak is observed. The nonexistence of any peak in tanδ defines the system as charge carrier-dominated system where the dominant contribution to polarization stems from electronic or ionic charges [53]. The similar type of behavior was also observed for $EuFeO_3$ [51].

3.4 AC conductivity

With the purpose of further studying the transport mechanism, the AC conductivity at different Ni contents is investigated. Figure 4 presents the frequency-dependent conductivity for different doping concentrations at room temperature. A plateau regime was observed at lower frequency while a dispersion region was found at higher frequency.

The Maxwell–Wagner two layers formalism can be applied to the observed phenomenon [54]. The plateau region characterizes the dc conductivity. The frequency-independent conductivity may be attributed to the random diffusion by activated hopping of ionic charge carriers. Therefore, the conductivity can be described by Jonscher's universal power law [55]

$$\sigma_{ac}(\omega) = \sigma_{dc} + D\omega^{s} \quad (1)$$

where the first part (σ_{dc}) of right-hand side is defined as the dc conductivity and the left part of the above equation is the frequency-dependent conductivity [56]. The frequency exponent 's' has the value in the range of $0 < s < 1$ while the of s is 1 suggesting an ideal Debye-type behavior. The value of s can be calculated from the $\log\sigma_{ac}$ versus $\log\omega$, and the calculated values are 0.55, 0.71, 0.65 for $x = 0.0$, 0.10 and 0.20, respectively.

A hopping mechanism for the direct current electrical conductivity is associated with a small value of activation energy. The small polaron can migrate by thermal activation from Fe^{2+} to Fe^{3+} sites contributing to the change in

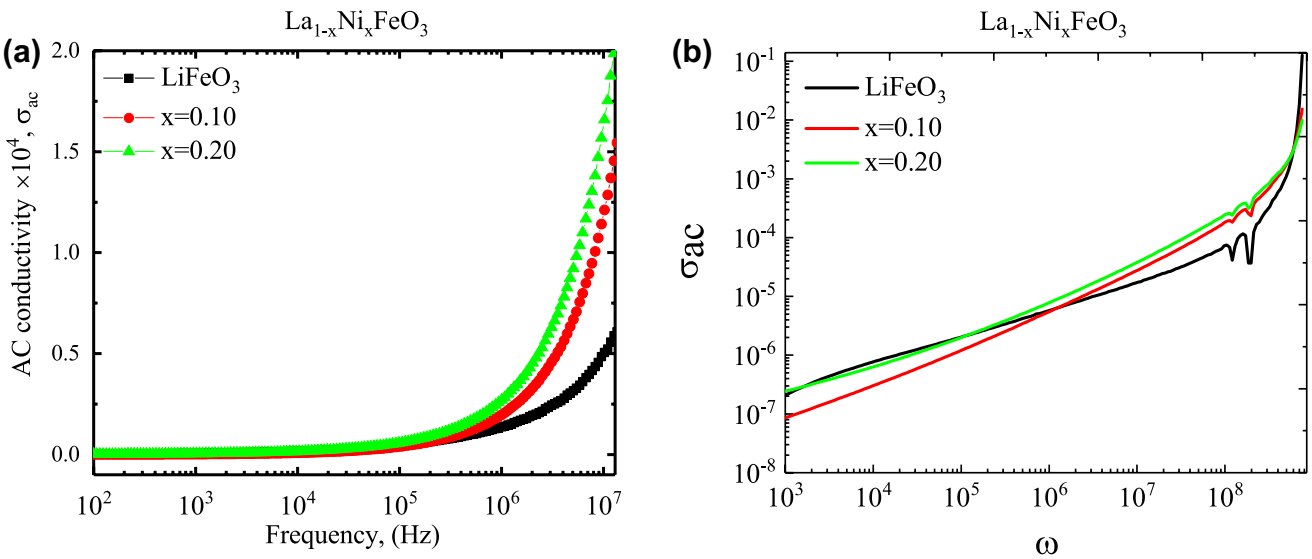

Fig. 4 **a** Frequency dependence of AC conductivity for $La_{1-x}Ni_xFeO_3$ ($x = 0$, 0.1, 0.2) ceramic and **b** $\log\sigma_{ac}$ vs $\log\omega$

mobility. This phenomenon requires the reduction in a small fraction of Fe^{3+} to Fe^{2+} with a simultaneous creation of coordinated oxygen vacancy to satisfy the neutrality of a γ-Fe_2O_3 crystallite [57]. Thus, the effect of trace secondary phase would not significantly deteriorate the dc conductivity (ac conductivity at low frequencies) of parent LFO.

3.5 Band gap tuning

With small band gap energy, LFO is a plausible attractive visible light irradiator. The photovoltaic (pv) absorption capacity of LFO is correlated with its electronic structure and thereby major contributor to their band gaps [58]. In this context, the diffused reflectance spectra captured by UV–Vis–NIR spectrophotometry (PE-1050) were used to obtain band gap energy, E_g of synthesized all nanomaterials. E_g was calculated by applying Kubelka/Munk function defined as: $F(R) = (1-R)^2/2R$, where R is the diffused reflectance. Shown in Fig. 5 are $[F(R)h\nu]^2$ versus $h\nu$ graphs.

E_g is obtained from the plots by drawing tangent lines at upper linear part of the curves. The point of intersection of a tangent at $[F(R)h\nu]^2 = 0$ is E_g [59]. Basically, E_g is the differentiate energy between (O:2*p*) valence band and (Fe:3*d*) conduction band of LFO. Table 2 summarizes obtained band gaps of pure LFO and Ni-doped LFO.

The observed reduction in E_g may stem from many contributors. The Fe–O octahedral restructuring of molecular orbital can reduce E_g [60]. The length scale of nanoparticles also affects the value of E_g [61], i.e., E_g scales with the size of particles. Theoretical calculation claimed that generation of new energy level between Fe:*3d* and O:*2p* by doping can reduce the effective E_g of LFO [62]. In addition, the Ni content in LFO might change the Fe–O bond length and Fe–O–Fe bond angle resulting in impact on E_g by modulating one-electron band width (*w*) [63]. E_{cb} of Ni^{2+} is – 4.39 eV, and E_{cb} of Fe^{2+} is – 4.33 eV; thus, $E_{cb}^{Ni} < E_{cb}^{Fe}$ [64] that may also reduce E_g. The experimental formula correlating *w* with bond length and bond angle reads

$$w = \frac{cos\omega}{d_{Fe-O}^{3.5}} \tag{2}$$

where d_{Fe-O} is Fe–O bond length and

$$\omega = \frac{1}{2}(\pi - (\mathrm{Fe} - O - Fe)) \tag{3}$$

E_g is linked to *w* by $E_g = \Pi$-w. Here, Π is the energy related to charge transfer [65]. Thus, the increase in bond angle, Fe–O–Fe, and the reduction in bond length, d_{Fe-O}, by Ni doping resulted in the increase in the value of *w* and thus the reduction in E_g. However, the band gap of γ-Fe_2O_3 is reported to be 2.0–2.2 eV [66, 67]; thus, it would interfere negatively with the purpose of lowering the band gap of LFO by doping.

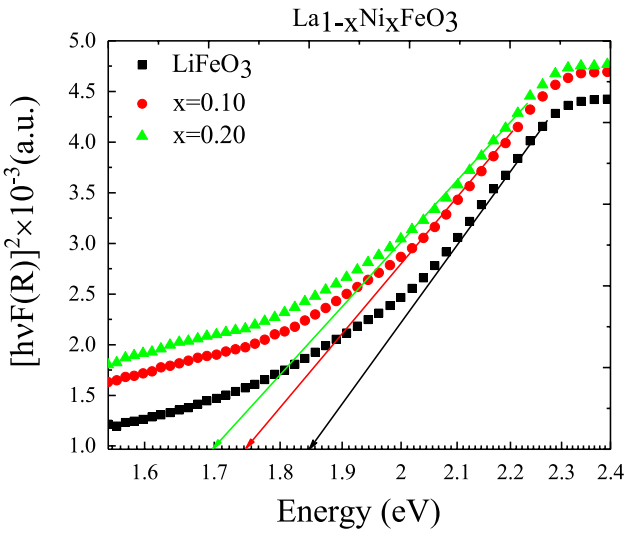

Fig. 5 $[h\nu F(R)]^2$ versus $h\nu$ (photon energy, eV) plots to calculate band gap energy for Ni-doped LFO

Table 2 Band gap, mean particle size and lattice strain of LFO and Ni-doped LFO

Samples ($La_{1-x}Ni_xFeO_3$)	Band gap (eV)	Mean particle size (nm)
$x = 0.0$	1.85	34
$x = 0.1$	1.75	31
$x = 0.2$	1.70	28

4 Conclusions

Pure LFO and $La_{1-x}Ni_xFeO_3$ (Ni-doped LFO) nanoparticles were successfully synthesized by conventional solid-state method. The parent crystal structure of LFO was found to retain in doped LFO. The size of crystallite was shown to decrease with increasing Ni doping level. The concurrently induced lattice strain increased with the reduction in crystallite size. However, the presence of clusters cannot be ruled out within the scope of XRD and FESEM experimental techniques. A significant agglomeration effect was thus presumably present in all synthesized nanostructured samples as confirmed by the disagreement between the crystallite size estimated from XRD patterns and the particle size calculated from FESEM images. Ni doping in LFO enhances its optical properties. The optical band gap was shown to decrease to 1.70 eV with 20 (at.)% Ni doping. Higher values of dielectric constant were obtained at low frequencies due to the conduction mechanism of heterogeneous nature. The observed dispersion at low

frequencies may resulted from electronic polarization by the underlying hopping mechanism of polaron. The AC conductivity (σ_{AC}) was found to increase with increasing Ni concentration. When the frequency of the applied field increases, the conduction process might be affected by 3d hopping electrons between Fe^{2+}/Fe^{3+} ions and Ni^{2+}/Ni^{3+} ions. The improved properties of synthesized Ni-doped LFO provide a scientific implication for its plausible applications in magnetically tunable filters and oscillators.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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