



Magnetic and dielectric behavior of terbium substituted $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ferrites

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ABSTRACT

The effects of concentration of terbium (Tb) on ferromagnetic resonance (FMR), relative initial permeability, morphology, and dielectric properties of MgFe_2O_4 ferrites were studied. It is found that FMR line-width and FMR position of magnesium–terbium (Mg–Tb) ferrites are found to fluctuate with the increase of Tb-content. The increase in FMR line-width is attributed to the charge transfer mechanism and inhomogeneity in the samples (TbFeO_3). The relative initial permeability decreases gradually as a function of Tb-content. The magnetic loss factors of substituted samples exhibit decreasing behavior in the frequency range 1 kHz to 10 MHz. Terbium substituted samples have lower dielectric constant as compared to the pure MgFe_2O_4 ferrite. The decrease in dielectric constant is attributed to the retardation in electron exchange mechanism caused by lockup among iron and terbium ions. An irregular behavior of the dielectric loss is found, which is related to the hopping probability of charges in these ferrites. A decrease in the ac conductivity is observed beyond 8 MHz and this corresponds to the occurrence of dielectric loss. The low magnetic and dielectric behavior makes these ferrite materials suitable for their applications in microwave devices.

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1. Introduction

Spinel ferrites are among the most widely used magnetic materials owing to their excellent electromagnetic and dielectric properties [1]. The miniaturization of the technology requires the development of new materials with optimum properties in order to cater for new demands. Magnesium ferrite is considered as an important candidate of the spinel family for its use in various potential applications of modern technology [2]. Many efforts have been made to improve the basic properties of ferrites by substituting various ions with different valence states. Rare earth substituted magnesium ferrites have attracted considerable attention in the field of technological applications extending from microwave to radio frequency. The rare earth ions are the promising substitute for the improvement of the properties of ferrites. Many research groups have studied magnetic and dielectric properties of magnesium ferrites with the addition of different rare earth ions in order to achieve optimized properties. The addition of rare earth metal ions like Gd, La, and Tb introduces changes in the structure and texture, allowing for significant modifications to be created in the magnetic and dielectric properties of these ferrites [3–7]. The aim of the present work is to investigate the effect of terbium ions substitution for magnesium ions on ferromagnetic resonance,

relative initial permeability, morphology, and dielectric properties of MgFe_2O_4 ferrites to make these ferrites suitable for microwave devices applications.

2. Experimental procedure

A series of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($x=0, 0.02, 0.04, 0.06, 0.08, 0.10, 0.12, 0.14, 0.16, 0.18, 0.20$) ferrites was prepared by ceramic method described in our earlier communication [7]. The ferromagnetic resonance measurements were performed on spherical shaped samples using a standard FMR spectrometer at X-band (9.5 GHz) in TE_{102} reflection cavity. The measurements were made under identical conditions on all the samples. In FMR measurements, the sample is always saturated due to static magnetic field required for resonance at microwave frequency. Field swept line-width was measured by sweeping the field from 60 Oe to 10 kOe.

Solatron 1260 impedance analyzer was used to measure the relative initial permeability. The ferrite samples fabricated in the form of toroid were used wound with Cu wire to serve as an inductor coil. The impedance analyzer was connected to a computer with GPIB interface. The whole operation of the analyzer was controlled by Solatron materials research and test, version 1.2 software. The microstructure of the samples was observed using a scanning electron microscope (SEM) model JEOL JSM-840. The average grain size was estimated from SEM micrographs using the line intercept method. The dielectric constant for the samples was calculated by using the formula:

$$\varepsilon' = \frac{Cd}{\varepsilon_0 A} \quad (1)$$

where C is the capacitance in F, d is the thickness of pellet in m, A is the cross-sectional area of the flat surface of the pellet, and ε_0 is the permittivity of free space. The ac electrical conductivity of all the samples was calculated using the relation:

$$\sigma_{ac} = 2\pi f(\tan \delta)\varepsilon'\varepsilon_0 \quad (2)$$

where $\tan \delta$ is the dissipation factor.

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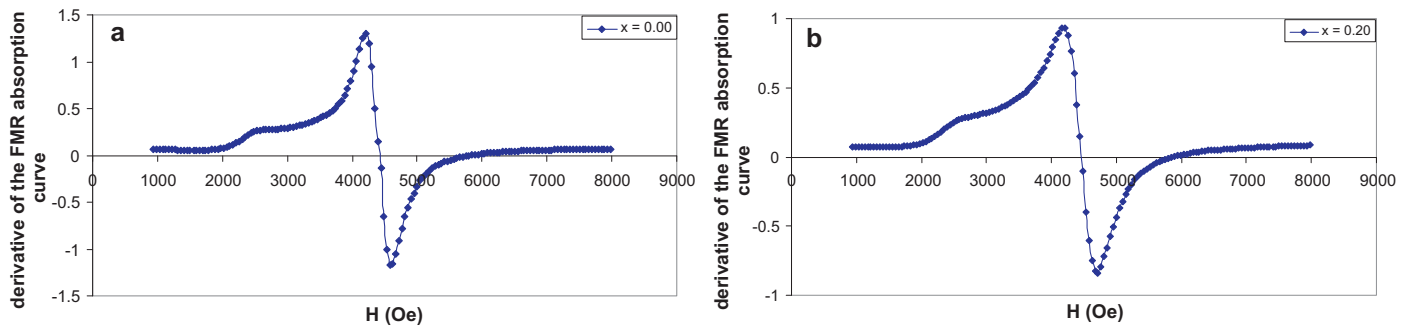


Fig. 1. FMR spectrum of (a) MgFe_2O_4 ferrite and (b) $\text{Mg}_{0.80}\text{Tb}_{0.20}\text{Fe}_2\text{O}_4$ ferrite.

3. Results and discussion

3.1. Ferromagnetic resonance

Typical ferromagnetic resonance (FMR) spectra of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($x=0, 0.2$) ferrites, taken at X-band (9.5 GHz) are shown in Fig. 1(a) and (b). A single resonance peak has been observed of the samples and all the profiles are slightly asymmetric. The spectra have been used to obtain the FMR line-widths (ΔH) and FMR position as listed in Table 1. The charge-transfer relaxation mechanism is important in spinel ferrites and it is the possible source of line-width in these ferrites. In the charge transfer mechanism, the energy is directly transferred to the lattice without going through either the degenerate or the thermal magnons. The line-widths of all the samples vary in the range (746–1146 Oe) except for $x=0.16$, which has the highest line-width (1146 Oe) as compared to rest of compositions. The composition $\text{Mg}_{0.98}\text{Tb}_{0.02}\text{Fe}_2\text{O}_4$ ($x=0.02$) has the minimum line-width (746 Oe) and highest saturation magnetization (32 emu/g) [7]. The line-widths obtained in the present investigation are much lower than the reported literature [8,9]. The substitution of terbium ions in these ferrites lowers the FMR line-width and this indicates better quality of the prepared samples. In the present samples a second phase (TbFeO_3) was observed in XRD patterns for $x \geq 0.06$ other than the fcc spinel phase. This second phase has the same effect as the surface pits in polycrystalline ferrites. A comparatively larger line-width has been reported [10] due to the presence of second phase. By using line-width the minimum relaxation time was calculated for these ferrites and it lies in the range $\sim 10^{-10}$ s. The intensity of the FMR profiles is sensitive to composition. The substitution of terbium ions in these ferrites varies the intensities of the FMR profiles. The intensities strongly depend on the gyromagnetic ratio (γ) and g -values of the cations present in each composition. All the cations involved like magnesium, ferric, and terbium have different values of gyromagnetic ratio (γ) because of the differences in the spectroscopic splitting g -factors. The FMR

profiles of all the Tb-substituted samples have lower intensities as compared to the unsubstituted sample MgFe_2O_4 as depicted in Fig. 1. The differences in the g -values of the cations involved are responsible for the observed variation in the intensity of the FMR profiles.

3.2. Relative initial permeability

The relative initial permeability, μ'_i , of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($0.00 \leq x \leq 0.2$) ferrites is measured in the frequency range 1 kHz to 10 MHz and the plot of μ'_i vs frequency is shown in typical Fig. 2. The plot indicates that the relative initial permeability is observed to decrease with increasing concentration of terbium ions. It is well known that [11] the magnetization process is considered as the superposition of domain wall motion and spin rotation. The domain wall-motions are affected by both the composition and microstructure. Domain wall-motions are dominant over the spin rotation; hence the μ'_i of ferrites is mainly attributed to the motion of domain walls [12–14]. In the present investigations the major contribution to μ'_i is due to the movement of domain walls in the low frequency region (Fig. 2). Our observations are in agreement with the Globus model [15]. The present samples have high μ'_i and it decreases with the increasing Tb-concentration. The decrease in μ'_i with the increasing concentration of terbium can be explained on the basis of following relation:

$$\mu'_i = M_S^2 \left[\frac{D_m}{K_1} \right] \quad (3)$$

where D_m is the average grain diameter, K_1 is the magnetocrystalline anisotropy constant, and M_S is the saturation magnetization. The saturation magnetization of the samples under investigation decreases [7] with the increasing concentration of terbium ions. So a reasonable decrease in μ'_i is consistent with saturation magnetization results. The average grain size decreases for all the substituted samples. Hence μ'_i is expected to decrease. A linear relationship of μ'_i vs grain size has been reported [16,17]

Table 1
FMR parameters of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ferrites measured at X-band (9.5 GHz).

Serial no.	Composition	FMR linewidth ΔH_{FWHM} (Oe)	FMR position (Oe)
1	MgFe_2O_4	752	4399
2	$\text{Mg}_{0.98}\text{Tb}_{0.02}\text{Fe}_2\text{O}_4$	746	4390
3	$\text{Mg}_{0.96}\text{Tb}_{0.04}\text{Fe}_2\text{O}_4$	799	4436
4	$\text{Mg}_{0.94}\text{Tb}_{0.06}\text{Fe}_2\text{O}_4$	954	4375
5	$\text{Mg}_{0.92}\text{Tb}_{0.08}\text{Fe}_2\text{O}_4$	790	4372
6	$\text{Mg}_{0.9}\text{Tb}_{0.1}\text{Fe}_2\text{O}_4$	1015	4203
7	$\text{Mg}_{0.88}\text{Tb}_{0.12}\text{Fe}_2\text{O}_4$	813	4365
8	$\text{Mg}_{0.86}\text{Tb}_{0.14}\text{Fe}_2\text{O}_4$	762	4420
9	$\text{Mg}_{0.84}\text{Tb}_{0.16}\text{Fe}_2\text{O}_4$	1146	4406
10	$\text{Mg}_{0.82}\text{Tb}_{0.18}\text{Fe}_2\text{O}_4$	991	4503
11	$\text{Mg}_{0.8}\text{Tb}_{0.2}\text{Fe}_2\text{O}_4$	1001	4438

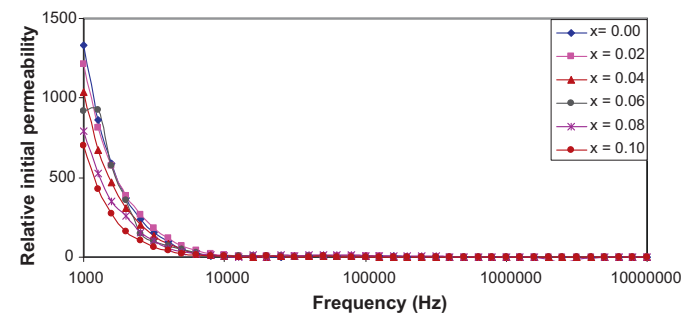


Fig. 2. Relative initial permeability as a function of frequency for $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($0.0 \leq x \leq 0.10$) ferrites.

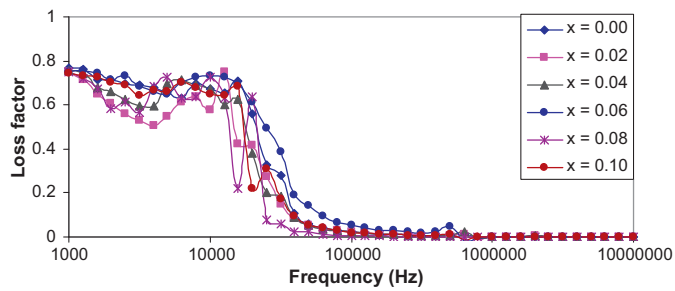


Fig. 3. Loss factor as a function of frequency for $Mg_{1-x}Tb_xFe_2O_4$ ($0.0 \leq x \leq 0.10$) ferrites.

and in some cases it deviates [18]. All the samples have almost similar decreasing trend of μ_i' except the samples having the terbium concentration, $x=0.12$ and $x=0.16$. Both these exhibit a drastic decrease in the values of μ_i' in the low frequency region. From Fig. 2, it is evident that there are no reasonable variations in μ_i' in the high frequency region.

3.2.1. Frequency dependent loss factor

Typical Fig. 3 depicts the plot of loss factor vs frequency for $Mg_{1-x}Tb_xFe_2O_4$ ($0.0 \leq x \leq 0.10$) ferrites. The loss factors of these samples have been observed to decrease with the increase in frequency. The samples with terbium concentration $0.00 \leq x \leq 0.10$ indicate very small peaks in the low frequency region and beyond 10 kHz a drastic decrease in the loss factors of these samples has been observed. The sample $x=0.08$ exhibits three loss peaks in the low frequency region and its value drops more rapidly as compared to neighboring compositions. The sample for the terbium concentration $x=0.06$ also exhibits a small loss peak in the high frequency region. The magnetic loss factor of the samples having terbium concentrations $0.12 \leq x \leq 0.20$ decreases more rapidly as compared to the compositions mentioned in Fig. 3. All the Tb substituted samples exhibit low values of loss factor as compared to the unsubstituted sample in the low frequency region. In the high frequency region the loss factor indicates very small values. The minimum loss factor has been observed for the compositions $Mg_{0.86}Tb_{0.14}Fe_2O_4$ and $Mg_{0.82}Tb_{0.18}Fe_2O_4$ as compared to all other compositions. The previously reported results indicate that [19] the partial substitution of rare earth ions (Eu, Ru, and Gd) in Ni–Zn ferrites significantly lowers the relative loss factor over a wide frequency range. All Tb substituted magnesium ferrites show smaller values of the loss factor over a wide frequency range.

3.3. Scanning electron microscopy

Fig. 4 shows representative SEM micrograph of the $Mg_{1-x}Tb_xFe_2O_4$ ferrite system for $x=0$. The micrograph exhibits inhomogeneous grain size distribution. The micrographs for $x=0$ and $x=0.08$ show monophasic microstructure along with some agglomerates. The micrographs for $x=0.14$ and $x=0.20$ depict biphasic microstructure. It can be observed that the second phase occurs in the form of precipitates located among the matrix of dark grains. These precipitates in the ferrite matrix were formed by either segregation to, or precipitating at the grain boundaries. With the increase of terbium concentration from $x=0.0$ to 0.2 the grain size found to decrease and lies in the range 7–4 μm . The decrease in the grain size can be explained on the basis of ionic radii of the terbium ions. It is assumed that some of the terbium ions may reside on the grain boundaries; these can hinder the growth [20] and also exert stress on the grains which causes the reduction of grain size. In our previous communication [7] EDX

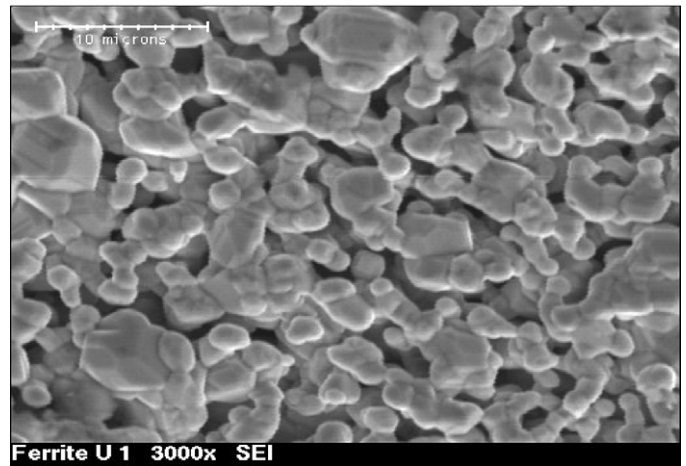


Fig. 4. SEM micrograph of $Mg_{1-x}Tb_xFe_2O_4$ ($x=0.00$) ferrite.

analysis have been presented which indicate the presence of Tb, Mg, and Fe in the prepared samples.

3.4. Dielectric properties

The dielectric constant decreases with successive substitution of terbium ions. The compositional variations of dielectric constant (ϵ') with frequency are shown in representative Fig. 5. It is known that in $MgFe_2O_4$ ferrite, most of the Mg ions occupy on the octahedral sites [21] while Fe ions occupy on both octahedral (B-sites) and tetrahedral (A-sites) sites. The increase of Tb ions substitution on B-sites replaces some Mg ions. The reduction of Mg ions on B-sites leads to the decrease in population of Fe^{2+} ions ($Mg^{2+} + Fe^{3+} \leftrightarrow Mg^{3+} + Fe^{2+}$) on B-sites. The electron exchange interaction happening at B-sites between the Fe^{3+} ions and Fe^{2+} ions retarded due to the presence of Tb ions on B-sites. Hence the hopping length of conduction electrons increases. Since the dielectric polarization mechanism in these ferrites is similar to the electronic conduction mechanism [22]. The number of electrons exchanged between the cations involved results in the local displacements in the direction of the applied external field which determines the dielectric polarization. This explains the decrease in the dielectric constant with the increasing concentration of Tb ions. Similar behavior of dielectric constant has been reported [23] when different rare-earths elements were substituted in Mn–Zn ferrites. It is also observed that the dielectric constant decreases with the increase of frequency. At the lower frequencies the samples have high values of dielectric constant of the order of 10^3 – 10^4 . These high values of dielectric constant at lower frequencies may be attributed to the inhomogeneous dielectric structure which is in agreement with Koops phenomenological theory [24]. The inhomogeneous dielectric structure is composed of two layers. The first

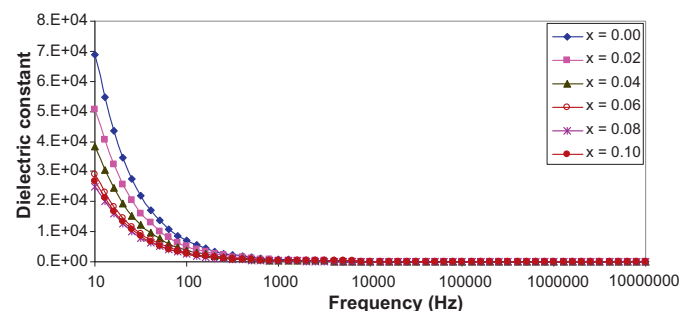


Fig. 5. Dielectric constant (ϵ') vs frequency of $Mg_{1-x}Tb_xFe_2O_4$ ($x=0.0$ – 0.10) ferrites.

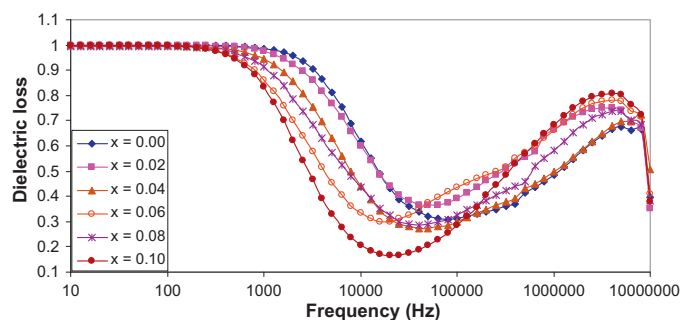


Fig. 6. Dielectric loss ($\tan \delta$) vs frequency of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($x=0.0\text{--}0.10$) ferrites.

layer consists of well conducting large ferrite grains and the second thin layer comprises poorly conducting grain boundaries. The ferrite grains having high conductivity and low dielectric constant are more effective in the high frequency region while the resistive grain boundaries are more effective in the low frequency region [25]. The frequency dependent behavior of dielectric constant in ferrites can be explained on the basis of space charge polarization. The charge carriers contributing in the polarizability exhibit lagging behind the applied field beyond a certain frequency [26] and cannot follow the alternation of the applied electric field and hence results in a decrease in the dielectric constant of these ferrites.

The variation of dielectric loss ($\tan \delta$) with frequency for $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($x=0.0\text{--}0.10$) ferrites is shown in representative Fig. 6. The figure revealed that the values of dielectric loss decrease with substitution of terbium ions in these ferrites. It is observed that the dielectric loss first decreases as the frequency increases and then increases rapidly beyond 100 kHz. Qualitatively the formation of dielectric loss peaks may be owing to the following reasons: a strong correlation has been reported [27,28] between the conduction mechanism and dielectric behavior. The conduction phenomenon in ferrites is considered due to the hopping of electrons between divalent and trivalent iron ions present over the octahedral sites. The dielectric loss peak is observed when the jump frequency of the local charge carriers ($f=1/\tau$ relaxation) is approximately equal to the frequency of external applied field [25,29,30]. The sharp peak is formed when both the frequencies exactly matched with each other. It is seen that the minima occurred in the dielectric loss for all the samples shifts towards the lower frequency region. The loss peak increase in high frequency region as the terbium concentration is increased from 0.00 to 0.10 as shown in Fig. 6. The dielectric loss peak decreases in high frequency region as the terbium concentration is varied from 0.12 to 0.20. The irregular behavior of the loss peak occurred owing to the jump or hopping probability of the cations involved. A decrease in the loss peak with increasing terbium concentration indicates that the hopping or jump probability between Fe^{3+} and Fe^{2+} is reduced. Similar trend of the dielectric loss ($\tan \delta$) has also been reported by other researchers [31,32]. Hence it can be inferred that the jump or hopping frequencies for all the samples showing peaking behavior lie in the range 1–10 MHz. The samples having nominal compositions $\text{Mg}_{0.82}\text{Tb}_{0.18}\text{Fe}_2\text{O}_4$ and $\text{Mg}_{0.84}\text{Tb}_{0.16}\text{Fe}_2\text{O}_4$ exhibit minimum dielectric loss and dielectric constant. The smaller value of dielectric loss is one of the characteristic parameters for microwave applications.

Representative Fig. 7 shows the variations of ac conductivity with frequency at room temperature for $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ferrites ($0.0 \leq x \leq 0.10$). The ac conductivity (σ_{ac}) increases with an increase in frequency. The magnitude of the ac conductivity decreases as the Tb-contents are increased. This decrease in magnitude of σ_{ac} can be explained on the basis that Tb-ions occupy on the octahedral sites in these ferrites. Tb-ions are expected to lockup with Fe^{2+}

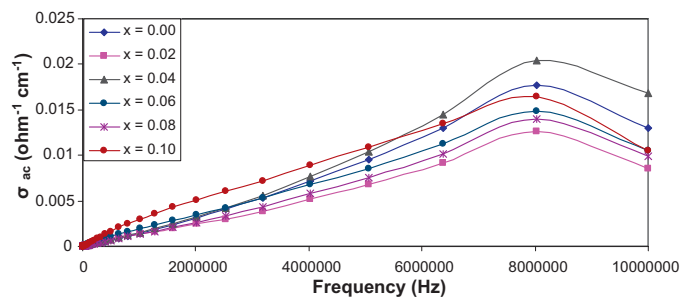


Fig. 7. The variation of ac conductivity with frequency for $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ($x=0.0\text{--}0.10$) ferrites.

ions and it reduces the hopping between the iron ions $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$. This decreases the probability of hopping of charge carriers; thus the conductivity decreases. The sample $\text{Mg}_{0.96}\text{Tb}_{0.04}\text{Fe}_2\text{O}_4$ exhibits high conductivity values in high frequency region (MHz) as compared to the neighboring compositions. The ac conductivity is observed to decrease in all Tb-substituted samples except $x=0.18$, which is anomaly in these plots. The decrease in conductivity beyond 8 MHz is attributed to the occurrence of loss peak in $\tan \delta$. A decrease in ac conductivity has been reported [23] when different rare-earths ions were substituted in Mn–Zn ferrites.

4. Conclusions

The FMR line-widths of $\text{Mg}_{1-x}\text{Tb}_x\text{Fe}_2\text{O}_4$ ferrites obtained in the present investigation are smaller as compared to the reported line-width of spinel ferrites. A decrease in relative initial permeability has been observed by the substitution of terbium ions. The substituted samples exhibit smaller values of the magnetic loss factors. The composition $\text{Mg}_{0.8}\text{Tb}_{0.2}\text{Fe}_2\text{O}_4$ exhibits smallest value of magnetic loss factor $\sim 1.02 \times 10^{-6}$. The dielectric constant and dielectric loss attain very low values at 10 MHz for higher concentration of terbium ions. Hence smaller magnetic loss factor and dielectric loss suggest the utility of these ferrites in microwave applications.

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