Structural and magnetic properties of erbium substituted spinel ferrites for microwave absorptions

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ABSTRACT
The strontium-based spinel ferrites were prepared by using the sol–gel technique. Generalized form for fabricated samples was written as: \( \text{SrEr}_x \text{Fe}_{2-x} \text{O}_4 \), \( x = 0.00, 0.025, 0.05, 0.075 \) and 0.10. Various techniques were used to reveal structural, electrical, magnetic and microwave characteristics. Each sample possessed a cubic spinel structure. The impedance was to be enhanced with the involvement of the erbium (Er) ion. Magnetic features were carried out by vibrating the sample magnetometer under the applied field of –2 kOe to +2 kOe. Magnetization values were declined from 70 to 30 emu/g with an increase in erbium contents in the strontium-based spinel ferrites. The cavity Perturbation method was used to obtain microwave measurements. Permeability and permittivity (real and imaginary parts) were reduced with a regular increase of erbium ions. Above-mentioned features of erbium substituted strontium ferrite exhibited their capabilities for microwave absorption applications.

1. Introduction
The various researchers investigated the properties of different magnetic oxides and ferrite substances. Many beneficial aspects of these magnetic substances like less losses, high magnetic susceptibility, Curie temperature and high magnetic induction value were found. The need for such materials also increased because of their astonishing features at normal temperatures [1]. There were two major categories of magnetic compounds; soft and hard ferrites. Soft ferrites comprise a wide range of uses such as microwave instruments, computer memories, medicine industry and catalysis. Some other field in which soft magnetic ferrites were needed includes information technology, biotechnology and large magnetic moment gadgets [2]. The strontium ferrites were classified as mixed spinel ferrite due to their mixed structure. In a mixed structure, the oxygen ions were closely organized at tetrahedral and octahedral (A and B) sites which are covered by metallic ions \( \text{Zn}^{2+}, \text{Sr}^{2+}, \text{Fe}^{3+} \), etc. The configuration of base strontium ferrites was modified by adding little quantity of erbium ions in them. However, the actual base crystalline structure does not change [3]. Cubic structure of Sr-ferrites was inspected with Fd-3m space group. The modification of some particular compound can be made according to desire by making compositional variations, varying ratios of reactants, sintering limits, manufacturing techniques and duration of calcination [4]. The dielectric and magnetic behaviour of prepared samples was evaluated under specific ranges of temperature and frequency to obtain optimum results for research purposes as well as for making ferrite-based devices. The dielectric study is very helpful when someone is focused on the ferrites for practical field. Dielectric behaviour was associated with the manufacturing method. Numerous papers with defined compositions were available for the study of dielectric behaviour with respect to preparation techniques. The rare-earth (RE) ion or transition element (TE) ion substituted spinel ferrites have astonishing properties as perceived in the literature [5].

According to numerous reports, rare-earth substituted spinel ferrites have been described in detail. Kadam et al. developed nickel–cobalt ferrites doped with dysprosium. Their magnetic, structural and electrical properties were investigated using the ceramic method. The saturation magnetization and magnetic moment decreased with Dy substitution, while the crystalline size increased [6]. Heiba et al. used the citrate method to create Gd-substituted nickel ferrites. The addition of Gd\(^{3+}\) resulted in non-monotonic lattice parameters. While gadolinium content increased...
coercivity at room temperature and saturation magnetization decreased [7]. The magneto-crystalline anisotropy of nickel–cobalt spinel ferrites was studied by Biasi et al. The magnetic anisotropy of the material increases monotonically as more Co is added to it, allowing it to be used as a magnet for practical purposes [7]. Zhang et al. [8] investigated the magnetic properties of sol–gel-synthesised spinel ferrites. Lanthanum effectively reduces coercivity while increasing magnetism. Srinivasamurthy et al. [9], on the other hand, investigated the magnetic properties of spinel ferrites. When they added rare-earth to the mixture, they revealed that the saturation magnetization decreased while the coercivity increased.

In the current study, spinel ferrites SrEr$_x$Fe$_{2−x}$O$_4$, $x = 0.00, 0.025, 0.05, 0.075$ and $0.10$ were prepared by sol–gel route and characterized under different techniques to evaluate the microwave, magnetic, structural and electrical behaviour. The benefits of this preparing technique include low cost, mixture homogeneity and fine quality grain size.

2. Experimental

Particular weights of raw metallic nitrates with 99.9% purity were taken from Sigma Aldrich. These include iron nitrate Fe(NO$_3$)$_3$, erbium nitrate Er(NO$_3$)$_3$·H$_2$O with 449.13 molecular weight and strontium nitrate Sr(NO$_3$)$_3$·4H$_2$O with 178.95 molecular weight. Measured weights of these metallic nitrates were thawed in DI (deionized) water. 99% pure citric C$_6$H$_8$O$_7$·H$_2$O was thawed to the solution beakers to achieve homogeneous solutions and obtain standard-sized nanoparticles. Continuous stirring was done at 80°C with a magnetic stirrer for 3 h. The solution of NH$_4$OH was added to set the solution’s pH at 7. The stirring process continues and when the solution changed into gel after 3 h, the soft gel is subjected to burning process for 2 h to attain ashes under 350°C. The Polyvinyl binder was added with ashes. The mixture was placed under hydraulic press to produce pellets within the applied pressure of 30 kPa. Binder present in the pellet was removed with the use of oven having 200°C. A tube furnace was used for final sintering with 900°C temperature. The sintering process continues for 7 h. Now, the pellets were ready for further characterization reports.

Various characterization tools were considered for structural, dielectric and magnetic examinations. The specimens were silver coated before the dielectric examination. Permeability and permittivity values were measured by using cavity Perturbation within microwave frequencies. The temperature was set at room temperature value during the examination. A rectangular cavity resonator and ideal Cu wave guide were used to construct the cavity. A network analyser (Model: Agilent 86510C) was enabled to excite the cavity. For the conduction judgement, Wayne Kerr Impedance analyser (Model: 6500B) was enabled.

3. Results and discussion

3.1. Structural analysis

Intensity versus angle ($2\theta$) graph obtained from XRD data were given in Figure 1. It can be seen clearly from Figure 1(a) that the samples possessed a cubic shape and have Fd-3m space group. There were no secondary phases in the present study. Each sample’s most intense (3 1 1) diffraction plane, as well as its intensity and location, are depicted in Figure 3. Due to erbium substitution, the peak shifts to the lower diffraction angle, which can be explained by the host and substituted ionic radius difference. Erbium has a larger ionic radius than iron because it was replaced for it. This substitution shifts the diffraction peak by increasing the d-spacing. In nickel ferrite, Meng et al. [11] discovered a similar peak shift for indium substitution. The presence of erbium, on the other hand, broadens the peak. The peak broadens as the erbium concentration rises. Peak broadening decreases with crystallite size, according to George et al. To put it another way, as substituent concentration rises, crystallite size decreases. As shown in Table 1, the lattice strain in pure strontium ferrite increases as the erbium content increases. It might be another explanation for the peak’s widening in the present case.

The crystallite size of samples was simply found through Scherrer’s relation and it ranged between 40 nm and 82 nm. The relation [12] was used to find lattice parameter “a”.

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \tag{1}$$

It was enhanced with addition of erbium ion in the crystal. The enhancement in the “a” value was due to the bigger size of erbium ions as compared to strontium ions. Lattice parameters for all samples were listed in Table 1. Following two relations were used to obtain X-ray and bulk density values [13].

$$D_x = \frac{8M}{N_A a^3} \tag{2}$$

$$D_m = \frac{M}{\pi l^2} \tag{3}$$

In these relations, $M$, $r$, $l$ and $N_A$ were the molecular weight, pellet’s radius, thickness and Avogadro’s number, respectively. A regular increasing behaviour was observed in the values of both densities with the substitution of RE ions. Table 1 consists of all these density values. Many other factors which were measured theoretically are listed in Table 1. These factors include theoretical lattice parameter “a”, discrepancy factor ($R_{wp}$),
expected values ($R_{\text{exp}}$), Bragg’s value ($R_{\text{Bragg}}$) and the goodness fit factor ($\chi^2$). These theoretical values were found by applying Rietveld Method. Rietveld refine of sample with $x = 0.00$ was given in Figure 1(b). Rietveld Model results have similarity with experimental data. Also, these theoretically measured values have good agreement with earlier chemist reports [14,15].

The EDX spectra were used to determine the elemental composition of SrEr$_x$Fe$_{2-x}$O$_4$ ($x = 0.0, 0.025, 0.05, 0.075$ and $0.10$) spinel ferrite. The images and results for EDX, a common spectrometer, are shown in Figure 2. It is impossible to tell if any impurities or other elements are present. The observed metal ion (Fe:Er) and strontium to oxygen (Sr:O) composition ratios are very similar. This shows that the sol–gel samples’ stoichiometry was maintained throughout the preparation process. The micrographs of samples were given in Figure 3. It showed that the grain size values were affected with erbium concentration variation. The grain size steadily falls with erbium concentration addition. This is due to the fact that the grain growth is restricted with erbium addition. So the average value of grain size is restrained.

### 3.2. Impedance analysis

The conduction mechanism of samples was understood through impedance and modulus spectroscopy. The conduction process in-between high and low conducting boundaries was focused. Many electrical conduction parameters such as impedance, relaxation time and resistance at grains and boundaries were attained. With the upswing of frequency of applied field, both the real and imaginary (Real & Imaginary) parts of impedance ($Z''$ and $Z'$) were declined as shown in Figures 4 and 5. The reason for this declination is that the transportation of charges restrained with applied frequency of AC field. The variations in both Real and Imaginary components of impedance ($M''$ and $M'$) were displayed in Figures 6 and 7. Both values of $M''$ and $M'$ were enlarged with the rise of frequency. Both $M''$ and $M'$ were linearly changed with the addition of RE content. These consequences showed that there are insufficient forces that control the charge carrier movements.

### Table 1. Different parameters of erbium substituted spinel ferrites.

| Parameters                        | $x = 0.00$ | $x = 0.025$ | $x = 0.05$ | $x = 0.075$ | $x = 0.10$ |
|----------------------------------|------------|-------------|------------|-------------|------------|
| Lattice constant (Å)             | 8.31       | 8.34        | 8.40       | 8.43        | 8.46       |
| Crystallite size $D_{\text{XRD}}$ (nm) | 82         | 71          | 63         | 51          | 40         |
| Bulk density (g/cm$^3$)           | 3.98       | 4.06        | 4.13       | 4.19        | 4.27       |
| X-ray density (g/cm$^3$)          | 5.12       | 5.21        | 5.29       | 5.40        | 5.45       |
| Discrepancy factor ($R_{\text{exp}}$) | 7.99       | 7.42        | 7.43       | 7.11        | 7.04       |
| Expected value ($R_{\text{exp}}$) | 7.01       | 6.71        | 6.45       | 6.11        | 5.99       |
| Bragg value ($R_{\text{Bragg}}$)  | 4.01       | 3.21        | 3.00       | 2.90        | 2.78       |
| Goodness fit factor ($\chi^2$)    | 2.86       | 2.74        | 2.69       | 2.61        | 2.51       |
| Lattice constant $a_0$ (Å)        | 8.32       | 8.35        | 8.42       | 8.46        | 8.48       |
| Average micro-strain              | 0.413      | 0.426       | 0.454      | 0.463       | 0.488      |
provide the information of electrical transportation [16]. The only one semicircle in the Cole–Cole plot explains the effect of electrical conduction due to bulk grains present in the lattice.

### 3.3. Magnetic properties

The M–H loops were drawn on behalf of the VSM data as displayed in Figure 9. The narrow shape of M–H loops tells the soft magnetic nature of prepared materials. The inspection of these loops offered various numerical values for saturation, remanence magnetization and the coercivity values which are listed in Table 2. The magnetization values were decreased with replacing of iron with erbium ion. Similar results were noted from the earlier published data [17,18]. The reduction in magnetization values was due to less magnetic moment of RE ion (Er\(^{3+}\)) as compared to iron ion (Fe\(^{3+}\)). When rare-earth ions are substituted in ferrites, the Reitveld refinement cation distribution confirms their preference for the octahedral B site. Spin–orbit coupling, as well as 3d-4f coupling, must be considered. In RE ions, the spin–orbit coupling effect is the most powerful [19]. The cation distribution clearly shows that Er\(^{3+}\) ions are displacing Fe ions in the octahedral site. The magnetic moment (\(\mu B\)) of the paramagnetic Er\(^{3+}\) ion in the current ferrite system under investigation is 3 BM, while that of the Fe\(^{3+}\) ion is 5 BM. The magnetization value of Er\(^{3+}\) ions should decrease in accordance with their magnetic moments, as shown in Table 2. With increasing substituted concentration, the saturation magnetization value decreased. Sodaei et al. [20,21] revealed a similar trend in substituted Tb\(^{3+}\) ions in cobalt ferrites. Spin–orbit coupling, as well as 3d-4f coupling, must be considered. In RE ions, the spin–orbit coupling effect is the most powerful [22].

There are three types of interactions in spinel ferrites which include A–B interactions, A–A interactions and B–B interactions. But, the A–B interactions were considered to be more effective than the other two types. These A–B interactions were considered to be responsible for lower magnetization values [23]. In soft spinel ferrites, the iron ions exist at both A and B (tetrahedral and octahedral) sites. But, the RE ions were only located at B sites. This happened due to the bigger size of RE ion. The possibility of staying RE ions at tetrahedral sites is very little. Because of the non-magnetic nature RE ion, the magnetic moment of B site is lesser than A site. Thus, the replacement of Fe ion by Er ion produces almost no change or very little change in magnetization value. Hence, net value magnetization was reduced. The shifting of iron ions from octahedral to tetrahedral sites also causes the reduction of saturation magnetization value.

The squareness ratio was found by dividing the remanence magnetization by saturation magnetization. These values were listed in Table 2. The \(M_r/M_s\) ratio increased with RE ion substitution. For a particular value; \(M_r/M_s = 0.5\), the samples were supposed to have solitary domain [24]. The substitution of RE ion in the lattice increased the value of its coercivity. The maximum coercivity value received for the sample having \(x = 0.10\) concentration of RE ion. Following factors such as domain size, grain size, porosity, morphology and magneto-crystalline anisotropy may disturb the coercivity value.

The saturation magnetization and coercivity behave oppositely as found from Brown’s relation. In the present case, the saturation magnetization reduced and coercivity (\(H_c\)) enhanced with addition RE ion. The other factor which is responsible for the enlarged coercivity is hampered grain size. Since the crystallite size behaves oppositely with coercivity value. The anisotropy constant \((k_1)\) was calculated from the following relation.

\[
k_1 = \frac{M_s \times H_c}{0.96}
\]
Three possible factors have influenced the magnetic properties of RE substituted spinel ferrites. The number of unpaired electrons, magnetic moment and spin–orbit coupling strength all contribute to the total number of electrons in a material. Myrtil et al. [25] discovered no correlation between the number of unpaired electrons in substituted ions and their properties. Additionally, there is no relationship between the magnetic properties of RE ions and their magnetic moment. The strength of the spin–orbital coupling determines the magnetic anisotropy of nanoparticles. Coercivity enhancement results in an increase in magnetic anisotropy. In the first row, the spin–orbital coupling between lanthanide and transition metal ions is typically much stronger. This is most likely the case, given the observed increase in coercivity of ferrites containing some RE ions. The strong electron spin–orbital coupling of the $\text{Er}^{3+}$ ion undoubtedly contributes to the increased coercivity of erbium substituted SrFe$_2$O$_4$. By doping strontium ferrites with Er, the coercivity of the materials was increased, resulting in an increase in magnetic hardness. Both parameters $k_1$ and $H_c$ were varied in the same fashion [26]. Since we have considered that the current system has a single domain, so, these ferrites should be unusual in sense of spin interactions. The quantity of Fe ions is reduced with the addition of RE ions which leads to an increase in anisotropy constant $k_1$. 

Figure 3. SEM of all the samples.
3.4. Microwave properties

The microwave inspection of samples was completed on the behalf of evaluation of permittivity and permeability. The inspection was done under 8–13 GHz frequency gap by adopting Cavity Perturbation technique. Both real and imaginary (Real and Imaginary) parts of permeability and permittivity were inspected with respect to RE ion concentration as well frequency. Both Real and Imaginary components of permeability and permittivity were noticed to be gradually reduced with the emergence of frequency. The complex permeability and permittivity were found through Cavity Perturbation under microwave frequency. It is a very sensitive measuring tool because it requires a complex permeability and very less amount of sample. The dissipative and dispersive terms of cavity Perturbation technique were linked with quality factor and resonance frequency, respectively. The suitable choice of cavity mode along the variation of quality factor and resonance frequency was used to find the complex permeability

**Figure 4.** Impedance (Real part) of Sr-based spinel ferrites.

**Figure 5.** Impedance (Imaginary part) of Sr-based spinel ferrites.
Figure 6. Electric modulus (Real part) of Sr-based spinel ferrites.

Figure 7. Electric modulus (Imaginary part) of Sr-based spinel ferrites.

\[(\mu - j\mu'')\text{ and complex permittivity (}\varepsilon' - j\varepsilon'\text{). The varying Real and Imaginary components of permittivity versus frequency under X-band were shown in Figure 10.\] Frequently, interfacial and dipolar polarization characteristics were noted to be missing within microwave frequency. However, the major donation of dielectric constant was obtained through electronic and ionic polarizations. The permittivity value linearly reduced with the rise of frequency value as expressed in Figure 10. The reason behind this behaviour is that the electron exchange \(\mathrm{Fe}^{2+} \leftrightarrow \mathrm{Fe}^{3+}\) does not follow the AC field frequency when the frequency of the microwave field crosses some specific limits [27]. In the stoichiometry of samples, the ferrous ions were not present and also
the preparation mechanism of the ferrites required a large level of sintering temperature to achieve solid form. At this temperature level, the oxygen dissociates due to the formation of nonstochiometric constitutions. Hence, the Fe$^{2+}$↔Fe$^{3+}$ electron exchange slowed down [28]. The Real part of permittivity ($\varepsilon'$) is shortened with the addition of RE ion in the lattice. The pure ferrites have larger $\varepsilon'$ value as compared to RE substituted sample. This is due to the fact that the pure sample has a larger quantity of Fe ions as compared to RE substituted samples. The ferrous ions on octahedral sites were reduced due to the phenomenon of polarization within the microwave frequency gap. So, the value $\varepsilon'$ continuously reduces with RE substitution. The sample with the highest amount of RE ion has lowest value of Re permittivity. The Imaginary component of...
permittivity ($\varepsilon''$) was lessened with the rise of frequency as shown in Figure 10(b). The polarization and Imaginary part of permittivity vary in the same way. Both $\varepsilon''$ and polarization were increased with the increase of ferrous ions and reduced with increase RE ions. The fact is that more number of $\text{Fe}^{2+}$ ions increased the electron exchange $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ mechanism happening on the other crystallographic site. Thus, these outcomes showed that both Real and Imaginary components of permittivity were associated with the number of $\text{Fe}^{2+}$ ions in the lattice. Within the microwave X-band frequency range, both Real and Imaginary permittivity behaved in the same manner. Within the specified frequency range, the spin rotations and domain wall displacement in nano-ferrites provide a healthy donation for real and imaginary parts of permeabilities. The magnetization factor leads the anisotropy field which is excited due to the AC magnetic field. The real part of permeability was measured by using the following relations [29,30]:

$$
\mu' = \left[ \frac{\omega_p^2}{\omega_p^2 - \omega^2 + jd\omega} \right] \varepsilon \cos \omega t
$$

(10)

Such type of formulation was applied when the considered system is present within the RF field. In the relation, Larmor frequency is represented by $\omega_p$, AC field frequency is denoted by $\omega$, precessional angle is indicated by $\varepsilon$, damping factor is denoted by “d” and damping term is denoted by $jd\omega$. Due to the presence of homogeneities and imperfections in the crystal, the damping factor is dependent on damping and precessional torques. For the particular case $\omega_p > \omega$, the value Re permeability linearly changed with AC field frequency. For the case $\omega_p = \omega$, the factor $\mu'$ attained maximum value. A sudden reduction in the value of $\mu'$ was observed when $\omega_p > \omega$. In the last condition, the RF magnetic field and magnetization vector were out of phase. The phenomenon is termed as resonance. The resonance is achieved when the natural and applied frequency become well matched [20]. The value of $\mu'$ inversely related with the frequency following Snoek’s Law as displayed in Figure 11(a). Formulation for the Snoek’s Law is given as

$$
f = \frac{\gamma M_s}{3\pi(\mu - 1)}
$$

(11)

In this formulation, the gyromagnetic ratio, resonant frequency and the saturation magnetization were represented by $F$, $M_s$ and $\gamma$, respectively. According to the above given relation, the value of $\mu'$ was found to be higher at the less resonant frequency value. The value of $\mu'$ was dependent upon the $M_s$ value. The net magnetization reduced gradually with the RE doping in spinel nanoparticles. It is also confirmed that the value of $\mu'$ is higher for pure strontium ferrite as compared to Er doped Sr-ferrites. The details of microwave losses can be easily guessed through Imaginary component of permeability “$\mu''$”. The resonance phenomenon for $\mu''$ can be observed over angular frequency. It also tells about the losses which appear from the spin lattice relaxation and magneto-crystalline anisotropy. The changes occurred in the $\mu''$ value with respect to frequency were displayed in Figure 11(b). This figure clearly depicts that the Imaginary part of permeability declined with increase of frequency at X-band. The main factor responsible for the losses of permeability is the lagging in AC frequency and magnetization.

**Figure 10.** (a) Permittivity (Real part) vs. frequency of Sr-based spinel ferrites. (b) Permittivity (Imaginary part) vs. frequency of Sr-based spinel ferrites.
Actually, the dielectric and magnetic features of RE-substituted spinel ferrites were changed with changes of microstructure like size, morphology, densification and precipitations. The permeability as well as permittivity was reliant on applied field frequency and stoichiometric compositions. In microwave range, both Real and Imaginary components of permeability and permittivity were gradually lessened with upturn of frequency. The electron exchange mechanism between ferric and ferrous ions is the chief reason for the variation of complex permittivity. In the case of complex permeability, it depends upon the saturation magnetization and resonance phenomenon. Normalized input impedance “Zin” was calculated by applying the equation [21].

\[ Z_{in} = \frac{1}{\mu_r \varepsilon_r} \]

In the equation, the “\( \mu_r \)” shows relative permeability, “\( \varepsilon_r \)” specifies relative permittivity and “c” signifies the speed of light. The reflection loss (RL) of electromagnetic waves measured in dBJs within normal frequency can be composed as a single layer [22]. Investigation of dependency of current system upon the RL thickness was done. Theoretical values of RL for whole prepared nanoparticles were investigated by using relation 13 and 14. The RL value for the system varies in-between 2 and 2.5 mm besides the thickness of the layer. The variational behaviour was presented in Figure 12. It was observed that the RL peaks moved towards thicker and
smaller frequency sections. Consequently, the width of absorption bands and their peak values initially rises and then falls. The thickness values (tm) were used to calculate the reflective loss. Wavelength in the absorber and incident frequency were denoted by $\lambda_o$ and $f$, respectively. The layer thickness and the frequency have correlation as depicted from the above relation. The pellet’s imaginary thickness largely affects the reflection loss. Similar trends of variation were observed by several previous material chemists as cited in the literature review [31]. The highest value of attenuation frequency was controlled properly by modifying the thickness as noticed in current case. Finally, we can say that the prepared samples with 2 mm thickness have greater absorption value and smaller losses.

4. Conclusion

The most common way called sol–gel was adapted to produce a series of RE substituted Sr-ferrite particles. The strongest peak at 3 1 1 was seen in the XRD patterns. This peak at the highest intensity is the confirmation of formation spinel nanoparticles. The dielectric constant was found to be reducing with RE doping. The electrical conduction was explained on behalf of impedance and modulus spectra. The electrical transportation and its effects at room temperature may be evaluated through the semicircles in the Cole–Cole plot. The complex permeability and permittivity values were changed with stoichiometric composition and frequency. With the rise of frequency, the real and imaginary components of permeability and permittivity values dropped within the microwave range. The behaviour of complex permittivity might be explained with the help of electron exchange $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ hopping. These evaluated parameters and modifications endorse their usage in the field of microwave absorption.

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Disclosure statement

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Data availability statement

Data sharing is not applicable to this article as no data sets were generated during the present study.

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