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Study on the influence of magnesium doping on the magnetic properties of spinel Zn-Mg ferrite

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ABSTRACT

Polycrystalline spinel ferrite powders of $\text{Zn}_2\text{Mg}_2\text{Fe}_4\text{O}_{12}$ ($x = 0.0, 0.4, 0.8$, and $1.0$) have been synthesized by solid-state reaction. An antiferromagnetic Néel temperature ($T_N = 25$ K) point is observed in $\text{ZnFe}_2\text{O}_4$ while $\text{MgFe}_2\text{O}_4$ shows a strong ferromagnetism. The magnetization value of $\text{Zn}_2\text{Mg}_2\text{Fe}_4\text{O}_{12}$ increases first and then decreases with the increase of $x$. When $x = 0.8$ ($\text{Zn}_0\text{Mg}_{0.8}\text{Fe}_{0.2}\text{O}_4$), the value of the saturation magnetization ($M_s$) reaches a maximum as 85.566 emu/g. The magnetization of $\text{Zn}_2\text{Mg}_2\text{Fe}_4\text{O}_{12}$ shows a very sensitive response to the Mg$^{2+}$ concentration at the tetrahedral sites ($A$-sites) or the octahedral sites ($B$-sites). I suggest that the $A-B$ super-exchange interaction is enhanced after Mg$^{2+}$ ions substituting Zn$^{2+}$ ions.

1. Introduction

As an important member of ferrite family, spinel ferrite has unique attraction. For a long time, spinel ferrites have attracted the extensive interests due to their remarkable properties reflected in various applications, such as data storage devices [1], microwave absorbing materials [2], ferrofluid and catalysis [3,4], gas sensing materials [5–7], anode materials of lithium-ion batteries [8], magnetic hyperthermia [9–11], optical and dielectric materials [12], fast frequency response in soft magnetic materials and biomedical fields [13], etc. In recent years, the research on $\text{ZnFe}_2\text{O}_4$ ferrite is fascinating. Due to its better chemical activity and thermal stability [14], $\text{ZnFe}_2\text{O}_4$ can be used as the electro-optical devices [15], magnetic hyperthermia materials in biomedical applications [16], photo-catalytic systems [17], contrast enhancers [18], and so on. Even the latest reports claim that it can be used as the potential detection of the corona virus disease (COVID-19) by S.B. Somvanshi et al. [19]. As known, the magnetic properties of such spinel ferrites strongly depend on their chemical compositions, element occupancies and substitutions. Even small amount of ions changing in the spinel ferrites can seriously impact the magnetic properties. Therefore, it is particularly important to select suitable metal cations to substitute. As a counterpart of $\text{ZnFe}_2\text{O}_4$, the $\text{MgFe}_2\text{O}_4$ is also applicable in the electronics and biomedical areas [20,21]. Meanwhile, these two ferrites have two variant lattice structures i.e. $\text{ZnFe}_2\text{O}_4$ (normal spinel) and $\text{MgFe}_2\text{O}_4$ (inverse spinel). Thus, a formation of mixed spinel lattice structure can give rise to superior properties than their individual parts. Furthermore, by controlling the stoichiometric ratio between zinc and magnesium, the magnetization parameters of zinc-magnesium ferrite can be fine-tuned to our expected values.

However, most of the synthesized magnetic nanoparticles are found to have mixed magnetic phases, so it is very important to understand the different magnetic phases in the synthesized materials for practical application [22]. In this paper, the microstructure, electronic valence state and magnetism of Zn-Mg ferrite are comprehensively explored and evaluated. These methods can be used to synthesize Zn-Mg ferrite such as ball milling [23], sol gel [24], solid state reaction [25], hydrothermal [26] and co-precipitation [27,28], etc. Considering the factors of low cost, no pollution and easy operation, the traditional solid sintering theory is used to synthesize it here. By measuring the low-temperature magnetic properties of the spinel ferrite powders, we found that the ion occupations of zinc ions and magnesium ions in the same crystal structure are very different, thus showing very different macroscopic physical properties. Therefore, further research and analysis of this kind of ferrite has attracted great interest and extensive attention of researchers.

2. Experimental

Zn-Mg spinel ferrite ($\text{Zn}_2\text{Mg}_2\text{Fe}_4\text{O}_{12}$, $x = 0.0, 0.4, 0.8, 1.0$) particles were synthesized by the solid state reaction method. $\text{Fe}_2\text{O}_3$, ZnO and MgO (purity of 99.9% for all) were used as the raw materials and mixed according to the calculated stoichiometric ratio. Then, the mixed raw materials were placed in a high-temperature sintering furnace to be gradually heated to 1373 K and sintered at this temperature for 2 h.
specific sample preparation process is shown in Fig. 1. X-ray diffraction (XRD, Cu Kα radiation, λ = 0.15406 nm, Philips) and scanning electronic microscopy (SEM) were used to analyze the crystal structure information and reveal the surface morphology of the samples at room temperature (300 K). The prepared Zn-Mg ferrite samples were authenticated by X-ray diffraction (XRD) patterns recorded within the angle scope of 20–90°. Sweep the samples at a scanning angle of 5 degrees per minute. The temperature dependent magnetization (M–T) curves were collected on a superconducting quantum interference device (SQUID) magnetometer from quantum design (MPMS). Both zero-field-cooled (ZFC) and field-cooled (FC) magnetization curves were measured from a very low temperature 2 K to a high temperature 400 K.

**Fig. 2.** The refined XRD patterns of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ at room temperature.

### Table 1
The achieved XRD Refinement Parameters at Room Temperature and Magnetic Parameters at 5 K of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ (x = 0.0, 0.4, 0.8, 1.0).

| Composition    | $R_p$ (%) | $R_wp$ (%) | $\chi^2$ | $M_r$ (emu/g, 5 K) | $H_C$ (Oe, 5 K) | Cation distribution |
|----------------|-----------|------------|----------|-------------------|-----------------|--------------------|
| ZnFe$_2$O$_4$  | 15.274    | 19.317     | 1.012    | –                 | 17.5            | Zn$_{0.00}$Fe$_{0.00}$[Zn$_{0.00}$Fe$_{0.00}$]O$_4$ |
| Zn$_{0.0}$Mg$_{0.0}$Fe$_2$O$_4$ | 17.265 | 24.803     | 1.060    | –                 | 100             | Zn$_{0.00}$Mg$_{0.00}$Fe$_{0.00}$[Zn$_{0.00}$Mg$_{0.00}$Fe$_{0.00}$]O$_4$ |
| Zn$_{0.2}$Mg$_{0.8}$Fe$_2$O$_4$ | 18.552 | 25.814     | 1.061    | 85.566            | 20              | Zn$_{0.00}$Mg$_{0.00}$Fe$_{0.00}$[Zn$_{0.00}$Mg$_{0.00}$Fe$_{0.00}$]O$_4$ |
| MgFe$_2$O$_4$  | 18.984    | 24.534     | 1.064    | 60.452            | 15              | Mg$_{0.00}$Fe$_{0.00}$[Mg$_{0.00}$Fe$_{0.00}$]O$_4$ |
3. Results and discussion

3.1. Structural analysis

The Rietveld refined XRD patterns for all the samples of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$, ($x = 0.0, 0.4, 0.8, 1.0$) spinel ferrite are shown in Fig. 2. All samples show the single phase of the standard face centered cubic structure (space group: Fd-3 m, 227). In order to fully investigate the structural information of Zn-Mg spinel ferrite, the values of the credible parameters ($R_p$, $R_wp$, and $\chi^2$) are obtained from the Rietveld refined XRD patterns of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$, ($x = 0.0, 0.4, 0.8, 1.0$) at room temperature. The detailed refinement parameters are shown in Table 1. As mentioned above, the samples obtained from our experiment are in accordance with the calculated stoichiometric ratio and is intrinsically effective.

In order to facilitate the understanding of their magnetic properties, the crystal structure of the samples and the occupancy of different ions therein need to be further analyzed and discussed. As shown as Fig. 3, the positions occupied by all ions in the unit cell and the positions occupied by substituted ions (metal cations) are clearly displayed. As we known, the formula unit of a cubic spinel ferrite can be expressed as $\left(A^{2+}\right)^{2}\left(Fe^{3+}\right)^{3}A^{2+}\left.A^{3+}\right)$, representing that the tetrahedral sites (A-sites) are occupied by the metal cations in parentheses and the octahedral sites (B-sites) are occupied by the metal cations in square brackets[13]. When $x = 1$, the divalent metal cations only occupy tetrahedral sites, resulting in spinel ferrite being a normal spinel ferrite. However, the divalent metal cations only occupy octahedral sites when $x = 0$, then spinel ferrite is an inverse spinel ferrite. If $0 < x < 1$, divalent metal cations occupy both tetrahedral and octahedral sites. At this time, spinel ferrite is in a state between normal spinel ferrite and inverse spinel ferrite[29, 30]. The distribution law of metal cations is generally related to factors such as ion radius, electron layer structure, ion valence bond balance and ion ordering, etc. Besides, it also depends on the heat treatment of ferrite[31, 32]. For example, metal cations such as Zn$^{2+}$ and Ni$^{2+}$ have exclusive A-sites and B-sites preferences[33–35], respectively. Thus, in a typical nickel ferrite, Ni$^{2+}$ cations only occupy the B-sites, forming an inverse spinel. While in a zinc ferrite, Zn$^{2+}$ cations only occupies the A-sites, forming a normal spinel. However, Mg$^{2+}$ ions occupy both the A-sites and B-sites in a spinel ferrite[1,13,29,32]. The variational proportion of cations distribution in A-sites and B-sites will greatly affect the structure, electricity and magnetism of spinel ferrite[29,32].

Fig. 4 shows the scanning electron microscope of the surface morphology of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.0, 0.4, 0.8$ and $1.0$). The pictures of (a), (b), (c) and (d) correspond to $x = 0.0, 0.4, 0.8$ and $1.0$, respectively. It can be seen from the Fig. 4 that the particle sizes of the obtained samples do not change significantly with the increase of the substitution amount of Mg$^{2+}$ ions for Zn$^{2+}$ ions. Meanwhile, the average particle size is mainly concentrated between 200 nm and 400 nm. This
fully shows that the samples obtained from the solid-phase reaction theory used in our experiment can also reach the level of nanometer size. The polyhedral morphology of these particles is due to the macroscopic stacking effect caused by the crystal structure of tetrahedron and octahedron structure. As mentioned above, the samples obtained from my experiments have good macro-structure morphology and standard Zn-Mg spinel crystal structure.

3.2. X-ray photoelectron spectroscopy

The presence of various elemental species and the oxidation state in the sample can be obtained by X-ray photoelectron spectroscopy (XPS), which is an important characterisation technique. The number of unpaired electrons, determined by the oxidation state, governs the magnetic properties of these ferrite materials. The XPS spectra for the Zn$_{0.6}$Mg$_{0.4}$Fe$_2$O$_4$ particles are displayed in Fig. 5. The full scan plot substantiates the presence of the elements Zn, Mg, Fe, and O as shown as Fig. 5(a). All the individual elemental spectra are fitted using XPS peak fitting software. In Fig. 5(b), the Mg1s peak is observed at 1304.1 eV with Mg$^{2+}$ in Zn$_{0.6}$Mg$_{0.4}$Fe$_2$O$_4$ ferrite. The lower energy peak attributed to $A$-site Mg$^{2+}$ and higher energy peak to $B$-site Mg$^{2+}$ [36]. It can be inferred from our experimental results that magnesium ions occupy $B$-site and force some iron into $A$-site. Fig. 5(c) displays the Zn2p peak with two distinct peaks at 1021.7 eV and 1045 eV corresponding to Zn2p$_{3/2}$ and Zn2p$_{1/2}$, respectively. This result confirms the valence state of zinc to be +2 oxidation state. The XPS spectrum for Fe2p, depicting two distinct peaks at 711.3 eV for Fe2p$_{3/2}$ and 725.3 eV for Fe2p$_{1/2}$, suggest the valence state of iron to be Fe$^{3+}$ with no Fe$^{2+}$. And the presence of a satellite peak of Fe2p$_{3/2}$ at around 719.0 eV, above the main peak, has confirmed it [36,37]. This is enough to show that the magnetism of experimental sample particles is caused by the change of iron oxidation state and the occupancy rate of different cations.
3.3. Thermogravimetric analysis (TGA)

In order to investigate the impact of the heat treatment on Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ samples, the thermal response recorded by TGA has been analyzed. The thermogravimetric (TG) curves for Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ have been displayed in Fig. 6. When $x = 0.0$, the weight loss is less than one percent, which has occurred in a very slow manner with the increase of temperature. For $x = 0.4$, 0.8, and 1.0 samples, the initial weight loss is occurred in very slow manner and further the sudden weight loss appears within a short range of temperature. The maximum value of weight loss are about 1.5 %, 2% and 2% when $x = 0.4$, 0.8, and 1.0 respectively. The maximum value and saturation point of weight loss reveal the formation of spinel phase with a second order phase transition, which is usually called as ferritization temperature [38]. For the Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ samples, the ferritization temperature is considered as 282.8 °C, 304.8 °C, and 341.4 °C for $x = 0.4$, 0.8, and 1.0 respectively. Above these temperature points, very little weight loss with the increase of temperature indicates the complete formation of spinel phase and a better thermal stability for higher temperature region. Considering that these samples belong to magnetic ferrite materials, it is necessary to study their low-temperature magnetic behaviors and the magnetic temperature-dependent magnetization curves are helpful for us to understand the weight loss of them with the change of temperature. The temperature-dependent magnetization curves are useful for us to understand their low-temperature magnetic behaviors and the magnetic phase transition process.

3.4. Magnetisation

Fig. 7 (a)–(d) show the magnetization curves of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.0$, 0.4, 0.8 and 1.0) under ZFC/FC processes ($M_{ZFC}/M_{FC}$) depending on the temperature from 5 K to 400 K. Here, in order to measure the temperature-dependent magnetization of the samples, the applied external field strength is $H = 100$ Oe. In Fig. 7 (a), the $M_{ZFC}$ curve and $M_{FC}$ curve of ZnFe$_2$O$_4$ show the peak due to strong magnetic interactions at low temperature of 25 K. It indicates that there is an antiferromagnetic super-exchange interaction caused by the reverse magnetic moment of Fe$^{3+}$ ions in the B-sites of ZnFe$_2$O$_4$ [39]. Meanwhile, the $M_{ZFC}$ curve and $M_{FC}$ curve of ZnFe$_2$O$_4$ show slight inversion due to the small particle size by the preparation techniques when the particle size decreases to nanoscale [40]. The spin-glass-like behavior may appear in the field of nanomagnetism when the size of magnetic particles decreases into nanometer-sized scale. From Fig. 7 (a)–(d), the $M_{ZFC}$ curve and $M_{FC}$ curve of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.4$, 0.8 and 1.0) are very different from that of ZnFe$_2$O$_4$. With the increasing of magnesium ions replacing zinc ions, the ferromagnetic interaction in the system is gradually strengthened, and the process of ferromagnetic and antiferromagnetic competition appears. It can be seen from Fig. 7 (c) that when $x = 0.8$, the magnetic susceptibility of the sample reaches the maximum. The competition between ferromagnetism and antiferromagnetism first strengthens and then weakens, and the system gradually enters a state where ferromagnetism dominates. As the temperature rise, the magnetization of MgFe$_2$O$_4$ increases slowly. And the spins of ions are frozen in their own easy-axes at a lower temperature, while the spin-like glass state will be destroyed by the thermal motion with the rise of temperature [40]. Compared with ZnFe$_2$O$_4$, Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.4$, 0.8 and 1.0) not only show B-B interactions, but also show stronger A-A interactions and A-B interactions. The $M_{ZFC}$ curve and $M_{FC}$ curve of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.4$, 0.8 and 1.0) show obvious bifurcation, which indicate that the samples show strong temperature-dependent magnetic behaviors. It can be clearly seen that Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.4$, 0.8 and 1.0) have large magnetization values, while the magnetization value of ZnFe$_2$O$_4$ basically approaches zero. This fully implies that the substitution of magnesium ions for zinc ions will lead to great changes in the magnetic properties of polycrystalline powder samples.

Fig. 8 (a) and (b) shows the magnetic hysteresis ($M$–$H$) loops of Zn$_{1-x}$Mg$_x$Fe$_2$O$_4$ ($x = 0.0$, 0.4, 0.8, and 1.0) at 5 K and 300 K, respectively.
Interestingly, the magnetization value of the sample increases first and then decreases with the increase of \( x \) when \( x = 0.8 \) \((\text{Zn}_{0.2}\text{Mg}_{0.8}\text{Fe}_{2}O_{4})\), the value of \( M_r \) reaches a maximum as 85.566 emu/g. As shown as Fig. 8 (c), the intrinsic coercivity \((H_c)\) of the sample also shows a change rule of increasing first and then decreasing at 5 K. All relevant values are listed in Table 1. Fig. 8(b) reveals that \( \text{ZnFe}_2\text{O}_4 \) shows a fully paramagnetic state at 300 K, while the ‘S’-like shape of hysteresis loops at 5 K as shown as Fig. 8(a). This phenomenon shows that due to the small size of the powder, zinc ions do not occupy all A-sites, and very few occupy B-sites.

In bulk \( \text{ZnFe}_2\text{O}_4 \) ferrite, \( \text{Zn}^{2+} \) ions fully fill A-sites and \( \text{Fe}^{3+} \) ions fully fill B-sites, respectively. Herein there is no A-B interaction and the negative B-B interaction caused by \( \text{Fe}^{3+} \) ions just has antiparallel moment resulting no net moment [24]. After \( \text{Mg}^{2+} \) ions substitute \( \text{Zn}^{2+} \) ions, \( \text{Mg}^{2+} \) ions can enters B-sites, forcing part of \( \text{Fe}^{3+} \) ions originally occupying B-sites to A-sites. \( \text{Fe}^{3+} \) ions concentration at A-sites, A-A interaction, A-B super-exchange interaction are enhanced and B-B interaction is weakened [41]. For this reason, the magnetization of \( \text{Zn}_{1-x}\text{Mg}_{x}\text{Fe}_2\text{O}_4 \) \((x = 0.0, 0.4, 0.8, 1.0)\) will increases with the increase of \( x \) and increased up to \( x = 0.8 \). As mentioned above, \( \text{Mg}^{2+} \) ions have no moment as same as \( \text{Zn}^{2+} \) ions. A-B super-exchange interaction and B-B interaction mainly contribute to \( \text{Fe}^{3+} \) ions. As the substitution amount of \( \text{Mg}^{2+} \) ions continues to increase, the \( \text{Fe}^{3+} \) ions in the B-sites enter the A-sites more and more, and then the \( \text{Fe}^{3+} \) ions in the B-sites decrease, which will eventually lead to the A-B interaction instead beginning to decrease. When \( \text{Mg}^{2+} \) ions continuously substitute \( \text{Zn}^{2+} \) ions, antiferromagnetism decays all the time, and ferromagnetism goes through the process of increasing at first and then decreasing, all of which are due to the change of magnetic \( \text{Fe}^{3+} \) ions occupation. The results of \( H_c \) shown in Fig. 8(c) also confirm this conclusion. Finally, the \( M_r \) of \( \text{Zn}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4 \) will decrease for \( x > 0.8 \), which is attributed to the decrease of \( \text{Fe}^{3+} \) ions of B-sites [24].

4. Conclusions

In conclusion, the magnetic properties of \( \text{Zn}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4 \) \((x = 0.0, 0.4, 0.8, 1.0)\) have been systematically investigated. The results of XPS, the curves of \( M-T \) and \((M-H)\) suggest that the substitution of \( \text{Mg}^{2+} \) ions for \( \text{Zn}^{2+} \) ions will cause \( \text{Fe}^{3+} \) ions to change from the original B-sites to the A-sites. Thus strengthened A-B super-exchange interaction and weaken B-B interaction. With the increase of \( x \), the ferromagnetism of the sample system increases and the antiferromagnetism decreases gradually. The magnetization value of \( \text{Zn}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4 \) \((x = 0.0, 0.4, 0.8, 1.0)\) increases first and then decreases with the increase of \( x \). when \( x = 0.8 \) \((\text{Zn}_{0.2}\text{Mg}_{0.8}\text{Fe}_{2}O_{4})\), the value of \( M_r \) reaches a maximum as 85.566 emu/g. The magnetic properties of \( \text{Zn}_{1-x}\text{Mg}_x\text{Fe}_2\text{O}_4 \) \((x = 0.0, 0.4, 0.8, 1.0)\) change obviously and show strong sensitivity to \( \text{Mg}^{2+} \) ions due to the different occupancy preference of \( \text{Mg}^{2+} \) and \( \text{Zn}^{2+} \) ions.

Declaration of Competing Interest

The authors reported no declarations of interest

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