Contents lists available at ScienceDirect

### Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

## Magnetic hysteresis loss crossover in Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrite

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#### ARTICLE INFO

Article history: Received 3 June 2015 Received in revised form 20 October 2015 Accepted 10 November 2015 Available online 1 December 2015

Keywords: Ni—Zn ferrites Magnetic hysteresis loss Crossover

#### ABSTRACT

 $Ni_{0.4}Zn_{0.6}Fe_2O_4$  and  $Ni_{0.4}Zn_{0.6}Fe_{1.95}Ti_{0.05}O_4$  ferrites were prepared by solid state reaction method and their magnetic properties were investigated. It was found that there is a magnetic hysteresis loss crossover around  $B_m = 4$  mT, the hysteresis loss coefficient from  $5.5 \times 10^{-2}$  T<sup>-1</sup> in  $B_m < 4$  mT jump down to  $4.0 \times 10^{-2}$  T<sup>-1</sup> in  $B_m > 4$  mT in  $Ni_{0.4}Zn_{0.6}Fe_{1.95}Ti_{0.05}O_4$  sample by measuring field dependence of relative loss factor. This crossover was verified by measuring field dependence of real part permeability and magnetic loops. The possible mechanism of this crossover was discussed, and it was regarded that the crossover was due to magnetic domain wall pinning by Ti cation doped in ferrite.

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#### 1. Introduction

High electrical resistivity, high cutoff frequency and low magnetic losses make Ni-Zn ferrite an obvious choice as a magnetic core material for high frequency application. Many works have shown that the properties of Ni-Zn ferrite were sensitive to the preparation method, chemical composition, microstructure, type and amount of additives [1,2]. Many oxides and compounds were added in soft ferrites to improve magnetic properties of material [3-7], and TiO<sub>2</sub> was not a popular oxide additive. TiO<sub>2</sub> was often doped into Mn–Zn ferrites to decrease magnetic loss and improve temperature stability of permeability [8]. There were some works on the properties of Ni–Zn ferrites doped with TiO<sub>2</sub> [9–14], and most of those works were investigated on microstructure and magnetic properties of ferrite [9-12], and some works on electrical and dielectric properties of ferrite [13,14]. Due to the lack of studies on the power loss characteristic of TiO<sub>2</sub> substituted Ni–Zn ferrites, systematic investigations of loss properties in those ferrites are demanding. We found an abnormal of the hysteresis loss in x = 0.05ferrite when the magnetic loss characteristic of the Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2-</sub> <sub>x</sub>Ti<sub>x</sub>O<sub>4</sub> ferrites was investigated. In this paper, novel hysteresis loss crossover behavior in Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrite was reported.

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#### 2. Experimental

Reagent grade Ni<sub>2</sub>O<sub>3</sub>, ZnO, TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> were taken in required proportion with chemical formula Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> and Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub>, the mixtures were milled thoroughly in agate mortar and then calcined at 900 °C for 3 h in air. The resultants were reground to reduce them into small crystallites. The obtained powders were pressed in a toroid shape in 20 MPa pressure using 5% PVA as binder. The pressed toroid samples were sintered for 3 h at 1220 °C in air followed by natural cooling to room temperature. The crystal structure of obtained ferrite samples was checked by X-ray diffractometer (Mac Science MPX18AHF, Cu K<sub> $\alpha$ </sub> radiation), and their magnetic properties were measured by BH analyzer (Iwatsu SY-8258).

#### 3. Results and discussions

Powder XRD patterns of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> and Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>-Ti<sub>0.05</sub>O<sub>4</sub> ferrites were shown in Fig. 1, the XRD patterns indicated that only spinel phase was detected in two samples, and no other phases were detectable. In the insert of Fig. 1, the details of (4 4 0) diffraction peak were displayed, it can be found that Ti<sup>4+</sup> substitution made the peak slightly shift to lower angle, which reveals that the lattice has a little expansion with Ti<sup>4+</sup> substitution [11]. When one Fe<sup>3+</sup> is substituted by Ti<sup>4+</sup>, another Fe<sup>3+</sup> becomes Fe<sup>2+</sup> due to charge balance. The sum of Ti<sup>4+</sup>(0.069 nm) and Fe<sup>2+</sup>(0.083 nm) radius was bigger than two Fe<sup>3+</sup>(0.067 nm) radius,





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Fig. 1. Powder XRD patterns of Ni\_{0.4}Zn\_{0.6}Fe\_2O\_4 and Ni\_{0.4}Zn\_{0.6}Fe\_{1.95}Ti\_{0.05}O\_4 ferrites. The details of (4 4 0) peak was shown in the insert of the figure.

relatively large cation substitution leads to lattice expansion. Expansion of lattice with substitution indicated that Ti<sup>4+</sup> was achieved on crystallographic sites. The initial permeability  $\mu_i$  of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> sample is 247, while  $\mu_i = 404$  for Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>-Ti<sub>0.05</sub>O<sub>4</sub> ferrite, which has about 60% increase than the former.  $\mu_i$  is inversely proportional to the magnitude of magnetic anisotropy constant  $K_1$ , and  $K_1$  of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> sample is negative. Small amount Ti<sup>4+</sup> substitution increases Fe<sup>2+</sup> cation concentration in ferrite and Fe<sup>2+</sup> has a positive contribution to  $K_1$ , which caused the decrease magnitude of  $K_1$  and lead to  $\mu_i$  increase.

The magnetic flux density ( $B_m$ ) dependence of power loss of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> and Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrites in 100 kHz was shown in Fig. 2, it could be found that the power loss of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrite was just about 1/5 of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> sample, which indicated that moderate substitution of Ti<sup>4+</sup> in Ni–Zn ferrites could dramatically decrease power loss of sample. The magnetic flux density dependence of relative loss factor (tan $\delta$ / $\mu$ ') in 100 kHz at room temperature was measured in order to



Fig. 2. Magnetic flux density dependence of the power loss of  $\rm Ni_{0.4}Zn_{0.6}Fe_2O_4$  and  $\rm Ni_{0.4}Zn_{0.6}Fe_{1.95}Ti_{0.05}O_4$  ferrites at 100 kHz.

analyze power loss mechanism in Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrite, the measured data was shown in Fig. 3. As a comparison, data measured for Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> sample under the same condition were shown in the insert of Fig. 3. It was found that the data in the insert of Fig. 3 obey a simple linearity, which was consistent with the Legg formula

$$\frac{\tan\delta}{\mu'} = \frac{1}{2\pi} (aB_m + ef + C) \tag{1}$$

where *f* is frequency, *a*, *e* and *C* are hysteresis loss coefficient, eddy current loss coefficient and residual loss coefficient, respectively. However, the experimental results for Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> shown in Fig. 3 indicated that the data could be fitted with two straight lines with different slopes which intersect around  $B_{\rm m} = 4$  mT, rather than a straight line just as Eq. (1). It could be found that the slope of the fitting line below 4 mT was  $5.5 \times 10^{-2}$  T<sup>-1</sup>, which was rather larger than  $4.0 \times 10^{-2}$  T<sup>-1</sup> of up 4 mT ranges. The Legg formula tells the slope represent hysteresis loss coefficient, the data shown in Fig. 3 revealed that the hysteresis loss has a crossover around  $B_{\rm m} = 4$  mT, from a rather large hysteresis loss coefficient *a* below 4 mT to a small *a* up 4 mT range.

One may argue that the kink around  $B_m = 4 \text{ mT}$  in Fig. 3 may be caused by experimental errors, more experimental evidences should be given to verify the observed crossover in magnetic loss measurement. It was known that Eq. (1) was valid only in low applied magnetic field; the field range was called Rayleigh range. In Rayleigh range, field dependence of real part of permeability satisfies

$$\mu' = \mu_{\rm i} + \eta {\rm H} \tag{2}$$

Here  $\mu_i$  is initial permeability and  $\eta$  is Rayleigh constant. The hysteresis loss coefficient *a* is proportion to  $\eta$  [14], so that the measured  $\mu'$  vs *H* should be constituted with two straight line with different slopes if the observed hysteresis loss crossover in Fig. 3 is intrinsic. The measured field dependence of  $\mu'$  data of Ni<sub>0.4</sub>Zn<sub>0.6</sub>-Fe<sub>1.95</sub>Ti<sub>0.05</sub>O<sub>4</sub> ferrite were shown in Fig. 4, and the  $\mu'$  vs *H* data of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2.04</sub> sample were also shown in the insert of Fig. 4 as a comparison. The  $\mu'$  data in the same field was measured five times and taken the average value into account in order to eliminate random errors in measurement. It was found that experimental



**Fig. 3.** Magnetic flux density dependence of relative loss factor of  $Ni_{0.4}Zn_{0.6}Fe_{1.95-}$ Ti<sub>0.05</sub>O<sub>4</sub> ferrite in 100 kHz at room temperature, the solid line was the fitted result with Eq. (1). The same data for  $Ni_{0.4}Zn_{0.6}Fe_2O_4$  ferrite were shown in the insert of the figure.

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