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Effects of SnO_2 addition on the microstructure and magnetic properties of NiZn ferrites

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ABSTRACT

The microstructure and magnetic properties of SnO_2-doped NiZn ferrites prepared by a solid-state reaction method have been investigated. Due to its low melting point (~1127 °C), moderate SnO_2 enhanced mass transfer and sintering by forming liquid phase, which accelerated the grain growth. However, excessive SnO_2 producing much of liquid phase retarded mass transfer and sintering, leading to a decrease in grain size. The diffraction intensity of the samples doped with SnO_2 addition was stronger than that of the sample without addition. The lattice constant initially decreased up to a content of 0.10 wt% and showed an increase at higher content up to 0.50 wt%. The initial permeability (μ_i) initially increased up to a content of 0.15 wt% and showed a decrease at higher content up to 0.50 wt%; however, losses (P_L) measured at 50 kHz and 150 mT changed contrarily. Both saturation induction (B_S) and Curie temperature (T_C) decreased gradually with increasing SnO_2. Finally, the sample doped with 0.10–0.15 wt% SnO_2 showed the higher permeability and lower losses.

1. Introduction

The downsizing of various electronic equipments has been possible due to the improved performances of transformers, inductors, choke coils and other electromagnetic components. Yet further miniaturization of components is in demand by the debut of super lightweight equipment including LCD, PDP and PDA. However, super lightweight equipment needs miniature DC–DC converters, inductors for filter and signal amplifying power inductors in circuit. Thus, it is necessary to realize miniaturization of electromagnetic components. At present, MnZn ferrites have been widely used in the mini DC–DC converters and inductors [1], because of their high saturation induction (B_S) and low losses (P_L). However, NiZn ferrites offer better miniaturization prospects for they show high electrical resistivity and can miniaturize magnetic components without a bobbin [2–5].

Besides chemical compositions [6] and sintering process [6–10], addition performs vitally here for it influences the microstructure and magnetic properties of NiZn ferrites. Recently, various additions, like $MnCO_3$ [11,12], MnO_2 [4], Cr_2O_3 [13], PbO [14], TiO_2 [15], WO_3 [16], V_2O_5 [17], etc., have been reported to obtain NiZn ferrites of high performance. In this work, the effects of SnO_2 addition on the microstructure and magnetic properties of NiZn ferrites are discussed.

2. Experimental procedures

NiZn ferrite, as a nominal composition of $Ni_{0.361}Zn_{0.639}Fe_{1.996}O_4$, was prepared by a solid-state reaction method. The analytical grade Fe_2O_3, NiO and ZnO were weighed following the composition and mixed for 2 h. After dried, the mixed oxide powders were homogenized and calcined at 950 °C in air for 2 h. The different contents of SnO_2 addition were 0, 0.05, 0.10, 0.15, 0.20, 0.25 and 0.50 wt%, corresponding labels were Sn000, Sn005, Sn010, Sn015, Sn020, Sn025 and Sn050, respectively. The calcined powders and SnO_2 addition were milled in deionized water for 6 h. After being further dried, the resulting ferrite powders were granulated with 8% polyvinyl alcohol. Then it was pressed into toroidal shapes with the dimensions of outer diameter = 20 mm, inner diameter = 10 mm and height = 7 mm. In the end, the samples were sintered at 1280 °C in air for 3 h and left to cool inside the furnace to the room temperature.

X-ray diffractograms of the samples were recorded using an X-ray diffractometer with Cu Kα radiation. The microstructure of the samples was observed by scanning electron microscopy (SEM). From enlarged SEM micrographs of the samples, average grain sizes (D), by applying the average value of 5 micrographs to each sample, were estimated by intercept method. The initial permeability (μ_i) was measured by TH2828 LCR meter at the frequency of 10 kHz. Curie temperature (T_C) of the cores was determined from inductance fading temperature. P_L and B_S of the specimens were measured by SY-8232 B-H analyzer, densities (d_b) by Archimedean method.

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3. Results and discussion

3.1. Microstructural and structural properties

Fig. 1 shows the typical SEM micrographs of NiZn ferrites doped with different SnO_2 contents. It is found that average grain size of the samples slightly increases with increasing SnO_2, maximizes at 0.10–0.15 wt%, and then decreases (see Table 1). The homogenous and dense microstructure tends to set in as the content of SnO_2 is 0.10–0.15 wt%. The standard deviation of grain size of all the samples ranges from 0.09 to 0.12 μm. The bulk density (d_b) and porosity (P) at various contents of SnO_2 are summarized in Table 1. It is observed that the density increases up to a content of 0.10 wt% and then decreases at higher content up to 0.50 wt%, however, porosity varies contrarily.

The microstructure of SnO_2-doped NiZn ferrites here suggests that SnO_2 should be a sintering flux in the ferrite during sintering at high temperature. The melting point of SnO_2 (~1127 °C) is lower than the sintering temperature (1280 °C) of ferrite. Thus moderate SnO_2 can form liquid phase during sintering, which consequently enhances mass transfer and sintering due to solid-state solubilization and segregation. Grains grow larger and grain boundaries become more evident, leading to an enhancement in densification and a decrease in porosity (see Table 1). However, excessive SnO_2 may result in much liquid-phase distributing on the surface of particles. Solid-state reaction among the particles attached excessive liquid phase is prevented, and as a result, the grain growth is suppressed and small grain occurs. Therefore, pores cannot be eliminated thoughtfully (see Fig. 1(d) and Table 1).

The typical XRD patterns of NiZn ferrites doped with different SnO_2 contents are shown in Fig. 2. It is observed that the patterns match well with the characteristic reflections of single-phase cubic spinel structure, and diffraction intensity of the samples doped with SnO_2 addition is stronger than that of the sample without addition. It is obvious that SnO_2 enhances the solid-state reaction by means of liquid phase sintering mechanism, thus the amount of spinel phase increases and the arrangement of crystal cells inside the grains becomes more regular.

The lattice constant (a) of NiZn ferrites doped with different SnO_2 contents is listed in Table 1. It implies that lattice constant

Table 1
The microstructural and structural properties of NiZn ferrites doped with different SnO_2 contents: lattice constant (a), bulk density (d_b), porosity (P), average grain size (D) and standard deviation of grain size (S_d)

No.	a (Å)	d_b (g/cm^3)	P (%)	D (μm)	S_d (μm)
Sn000	8.4118	5.18	3.00	5.26	0.11
Sn005	8.4076	5.21	2.25	5.68	0.10
Sn010	8.4038	5.26	1.50	6.24	0.09
Sn015	8.4049	5.22	2.25	6.13	0.10
Sn020	8.4085	5.16	3.19	4.23	0.12
Sn025	8.4102	5.10	4.32	4.14	0.11
Sn050	8.4118	5.03	5.45	3.49	0.09

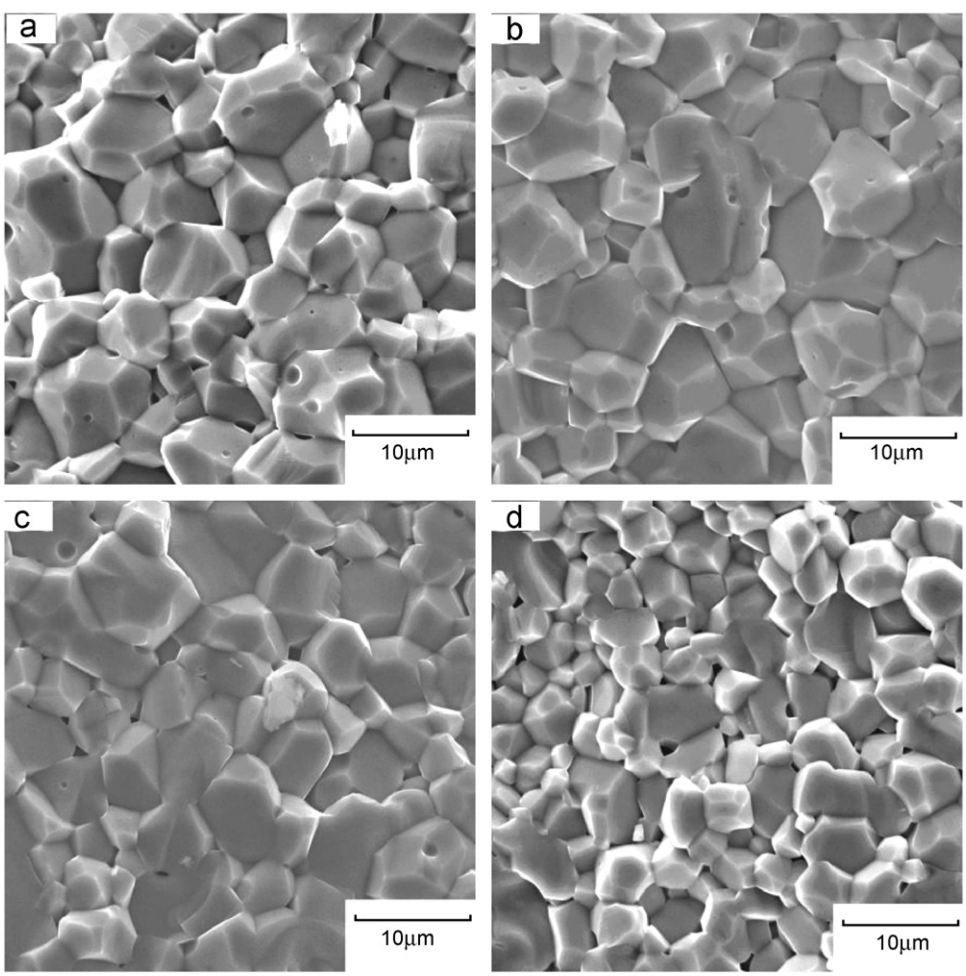

Fig. 1. Typical SEM micrographs of SnO_2-doped NiZn ferrites: (a) $w(SnO_2) = 0$, (b) $w(SnO_2) = 0.10$ wt%, (c) $w(SnO_2) = 0.15$ wt% and (d) $w(SnO_2) = 0.50$ wt%.