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I.-N. Lin, R.K. Mishra, and G. Thomas

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Controlled Atmosphere Annealing of High Permeability

Manganese Zinc Ferrite

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Abstract

Annealing of sintered $(\text{Mn,Zn})\text{Fe}_2\text{O}_4$ in a controlled atmosphere improves the apparent electrical resistivity through the reduction of the ferrous ion content. The initial permeability, however, is degraded as a result of annealing. It is suggested that stringent control of the oxygen partial pressure at an earlier stage of processing rather than post sintering annealing is necessary to raise the intrinsic electrical resistivity of the bulk materials by reducing the ferrous ion concentration without affecting the magnetic permeability detrimentally.

Introduction

The initial permeability and electrical resistivity of MnZn-ferrites are microstructure dependent and hence sensitive to the processing variables [1]. Over the years, researchers have been able to improve these properties by adjusting the chemical compositions and optimising the processing parameters [2,5]. Recently, emphasis has been placed on understanding the influence of grain boundaries on the electrical and magnetic properties and engineering the grain boundaries to improve these properties [6-8]. Additives such as CaO have been shown to preferentially segregate at the grain boundaries, probably forming an insulating layer between the grains [6-9]. These additives improve the d.c. electrical resistivity of the bulk sample but lower the initial permeability [6]. At high frequencies (over 1 MHz), the insulating layer is rendered useless due to the space charge polarisation effect. In the materials with no CaO addition, permeabilities of ~20,000 have been attained [6] but generally such materials have poor electrical resistivity. Thus it is necessary to increase the bulk resistivity of MnZn-ferrite grains rather than that of the grain boundary layers to prepare ferrites with high electrical resistivity and good soft magnetic properties.

The basic electron conduction mechanisms in ferrites have been studied by many investigators [10-12] and reviewed by Klinger et al. [13]. Among the various models proposed, such as the standard band theory model, a small-polaron mechanism, and the thermally activated hopping model, the latter model has been well applied to explain qualitatively the electrical behavior of MnZn-ferrites. The extra electron on a ferrous (Fe^{2+}) ion requires little energy to move to an adjacent ferric (Fe^{3+}) ion sitting in a similar interstitial site. Under the influence of the electric field, these extra electrons, hopping from one iron ion to the next, constitute the electrical conduction. The ferrous ions are thus acting as donors in this semiconducting material. The minimization of the divalent iron content in MnZn-ferrite is, therefore, a basic approach to increase the intrinsic resistivity of the bulk materials. Furthermore, it eliminates side effects such as the localized strain induced by CaO additions.

The equilibrium Fe^{2+} content in MnZn-ferrite is known to be sensitive to the oxygen partial pressure of the environment at high temperatures [14]. Control of the amount of divalent iron in polycrystalline material can thereby be achieved by annealing the specimens in an atmosphere with controlled P_{O_2} level. Specimens having low resistivity (high divalent iron content) were annealed in a controlled atmosphere to reduce the Fe^{2+} content. The results of the heat treatment, the effect of annealing upon electrical and magnetic properties, and the significance of these observations are discussed in this paper.

Experiment

Commercial grade MnZn-ferrite of nominal composition $\text{MnO}:\text{ZnO}:\text{Fe}_2\text{O}_3 = 26.9:19.8:53.3$ (mole%) containing no added impurities [6] were used for all the annealing experiments. A few samples of the same approximate composition but containing ~2500ppm of CaO were also used in the study for comparison purposes. These two materials with and without CaO will be referred to as type A and type B materials respectively in the text. Magnetic measurements were made on toroidal samples of

dimensions 4.76mm i.d, 9.52mm o.d. and 1.5mm thickness. Electrical resistivity was measured on rectangular samples of dimensions 2.67mm x 5.5mm x 10.53mm. In both cases, the ac impedances were measured by an HP gain-phase meter. The initial permeability (μ') and magnetic loss (μ'') [15] or the apparent resistivity (ρ_p) and dielectric constant (ϵ_p) [16] were calculated from the measured impedances.

The annealing experiments were done in a platinum tube furnace at 1100°C. The oxygen partial pressure was varied by adjusting the flow rate of the inlet N₂ and O₂ gases and the oxygen pressure was monitored before and after the gas mixture reacted with the specimens. The samples were held at the annealing temperature for eight hours followed by cooling at a rate of 200°C/min in the flowing gas (at const. P_{O_2}). The ferrous ion content of the specimens was determined by titration in deoxygenated conc. HCl in an inert atmosphere [17].

Results and Discussion

(a) Effect of annealing on ferrous (Fe^{2+}) ion content. As shown in Fig. 1, the ferrous ion content of the specimens changes as a result of annealing. It decreases when a high oxygen partial pressure (P_{O_2}) is used for annealing; a low oxygen partial pressure results in increased ferrous ion concentration. The Fe^{2+} concentration of an unannealed specimen, labelled S in the figure, is included for comparison. The oxidation of ferrous ions is more sensitive to the P_{O_2} level in the low Fe^{2+} region than in the high Fe^{2+} region. Only a small change in the degree of oxidation results from a large change of the P_{O_2} level in the high Fe^{2+} region, while a change in the degree of oxidation is produced by the same change of the P_{O_2} level in the low Fe^{2+} region. The control of divalent iron content at a low level is more difficult; stringent control of the P_{O_2} level during annealing is necessary in this region.

A model for the oxidation-reduction equilibria in MnZn-ferrite has been proposed by

Morineau [18]. In the model, equilibrium equations between ions, defects and oxygen partial pressure are suggested, taking into account the site preference of different cations. The model fits their data very well [19]. An $\text{Fe}^{2+}/\text{P}_{\text{O}_2}$ response curve corresponding to 1100°C annealing temperature obtained by the same authors [14] using thermogravimetric measurement is shown as a dashed line in Fig. 1. The trends in the change of ferrous ion content with P_{O_2} levels in their study are similar to those reported here. Discrepancies arise mainly from the difference in specimen composition and morphology. The specimens used in this study contain smaller amounts of MnO and are polycrystalline. The presence of multivalent Mn ions affects the oxidation-reduction behavior of iron ions [20] and the reaction in polycrystalline materials is much slower than in powder due to the slow diffusion through the sintered specimens.

The annealing temperature can also affect the degree of oxidation of the heat treated specimens. For a constant oxygen partial pressure, the ferrous ion content is higher for a specimen annealed at higher temperature (light dashed line in Fig. 1). It also has been observed that there exists a phase boundary corresponding to each annealing temperature, indicated as the shaded area in Fig. 1. The secondary phases such as Fe_2O_3 (hematite) or Mn_2O_3 might precipitate if the P_{O_2} level of the annealing atmosphere is higher than that of the phase boundary. When the annealing temperature increases, the critical P_{O_2} level also increases and the critical divalent ion content decreases, as indicated by the arrows in Fig. 1.

The consequence of this behavior is that the divalent iron content of the specimen will decrease when it is cooled from high temperature with oxygen partial pressure kept at a constant level. In the extreme case where the Fe^{2+} content of the specimen is too low, the phase boundary will be encountered during cooling and either precipitation will occur or the high temperature structure will remain as a metastable phase at room temperature. Both enhanced oxidation and precipitation of the secondary phase will have detrimental

effects of the magnetic properties of the material; both are likely to occur preferentially along grain boundaries in polycrystalline specimens. This will be discussed shortly.

(b) Effects of annealing on electrical resistivity. The apparent resistivities (ρ_p) of the annealed type B specimens are plotted versus operating frequencies (the dispersions) and are shown in Fig. 2 as solid lines with the annealing P_{O_2} level indicated. The dispersion curves of unannealed type A and type B materials are also included for the convenience of comparison and are plotted as dotted and dash-dotted lines respectively. In general, a higher apparent resistivity (ρ_p) was obtained for the specimen annealed in a higher P_{O_2} level. The resistivity has been raised as much as two orders of magnitude for the specimen annealed in 7000ppm oxygen partial pressure. It is even higher than the resistivity of MnZn-ferrite (type A). On the other hand, the resistivity is lowered when the specimen is annealed in a P_{O_2} level lower than 300ppm.

The increase in apparent resistivity as a result of annealing is attributed to the oxidation of ferrous (Fe^{2+}) ions to ferric (Fe^{3+}) ions, while the decrease in apparent resistivity is ascribed to the reverse reaction, i.e. reduction of ferric ions to ferrous ions. The variation of apparent resistivity with divalent iron content is shown in Fig. 3 for low and moderately high operating frequencies. The data labelled Sa and Sb are the resistivities of type A and type B untreated materials respectively. The accompanying question marks (?) indicate that the data are included only for comparing the resistivity of the material, while the amount of Fe^{2+} contained in the specimen is not the same as the corresponding value represented by the datum point. The behavior by which the apparent resistivity of the specimen increases as the amount of divalent iron decreases, is in agreement with the hopping model of electrical conduction in MnZn-ferrite [13]. The ferrous ions can be considered as donors containing an extra electron, which will jump to the adjacent ferric ions easily and constitute the electrical conduction. The oxidation of divalent iron by annealing reduces the donor concentration in the material such that the electrical resistivity increases.

The apparent resistivity drops off in the high frequency regime (Fig. 2) and is more prominent for high resistivity material. This behavior is similar to that of type A, and is an indication that the annealed type B material consists of low resistivity bulk materials separated by high resistivity layers, presumably the grain boundary phase. Space charge polarization occurs at high operating frequencies and short circuits the high resistivity layers causing the apparent resistivity to drop off rapidly.

The electrical behavior of the grain boundary layers [6,9] can be separated from the bulk materials by the complex-impedance technique [16,21]. Analysis of the impedance data of the specimens using the above-mentioned technique shows that both the bulk and grain boundary resistances of the annealed specimens increase as the Fe^{2+} concentration decreases (Fig. 4). The improvement of the electrical behavior due to annealing is much more prominent in the grain boundary phases than in the bulk materials. This marked difference is even more clearly demonstrated when the specific resistance (resistivity) of the two phases are plotted against divalent iron content, as shown in Fig. 5. The grain boundary resistivity is seen to be at least three orders of magnitude higher than that of the bulk resistivity, and it increases much more rapidly as the ferrous ion content of the specimen decreases. The fact that the grain boundary phases contain less divalent iron than the bulk materials as a result of annealing is strongly implied. Therefore it can be concluded that preferential oxidation along grain boundaries occurs during annealing.

The dielectric constant of the grain boundary phases, as analyzed by the complex impedance technique, does not change appreciably with the specimens and is in the range of $15 \epsilon_0$ to $40 \epsilon_0$, where ϵ_0 is the dielectric constant of free space. The resistivity of the bulk materials is much lower than that of the grain boundary phases (Fig. 5). These observations are consistent with the assumptions made in modelling the electrical response of the polycrystalline MnZn-ferrite [16,21], that is, the grain boundary phases are thin layers of high resistivity materials while the bulk materials are low resistivity materials.

(c) Effect of annealing on magnetic properties. A high magnetic permeability (μ') and low magnetic loss (μ'') are the two most important magnetic properties desired for MnZn-ferrite. While the eddy current loss is improved due to the increase in resistivity, the magnetic permeability of the material is, unfortunately, lowered as a result of controlled atmosphere annealing (Fig. 6a). The $\tan \delta$, which describes the ratios of dissipated energy to stored energy, and the magnetic loss (μ'') are improved by annealing (Fig. 6b,c).

In the figures showing the dispersion of magnetic properties, the curves are plotted as solid lines, with the P_{O_2} level indicated, for annealed samples and are plotted as dotted and dash-dotted lines for unannealed type A and type B specimens respectively. The magnetic permeability (μ'), loss (μ'') and $\tan \delta$ are all decreased as a result of annealing; the amount of change is larger for specimens heat treated at higher oxygen partial pressures. In other words, all the magnetic responses are suppressed when the ferrous ion concentration in the specimens is reduced. The dependence of these magnetic properties on the divalent iron content is shown in Fig. 7. The magnetic permeabilities (μ') are plotted as solid lines for two operating frequencies (1 KHz and 100 KHz); the maximum magnetic loss ($\mu''_{\max}$) is plotted as a dash-dotted line; and the $\tan \delta$ is shown as a dotted line for 6 Meg.Hz operating frequency.

It is interesting to notice that for the specimen annealed in 4800ppm oxygen partial pressure, all the resultant magnetic properties are the same as those of the unannealed type A material, within the error of measurement. The effect of annealing on the grain boundary structure/composition is thus suggested to be similar to that of CaO addition. The degree of modification of the grain boundary characteristics and properties can, however, be controlled by using the appropriate P_{O_2} level of the annealing atmosphere.

While the dependence of electrical resistivity on ferrous ion content can be well explained by the electron hopping mechanism, the dependence of magnetic permeability

on the divalent iron content is less clear and a mechanism which can predict this behavior is not well-established. Although there are reports concerning the variation of permeability with Fe^{2+} concentration [15,22,23], the observations are inconclusive. The degradation of the permeability as a result of annealing observed in this study can, however, be explained by preferential oxidation of ferrous ions at regions near grain boundaries. In polycrystalline materials, the diffusion of ionic species along grain boundaries is known to be much faster than that in the bulk [24]. The grain boundary regions are, therefore, expected to be oxidized more readily when sintered MnZn-ferrite specimens are annealed in a controlled atmosphere. Since the formation of cationic vacancies is inevitable when oxidation takes place in the spinel lattice, as more anions are bound to ferric ions than to ferrous ions, a dilation of lattice parameter (a_0) in the oxidized regions is expected [25]. Localized strain will be induced when there are regions which are oxidized preferentially to their adjacent regions [26]. The magnetic anisotropy energy in those regions will thus be increased and domain wall mobility lowered.

The preferential oxidation of grain boundary phases is enhanced when the oxygen partial pressure is kept at a constant level during cooling. The equilibrium Fe^{2+} concentration is decreasing with the temperature and diffusion is fast enough to reach equilibrium only in the grain boundary region. This will lead to a higher magnetic anisotropy energy in the regions near the grain boundaries. Although the separate measurement of divalent iron content in the bulk material and grain boundary phase is practically impossible by current techniques, the fact that the grain boundary phase contains fewer ferrous ions is strongly implied by the overwhelmingly high resistivity of the grain boundary phase. The decrease in permeability due to annealing can, therefore, be ascribed to localized elastic strains in the regions near the grain boundaries induced by preferential oxidation.

Conclusion

Controlled atmosphere annealing is capable of improving the apparent resistivity (ρ_p) of the MnZn-ferrite sintered material through the reduction of the ferrous ion content. The annealing effect is more pronounced on grain boundary phases than on bulk materials. The magnetic permeability (μ') is, however, degraded due to annealing and is ascribed to the preferential oxidation along grain boundaries in polycrystalline specimens.

The electrical resistivity of the materials could be improved without seriously deteriorating the magnetic permeability if the inhomogeneity in oxidation could be eliminated. In other words, a stringent control of the oxygen partial pressure at an earlier stage of processing rather than post fabrication annealing is called for, in order to raise the intrinsic electrical resistivity of the bulk materials by reducing Fe^{2+} concentration and not affect the magnetic permeability severely.

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Figure Captions

- Fig. 1. Oxidation/reduction of ferrous (Fe^{2+}) ions in type B MnZn-ferrite caused by controlled atmosphere annealing. The divalent iron content decreases as either the P_{O_2} level of annealing atmosphere increases or as the annealing temperature decreases for the same P_{O_2} level. Precipitation might occur when the P_{O_2} level exceeds the phase boundary limit.
- Fig. 2. Dispersions (frequency dependence) of apparent electrical resistivity for annealed and unannealed MnZn-ferrite specimens. A higher resistivity is obtained for a specimen annealed in a higher P_{O_2} level. The resistivities drop off in the high operating frequency regime due to the grain boundary capacitance effect.
- Fig. 3. Fe^{2+} dependence of apparent electrical resistivity of the same materials as in Fig. 2. The resistivities are higher for specimens containing smaller amounts of ferrous (Fe^{2+}) ions. The annealing atmospheres are indicated in Fig. 2.
- Fig. 4. Fe^{2+} dependence of grain boundary and bulk resistances. They are comparable in magnitude and increase as the amount of divalent iron decreases. The change of grain boundary resistance is more pronounced as a result of annealing.
- Fig. 5. Fe^{2+} dependence of grain boundary and bulk resistivity. The grain boundary resistivity is overwhelmingly higher than the bulk resistivity and it increases more rapidly.
- Fig. 6. Dispersions of (a) magnetic permeability μ' , (b) loss μ'' , and (c) $\tan \delta$ for annealed and unannealed MnZn-ferrite. The magnetic properties decrease as the annealing P_{O_2} level is increased. Vertical scale is only relative.
- Fig. 7. Fe^{2+} dependence of magnetic permeability (at 1KHz and 100 KHz), maximum magnetic loss and $\tan \delta$ (at 6MHz) of the same specimens as Fig. 6. The magnetic properties decrease with the content of ferrous (Fe^{2+}) ions.

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Fig. 1

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Fig. 2

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Fig. 3

Fig. 4

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Fig. 5

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Fig. 6a

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Fig. 6b

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Fig. 6c

Fig. 7

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