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The Dependence of Magnetic and Mechanical Properties on Composition and Processing in Air Fired MnZn Ferrites

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Abstract: The relationship between magnetic and mechanical properties has been studied as a function of composition, atmosphere and temperature for a series of MnZn ferrites. When processed in air, strength and permeability are critically dependent upon the formation of Mn^{2+} and Fe^{2+} on sintering, and residual stresses introduced via subsequent (re)oxidation upon cooling. In Fe deficient ferrites, properties are determined by the reoxidation of Mn^{2+} , which introduces residual tensile stresses on cooling, so that low strength/high μ_i are observed. In the Fe excess region, reoxidation of Fe^{2+} on cooling introduces compressive stresses, resulting in high strength/low μ_i . Both strength and permeability increase with increasing sintering temperature in Fe excess materials. Cooling in N_2 prevents reoxidation, and results in increased permeability for Fe excess ferrites.

1. INTRODUCTION

MnZn ferrites used as deflection yokes in T.V./monitor applications, differ in two important areas from ferrite grades which are used for power or inductor applications. Precursor materials are less pure, so that the final ferrite compositions can contain up to 1.0 wt% of a (glassy) grain boundary phase based upon CaO and SiO_2 ; in addition yoke ring grades are processed entirely (i.e. sintered and cooled) in air. Previous investigations have shown that, for both MnZn [1] and MgZnMn [2] ferrites processed in air, strength is determined primarily by the extent of formation of Mn^{2+} relative to Fe^{2+} on sintering, and the stresses introduced via (re)oxidation on cooling. This investigation was carried out in order to assess if the mechanisms which control strength would also determine magnetic properties in air processed MnZn ferrites.

2. EXPERIMENTAL

A series of Mn ferrite compositions were prepared via standard laboratory conditions, ranging from Fe deficient to Fe excess relative to stoichiometry. For Fe deficient grades, the general composition was according to $\text{Mn}_{0.6}\text{Zn}_{0.4}\text{Fe}_x\text{O}_{1+1.5x}$, where x was varied from 1.7 to 2.0. In the Fe excess region, the Fe:Mn ratio was varied according to $\text{Mn}_{0.65-x}\text{Zn}_{0.35}\text{Fe}_{2+x}\text{O}_4$, where x ranged from 0.06 to 0.14. Starting materials were Mn_2O_3 , ZnO, and Fe_2O_3 , dry mixed with sinter aids (CaO and SiO_2 , added in the range 0.1-1.0 wt%), pre-fired (900°C , 2 hours), ball milled (2kg, 15 hours), and granulated with 0.4 wt% PVA binder. Toroids (27mm O.D. x 16mm I.D. x 8mm) were pressed to a (dry) density of $2,800 \pm 50 \text{ kg.m}^{-3}$ and sintered at temperatures in the range 1210 to 1350°C , (heating/cooling rates of 155°C/hr , 1 hour at peak temperature). Sinter atmospheres were air, followed by air or N_2 cooling. Magnetic/electrical measurements on fired toroids included saturation flux density (B_{sat} , 800A.m⁻¹, 0.1Hz, 25°C), initial permeability (μ_i , $<0.1\text{mT}$, 1kHz, 25°C), and resistivity ($\Omega\text{.m}$, d.c., 25°C). Strengths (diametral compression to produce tensile failure) were also measured on sintered toroids.

3. RESULTS AND DISCUSSION

Fig 1 shows the relationship between permeability and strength as a function of Fe content for ferrites which range from Fe deficient to Fe excess relative to stoichiometry. Low strength/high permeability are observed upto $[\text{Fe}] = 2$, after which σ increases whilst μ_i decreases for compositions in the Fe excess region (shown in detail in Fig 2). Electrical resistivity decreases from $10^4\Omega\text{.m}$ for Fe deficient materials to $1\Omega\text{.m}$ for Fe excess compositions, whilst B_{sat} increases with increasing

Fe content, from 265 to ~370 mT across the range of compositions studied. Microstructural observations show that hematite (α -Fe₂O₃) is precipitated on cooling from the spinel phase in the Fe excess region. These results (Figs 1 & 2) are explained

Figure 1: μ_i and strength as a function of Fe content for $\text{Mn}_{0.6}\text{Zn}_{0.4}\text{Fe}_x\text{O}_{1+1.5x}$ (1270°C)

Figure 2: μ_i and strength as a function of Fe (relative to Mn content) for $\text{Mn}_{0.65-x}\text{Zn}_{0.37}\text{Fe}_{2+x}\text{O}_4$ (1270°C)

by the reoxidation on cooling of Fe²⁺ to Fe³⁺ with the formation of cation vacancies and an accompanying decrease in lattice constant [3,4]. This contraction introduces residual compressive stresses on cooling which enhance σ and decrease μ_i . In the Fe deficient region, the reoxidation of Mn²⁺ to Mn³⁺ predominates and results in an expansion which gives rise to residual tensile stresses, decreasing σ and increasing μ_i , in accordance with the observed results. These mechanisms are discussed in detail in a previous publication [1]. For any given composition in the Fe excess region, σ and μ_i increase with increasing sinter temperature (Fig 3), due respectively to increased Fe²⁺→Fe³⁺ formation (promotes σ) and increased grain size (enhances μ_i). If reoxidation is prevented via cooling in N₂, permeability is further increased (Fig 4).

Figure 3: μ_i and strength as a function of sinter temperature for $\text{Mn}_{0.57}\text{Zn}_{0.37}\text{Fe}_{2.08}\text{O}_4$ (1270°C)

Figure 4: μ -T curves for $\text{Mn}_{0.57}\text{Zn}_{0.35}\text{Fe}_{2.08}\text{O}_4$ fired at 1350°C and cooled under different atmospheres

4. CONCLUSIONS

For the series of air processed MnZn ferrites studied, strength and permeability are determined by the reoxidation of Mn²⁺ relative to Fe²⁺ on cooling. Low strength/high μ_i are observed in Fe deficient materials; high strength/low μ_i is observed in Fe excess compositions. Both strength and permeability increase with sinter temperature for a given composition.

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