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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 $\mu_i$ are observed. In the Fe excess region, reoxidation of Fe$^{2+}$ on cooling introduces compressive stresses, resulting in high strength/low $\mu_i$. 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 Mn$_{6-x}$Zn$_x$Fe$_{2x}$O$_{3+0.5x}$, where $x$ was varied from 1.7 to 2.0. In the Fe excess region, the Fe:Mn ratio was varied according to Mn$_{0.65-x}$Zn$_{3.3x}$Fe$_{2x+0.5x}$O$_6$, where $x$ ranged from 0.06 to 0.14. Starting materials were Mn$_2$O$_3$, ZnO, and Fe$_2$O$_3$, dry mixed with sinter aids (CaO and SiO$_2$, added in the range 0.1-1.0 wt%), prefired (900°C, 2 hours), ball milled (1 kg, 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 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 ($B_s$, 800A.m$^{-1}$,0.1Hz,25°C), initial permeability ($\mu_i$, <0.1mT,1kHz,25°C), and resistivity (Ω.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 up to [Fe] =2, after which $\sigma$ increases whilst $\mu_i$ decreases for compositions in the Fe excess region (shown in detail in Fig 2). Electrical resistivity decreases from $10^4$Ω.m for Fe deficient materials to 1Ω.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 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. 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, 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 increase with increasing sinter temperature (Fig 3), due respectively to increased Fe²⁺→Fe³⁺ formation (promotes σ) and increased grain size (enhances μ). If reoxidation is prevented via cooling in N₂, permeability is further increased (Fig 4).

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 are observed in Fe deficient materials; high strength/low μ is observed in Fe excess compositions. Both strength and permeability increase with sinter temperature for a given composition.

References