



The role of prefiring in the development of Mn–Zn spinel ferrites for inductive power transfer

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Abstract

As soft magnetic materials prevail in the implementation of shielded planar coils with high inductance for wireless power transfer applications, the fabrication of efficient flat magnetic ceramics has become a critical objective. To this end, we explore the effects of the calcination top temperature (T_{CALC}), during the first firing cycle of the solid state reaction method, on the pressing characteristics of ferrite granules and the magnetic properties of sintered Mn–Zn ferrites. The composition of the prefired powders was qualitatively and quantitatively analysed by means of X-ray diffraction, as a function of T_{CALC} . This has revealed that, the altering phase composition determines the non-linear dependence of the particle size distribution in the milled mixture on T_{CALC} . Further characterization of the powders and the respective granulates has shown that, the reactivity is basically reduced and compressibility increases with T_{CALC} . However, certain local differentiations from these trends occur, on account of the varying ratio of fine to coarse particles. Thus, an explanation model is proposed to integrate the relation between the calcination degree and the monitored properties. Moreover, the sintered cores were investigated in terms of their microstructure and magnetic properties. The optimal combination of high flux density (550 mT at 1200 A/m, 10 kHz, 25 °C) and low power loss density (130 mW/cm³ at 100 kHz, 100 mT, 25 °C) is thus achieved by the powder prefired at 850 °C, in conjunction with the low required compaction force, high sintered density and homogeneous density distribution.

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1. Introduction

The principles of inductive power transfer are already utilized to achieve the contactless power supply or charging of a wide variety of systems, extending from robotic platforms, biomedical systems and industrial sensors to electronic appliances, hand-held devices and electric vehicles [1–4]. In these applications, flat magnetic cores or sheets are usually employed for the enhancement of power transfer efficiency between the transmitting and receiving coils and for the suppression of electromagnetic interference (EMI) with the surrounding environment. To this

end, high saturation magnetic materials with low core losses are mainly required, so that they can cope with the input power with the minimum heat generation [5–6]. In specific, according to the worldwide “qi” standard, which applies to the wireless power transfer up to 5 W in the frequency range 100 kHz - 200 kHz [7], materials of the Mn–Zn ferrite family are highly suggested for use as magnetic susceptors and shielding materials.

Additionally, the versatility of the design and the miniaturization of the coils setup necessitate ferrite cores with planar geometry of high aspect ratio. Therefore, as the general objective is to form nearly net-shape compacts before sintering, the consolidation of ceramic powders or granules to produce the proper flat green body may prove a challenging task. Specifically, one of the key processing difficulties, met after the granulation process, is the achievement of high and uniform green density with the lowest possible compaction

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force [8]. In reality, any friction phenomena between the powder particles and the die walls should also be diminished, to avoid density gradients and wear of die components [9].

During the first firing process (calcination/prefiring) of the typical mixed oxide route, which is applied in ferrite production, the ferrite lattice is formed, the powders coarsen considerably and the driving force for sintering is established [10–11]. Consequently, some critical material aspects are determined at this stage, which affect the ensuing powder pressing behaviour and the final magnetic performance. Moreover, as any changes of this heating cycle are relatively easy to incorporate in large-scale production, we have selected to optimize the calcination degree of a Mn-Zn ferrite composition, with regard to its magnetic and compaction properties.

2. Material and methods

The present study was carried out on Mn-Zn ferrites with the nominal chemical composition $\text{Mn}_{0.7}\text{Zn}_{0.2}\text{Fe}_{2.1}\text{O}_4$, which have been fabricated with the conventional solid state reaction process. Initially, the appropriate amounts of the precursor oxides Fe_2O_3 , Mn_3O_4 and ZnO were mixed for 9 h in distilled water, dried overnight and calcined in air at various top temperatures for 4 h (T_{CALC} : 700 °C to 1100 °C with 50 °C intervals). The calcined powder mixtures were further wet-pulverized in a ball mill for 9 h, dried and subsequently roll-granulated with the addition of a plasticizer (10 wt% aqueous solution of polyvinyl alcohol). The granulated samples were then pressed under axial compaction (100 MPa) to form toroidal samples (outer/inner diameter: 14 mm/9 mm) and finally sintered at 1325 °C for 3 h under controlled oxygen partial pressure. Especially during the cooling stage, the equilibrium relation between oxygen partial pressure and temperature was followed to ensure phase stability, as it was first proposed by Morineau and Paulus [12].

During the preparation process, samples were taken from different stages and examined with various characterization techniques. Specifically, the phase compositions of the as-calcined and as-sintered materials were qualitatively and quantitatively analysed as a function of T_{CALC} , by means of X-ray diffraction. To this effect, the respective X-ray diffractograms were obtained between 15° and 75° with 0.04° step and a 5 s dwell time at each step (Siemens, D-500, CuK α radiation). For this investigation we also applied the Rietveld refinement method, by utilizing the ICDD PDF-2 database [13,14]. Furthermore, the milled powders were characterized in terms of their particle size distribution (D_{v10} , D_{v50} , D_{v90}) with laser diffraction method (Malvern Instruments, Mastersizer-S with software version 2.17), skeletal density d_{SK} with He pycnometry (Micrometrics, AccuPyc-1330) and specific surface area s with the BET method (Quantachrome, Autosorb-1). The compaction ability of the prepared granules was investigated by testing and averaging over multiple specimens per each powder with an instrumented compaction die (KZK, PTC-03DT). This mechanical test apparatus has a cylindrical die cavity with fixed diameter of 12.8 mm. After sintering of the compacted cores, the microstructure of the specimens was observed by means of

scanning electron microscopy (JEOL, JSM6300, 20 kV). Finally, the sintered cores were evaluated by the measurement of initial permeability μ_i (100 kHz, 0.1 mT), power loss density P_V (100 kHz, 100 mT) and saturation flux density B_{SAT} (10 kHz, 1200 A/m) at 25 °C and 100 °C. The magnetic measurement setup comprises the precision LCR meter (Agilent, 4284 A), a digital oscilloscope (Tektronix, TDS 714 L), a waveform generator (Agilent, 33120 A) and a power amplifier.

3. Results and discussion

3.1. Calcined powders

3.1.1. Crystal structure and phase composition

By altering the calcination temperature of the same powder mixture from 700 °C to 1100 °C, as expected, the occurring reactions and the resulting phase composition are directly affected. Fig. 1 (a) depicts the X-ray diffractograms obtained from all the calcined powders, as a function of T_{CALC} . In particular, after prefiring at 700 °C the mixture was identified to contain the oxides Fe_2O_3 (JCPDS No.: 89-8104) and FeMnO_3 (JCPDS No.: 75-0894), zinc manganite ZnMn_2O_4 (JCPDS No.: 71-2499) and zinc ferrite ZnFe_2O_4 (JCPDS No.: 65-3111). Further increase of T_{CALC} above 750 °C and 950 °C leads gradually to the formation of MnFe_2O_4 (JCPDS No.: 88-1965) and mixed Mn-Zn ferrite phase (JCPDS No.: 89-7553), respectively. In order to quantify the described compositional changes, the obtained diffractograms were analysed with Rietveld method (Rietveld quality factors: $R_p \leq 8.1$, $R_{\text{WP}} \leq 10.5$ and $R_{\text{EXP}} \leq 8.4$). The agreement between the observed I_O and calculated I_C diffraction profiles of the powder sample prefired at 700 °C is indicatively shown in Fig. 1 (b). The quantitative analysis of diffraction data has revealed the nonlinear variation of the phases' weight fractions with T_{CALC} , as it is illustrated in

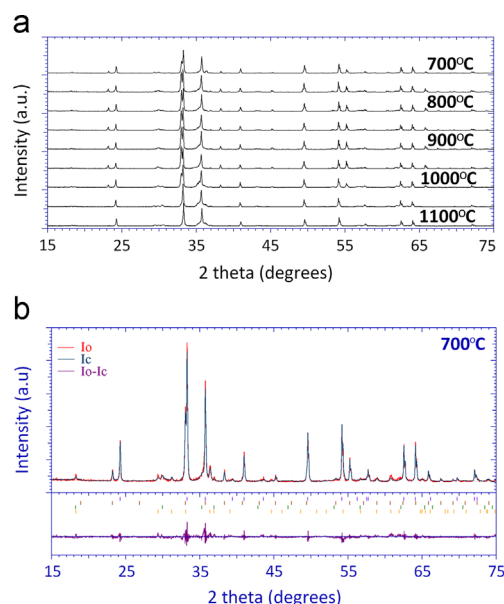


Fig. 1. (a) X-ray diffraction patterns of the powders for different T_{CALC} . (b) Observed I_O and calculated I_C XRD patterns with Rietveld refinement of the sample prefired at 700 °C.

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