Powder Injection Moulding of Mn-Zn Ferrite

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Abstract

Ceramic Injection Moulding (CIM) technology has been successfully used for the fabrication of Mn-Zn Ferrite part. The binder was composed by polypropylene and paraffin wax. The optimal powder loading (58% vol.) was determined by means of rheological measurements. Threedifferent parts, toroids, bending and tensile probes were injected. Thermal and solvent-thermal debinding was designed based on DSC and TGA studies of the binder. The time of the debinding cycle was reduced using n-heptane to dissolve previously the paraffin wax. Final properties have been determined and compared with uniaxial pressure parts values. The densities obtained were slightly higher than those of uniaxial pressure parts and the magnetic properties presented similar values.

Keywords: Mn-Zn Ferrite, CIM, PIM, rheology

1. Introduction

Manganese-Zinc ferrites are a family of soft magnetic materials with interesting magnetic properties such as high initial permeability, high electrical resistivity, which allows their use at higher frequencies (compared to other metallic soft magnetic materials) and low power losses. These ferrites are widely used at the electronic industry as inductors in general purposes and power transformers, resonant and filtering circuits and many other high-frequency (few kHz- few hundreds of MHz) electronic applications. The miniaturization of magnetic and electronic devices has demanded advanced materials with new forms and complicates shapes, so it is necessary alternative ways to the uniaxial or extrusion conventional ferrite manufacturing process.

Powder Injection Moulding (PIM), as a forming technology, offers a great number of advantages for the manufacture of metallic and ceramic components and it is widely used for the production of relatively complex and small parts. This process has been widely studied in structural ceramic[1] such as Al_2O_3 or ZrO_2 however up to now we have not found more than a few works to produce magnetic ceramics by PIM and less applied to Mn-Zn Ferrite[2,3].

In this work, the manufacturing of Mn-Zn Ferrite by Powder Injection Moulding is reported. Final density, magnetic properties and microstructure of the sintered parts are compared to the ones achieved by means of extended industrially practice.

2. Experimental and Results

The starting raw material was a granulated pre-sintered Mn-Zn Ferrite powder which is normally used for the industrial manufacturing of ferrites by uniaxial pressure. The binder was a mixture composed by polypropylene (PP), paraffin wax (PW) and stearic acid (SA). Feedstocks composition and some physical properties of the binder components are provided in Table 1.

Table 1. Feedstock composition and properties of their constituents.

	%Vol.(%wt)	ρ (g/cm^3)	T_m (ºC)
Powder	58 (88.4)	5.1	
Agglutinative	2.3 (1.1)	1.5	
PP	19.8 (5.2)	0.886	163.8
PW	18.6 (4.9)	0.887	56.8
SA	1.2 (0.36)	1.012	73.7

For the first blending all the constituents in powder form were pre-mixed in a turbula mixer during 20 minutes. Afterward the mixture was introduced into the twin-screw extruder three times to ensure good homogeneity of feedstock.

Rheological behaviour of feedstocks with different powder loading was evaluated with a capillary rheometer (Rheoflixer-ThermoHaake) between 160 and 200 ºC over a range of shear rates from 10 to 10000 s^{-1} and. The dimensions of the die were 30 mm length (L) and 1 mm diameter (D) (L/D ratio of 30).

In order to evaluate the critical powder volume concentration (CCPV) we have applied different models usually employed for fluid filled systems: Eilers, Chong, Mooney, Maron and Janardhana models. The first four can follows experimental results and overestimated the viscosity raise of the PIM mixture. Janardhana´s model provides the best result according with experimental

behaviour and CCPV was 62%vol, so we have estimated 58%vol powder the optimal loading.
Figure 1 shows the viscosity evolution with shear rate of the feedstock with 58% vol. In all cases, the viscosity decreases as the shear rate increases according to a pseudoplastic behaviour. Viscosity decreases with increasing temperature. This behavior is the most adequate for the injection process due to the viscosity of the mixture decreases when it is approached to the nozzle and when the mould is filling up the viscosity increases.

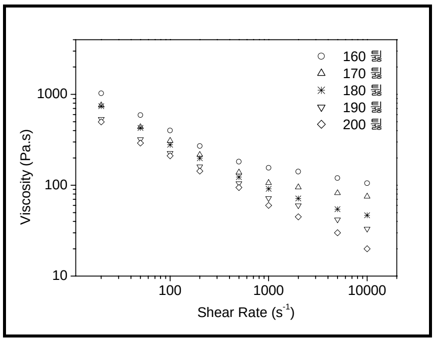

Fig. 1. Viscosity curves of feedstock with 58%vol. at different temperatures.

Flow index “n” was determined by a log-log plot of Ostwald-de Waele equation. As powder loading and temperature increase “n” decrease and the mixture is more pseudoplastic. For example, at 180 ºC, “n” changes from 0.62 to 0.45 for 48%vol to 58%vol. powder respectively.
In view of rheological results, the mixture with 58%vol powder was injected to obtain three geometry parts (toroids, bending and tensile probes).
Binder removal was carried out with two different processes, thermal and a combination of solvent-thermal debinding. The elimination of binder was optimized by means of the TGA and DSC analysis of the binder. Thermal cycle employed 23 hours for the full elimination of the binder. In order to reduce the debinding time, a combination of solvent and thermal debinding was used. The solvent was n-heptane, in which the paraffin is soluble. The temperature of the bath was 60 ºC. Afterward, the thermal decomposition of the remaining organic component takes place through a short thermal cycle considerably reduced to 9 hours. The full elimination of the polymeric part was again confirmed through LECO analysis.
Finally, a sintering study was carried out. Figure 2 shows the evolution of density with temperature. PIM parts overcome uniaxial densities and they achieve the same value (95% Th) at 1330 ºC with 40% vol. shrinkage. Initial permeability at 10 kHz follows the same behaviour than density, achieving values about 2100 at the maximum temperature (Figure 3).

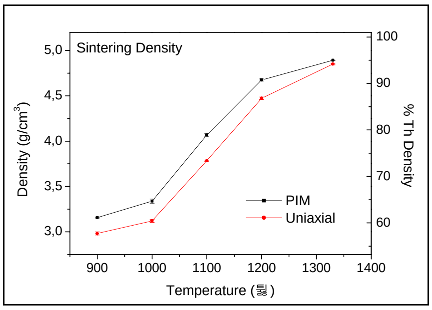

Fig. 2. Evolution of final density with sintering temperature.

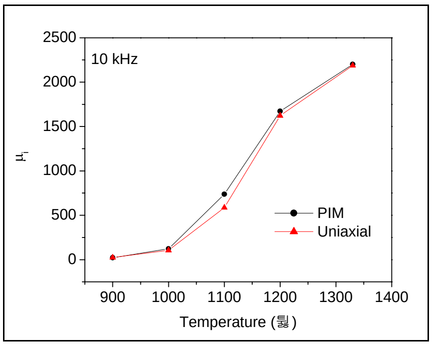

Fig. 3. Evolution of initial permeability with sintering temperature.

3. Summary

We have developed a successfully PIM process to obtain parts with expected final magnetic properties as alternative for conventional uniaxial pressure of Mn-Zn ferrite parts.

4. References

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