Journal of Korean Magnetics Society, Volume 5, Number 5, October 1995

THE EFFECT OF PROCESSING PARAMETERS ON THE MAGNETIC PROPERTIES OF Mn-Zn FERRITE FOR SMPS

J. H. SUK, K. K. JUNG, H. J. SHON and W. D. HUR
R & D LAB., Samwha Electronics Co., LTD., Osan, Korea

Abstract－The effect of processing parameters such as milling, additives and sintering atmoshpere on the magnetic properties of Mn－Zn ferrite was investigated. The experiment was followed by general ceramic fabrication process and added additives were $CaCO_3$, SiO_2, V_2O_5, ZrO_2, and Nb_2O_5. The effects of additives could be divided into three categories which were formation of liquid phase, substitution in lattice and inducing stress. Core loss among the magnetic properties was dependent mainly on the additives and also correlated with processing parameters. As a result, an optimum condition of preparing process for a high quality Mn－Zn ferrite was suggested by controlling the correlation of each processing parameters.

I. INTRODUCTION

As the electronic industry has been developed, it was eagerly required that electronic equipment should be made smaller size with high performance. To correspond with current trends of electronic equipment, reducing the core loss value was essential for making switching power supplies. Recently, new process such as Co－spray roasting has been suggested to develop low loss Mn－Zn ferrtie [1] . It is difficult to adopt a new process in view of economy or mass production. Therefore, realizing good magnetic properties using conventional process is urgently needed.

In conventional process, even though it has been well known that processing parameters affect the magnetic properties of Mn－Zn ferrite, the effects of parameter on magnetic properties were not investigated systematically yet. Among the processing parameters, additives were thought as the most important factor on magnetic properties. CaO and SiO_2 have been regarded as basic additives in Mn－Zn ferrite for application of transformer, thus there were many studies about the effects of CaO and SiO_2 [2] , [3] . Besides, it has been reported that other additives, such as V_2O_5, Nb_2O_5, ZrO_2 tend to promote magnetic properties. [1]

In this study, the effect of processing parameters such as additives, milling and sintering atmosphere on magnetic properties in Mn－Zn ferrite was simultaneously investigated. To obtain the optimum condition for lower core loss, we tried to understand the mechanism of additives and correlation of each process.

II. EXPERIMENTAL

Mn－Zn ferrite samples were prepared by conventional ceramic processing. The composition was 53 mol% Fe_2O_3, 37 mol% MnO, and 10 mol% ZnO. After calcination the powder was milled in an attrition mill for various times with additives. The basic additives, $CaCO_3$ and SiO_2, were added 0.1 wt% and 0.01 wt% at all specimens, respectively. In addition to basic additives, V_2O_5, Nb_2O_5 and ZrO_2 were added in the range of 0.01 wt% － 0.05 wt%. Pressed samples were sintered at 1350 ℃ for 4 h in various oxygen partial pressure.

Core loss was measured by an Iwatsu SY－8232 with variation of temperature. Microstructure of fractured surface was observed by SEM and K_{IC} was measured by the microindentation technique.

III. RESULTS

1. The effect of additives

Fig.1 shows the sintered density of Mn－Zn ferrite sintered at 1350 ℃ for 4 h in 4 % oxygen partial pressure. The density increased with addition of additives V_2O_5, Nb_2O_5 and ZrO_2, respectively. Among the various additives, V_2O_5

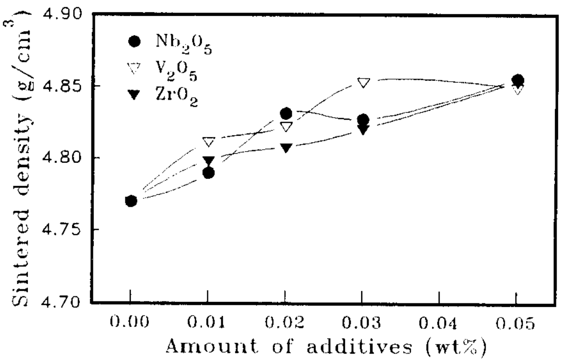

Fig. 1. The effect of additives on sintered density. The samples were sintered at 1350 ℃ in 4 % oxygen.

was the most effective sintering agent for densification of Mn−Zn ferrite system.

Fig.2 is the SEM photographs of fractured surface of specimens. As shown in Fig.2 a), when additives were CaO and SiO_2, the grain size was measured 9.7 μm and fracture mode was intergranular fracture. In the case of addition of V_2O_5, besides CaO and SiO_2, as shown in Fig.2 c), the fractographs were similar. In the case of Nb_2O_5, b), the grain growth occurred and there were a few transgranular fractures. As ZrO_2 was added, many transgranular fracture were observed in d), without variation of grain size.

Fig.3 shows the variation of K_{Ic} with addition of various additives. When V_2O_5 was added to the specimens, the K_{Ic} values of specimen slightly increased. On the contrary, the K_{Ic} decreased as adding Nb_2O_5 or ZrO_2. Especially ZrO_2 made a sharp decrease of the K_{Ic} value.

Fig.4. shows the effect of additives on core loss at 90 ℃, 2000 G and 100 kHz. Core loss

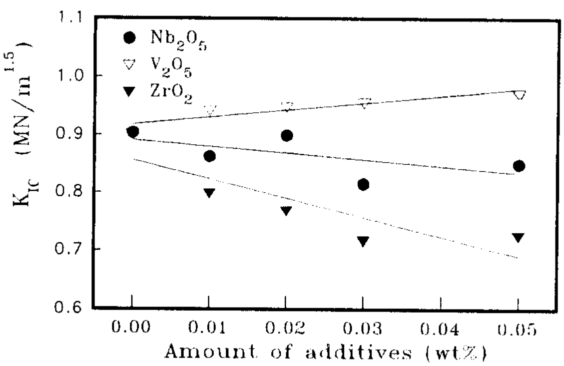

Fig. 3. The effect of additives on K_{Ic} of sintered Mn−Zn ferrite.

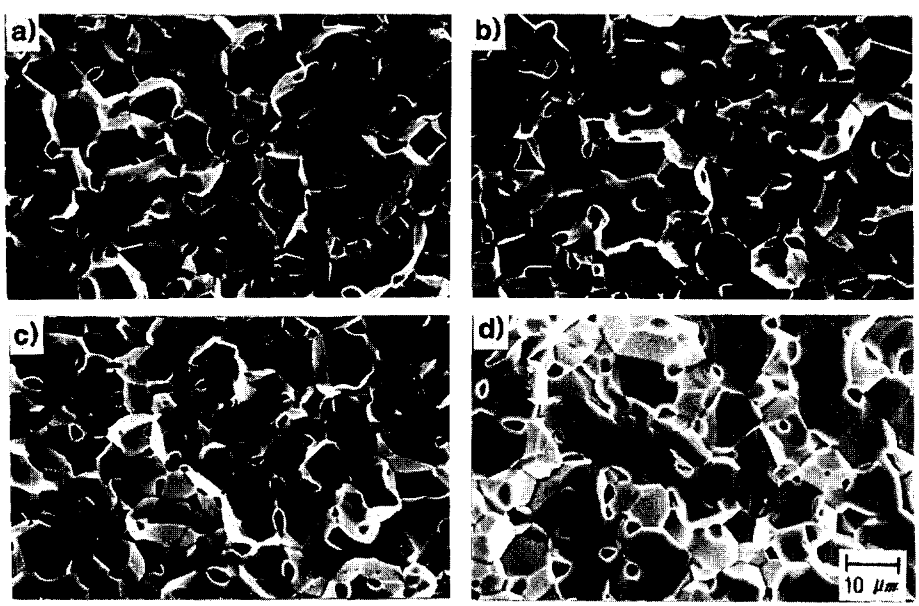

Fig. 2. The SEM photographs of fractured surface of Mn-Zn ferrite. The additives were a) 0.1 wt% $CaCO_3$ and 0.01 wt% SiO_2, b) 0.03 wt% Nb_2O_5, c) 0.03 wt% V_2O_5, and d) 0.03 wt% ZrO_2. 0.1 wt% $CaCO_3$ and 0.01 wt% SiO_2 were added to b), c) and d).

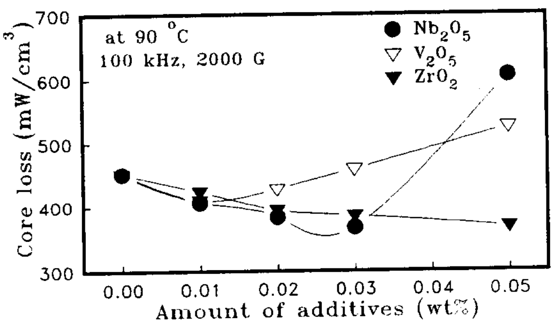

Fig. 4. The effect of additives on core loss of Mn–Zn ferrite., wt%.

was decreased with the addition of V_2O_5 up to 0.01wt%, and then core loss increased continuously when amount of V_2O_5 exceeded 0.01 wt%. With addition of Nb_2O_5 up to 0.03 wt%, core loss decreased. Further addition of Nb_2O_5 over 0.03 wt% caused core loss to increase remarkably. In the case of ZrO_2, core loss decreased, and the value of core loss was very stable in the range from 0.01 wt% to 0.05 wt%

2. The effect of oxygen partial pressure during sintering

Fig.5 shows the effect of oxygen partial pressure during sintering on the core loss. Core loss tended to decrease as atmosphere closed to oxidation condition. When the oxygen partial pressure increased up to 2 %, the core loss decreased, regardless of additives. In the case of V_2O_5 and ZrO_2, the core loss did not show a big difference between 2 % and 4 %. On the other hand, the Nb_2O_5 added sample kept decreasing the core loss with increase of Nb_2O_5 contents.

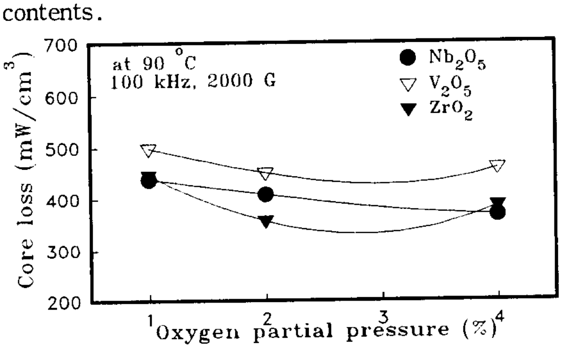

Fig. 5. The effect of oxygen partial pressure on the core loss during the sintering.

3. The effect of milling time

Fig. 6 is the effect of milling time on the core loss with variation of additives. In the case of V_2O_5, the core loss was higher than any other additives. Nb_2O_5 showed low core loss at only very narrow region and the extending milling caused core loss to deteriorate remarkably. Compared to other additives, the addition of ZrO_2 resulted in better core loss and core loss was less dependent on milling time.

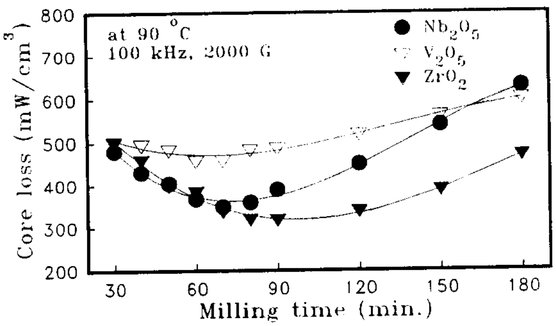

Fig. 6. The effect of milling time on the core loss with variation of additives.

IV. DISCUSSION

It is well known that V_2O_5 is melted during the sintering and then it enhances densification [4]. Liquid phase increased the resistivity of grain boundaries and decreased power loss value as shown in Fig.4. However, when V_2O_5 was added over 0.01 wt%, the power loss increased in spite of increasing density. It is due to thickening of the grain boundary layer. When the glassy phase became thickened too much in the grain boundary, it caused internal stress and the magnetic property deteriorated.

The behavior of Nb_2O_5 on Mn–Zn ferrite could be divided into two mechanisms. Firstly, enhanced densification might be liquid phase. Compared with V_2O_5, Nb_2O_5 has a higher melting temperature, 1485 ℃ and it was not expected to form liquid by itself during the sintering. But, eutetic melting might be probable. There was a report in which the enhanced densificaton was due to liquid phase formed by eutetic melting [4]. Secondly, Nb_2O_5 might be regarded as vacancy maker. When Nb_2O_5 dissolved into the spinel lattice, it could change the vacancy concentration. The densification and grain growth could be promoted by increase of volume diffusion, which resulted from increase of vacancy concentration. [5] At this time it is not clear which mechanism is dominant in Mn−Zn ferrite. The power loss tended to decrease with addition of Nb_2O_5. Finally, it worked on power loss decreasing bigger than V_2O_5 component.

Generally, the effect of tetravalent cation oxide, such as ZrO_2 on Mn−Zn ferrite was thought to be formation of cation vacancies [4] . Therefore ZrO_2 might act as a creator of cation vacancy or grain growth inhibitor. In this study, the densification was enhanced without grain growth, as shown in Fig.1 and Fig.2 and it indicates that the role of ZrO_2 might be grain growth inhibitor. It is believed that the enhanced densification without grain growth and increased resistivity resulted in the improvement of core loss. Many transgranular fractures were observed with addition of ZrO_2 and it was conventional effect of ZrO_2 in Mn−Zn ferrite. It has been proposed that grain boundaries are reinforced by segregation of ZrO_2, responsible for inducing compression stress near the grains and transgranular fracture occur [6] . On the contrary, the K_{Ic} dramatically decreased as adding the ZrO_2 in this experiment. Regarding the fracture mode, even though the effect was contrary to other reports, it is true that ZrO_2 induced stress in grain or grain boundary. The induced stress seems to result from transformation of ZrO_2 or big difference of thermal expansion coefficient between ZrO_2 and ferrite matrix. The induced stress by ZrO_2 enhanced the boundary segregation of CaO or SiO_2, which resulted in increase of resistivity. Although the mechanism of ZrO_2 was not clearly explained in the view of mechanical property, it is believed that the decreasing K_{Ic} originated from induced stress by ZrO_2.

The dependence of core loss on milling and sintering atmosphere was different form each additive. Therefore, to obtain a lower core loss material, even though it is very complex to understand the correlation of each processing parameters, all the parameters should be regarded simultaneously.

V. SUMMARY

The effect of processing parameters such as milling, additives and sintering atmoshpere on the magnetic properties of Mn−Zn ferrite was investigated. The effects of additives could be divided into three categories which were formation of liquid phase, substitution in lattice and inducing stress. Additives influenced microstructure, resistivity and other properties. Core loss among the magnetic properties was dependent mainly on the additives and also correlated with processing parameters. As a result, a high quality low loss Mn−Zn ferrite was obtained by controlling the correlation of each processing parameters (Fig.7).

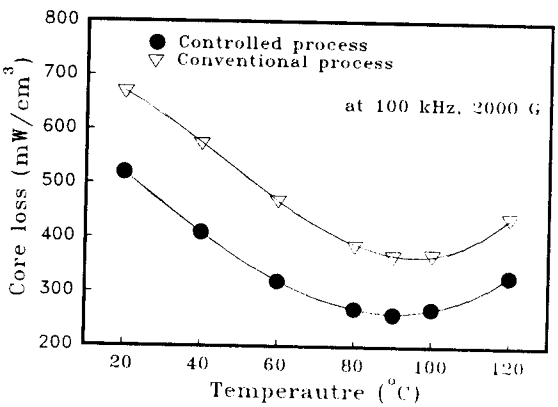

Fig. 7. Temperature characteristics of core loss with variation of process.

REFERENCES

[1] A.Goldman, Mordern Ferrite Technology, 1990.
[2] Y.Chiang et al., Proc. ICF4, 300 (1980).
[3] P.Franken et al., J. Am. Ceram. Soc., 63, 315 (1980).
[4] M.Yan et al., J. Am. Ceram. Soc., 61, 342 (1978).
[5] H.T.Kim et al., IEEE Trans. on Mag., Mag-18, 1541(1982).
[6] K.Hirota et al., Proc. ICF4, 385 (1980).