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Microstructural Control of Mn-Zn Ferrite with Addition of Seed Grains

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Abstract. : In order to obtain desired electromagnetic properties, high initial permeability and good frequency dependance of initial permeability, seeding effects are experimented in Mn-Zn ferrite system. As adding seed grain, calcined at sintering temperature, abnormal grains are disappeared clearly, but the density of sintered Mn-Zn ferrite body and initial permeability of this ferrite body are decreased. On the contrary, in case of adding seed grain which were calcined below the calcination temperature of matrix particle, abnormal grains were remain but the initial permeability is increased with proper seed content. Compared these results with those of experiments on additive variations, mean particle size controls, sintering conditions, seeding affects on the microstructure of Mn-Zn ferrite similarly.

1. Introduction

Desired magnetic properties of ferrite could be achieved by controlling microstructure. At present the control of particle size distributions, mean particle size, additions of proper additives, optimizing the sintering condition would be employed to obtain desired mechanical and electromagnetic properties. However abnormal grain growth is easily The growth of abnormal grain is linearly increased with sintering time and results in pore observed in ferrite. captivation and broad grain size distribution, consequently inhomogeneity of microstructure can degrade mechanical Especially the frequency dependence of initial permeability and the strength and electromagnetic properties. variations of initial permeability on induced magnetic field in high permeability Mn-Zn ferrite. Much effort has been devoted to investigate the behavior of abnormal grain growth and many ways have been suggested to cope with this problem[1-2]. In particular, large or small particle of seed grains were added to matrix powder in order to increase the number of abnormal grains at initial stage of sintering and then led to fine microstructure or large grain size. Small and uniform grain size is favourable to low power loss material but large grain size is favourable to high permeability materials. Therefore, addition of seed grains is applicable to the control of microstructure in sintering Mn-Zn ferrite system.

2. Experimental Procedure

In this experiment, 52.15mol% Fe₂O₃, 26.75mol% MnO, 21.10mol% ZnO were mixed with ball mill for 10hr and calcined at 950°C for 2hr. Calcined Mn-Zn ferrite materials as a matrix material were milled with attritor to its mean particle size of $1.0\,\mu$ m. As for seeding materials, the mixed Powder were calcined at 750°C, 850°C, 950°C, 1300°C for 2hr and milled with ring mill to its mean particle size of $0.5 \sim 1.0\,\mu$ m and $5 \sim 10\,\mu$ m. This seed grains are added to the matrix material at 0, 0.5, 1.0, 3.0wt% and pressed with mechanical hand press. Its green density was $2.55 - 3.00g/\text{cm}^3$. These green bodies were sintered at 1330°C for 3, 5, 7hr and annealed at 1250°C for 2hr. The electromagnetic properties of these toroidal cores were measured with HP 4194A network analyzer. And these properties were compared with those obtained from experiments to the variations of additives, particle size distributions and sintering conditions. And microstructures of these sintered Mn-Zn ferrite bodies were observed with SEM photographs after polishing with sand paper and chemical etching. From these results, the effect of seeding in the sintering process could be deduced. Also, relations of microstructures of sintered Mn-Zn ferrite bodies were body with the electromagnetic properties, initial permeability and frequency dependence were understood.

3. Results and Discussion

The mean particle size of matrix was $1.0\,\mu$ m and those of seed grains were $0.5 \sim 1.0\,\mu$ m and $5 \sim 10\,\mu$ m.

In case of adding seed grains calcined at 1300°C, the abnormal grains were disappeared and then Mn-Zn ferrite body had uniform grain size distributions. However, density and grain size were decreased. As a result, initial permeability of this sintered body was much lower than that of specimens without seed grains. But the cut off frequency of initial permeability was increased. By the way, as adding seed grains calcined below the calcination temperature of matrix particles, the abnormal grains were not disappeared. On the contrary, the abnormal grain was much larger than that of specimens without seed grains. Therefore the initial permeability of this sintered body at low frequency was slightly higher than that of specimens without seed grains, and its sintered density was increased with proper seed contents. The cut off frequency of the initial permeability, accordingly, was decreased.

From these results, the added seed grains of small or large particle size, calcined at 1300°C, retarded densifications by the scaling law. The added seed grains of small or large particle size and calcined below the calcination temperature of matrix particles had slightly higher driving force and assisted densifications at the initial stage of sintering process.

It might be proposed that the calcination temperature of seed grains affects the driving force in the initial stage of sintering(see Fig.1 b) That is, the seed grain calcined at sintering temperature has thermodynamically lower driving force in the initial stage of sintering, therefore it could be an inhibitor, which resulted in decreasing driving force for densification in the initial stage of sintering and finally retarded densification. But the seed grains calcined at below the temperature of matrix was vice versa.

As we can see at Fig. 1 a) a certain additive makes it possible to increase the initial permeability with its content only but decrease the cut off frequency of initial permeability. Also another additive can make it possible to increase the cut off frequency of initial permeability with minor change in original initial permeability.



Fig. 1 Characteristics of initial permeability (a) Frequency dependence of initial permeability for specimens manufactured at different conditions (b) Initial permeability variations according to calcination temperature of seed grains.

4. Conclusions

Seed grains of different particle size and calcined at different temperatures, have different driving force in the initial stage of sintering. Therefore these thermodynamic variations of seed grains could affect the grain growth of Mn-Zn ferrite. Microstructural control of Mn-Zn ferrite can be achieved by controlling calcination temperature and mean particle size of seed grains. These results can be applied to manufacturing of high permeability Mn-Zn ferrite material.

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