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AGGLOMERATION AND NATURE OF PARTICLE SIZE AND SOME CHARACTERISTIC PROPERTIES OF Zn-FERRITES

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ABSTRACT

Powder characterization and its influence on both sintering and quality of final product have been studied. The powder of Zn-ferrite of particle size ranging from 0.13 to 0.43mm were ball-milled at different times and characterised. All samples were dispersed in an ultrasonic bath for sedimentation test using water as dispersing agent. Dry and mixed sample powders were processed and sintered at relevant temperatures.

It was noticed that sample A (that was ball-milled for a longer period) had very high agglomeration, high density and above all partly consists of spherical aggregates. These were responsible for the comparable high quality product obtained. This implies that the nature of the starting powder and processing techniques influenced the finished product. Also the characteristic properties of the materials were greatly improved by the powder processing time. The extensive pore growth and broad open-pore size distribution observed in aggregated powder have been explained in terms of preferential sintering within the aggregates.

Key words: Aggregates, Powder, Characteristics, Particle- Size.

INTRODUCTION

The sintering behaviour and aggregation of particles of powder have great impact on the characteristics of most ferrite products {Sokol and Bromberg (1971) and kato (1976)}. This has been the subject of study by many workers such as Gallgher and Johnson; (1996) and Nomura (1979). Similarly, the effects of changes in process parameters on the magnetic properties of Ni-Zn ferries were thoroughly investigated by Owate(1993).Inspite of the recent development in the theory and practice of sintering, it is considered that little impact has been made terms of studying the influence of powder properties on both sintering performance and characteristics of the final product. Among many factors responsible for the quality of the products; sintering circle, and the state of the primary particles are highly important. Other researchers like Nomura et al (1978), Watanable et al (1980), Buchanan (1986) and Snelling (1988) have reported that the sintering of BeO and UO_2 depends upon the size and shape of the aggregate rather than on those of crystallite. Gallagher et al (1996), defined the agglomeration [1]parameter for expressing the degree of agglomeration of primary particles. The definition and methods of analysis did not appear to be sufficient for a comprehensive understanding of the sintering behaviour of most materials. It is deemed right to consider information on the nature of agglomeration as an equally important parameter in characterising the sintering behaviour of a system {Iryine et al (1990) Hokazono et al (1972), Elder et al (1992) and Snelling (1988)}. The present work consider the characterization of Zn-ferrites raw material powders. This material was chosen because of its special reference arising from its useful application in the area of magnetic core characteristics. Some importance properties for this application include eddy current loss, loss tangent, permeability, resistivity and density of product.

The effects of agregation state of primary particle on sintering as well as on the solid state reaction is given due consideration. Consequently the influence of agglomeration of particle size on the magnetic characteristics of Zn-ferrites is analysed.

EXPERIMENTAL PROCEDURE

Fe_2O_3 powders of particle size ranging from 0.13mm obtained from Satford Electrics limited, United Kingdom were ball-milled at three different selected periods of 15 hours, 10 hours and 5 hours. The sample were classified as A, B and C according to milling time and composition. The basic Zn–ferrite composition were prepared by the usual ceramic conventional methods using the mixed powder oxides of Fe_2O_3 (66.7 wt%) ZnO (15.9 wt%)CO_3O_4 (0.9 wt%) and V_2O_5 (0.3 wt%) respectively.

The aim of ball-milling at different times was to breakdown the agglomeration of aggregation state of the primary particle. Subsequent study of the

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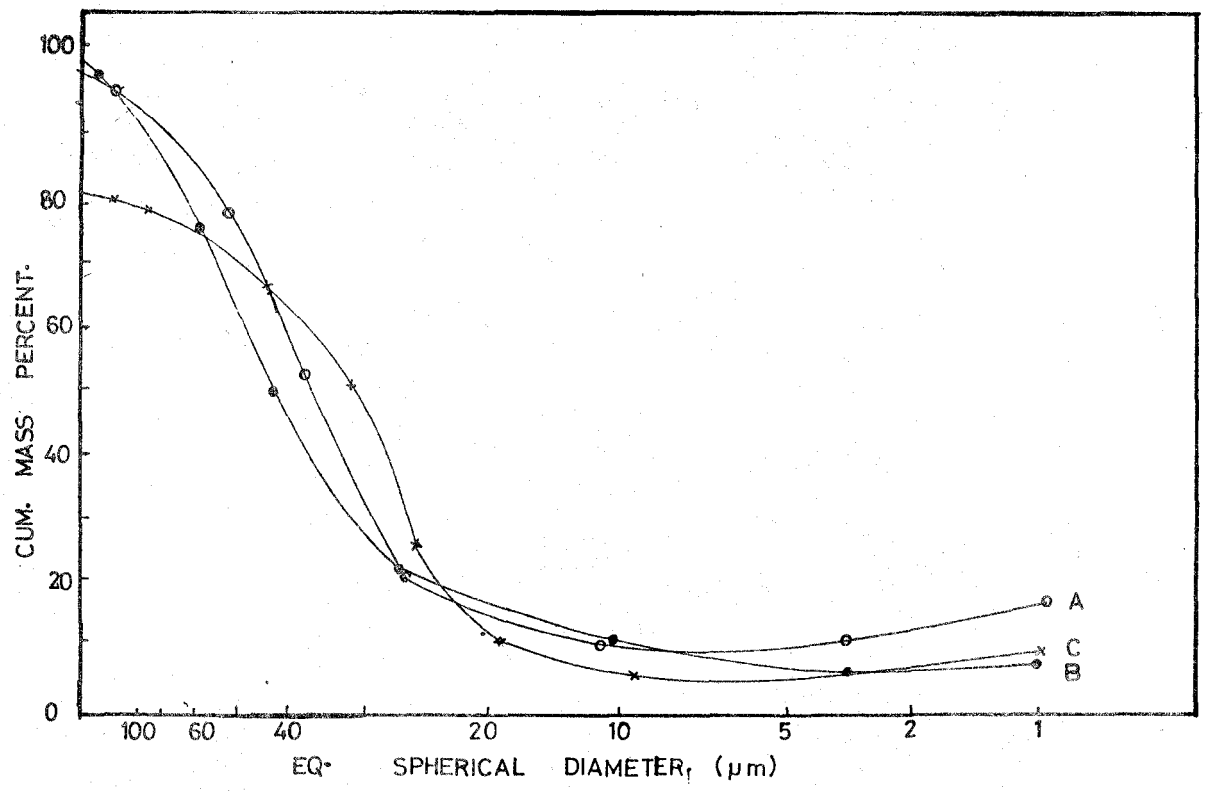

Fig.1 Particle size distribution for sample A, B and C.

TABLE I: CHARACTERISTIC DENSITY OF Zn FERRITE

Sample	Density (g/cm^3)			
	Final Density	Calcined	Green	Milling-Ball Time (Hrs)
A	5.08	3.41	1.27	15 Hrs
B	4.47	2.63	1.19	10 Hrs
C	3.77	2.06	1.37	5 Hrs

effects of particle-size distribution and aggregation state on the characteristic properties of the final product suggested the procedure All the samples were dispersed in an ultrasonic bath for sedimentation test using water as dispersing agent. The particle size distribution and densities were determined. Each mixture was wet-milled and mixed again for 30minutes using stainless steel balls, a dispersant and water in the ratio of 1:3 were now applied. The dispersant (polyacrylic acid salt) was added to the mixture to fluidise and stabilize the specimens. The mixture was then dried at 200°C far 8 hours, ball-milled again for another 3 hours and then granulated. An X-ray diffraction technique with Cu_aK radiation and a modern Philips diffractometer was employed to study the mixed powder sample and final product.

The powder for each specimens were cold pressed in a die of 20.00mm diameter and sintered using a semi-automatic furnance system. The density was calculated from the weight determined by established methods used elsewhere by Owate (1993).

RESULTS AND DISCUSSION

The particle size distribution for specimens A, B and C are presented in Figure 1 whereas the densification process and densities at the different stages of processing are shown in Fig. 2 and Table1. X-ray diffraction patterns for ZnO and Fe_2O_3 were identified in the mixed powders whereas the sintered product had the usual spinel structure of $ZnO_{0.49}$ Fe_2O_4 with a lattice constant of 8.61A. Similar methods applied in this work have been applied by Watababe (1980).

The particles sizes determined indicated the degree of aggregation. Agglomeration appear to be highest for powder A that was ball-milled for 15 hours, followed by B and C. Importantly, the particle size

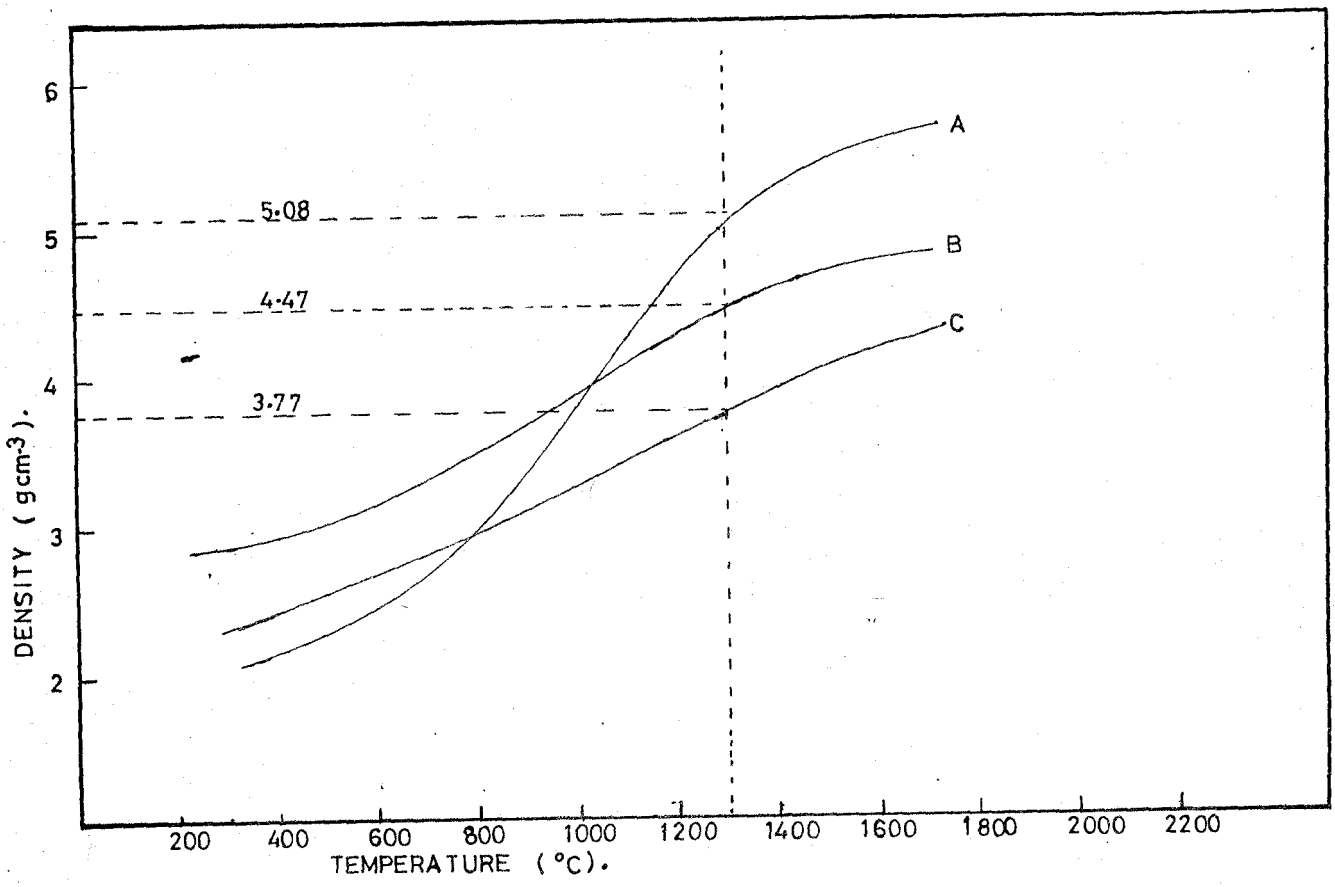

Fig.2 The densificaion process for the samples.

distribution was initially very sharp for all the specimens at the preliminary particles. Generally there was an increasing density from samples C to A for both final and calcined products. Thus the quantitative bulk densities were closely related to the milling time and hence the aggregation state. It would appear that the higher the bulk density, the smaller the aggregates and the more homogenous the specimens. The implication of the result is that sample A powder was in a very loose packed state than B. and C.

Similar pattern of characteristics was reflected during the densification process of the specimens at sintering stage. It could be deduced that the densification processes in the early stages of sintering depended upon particle size distribution and the temperature at which the densification starts. Figure 2 shows that densification started between 600° and 800°. The starting of densification process was lowest for sample A followed by C and very high for B. There was no clear and well-defined pattern for the green densities of the specimens. In summary it was obvious that prolonged milling of powder samples assisted in producing high quality products with good densification products.

Figures 3,4,and 5 show the electron micrographs of the influence of the aggregation after milling and sintering of samples A, B, and C. The microscopic analysis revealed that power A consists in of party spherical aggregates between 15 –25 um in diameter and these particles were very much smaller than those of B and C. Indeed, the porosimetry of the pressed body of power A detected large pore greater than 1 um which have been produced by the packing of uncollapsed aggregate. This observation agrees with the work

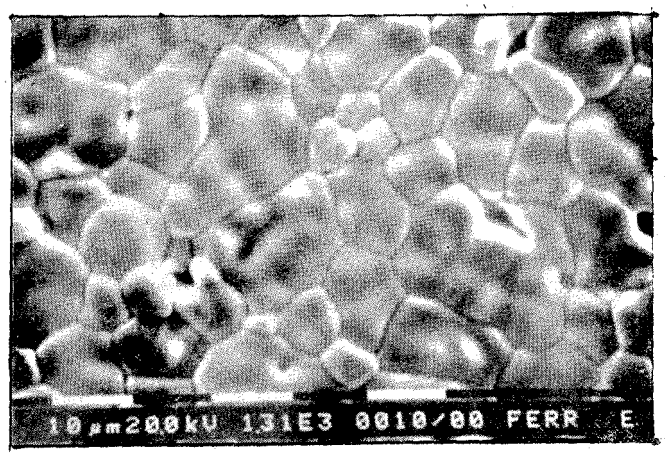

Fig.3 The Electron micrograph of sample A

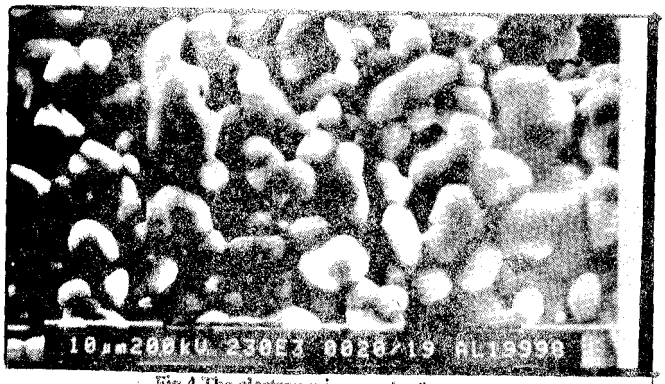

Fig.4 The electron micrograph of sample B

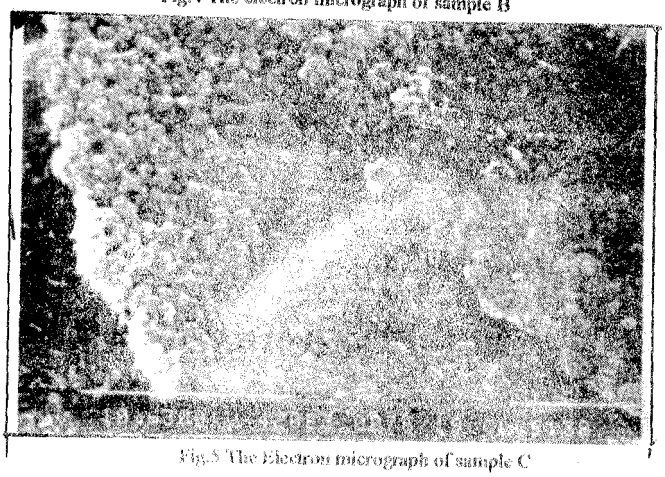

Fig.5 The Electron micrograph of sample C

of other investigators (Buchanan(1986) and Snelling (1988)).

Table 2 presents the value for permeability, resistivity, shrinkage percentage, Eddy current loss, Residual losses, loss tangent and densities. Generally, it was observed that sample A showed good characteristics than sample B and C. This could be due to the final nature and good powder preparation method of the starting powder. The prolonged powder processing time enhanced the quality of product. This is reflection of the importance of powder processing in most sintering process, enhances the credit of power metallurgy. The composition of the ferrite used in the present work has been previously applied as magnetic core material and the value of the parameters obtained are in good agreement with that of other investigators for the application area (Snelling, 1988).

CONCLUSION

The nature of the starting powder and processing techniques influenced the final product and densification process. This means that the densification behaviour did show some significant dependence on the primary particles size and was affected by the nature and State of aggregation. Also the characteristic properties of the materials were greatly improved by powder processing time and technique.

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TABLE 2 [illegible] CHARACTERISTIC PROPERTIES

	Perm.	Eddy Current Loss X 10^{-6}	Loss tangent	Residual loss	Shrinkage & (%)	Resistivities X 10^5 (ohm-cm)
C	113	14.08	158.3	0.099	14.2	2.72
B	101	6.09	114.7	0.071	13.1	3.34
A	75	4.15	99.4	0.037	12.4	4.62