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Effect of post sinter cooling rates on manganese zinc ferrites

It is well known that the important technical and commercial parameters of soft ferrites are the permeability, the quality factor, hysteresis loss, temperature factor and disaccommodation factor. To satisfy requirements all these parameters must simultaneously be within certain well defined limits. Unfortunately due to the inter-dependence of these parameters and their conflicting requirements during powder preparation and heat-treatment, it is extremely difficult to obtain a sintered ferrite with all five components fully maximized. In practice some degree of optimization must occur e.g. a high quality factor (μQ) requires that MnZn ferrites be sintered under an atmosphere giving approximately 96% stoichiometry [1], while to obtain a low temperature factor (TF) a somewhat higher oxygen content is necessary in the sintering atmosphere [2] resulting in a lower stoichiometry in the sintered ferrite [3]. This in turn can produce quite high values for the disaccommodation. The temperature factor and disaccommodation factor (DF) have sometimes been regarded as secondary factors and many workers have concentrated on the other three parameters. Unfortunately, once these parameters have been brought into line, there is no guarantee that the TF and DF will be within limits. The only existing method of controlling the latter is to alter either the firing conditions, or the basic composition of the powder and, as outlined earlier, this could invalidate the earlier maximization of the primary parameters. Quite clearly any technique which enables the separate control of any of these parameters is particularly welcome.

A number of workers [4–7] have shown that in MnZn ferrites the DF depends on the Fe^{2+} ion concentration and the vacancy concentration. The TF, however, has been shown to depend on Fe^{2+} content and also on the degree of cation order–disorder in the final sintered samples [8]. Thus it may be possible to vary one of these parameters by varying the degree of cation order–disorder. A MnZn ferrite having an initial chemical composition of 53.00 mol. % Fe_2O_3, 30.10 mol. % MnO_x, 16.30 mol. % ZnO, 0.60 mol. % TiO_2 was prepared by standard ceramic processing methods and toroidal specimens were dry pressed at 155×10^6 Pa (10.05 tons $in.^{-2}$). Two sintering schedules were carried out by means of a programmable furnace. Initially the specimens were heated from room temperature at a rate of $100° C^{-1}$ under an atmosphere of air and then sintered for 5 h at 1255° C under an atmosphere of 95% N_2 and 5% O_2. As the furnace cooled the oxygen level was continuously decreased according to the equation $\log P(O_2) = KT^{-1}$. At a temperature of 960° C the oxygen content of the furnace atmosphere was less than 0.001% and the remainder of the cooling schedule took place under pure nitrogen. This "phase rule" cooling maintains the Fe^{2+} level at the value dictated by the oxygen/nitrogen ratio and the temperature in the sintering stage [9, 10]. In the first schedule the toroids were cooled at $300° C h^{-1}$, while in the second the cooling rate was $150° C h^{-1}$. In both cases the oxygen content of the atmosphere was monitored using a high accuracy semiconductor probe [11]. The inductance of a toroidal specimen was measured with a Boonton Inductance Bridge at a frequency of 100 kHz and a field of 0.5 mT. In the case of the DF, measurements were made 1 min and 10 min after demagnetization. In all cases the results quoted are the mean of those obtained on 10 specimens.

	Cooling rate	
	$150°\,C\,h^{-1}$	$300°\,C\,h^{-1}$
μ	2110	2150
$\mu Q \times 10^{-3}$	340	370
$h\mu^{-2} \times 10^6$	520	483
DF $\times 10^6$	4.05	1.26
TF(23 to 55° C) $\times 10^6$	0.28	0.21

It appears that μ, μQ and $h\mu^{-2}$ are all slightly improved by cooling at the faster rate. However, the difference in the values is not large and is just within the limits of accuracy of the bridge. The shape of the μ–T curve is unaffected by cooling rate, although the TF (between 23 and 55° C) is slightly lower at the higher cooling rate. It is observed, however, that the DF depends markedly on cooling rate and a significant improvement is obtained with the more rapid cool. The DF of the rapidly cooled specimens drops to about 30% of those specimens which were slow cooled, and this improvement is obtained with no significant effect on the other parameters.

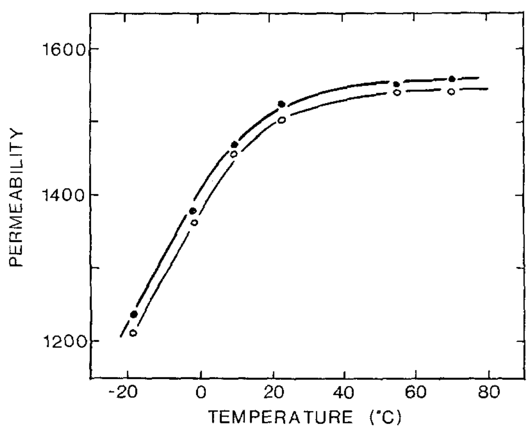

Figure 1 Relation between permeability and temperature for specimens cooled at two different rates: ● $300°\,C\,h^{-1}$; ○ $150°\,C\,h^{-1}$;

The two firings involved exactly the same sintering schedules and cooling took place under phase rule conditions. The Fe^{2+} content should, therefore, be the same in each set of specimens. Also, since there is very little difference between the temperature factors, it may be assumed that the distribution of Fe^{2+} ions is similar in each case. An explanation of the observed change in DF will thus involve only the vacancy concentration or distribution. The nature of this relationship will be the subject of a later paper.

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