Manganese-Zinc-Ferrites with Improved Magnetic and Mechanical Properties

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Abstract: The 3-component system of manganese-zinc-ferrites is being thoroughly investigated as regards its intrinsic properties. The macroscopic properties of these spinel are variable within broad limits over the process of microstructure formation. Oxides of the 3d and 4d elements are frequently used as additives to determine the temperature dependence of the initial permeability and the electrical conductivity of manganese-zinc-ferrites. Additives for acceleration of sintering based on oxides of the elements of the third, fourth and fifth main group of the periodic system require an improved knowledge about the process of microstructure formation of 8 - 10 components systems especially as regards grain growth, microchemistry and the structure of the grain boundary. Due to the increased application of silicon power semiconductors (IGBT, power MOSFET) high demands are made as well on the mechanical properties of ferrite components, especially on tensile strengths, crack toughness and thermal shock resistivity. Proceedings from the basics of linearly elastic fracture mechanics experimental results are presented as regards increase of tensile strength, reduction of the mechanical stress dependence of the initial permeability and improvement of the thermal shock resistivity of manganese-zinc-ferrites for commercial applications. An interpretation of the microstructure-properties ratio is carried out by means of microstructural and microchemical investigations using the high-resolving transmission electron microscopy.

1. MANGANESE-ZINC-FERRITES WITH IMPROVED MAGNETIC AND MECHANIC PROPERTIES

New fields of application regarding manganese-zinc ferrites as for instance in drive technology and industrial automatization nowadays require besides defined magnetic properties the fixing of mechanical parameters. The mechanic and magnetic properties of ferrites are defined by their microstructure. The improvement of properties concentrates mainly on the increase of the tensile strength, break toughness and thermal shock resistivity. These properties are determined by grain and pore size as well as by their distribution, composition and structure of the grain boundary, microcracks and distribution of crack lengths as well as by dislocations, punctual, planar and pile defects in the spinel-type lattice.

The possibilities of microstructural designing in order to improve mechanic properties include the use of common reinforcing techniques as there are dispersing reinforcement, tension-induced phase transformation, the generation of gradients of the chemical composition and the increase of the sintering density with a defined interface structure. These reinforcing techniques contribute to the energy dissipation of crack propagation and therefore increase the crack propagation resistivity. On the other hand they lead to worse magnetic parameters making a optimum compromise necessary.

1.1 Sample preparation

The investigations for an improvement of properties have been carried out on manganese-zinc ferrites with initial permeability of $6000 \pm 20\%$. The preparation of the samples was based on common ceramic processing techniques. The fine milling of the calcined powders was carried out with a ball mill whereas the ultra fine milling was done with an attritor mill. Sintering was carried out in air at temperatures between 1200 and 1350°C, varying at the same time the isothermic sintering times between 3 and 18 hours. Oxygen partial pressure of the sintering atmosphere and the cooling regime were selected such as to achieve a defined degree of oxidation in the samples and to avoid the separation of secondary phases. The heating and cooling rate was about 5 K /min

1.2 Improvement of mechanic properties

For the evaluation of the mechanic properties of the examined ferrites, tensile strength, break toughness and hardness are used. As shown in figure 1, the tensile strength of high permeable ferrites can be improved considerably by decreasing the dense sintering temperature. The increase in strength is coupled with a slight decrease of the initial permeability and the density (increase of porosity). The average grainsize reduces slightly from about $15\mu\text{m}$ down to $12\mu\text{m}$. When isothermic sintering times are prolonged from 13 to 17.5 hours the permeability rises again and the tensile strength increases too at the same time.

1.3 Investigations with Electron Microscopy (TEM and HREM)

The high resolution transmission electron microscopy was used to explain the reasons for fracture. The detected principal reasons are intra-crystalline formations of cracks. Therefore further processing improvements for the fabrication of ferrite is to be concentrated on the reduction of process-related crack lengths. Figure 2 shows the intracrystalline formation of cracks in a dense sintered structure.

Further investigations were based on the structure of grain boundaries. In the examined ferrites, crystalline or amorphous boundaries were formed in a range approx. 2 to 5 nm. Amorphous phases represent segregations of the sintering additives or result from impurities of the raw materials.

Figure 3 shows amorphous phases in the grain boundary between the grains of similar orientation. The transition to amorphous phases features a higher number of pile defects and dislocations.

Fig 2: Intracrystalline microcrack (photographed with TEM)

Fig 3: Amorphous grain boundary (photographed with HREM, numerically manipulated)

1.4. Summary

The results of investigation between microstructure, magnetic and mechanic properties show that permeability and mechanical strength are contrariely dependent on the grain size. The modification of the grain boundary by amorphous phases leads to lower remagnetization losses at high frequencies. The intergranular glass phase causes a weakening of the microstructure when exposed to mechanical stress and relaxation cracks appear. The experimental interdependence can be explained adequately by means of linear elastic fracture mechanics.

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