

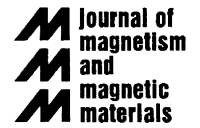


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Effects of mixing procedure and Bi_2O_3 content on structural and magnetic properties of hexaferrites sintered at low temperature

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Abstract

Z-type hexaferrites with composition of $\text{Ba}_3(\text{Co}_{0.4}\text{Zn}_{0.6})_2\text{Fe}_{24}\text{O}_{41} + x \text{ wt}\% \text{ Bi}_2\text{O}_3$ (where $x = 0\text{--}2$) were prepared by a solid-state reaction method. The effects of different mixing procedures and Bi_2O_3 content on the sintering behavior, phase composition, microstructure and magnetic properties of the samples were investigated. It was confirmed that with the same secondary mixing time (t_2), increasing the primary mixing time (t_1) leads to a decrease of the average grain size and an increase of the initial density. The initial density and the distribution of particles and pores determined the sintering density. With $t_1 = 24 \text{ h}$ and $t_2 = 6 \text{ h}$, the sintering density reached its maximum. The initial permeability and Q -factor also peaked, which could be attributed to the highest sintering density and compact microstructure. Further studies showed that with increasing Bi_2O_3 content, the major phase changed to the Z-phase; simultaneously, the remnants content of intermediate phase decreased. The Bi_2O_3 additive, as a reaction center, promoted the grain growth at lower temperatures. The decrease of the sintering temperature and the partial agglomeration of Bi_2O_3 on the grain boundaries restrained abnormal grain growth, thus closed pores in grains were not formed. A maximum initial permeability, a higher Q -factor, a cut-off frequency of 900 MHz and a resonance frequency above 1.8 GHz were obtained for the sample with 0.6 wt% Bi_2O_3 sintered at low temperature.

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Keywords: Hexagonal ferrite; Bi_2O_3 ; Mixing; Sintering behavior; Microstructure; High-frequency magnetic property

1. Introduction

Nickel–zinc ferrites are the widely used soft magnetic materials for medium high frequency up to 100 MHz. However, beyond 100 MHz, they cannot be used because they typically exhibit severe property changes due to Snoek's limit. Presently, hexagonal ferrites with planar magnetic anisotropy are of great interest for application in the hyper high-frequency range such as chip inductors, LC filters, and microwave absorbers. Especially, Z-type hexaferrites are among the most widely used soft magnetic materials, instead of spinel ferrite, for high-frequency application due to their higher initial permeability, excellent thermal stability ($T_c = 410 \text{ }^\circ\text{C}$) and ferromagnetic resonance frequency up to the GHz region [1–4]. From the

crystallographic point of view, Z-type hexaferrite is among the most complex compounds in the families of hexaferrites with planar hexagonal structure. The extremely large elementary cell and the “strongly anisotropic” Co^{2+} ions of Z-type hexaferrite lead to preparation difficulty and complex structure and magnetic properties [5,6]. Magnetic properties of these ferrite materials strongly depend on their chemical compositions and additives. For example, Zn element was required to obtain higher initial permeability [7], Bi_2O_3 additive was often doped into ferrites to realize a decrease of the sintering temperature and an increase of the density [8]. Considering the demand for high initial permeability and low-temperature sintering, the befitting content of ZnO in the composition formula was 1.2, and the content of Bi_2O_3 additive was from 0.1 to 2.0 wt%. However, properties of these ferrite materials are also very sensitive to the preparation conditions, such as mixing procedure, sintering temperature, etc. In this study,

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the effects of mixing procedure and content of Bi_2O_3 additive on the sintering behavior, phase composition, microstructure and magnetic properties of Z-type hexaferrites are investigated. The relationships between the mixing procedure, densification, microstructure evolution and magnetic properties of Z-type hexaferrite with various amounts of Bi_2O_3 were also presented.

2. Experimental procedure

The hexaferrites with composition of $\text{Ba}_3(\text{Co}_{0.4}\text{Zn}_{0.6})_2\text{Fe}_{24}\text{O}_{41} + x \text{ wt}\% \text{ Bi}_2\text{O}_3$ (where $x = 0\text{--}2$) were prepared by a standard solid-state reaction method. The raw materials, BaCO_3 , $\alpha\text{-Fe}_2\text{O}_3$, ZnO and Co_3O_4 , of high purity, were mixed and milled for various time ($t_1 = 4\text{--}60$ h). The mixed powders were calcined at 1280°C for 6 h in the air. Then the powders were mixed ($t_2 = 6$ and 12 h) with a small amount of Bi_2O_3 , and milled, dried and pressed. The pressed toroidal samples (outside diameter 18 mm, inside diameter 8 mm, thickness 4–6 mm) were sintered at $950\text{--}1000^\circ\text{C}$ for 6 h in the air and then cooled in the furnace. From room temperature to 600°C , the heating rate was $1.5^\circ\text{C}/\text{min}$, and then the temperature was kept at 600°C for 30 min to burn out the binder. From 600°C to the final sintering temperature, the heating rate was $2^\circ\text{C}/\text{min}$. Serial number of obtained samples is Z*****. Among the six numbers after letter Z, the anterior four numbers refer to the sintering temperature of the sample, and the latter two numbers refer to Bi_2O_3 content.

Hexagonal structure of the samples was identified by means of X-ray diffraction (XRD; Philips X'Pert) using $\text{Cu K}\alpha$ radiation in the range of $2\theta = 25\text{--}75^\circ$. A scanning electron microscopy (SEM; S-530) was used to observe the microstructure of the fracture surface of the samples. An HP4291B impedance/materials analyzer in the frequency range of 1 MHz–1.8 GHz measured the complex permeability of the samples. The bulk density was determined by the Archimedes method.

3. Results and discussion

The effects of different mixing procedures on the sintering behavior, microstructure and magnetic properties of hexaferrites with 2.0 wt% Bi_2O_3 are shown in Figs. 1–3, respectively. t_1 denoted the primary mixing time and t_2 the secondary mixing time. It is obvious that these properties were affected by the mixing procedure significantly.

The typical microstructures of the samples with different mixing time are shown in Fig. 1. Obviously, the mixing procedures influence not only the densification, but also the sample microstructure. As the mixing time increases, the average grain size decreases. Especially, the sample with $t_1 = 24$ h and $t_2 = 6$ h has a very uniform and compact hexagonal sheet structure with average grain size of about $3\ \mu\text{m}$. The sample with $t_1 = 6$ h and $t_2 = 12$ h has the largest average grain size of over $5\ \mu\text{m}$. This result suggests that

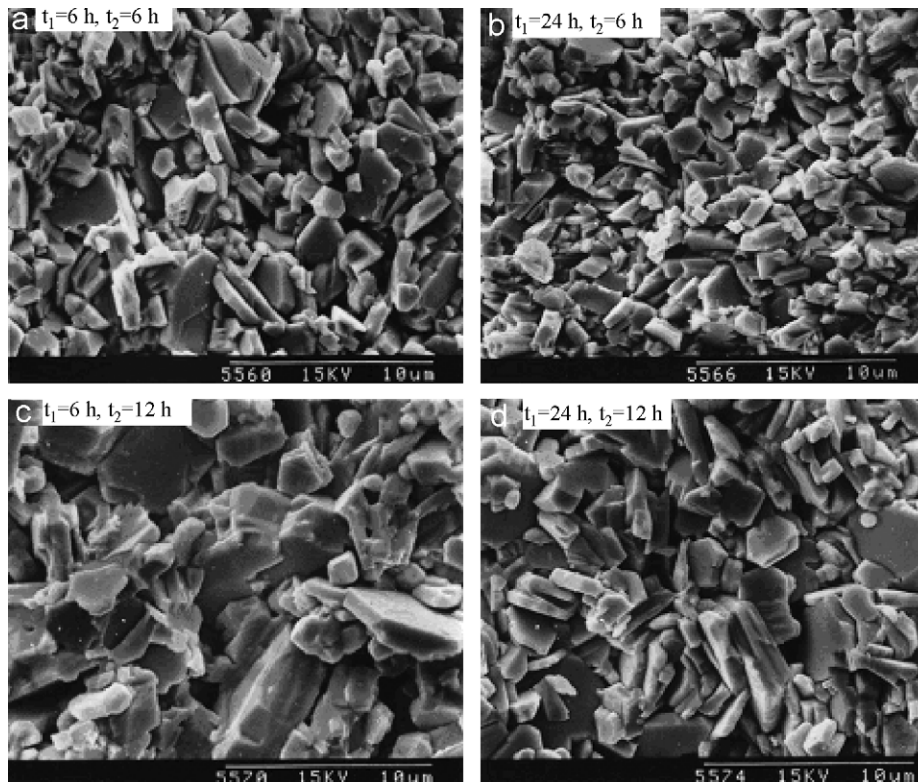


Fig. 1. SEM micrographs of the specimens prepared in different mixing conditions.

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