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Effects of Li_2CO_3 addition on the microstructure and magnetic properties of low-temperature-fired NiCuZn ferrites

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ABSTRACT

NiCuZn ferrites doped with 0.5 wt% Bi_2O_3 and different Li_2CO_3 contents (0–0.25 wt%) were sintered at 900 °C. The microstructure and magnetic properties of these materials were investigated. The addition of low-melting-point Li_2CO_3 led to large and uniform grains. However, excess Li_2CO_3 addition produced abnormal grains and many closed pores, thereby reducing density. Permeability initially increased and then decreased at the Li_2CO_3 content of >0.2 wt%. Maximum magnetic flux density (431.1 mT at room temperature, 339.6 mT at 100 °C) and minimum power loss were achieved at 0.2 wt% Li_2CO_3. These findings suggested the suitability of 0.2 wt% Li_2CO_3 for applications in low-temperature co-fired ceramic magnetic power components and modules.

1. Introduction

Miniaturised, lightweight, integrated and multifunctional electronic devices are rapidly being developed. Portable devices are continuously fabricated with multifunctional and high-frequency applications [1]. Thus, the integration of low-temperature co-fired ceramic (LTCC) with inductors, capacitors and resistors, among others, into one monolithic chip component will offer enhanced performance and save space on the printed circuit board during assembly [2–5]. Low-temperature-fired NiCuZn ferrites have been widely used in manufacturing LTCC magnetic components, such as inductors and transformers, because of their superior magnetic properties. Ag has been chosen as the material for the internal conductor of multilayer chip magnetic components because of its low resistivity; thus, high-quality components are produced [6,7]. The interfacial reaction between Ag and ferrites can be inhibited by co-firing at a temperature lower than the melting point of Ag. Several methods such as ion exchange, fine powder and flux have been introduced to lower the sintering temperature of NiCuZn ferrites [8–15]. An increase in power density is important to further improve the integration of LTCC components and modules. However, few studies have investigated the power characteristics of low-temperature-fired NiCuZn ferrites to produce LTCC power magnetic elements and modules. In this study, we mainly analysed the influences of Li_2CO_3 addition on the microstructure and magnetic properties, especially power-related properties, such as magnetic flux density (B_s) and power loss (P_{cv}), of low-temperature-fired NiCuZn ferrites.

2. Experiment procedures

NiCuZn ferrite samples ($Co_{0.01}Ni_{0.32}Zn_{0.49}Cu_{0.18}Fe_{1.98}O_4$) were prepared by a solid-state reaction method. Analytical grade Fe_2O_3, NiO, CuO, ZnO and Co_2O_3 were weighed following the required composition and then mixed for 4 h. The mixed oxide powders were calcined at 780 °C in air for 2 h. Different Li_2CO_3 contents (0, 0.05, 0.10, 0.15, 0.20 and 0.25 wt%) and 0.5 wt% Bi_2O_3 were added to the calcined powder, and the mixtures were further milled in ball mills. The resulting powders were granulated with 8% polyvinyl alcohol after they were dried. The powders were then pressed into toroidal shapes with the size of 18 mm (outside diameter) $\times$ 8 mm (inside diameter) $\times$ 4 mm (height), which were finally sintered at 900 °C with the heating rate of 2 °C/min and held for 3 h in air.

To test permeability values of the samples, we first measured the outside diameter (R), inside diameter (r) and height (h) values of the sintered samples by vernier caliper. Then the samples were winded 20 turns wires respectively and measured by a precision LCR mete(Agilent 4284A) at the frequency of 10 kHz. Permeability

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values could be calculated according to the following formula:

$$\mu = \frac{L \times 10^4}{2N^2 \cdot h \cdot \ln(R/r)}$$

in which the unit of R, r and h was mm, N was the wire turns, L (μH) was inductance values of the sample, which were directly measured by the LCR meter. B_s and P_{cv} were measured using a B-H analyser (IWATSU, SY-8232) at room temperature and 100 °C. The density of the sintered samples was calculated as the mass/volume ratio. The morphology of the samples was investigated using a scanning electron microscope (SEM, JSM-6490LV). X-ray diffractograms of the samples were recorded using an X-ray diffractometer with Cu Kα radiation.

3. Results and discussion

Fig. 1 shows the typical SEM micrographs of the samples with different Li_2CO_3 contents. The sample without Li_2CO_3 presented a microstructure with some large grains and very small grains. The ratio of large grains gradually increased with the Li_2CO_3 content. The sample with 0.2 wt% Li_2CO_3 presented a microstructure with uniform and large grains, which were favourable to improve permeability and density. Subsequently, some large grains and many closed pores appeared, which might negatively affect the magnetic properties of the sample. The variation in microstructure was due to the ability of low-melting-point Li_2CO_3 (~723 °C) to form a liquid phase during sintering, which further enhanced mass transfer and sintering by solid-state solubilisation and segregation [16–18]. However, an excess liquid phase for the sample with 0.25 wt% Li_2CO_3 might lead to the rapid growth of grains, resulting in

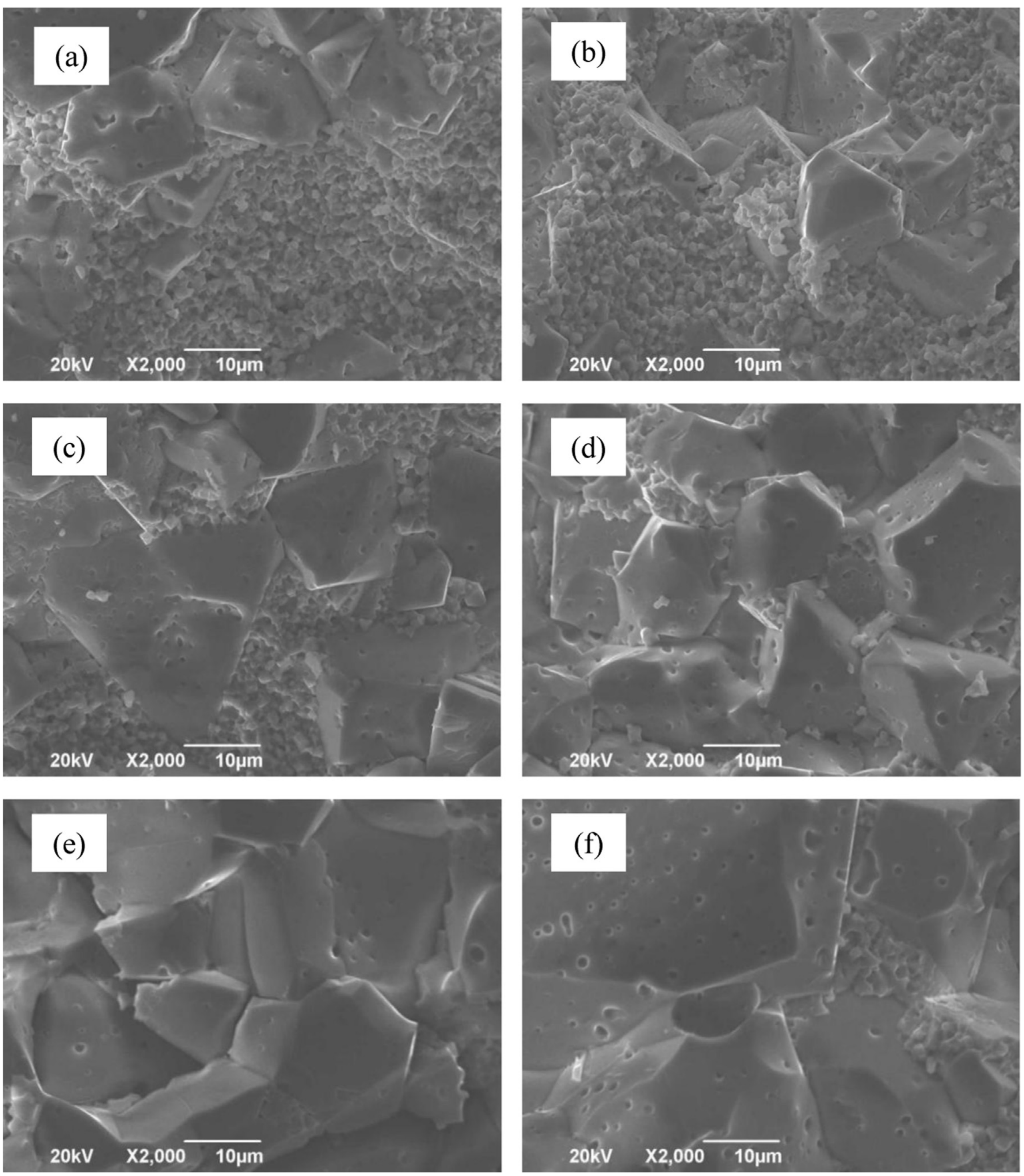

Fig. 1. Scanning electron micrographs of the samples with different Li_2CO_3 contents.