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Structural and electrical properties of sintered zinc-titanate ceramics

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Abstract

The aim of this work was an investigation of structural and electrical properties of sintered zinc-titanate ceramics obtained by mechanical activation. Mixtures of ZnO and TiO_2 were mechanically activated in a planetary ball mill up to 90 min and sintered isothermally in air for 120 min at 1100 °C. The phase composition in the ZnO–TiO_2 system after milling and sintering was analyzed using the XRD method. Microstructure analyses were performed using SEM. The results of electric resistivity, capacitance and loss tangent of the sintered samples were obtained. The existence of zinc-titanate as a dielectric was proved ($\varepsilon_r = 12.5$, $Q = 386.1$, $\mathrm{tg}\delta = 0.0026$, $\rho = 1.02\ \Omega$m).

Keywords: A. Milling; A. Sintering; B. X-ray methods; C. Electrical properties

1. Introduction

Oxides with a spinel structure are some of the most studied compounds in solid-state sciences due to their wide range of applications. Commonly, they are used as humidity sensors, semiconductors, magnetic materials, catalysts, microwave dielectrics and others [1,2].

A variety of titanates, such as $ZnTiO_3$, $Zn_2Ti_3O_8$ and Zn_2TiO_4, can be simultaneously obtained by a classic solid-state route [3,4]. It is well known that mechanical activation is a widely used method, which enhances the mixture homogeneity of the starting components; their reactivity and can remarkably lower the reaction temperature. Unfortunately, only a few researchers applied this method for obtaining zinc-titanate [2,5]. Zn_2TiO_4 prepared by the conventional solid-state reaction between 2ZnO and $1TiO_2$ was also reported in our previous study [6].

In this study, the authors have attempted to reveal the influence of milling conditions on structural and electrical properties of sintered zinc-titanate ceramics.

2. Experimental procedure

Mixtures of ZnO (99.9% Kemika-Zagreb) and TiO_2 powders (99.9% Alfa product-Ventron) with a molar ratio of ZnO:-TiO_2 = 2:1 were mechanically activated by grinding in a planetary ball mill (Fritsch Pulverisette 5). The milling process was performed in air during 5, 30 and 90 min at the basic disc rotation speed of 320 rpm and a rotation speed of bowls of 400 rpm.

Zirconium oxide balls (approx. 10 mm in diameter) and bowls (500 cm^3) were used with a ball to powder mixture mass ratio of 40:1. Samples were denoted as ZTO-005 to ZTO-090 according to the milling time. The binder-free powders were compacted using the uniaxial double action pressing process in an 8 mm diameter tool (Hydraulic press RING, P-14, VEB THURINGER). Compacts were placed in an alumna boat and heated in a tube furnace (Lenton Thermal Design Typ 1600). The heating rate was 10 °C/min and when the temperature of the furnace reached 1100 °C, compacts were sintered isothermally in air atmosphere for 120 min. The density of specimens was calculated from precise measurements of specimen's diameter, thickness and mass.

X-ray powder diffraction patterns of the milled powder mixtures and sintered samples were obtained using a Norelico-Philips PW-1050 diffractometer, with a Cu Kα radiation and a step scan mode of 0.02°/0.4 s.

The morphology of obtained powders was characterized using scanning electron microscopy (JSM 5300-JEOL, 30 kV).

The measurements of electrical resistivity, capacitance and loss tangent of samples were measured in the frequency range from 400 Hz to 4 MHz frequencies with a HIOKI 3532-50 LCR

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