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Influence of MgO addition on the synthesis and electrical properties of sintered zinc–titanate ceramics

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ABSTRACT

Starting mixtures of ZnO, TiO_2 and MgO (0, 1.25 and 2.5 wt.% MgO) powders were mechanically activated for 15 min in a planetary ball mill. The powders obtained were sintered non-isothermally to temperatures between 800 and 1100 °C and then held at those temperatures for 120 min. Analysis of the influence of MgO addition on the synthesis of zinc–titanate ceramics showed that its addition increased slightly the temperature at which the reaction process started, accelerated the reaction and resulted in higher sample densities. These results were correlated with the results of structural characterization using X-ray powder diffraction method and SEM analysis. Also, the results of electric resistivity, capacitance and loss tangent of the sintered samples were obtained.

1. Introduction

Spinels have awakened great interest particularly in the study of the physico-chemical properties of binary compounds and solid solutions [1]. Much attention has been paid to the synthesis and electrical properties of zinc titanate due to its attractive applications in microwave dielectrics [2,3]. Zinc titanate (Zn_2TiO_4) is an inverse spinel, which has been used as a catalyst and pigment [4].

Phase transitions in the ZnO–TiO_2 system are relatively complex, and sensitive to the starting material, additives and the preparation process [5]. The formation temperature for each of the $ZnTiO_3$, $Zn_2Ti_3O_8$ and Zn_2TiO_4 phases was shown to vary significantly with the preparation method and the Zn/Ti molar ratio of the starting materials [6–11]. A number of studies up to now have been devoted to the preparation, compound formation, crystal structure, stability, additives, as an effective way to simplify the synthesis process and improve the microwave dielectric properties, as well as the electrical properties of zinc titanates [4,12–18].

In the present work, Zn_2TiO_4 ceramics with MgO addition were prepared by a solid-state reaction. MgO has been proved to be an effective sintering aid for zinc–titanate ceramics [19]. Having in mind recent accomplishments made in the field of oxide addition to the ZnO–TiO_2 system, we presumed that MgO addition would form a solid–solution $(Zn, Mg)_2TiO_4$ and therefore stabilize the crystal structure, improve both densification and microstructures after the sintering process. The phase structure, microstructures along with electrical properties as well as dilatometer investigations of reactive sintering of zinc–titanate ceramics with MgO addition was investigated.

2. Experimental procedure

Zinc–titanate samples with addition of MgO = 0–2.50 wt.% were prepared by a conventional solid-state reaction method using ZnO (99.99% p.a. Aldrich), TiO_2 (99.99% p.a. Aldrich) and MgO (97% p.a. Merck) powders. The starting materials were mixed in ethanol with a magnetic whisk for 2 h in order to obtain homogeneity of the mixture and then dried at 120 °C for 2 h. The powders were submitted to mechanochemical treatment for 15 min in a planetary ball mill device (Fritch Pulverisette 5) at the angular speed of the supporting disc set on 400 rpm. Samples were denoted according to the added oxide, ZMTO-0, ZMTO-1.25 and ZMTO-2.50. Powders were then sieved through a 0.2 mm sieve.

X-ray diffraction patterns of powder mixtures after milling and sintering were obtained using a Philips PW 1050 diffractometer with λCu Kα radiation and a step/time scan mode of 0.05°/1 s. The morphology of obtained pow-

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