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Mechanical properties of sintered $La_{9.33}Si_2Ge_4O_{26}$ oxyapatite materials for SOFC electrolytes

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Abstract

Mechanical properties of $La_{9.33}Si_2Ge_4O_{26}$ prepared by mechanical alloying and subsequent sintering at 1300–1400 °C for 1 h were evaluated. Hardness and Young's modulus values in the range 7.3–9.6 GPa and 106–135 GPa, respectively, were obtained from nanohardness tests. The fracture toughness values derived from the Palmqvist method varied between 3.5 and 3.9 MPa $m^{1/2}$ from classical microindentation test with an indentation load of 9.8 N. Yield stress (σ_y) was determined by inverse analysis from microhardness tests. The maximum value of σ_y (1829 MPa) was obtained for the sample sintered at 1400 °C showing the highest density (5.42 g/cm^3).
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Keywords: C. Mechanical properties; Lanthanum-based oxyapatite; Mechanical alloying; SOFC

1. Introduction

M-doped lanthanum oxides of general formula $La_{10}(MO_4)_6O_2$ where M is an element such as Ge, Si, Al, are being considered for a range of technological applications, such as electrolytes in solid oxide fuel cells (SOFCs). These materials have been obtained by different techniques, such as solid state reaction [1], sol–gel synthesis [2], hot-pressing [3], mechanical milling [4], precipitation combined with an azeotropic-distillation process [5], colloidal processing [6] and floating zone methods [7]. All these processes require high temperature consolidation or heat treatment processes in order to obtain dense materials with apatite-type structure, with suitable high ion conductivity. In addition, these materials must be chemically stable and possess adequate mechanical properties such as high hardness, rigidity and fracture toughness in order to assure a desirable performance of the anode-electrolyte-cathode final assembly.

In a recent study [8], powders of La_2O_3, SiO_2 and GeO_2 were used to obtain $La_{9.33}Si_2Ge_4O_{26}$ materials with an apatite-type structure. Mechanical alloying was used as synthesis technique for obtaining the desired structure with doping Ge atoms in the tetrahedral sites of the apatite phase. The mechanically alloyed powders were subsequently consolidated at lower temperatures than those mentioned in the literature (1500–1600 °C). Dense samples were obtained at sintering temperatures of 1400 °C.

The present study evaluates the mechanical properties of $La_{9.33}Si_2Ge_4O_{26}$ samples obtained under different conditions of mechanical alloying and subsequent sintering. For this purpose, depth-sensing indentation techniques were used to evaluate hardness (H), Young's modulus (E), yield stress (σ_y) and fracture toughness (K_c) of the sintered samples.

2. Experimental

2.1. Sample preparation

Oxyapatite-based $La_{9.33}Si_2Ge_4O_{26}$ electrolytes have been prepared in a previous work [8] by conventional

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