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The effect of La_2O_3 on the microstructure and room temperature mechanical properties of t-ZrO_2

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ABSTRACT

Structural and mechanical properties of La_2O_3 added (up to 5 wt%) t-ZrO_2 compacts were examined with the aim of optimizing hardness and fracture toughness for room temperature applications. Structural examinations were performed using an X-ray diffractometer and a scanning electron microscope. Mechanical properties of the compacts were determined as modulus of elasticity, hardness and fracture toughness by conducting Vickers indentation tests under test loads of 0.5, 1 and 10 kg. Addition of 0.5 wt% La_2O_3 deteriorated the room temperature stability of t-ZrO_2 by forming m-ZrO_2 and coarse polygonal grains in the matrix. Higher concentrations (2 and 5 wt% La_2O_3) caused precipitation of $La_2Zr_2O_7$ at the grain boundaries, which also accompanied by a reduction in hardness. Fracture toughness increased with increasing La_2O_3 content of the compact. Finally, an optimum combination between hardness and fracture toughness was obtained for the 0.5 wt% La_2O_3 containing compact.

1. Introduction

Zirconia (ZrO_2) is an attractive ceramic material for various advanced technology structural applications, because of its stress induced phase transformation characteristic promoting exceptional fracture toughness among the other ceramic materials. Stress induced phase transformation is based on the transformation of tetragonal zirconia (t-ZrO_2) into monoclinic zirconia (m-ZrO_2) under external stresses, which hinders crack propagation via generation of compressive stresses around the cracks as the result of the increase in crystallographic volume [1–3]. In pure ZrO_2, t-ZrO_2 is stable over 1173 °C and can be retained at room temperature upon addition of stabilizing oxides [4], such as magnesium oxide (MgO), calcium oxide (CaO), cerium oxide (CeO_2) and yttria (Y_2O_3) Among these stabilizing oxides, Y_2O_3 is the most popular one and the fraction of t-ZrO_2 retained in the microstructure of yttria stabilized zirconia (YSZ) at room temperature depends on the grain size as well as the Y_2O_3 content. For example, room temperature microstructure of the 2–3 mol% Y_2O_3 containing YSZ can completely consist of the t-ZrO_2 phase at grain sizes lower than 0.8 μm [5]. Decrease in grain size reduces the transformability of t-ZrO_2 into m-ZrO_2 under external applied stresses [1,6]. Thus, the operation of stress induced phase transformation toughening mechanism is more likely for coarse grained metastable t-ZrO_2.

The other important property of YSZ is its thermal characteristic [7]. Its better deposition efficiency by thermal spray processes in comparison to other refractory oxides, make YSZ an attractive thermal barrier coating (TBC) material for high temperature engineering applications [7]. Since lanthanum oxide (La_2O_3) has significant influence on the densification characteristics of YSZ, some attempts have been made with the aim of reducing the thermal conductivity of TBCs while preserving their structural stability at elevated temperatures by modifying YSZ with La_2O_3 [8–11]. In this context, high temperature mechanical behavior and the changes in microstructure of La_2O_3 added YSZ have been reported recently [12]. However, to the best knowledge of the authors, studies focusing on the effect of La_2O_3 on the room temperature mechanical behavior of YSZ are scarce in the open literature. The objective of this study is to investigate the microstructure, hardness and fracture toughness of conventionally sintered YSZ incorporated with different amounts of La_2O_3.

2. Experimental studies

Commercially available 3 mol% (5.2 wt%) Y_2O_3 doped ZrO_2 powders having an average particle size of 45 μm and commercially available La_2O_3 powders having an average particle size of 40 μm were used in this study [13]. YSZ powders were mixed with

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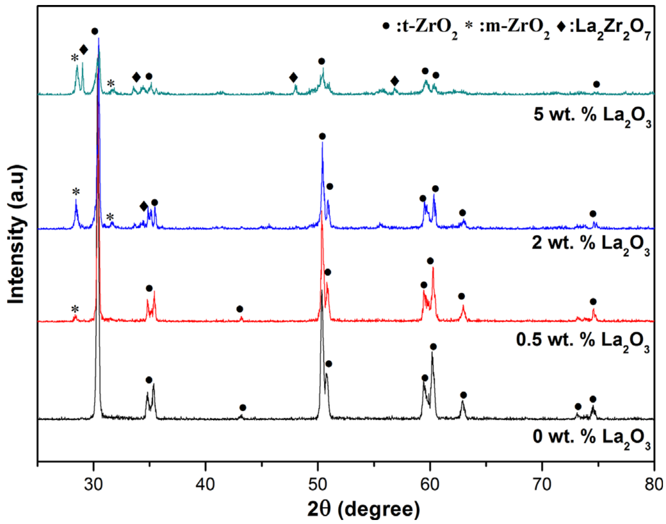

Fig. 1. XRD patterns of the t-ZrO_2 compact and those containing 0.5 wt%, 2 wt% and 5 wt% La_2O_3.

the La_2O_3 powders at different weight percentages (max. 5 wt%) for 1 h. Powder mixtures were then compacted using an uni-axial hydraulic press under a pressure of 400 MPa to obtain disc shaped green bodies having 12.7 mm diameter and 10 mm height. Green bodies were then sintered at 1500 °C for 2 h in a normal atmospheric electric furnace. Heating and cooling rates of the furnace were set at 2 °C/min. After removal from the die, the densities of the compacts were measured utilizing the Archimedes' principle.

Microstructural characterization of the sintered compacts was carried out using X-ray diffraction (XRD) and scanning electron microscope (SEM) techniques. XRD analyses were conducted using a Bruker™ D8 Advance diffractometer under Cu Kα radiation. SEM investigations were performed on the polished and thermally etched surfaces of the sintered compacts in a Hitachi™ TM-1000 Model table top SEM. For SEM examinations compacts were prepared according to the ceramic sample procedures described by Elssner et al. [14]. Thermal etching was conducted by holding the polished compacts at 1425 °C for 45 min in air atmosphere.

Mechanical properties of the sintered compacts were determined by indentation tests having standard Vickers indenter under the indentation loads of 0.5 kg, 1 kg and 10 kg using a CSM Instruments™ depth sensing microhardness tester, a Shimadzu™ conventional microhardness tester and a Zwick™ ZHU 2.5 macrohardness tester, respectively. At least ten successive test results were averaged for the quantification of hardness of each compact under these three different loads.

The fracture toughness of the sintered compacts was evaluated by measuring the indentation induced crack length (2c) and imprint diagonal (2a) formed under indentation load of 10 kg. Since the c/a ratio of the compacts ranged from 1.9 to 2.2, the equation proposed by Anstis et al. [15], which is valid for fully developed cracks having c/a ratio of about 2, have been utilized. It should be noted that the equation proposed by Anstis et al. [15] in the form of:

$$Kc = 0.016(E/H)^{1/2}\left(P/c^{3/2}\right)10^{-6}$$

is the widely used as the Vickers indentation fracture toughness equation [16], which is also applicable to the ZrO_2 based ceramics [17, 18]. In this equation, Kc is the fracture toughness (MPa $m^{1/2}$), E is the modulus of elasticity (GPa), H is the hardness (GPa), and c is

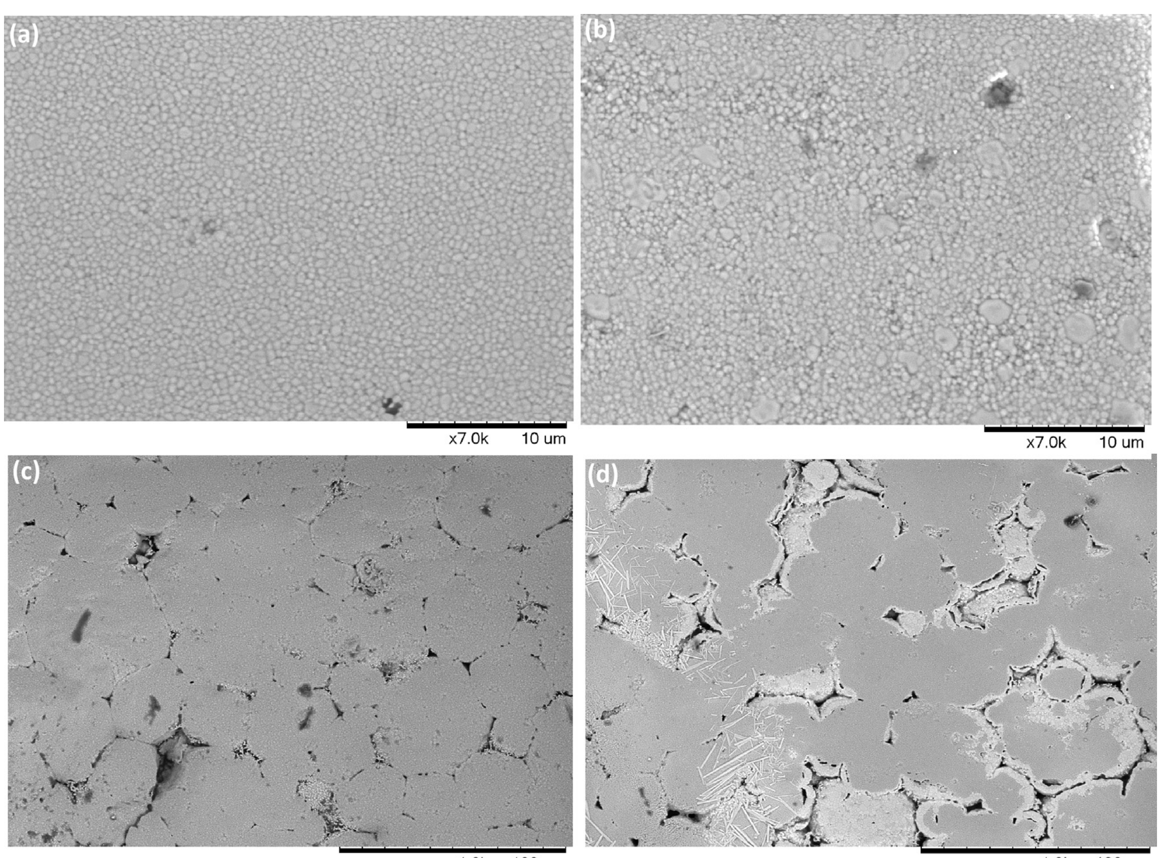

Fig. 2. SEM micrographs of sintered (a) La_2O_3 free and (b) 0.5 wt%, (c) 2 wt%, (d) 5 wt% La_2O_3 containing compacts.