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Review article

Nanomechanical properties of zirconia- yttria and alumina zirconia- yttria biomedical ceramics, subjected to low temperature aging

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ABSTRACT

Samples of ZTA composites {80 wt%Al_2O_3+20 wt%TZ-3Y}, ATZ composites {20 wt%Al_2O_3+80 wt%TZ-3Y}, tetragonal polycrystalline zirconia (3Y-TZP), and cubic stabilized zirconia (8Y-CSZ) were prepared to study the nanomechanical properties by nanoindentation before and after low temperature aging or degradation. Moreover, structural properties and crystalline present phases were evaluated by X-Ray Diffraction (XRD) and Electron Diffraction Patterns from transmission electron microscopy (TEM). The 8Y-CSZ ceramic showed the best mechanical behavior among all the analyzed materials (ZTA, ATZ and 3Y-TZP), the 8Y-CSZ sample did not showed any phases transformations when subjected to low temperature degradation (LTD). Absolute nanohardness, Young's modulus and fracture toughness after the LTD were carried out in different samples, the obtained results, in a decreasing order were: 8Y-CSZ > ZTA > 3Y-TZP > ATZ, 8Y-CSZ > 3Y-TZP > ZTA > ATZ and 8Y-CSZ > 3Y-TZP > ATZ > ZTA, respectively. The 8Y-CSZ ceramic, did not showed any variations in nanomechanical properties due to the absence of anisotropic behavior, manifesting high hardness, elastic modulus and relative values of fracture toughness. Perhaps this material could be candidate for biomedical applications.

1. Introduction

During the past years, ceramic materials based on zirconia (ZrO_2) have received special attention because they are promising materials for structural applications, some of their excellent physical and chemical properties are, large hardness, high elastic modulus, high melting temperature, resistance to chemically aggressive media [1,2]. Zirconia was originally referred to as ceramic steel by Garvie because its Young's modulus and thermal expansion coefficient are similar to those of steel. Recently some authors reported advances in the development of zirconia based ceramics with high strength properties obtained through the transformation of tetragonal to monoclinic phase (t→m) that increases the mechanical properties of these materials [3]. Gupta and collaborators [4] mentioned the benefits of the application of this phenomenon that would prevent the propagation of cracks. However, pure ZrO_2 has been limited in its use as a structural ceramic due to (t→m) phase transformations during cooling, making manufacturing almost impossible [5]. This transformation is accompanied of an increase in volume that can reach up to about 5%, producing structure instability [6]. To avoid this, the ceramics based on ZrO_2 must stabilized with other oxides such as yttria which stabilizes total or partially, the tetragonal and cubic phase [7]. The systems, zirconia ceramic based, alumina-zirconia composite ATZ (alumina toughened zirconia) and ZTA (zirconia toughened alumina) are promising material for many applications [8]. Zirconia (ZrO_2) is a material with adequate mechanical properties for manufacturing medical devices, when a stress occurs on zirconia stabilized with Y_2O_3; a crystalline modification opposes the propagation of cracks [9]. Zirconia implants seem to have good biological and mechanical properties [10]. The hydrothermal conditions of the human body could affect the structure of zirconia ceramics, promoting the transformation from tetragonal phase to monoclinic (t→m) [2,11]. Therefore, it is clear that the mechanical properties and microstructural characteristics of the zirconia, presents notorious changes when it is subjected to different

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chemical and physical treatments such as hydrothermal conditions. On the other hand, some documents discussed the low temperature aging or degradation (LTD) treatment of ATZ, ZTA and 3Y-TZP [1,2,11–17]. However, there is not study in the literature in comparison with 8Y-CSZ over the nano-mechanical properties after the LTD. The aim of the present work is to evaluate the nanomechanical properties, such as elastic modulus, stiffness, hardness and fracture toughness in the ATZ, ZTA, 3Y-TZP and 8Y-CSZ samples, before and after a LTD treatment. The structural of the transformation from the tetragonal to monoclinic phases affected by the LTD were evaluated by X-Ray diffraction and transmission electron microscopy diffraction patterns.

2. Materials and methods

2.1. Synthesis of ceramics materials

Powders of Al_2O_3 (Baikalox SM8 Bikowki USA) and ZrO_2+3 mol% Y_2O_3 (thereafter abridged as TZ-3Y, Tosoh); all of them with a purity > 99.99% were used as starting materials to prepare homogeneous mixtures of 80.wt%Al_2O_3+20%TZ-3Y. This formula was used for the ZTA composite, and 20 wt%Al_2O_3+80%TZ-3Y for ATZ composite. Also 3Y-TZP (97% mol ZrO_2 +3% mol Y_2O_3) and 8Y-CSZ (92%mol ZrO_2 +8% mol Y_2O_3) to prepare from TZ-3Y and TZ-8Y Tosoh powders. The all powders were milled using a Spex Certiprep 8000 M Mixer/Mill during of 5 min. On the one hand, The powder of mixtures 80.wt% Al_2O_3+20%TZ-3Y and 20 wt%Al_2O_3+80%TZ-3Y were first compacted under a uniaxial pressure of 50 MPa applied at a constant rate in an Elvec Hydraulic Press (ELVEC S.A. de C.V. Mx) steel mold (16 mm in diameter size). Later these green samples were sintered at 1400 °C for 2 h in air at a heating rate of 10 °C/min in the oven Carbolite model HTF 18/15. For sintering, the green samples were placed into an alumina crucible on ZrO_2+Al_2O_3 bed powders. This heat treatment has been recognized as the best sintering conditions by several authors [18,19]. After sintering ATZ and ZTA composites, the furnace was turned off and left to cool down. On the other hand, the samples 3Y-TZP and 8Y-CSZ were uniaxial pressed at 2 MPa in a Desk Top Electromotion Presser with the finality to obtain the cylindrical shape sample. Later these green samples were introduce in the camera with oil of the press isostatic in cold CIP 50 M mark TMI, introducing at 30 MPa during 8 min in side a container of latex. The sintering process of 3Y-TZP and 8Y-CSZ samples, were carried out at the temperature of 1400 °C for 1 h in the oven Carbolite model HTF 18/15. The heat up and cooling down rate was 5 °C/min. After the sintering process, the samples were first hand- and after machine-polished in a diamond sandpaper. Four samples obtained: ATZ composite, ZTA composite, 3Y-TZP tetragonal polycrystalline zirconia, and 8Y-CSZ cubic stabilized zirconia.

Aging treatment:All samples were subjected to a Low Temperature aging or Degradation (LTD) in a solution of artificial saliva. The artificial saliva was prepared in accordance with the formula of Macknight-Hane and Witford [11,20]. Polished samples were put in steam autoclave (Autoclave Yamato SN510C), whit temperature aging of 134 °C (under 2 bar pressure) and pH 5.5 (with 0.5 mol/L natrium hydroxydatum and 30% acetic acid).

2.2. Experimental details of XRD and TEM

Crystalline structure and phases of the ceramics samples were analyzed by XRD in a Panalytical X-Pert system, the patterns were obtained using Cu Kα radiation at 40 kV and 35 mA. Diffracted beam path included a graphite flat crystal monochromator. The scanning angle 2θ was varied between 20° and 100°, at steps of 0.02°. Moreover, the structure was corroborated by high-resolution transmission electron microscopy (HRTEM) in a JEOL JEM-2200FS+Cs equipped with a spherical aberration corrector in the condenser lens and operated at an accelerating voltage of 200 kV. Selected area electron diffraction patterns (SAED) were also employed in the microstructural characterization, and the elemental composition was determined. Samples for HRTEM were prepared using a JEOL JEM-9320 focused ion beam (FIB) system operated at 30 kV.

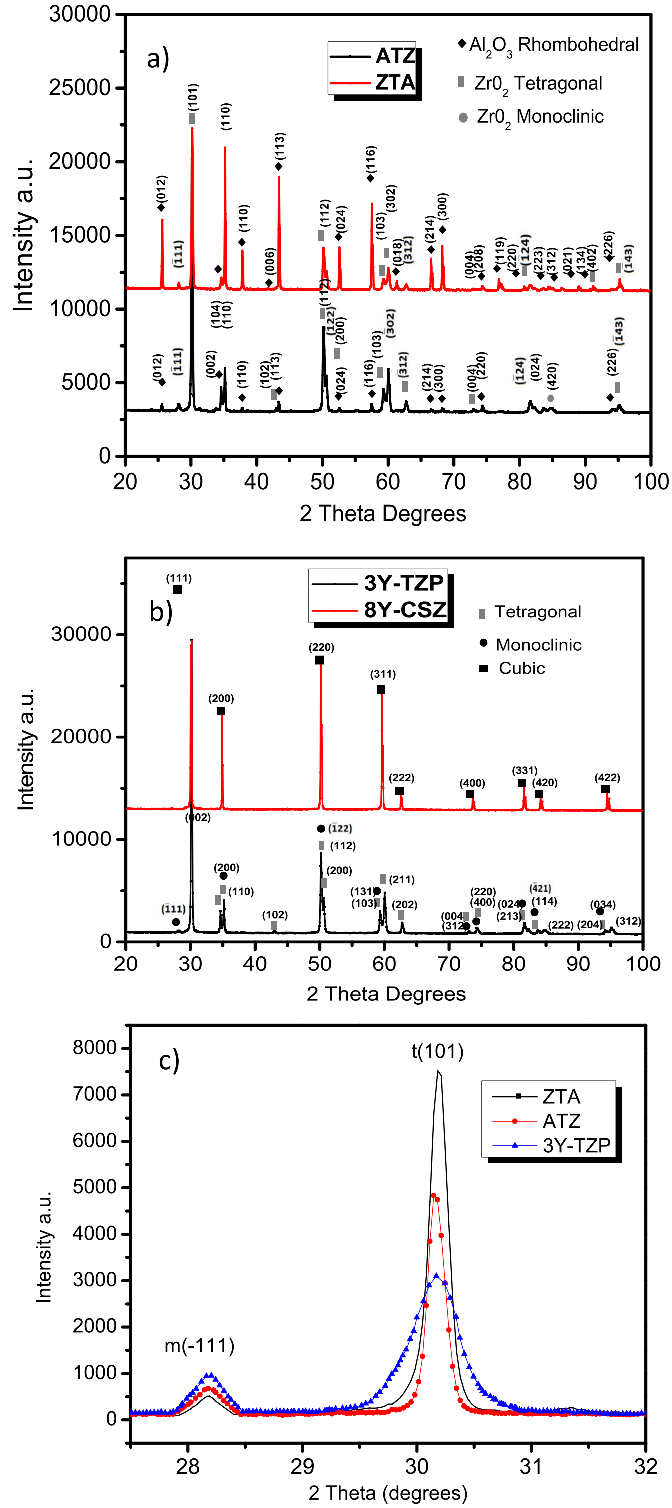

Fig. 1. XRD patterns of the different ceramics samples; a) ATZ, ZTA, b) 3Y-TZP, 8Y-CSZ and c) the monoclinic concentration from grazing incident angle XRD.

2.3. Nanomechanical properties

Nanomechanical properties such as hardness (*H*), stiffness (*S*)

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