

# Microstructure, mechanical and tribological properties of microwave sintered calcia-doped zirconia for biomedical applications

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## Abstract

Among the available ceramics materials for load bearing bio-implant applications, Y-TZP is superior (fracture toughness:  $\sim 10 \text{ MPa m}^{0.5}$ ) for its better mechanical properties. However, due to concerns related to property degradation of Y-TZP during long exposure in body fluid, the current work is taken up to study the feasibility of developing stabilised zirconia ceramics in CaO–ZrO<sub>2</sub> system, using microwave sintering (MW) technique. The present paper reports the processing, microstructure and tribological properties of microwave sintered Ca-doped ZrO<sub>2</sub> based ceramics. An important experimental result is that MW sintering to greater than 90% theoretical density can be achieved in Ca-PSZ (8 mol% CaO) and Ca-FSZ (16 mol% CaO) ceramics by sintering at 1585 °C for 1 h. The sintered materials exhibit Vickers hardness  $\sim 8\text{--}10 \text{ GPa}$ , which would allow them to be used as load bearing implants. Also, a modest fracture toughness ( $\sim 6 \text{ MPa m}^{0.5}$ ) was measured for Ca-PSZ, which is better than commercial grade alumina. So, it is possible to synthesize a material which has better combination of hardness and toughness than other commercially available bioceramics like alumina, hydroxyapatite, TCP, etc. Considering its specific application for THR (total hip replacement), tribological experiments using fretting wear tester serve to provide data about the wear behaviour of the proposed materials. The fretting experiments were conducted against a bearing-steel counterbody in air as well as in a SBF (simulated body fluid) environment. The wear behaviour of the investigated tribocouple is dominated by the formation of Fe oxide/chloride layer at the worn surface.

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## 1. Introduction

Among various structural ceramics, zirconia-based ceramics have been widely researched in the last three decades, because of their better toughness and strength properties [1]. The applications of ZrO<sub>2</sub>-based ceramics include mechanical seals, engine components, cutting tools, sensors, and thermal barrier coatings and lately, as biomedical implants [2]. It is known that among the bioceramics, bioinert materials like alumina and zirconia are candidate materials for load bearing implants, e.g. total hip joint replacement (THR), knee joints, etc. [3]. Various combinations of ceramics have been studied for hip joint prostheses, these include alumina on alumina, alumina on zirconia to assess the relative performance/durability in physiological conditions [4]. It was found that the wear rate

of Al<sub>2</sub>O<sub>3</sub> is  $\sim 8$  times higher than that ZrO<sub>2</sub> under identical testing conditions [5]. Such high wear resistance for ZrO<sub>2</sub> would evidently ensure longer implant life. It was additionally demonstrated that the presence of zirconia ceramics did not lead to any local or systemic adverse reactions or cytotoxic effects [3].

It is well known that pure monolithic/undoped zirconia has very poor mechanical properties due to volume expansion induced cracking, resulting from the tetragonal to monoclinic ZrO<sub>2</sub> transformation at 1170 °C. CaO, MgO and several other oxides in appropriate amounts are widely used to retain/stabilize the cubic zirconia phase at room temperature. Among stabilized ZrO<sub>2</sub> ceramics, the cubic zirconia phase containing ceramics are widely used for oxygen sensors and as anodes in solid oxide fuel cells [6]. For load bearing biomedical applications, the mechanical properties, in particular hardness and fracture toughness are the important parameters. Until date, yttria stabilized zirconia (Y-TZP) ceramics are popular for biomedical applications due to their high fracture toughness

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(2Y-TZP:  $\sim 10 \text{ MPa m}^{0.5}$ ) [7,8]. In spite of good mechanical properties, Y-TZP materials often behave in unpredictable manner as implants [9]. Such problems have been attributed to the susceptibility of Y-TZPs towards aging induced property degradation. Therefore, other partially and fully stabilized zirconia (CaO, MgO doped) may be used as suitable replacement for Y-TZP and other bioceramics in biomedical applications.

It is now well recognized that the microstructure and properties of ceramics critically depend on processing routes and densification parameters. For partially stabilized zirconia, a high toughness microstructure is generally obtained by sintering, followed by high temperature solid solution treatments for longer time and a controlled cool to room temperature. For example, Wu and Brook [10] densified Ca-PSZ ceramics via conventional sintering in air at  $1560^\circ\text{C}$  for 13 h. They also reported the sintering of Ca-PSZ ceramics at  $1700^\circ\text{C}$  by fast heating using a zone sintering technique. Although such a longer processing time causes extensive grain growth, it helps to precipitate tetragonal phase (often cuboid shaped) in the cubic matrix. It is therefore important to investigate whether the faster heating rate, in combination with shorter sintering time in microwave sintering, can produce microstructure in CaO stabilized  $\text{ZrO}_2$  with good material properties, as often obtained by conventional route with a combination of sintering and post-sintering annealing treatment. One of the major aspects of the present work is to address this issue.

For biomedical applications, the tribological properties play an important role, particularly when two similar or dissimilar materials come in contact during applications. For example, in THR application, zirconia ball head is attached with the metallic (Steel/Ti alloys) stent. The joint of these zirconia and metallic part can undergo micro-movement (fretting fatigue) during various external activities. Therefore, it is important to evaluate the material degradation (corrosion, wear) for such type of applications. The evaluation of fretting wear properties of the stabilized  $\text{ZrO}_2$ s constitutes another goal of the present work.

In order to achieve the above-mentioned objectives, the MW sintering technique is used in the present work for densification of Ca-doped  $\text{ZrO}_2$ . Controlled sintering of ceramics, by utilizing the microwave has been successfully used to densify  $\text{Al}_2\text{O}_3$  [11]. The interaction of high frequency electromagnetic fields with polycrystalline dielectric materials leads to volumetric, selective and rapid heating. Since the 1980s, microwave processing of monolith and composite ceramic materials has been recognized to offer a number of advantages over conventional sintering approaches. The increase in densification rate, as well as relatively low sintering temperature and time make the MW fabricating technique significantly faster than the conventional sintering process. Earlier, Upadhyaya et al. used MW sintering to densify 3Y-TZP [12] and 9Y-CSZ [13] ceramics. For TZP(Y) 20 wt.% alumina ceramics, a very fine grain ( $< 0.5 \mu\text{m}$ ) structure is obtained after microwave sintering [14]. Interestingly, Xie et al. [15] reported that microwave sintering does not always produce fine-grained ceramic microstructure and in fact, due to rapid diffusion rate grains grow faster during microwave sintering than conven-

tional heating. Based on the above observations, the goal of the present work is to produce calcia-doped zirconia via microwave sintering route. The microstructures as well as the mechanical, tribological properties are characterized.

## 2. Experimental procedure

### 2.1. Processing

The ceramic compositions investigated in this study include Ca-PSZ (8 mol% CaO) and Ca-FSZ (16 mol% CaO). Fig. 1 shows the  $\text{ZrO}_2$  rich end of the  $\text{ZrO}_2$ –CaO phase diagram and from this diagram, the composition of investigated materials of the present work has been identified. In this paper, Ca-PSZ will always indicate the material composition of 8 mol% CaO in  $\text{ZrO}_2$ , whereas Ca-FSZ will indicate the composition of 16 mol% of CaO in  $\text{ZrO}_2$ , irrespective of the phases present after MW sintering. In order to synthesize Ca-PSZ and Ca-FSZ materials, 8 and 16 mol% CaO is alloyed with monoclinic  $\text{ZrO}_2$ , respectively. Commercial high purity monoclinic  $\text{ZrO}_2$  powder ( $d_{50} = 7.62 \mu\text{m}$ , Tosoh TZ-O grade, Japan) was mixed in appropriate proportion with precursor  $\text{CaCO}_3$  powder (Rankem C0050 grade, India) to obtain a final compositions of 8 and 16 mol% CaO–zirconia ceramics. Because of the use of  $\text{CaCO}_3$  starting powders, it is expected that the carbonate salt would be decomposed into CaO and  $\text{CO}_2$  during calcination/early stage sintering of the mixed powders. The starting powders were mixed on a multidirectional (planetary ball mill) mixer for 24 h in acetone, using tungsten carbide milling balls with powder: balls in 1:5 ratio to break the agglomerates and reduce the particle size further. The mixture thus obtained was calcined at  $450^\circ\text{C}$  for 3 h in air to remove the moisture and other low temperature volatiles. This was followed by cold

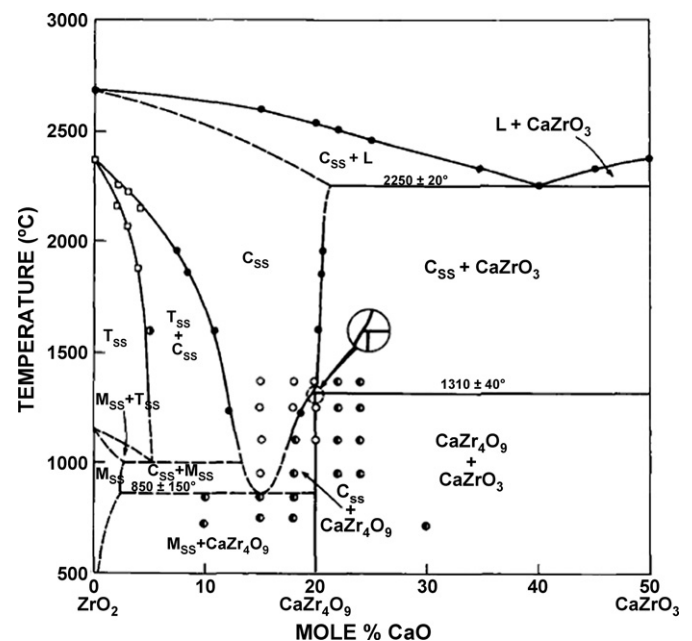


Fig. 1.  $\text{ZrO}_2$  rich part of the  $\text{ZrO}_2$ –CaO binary phase diagram, showing the different phases of  $\text{ZrO}_2$  as a function of temperature and mole percent of CaO addition. The details of the phase diagram description can be found in Ref. [32].

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