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Investigation the effect addition of nano Al_2O_3 on microstructure of Yttria tetragonal Zirconia polycrystalline ceramic

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ABSTRACT

Purpose: This research aimed to prepare tetragonal zirconia polycrystals powder by coprecipitation method and study effects of addition of different amounts of nano Al_2O_3 (1, 2 and 4) wt.% on its microstructure and mechanical properties of (5Y-TZP) composite.

Design/methodology/approach: The powder was uniaxial pressed at a pressure of 150 MPa and held for 60 s, and sintered at the 1500°C, held for two hours and then cooling down at 5°C/min to room temperature. Microhardness and fracture toughness tests were utilized to evaluate the mechanical properties of yttria tetragonal zirconia polycrystal composite. The microstructure has been observed using field emission scanning electron microscopy(FESEM).

Findings: The results showed an addition of nano Al_2O_3 has a great influence on hardness and microstructure, the increase in Vicker's microhardness of composite samples with the increase in the nano Al_2O_3 wt.% and microstructure were characterized with homogeneous zirconia distribution, grain growth destruction with the increasing percentage of nano Al_2O_3. The most important influence is the enhancing of the densification process as the porosity decreased. The highest hardness and maximum fracture toughness were recorded at 4 wt.% nano Al_2O_3.

Research limitations/implications: Ceramic matrix composites are developed to overcome the brittleness of ZrO_2 and the low toughness of alumina by formation, a large difference of elastic behavior between matrix and particles(dispersion phase)which disturbs the stress field as a dislocation comes near a particle.

Practical implications: Zirconia has mechanical properties similar to those of stainless steel. Yttria-stabilized tetragonal Zirconia (Y-TZP) is growing used in dentistry due to its good mechanical properties such as hardness and fracture toughness. Thus, controlling of microstructure by adding nanoalumina plays an important role in enhancing these properties.

Originality/value: Study the adding bitty percentage from nano alumina on microstructure and mechanical properties of (5Y-TZP) ceramic.

Keywords: Tetragonal, Zirconia, Composite, Nano Al_2O_3, Microstructure, Grain size

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MATERIALS

1. Introduction

Ceramics and ceramic composites are used in almost all areas of technology and have the potential for even greater and more widespread applications. To make this a truth, it is necessary to comprehend the microstructure of the material and its relationship with properties and performance [1].

Over the past several years, the manufacturing of all-ceramic dental restorations is the most interesting biomedical application of zirconia in the form of tetragonal zirconia polycrystals (TZP) because of their outstanding low-temperature mechanical properties, biocompatibility as well as aesthetic benefits [2]. The application of zirconia-based materials in the biomedical field, especially in dentistry, is currently increasing [3]. To overcome the low toughness of alumina and the aging sensitivity of zirconia, alumina-zirconia composites have been proposed for biomedical applications [4].

Alumina-Zirconia composite is an important class of toughened structural ceramic in which the strength and toughness have been improved due to stress-induced t-m transformation [5-7]. A major problem in achieving high-density Al_2O_3-ZrO_2 composites is the coarsening of ZrO_2 particles during high-temperature processing. The coarsening of ZrO_2 will not only adversely affect the densification behaviour but also will reduce the retained tetragonal ZrO_2. Ceramic matrix composites (CMC) are developed to overcome the brittleness and low reliability of monolithic ceramic materials. The main advantages of these materials include high strength at high temperatures, low weight ,and flaw tolerance [8]. 3Y-TZP is one of the most common zirconia-based ceramics used in dentistry. It consists of the tetragonal (T) phase which is the only phase that can be transformed to (M) phase [9,10].

Y-TZP has many outstanding properties, such as high strength, toughness, wear resistance, chemical resistance, and biocompatibility, which make it as a promising candidate for various structural and biomedical applications [11]. The mechanism of toughening is connected to the stress-induced tetragonal to monoclinic phase transformation; as a result, high fracture toughness can be reached in this material when containing transformable tetragonal precipitate [12]. When Y_2O_3 is added into Mg-PSZ, the t-ZrO_2 tends to stabilize, so that the process of martensitic transformation from t- ZrO_2 to m-ZrO_2 is present [12,13]. As a hard-to-sinter ceramic, polycrystalline Y-TZP usually is cloudy even after high-temperature sintering mainly because of the existence of residual pores. These pores can considerably scatter incident light and deteriorate optical properties. In addition ,the nanometric microstructure is decisive to accomplish good transparency for Y-TZP since this material has serious birefringent scattering [11,14]. The densification behaviour and microstructural development during sintering of Y-TZP doped with alumina have been investigated by numerous researchers [15-17]. This research aimed to study the effects of the addition of different amounts of nano Al_2O_3 (1, 2 ,and 4) wt.% on its microstructure and mechanical properties of (5Y-TZP) composite.

2. Materials and methods

2.1. Materials used

5 mol % yttria zirconia polycrystals were prepared by using the coprecipitation method in an aqueous solution from Zirconium oxychloride ($ZrOCl_2.8H_2O$) and anhydrous yttrium chloride(YCl_3) as starting materials and NH_4OH as precipitant. Ethanol absolute (99.9% purity, Scharlau, Spain) and distilled water was used to remove the excessive NH_4OH.

2.2. Preparation of samples

The dissolving of the materials is associated with energetic stirring and heating. To assure the homogenizing of the suspension, drop of NH_4OH solution was added to the mixture during stirring. After that a white gel is precipitated, then washed with distilled water and ethanol, to remove the excessive NH_4OH and filtered, dried at 110°C for 12 hr. This leads to the formation of hard agglomerates from 5 yttria zirconia polycrystals that were milling using pestle and mortar. To transform the 5Y-TZP powder from the amorphous structure to the crystalline structure a heat treatment such as calcination should be accomplished [18]. The electrical resistance furnace was used at temperature 800°C for 2 hours with a heating rate of 5°C/min. The powder was milled in a planetary ball mill (SMF-Disk-Top Planetary Ball Miller) for 6 hr. and the speed of milling at 500 r.p.m. Lastly, composite materials were prepared by adding 5Y-TZP and different compositions of nano Al_2O_3 particles (1, 2, and 4) wt.% with grain size 50 nm to the ethanol with stirring for 60 min. to ensure the distribution of the nano Al_2O_3 in the 5Y-TZP matrix. After that, the slurry was treated by using ultrasonic mixing (ultrasonic presser MSK, MTI Corporation, USA) to ensure the dispersion of the nano Al_2O_3 in the 5Y-TZP matrix. Then, the slurry was dried for one hour at 80°C to eliminate the residue ethanol.

Mixing and compacting of powder

The prepared composite powder was mixed with the binder polyvinyl alcohol (PVA). Approximately 10 g of powder was uniaxial pressed using a cylindrical die at a pressure of 150 MPa at room temperature and held for 60 s by using (CT340 – Automatic - 2000kN – Cube - and Cylinder - Compression Machine).

Sintering of green compact

To remove the binder from the green compact at slow rate, the specimens were heated to 450°C and cooling rate of 5°C/min. To attain dense zirconia-based materials, it is necessary to using high heating rates to overturn grain boundary diffusion at lower temperatures and stimulate bulk diffusion at higher temperatures. The green compact specimens are heated to the 1500°C, held for two hours ,and then cooling down at a rate of (5°C/min) to R.T. The sintering treatment was fulfilled in Zirkonofen 600 furnace, Zirkon Zahn company). Figure 1 shows the sintered compacted sample.

Fig. 1. Shows the sintered compact sample

2.3. Characterizations of microstructure

In order to observe the morphology, grain boundaries, phases, grain sizes, and other microstructural details in ceramic specimens. The surface of the sample was polished and thermally etched at 1350°C for one hour. Then the samples were examined using Field Emission Scanning Electron Microscopy (FESEM).

2.4. Vickers hardness

The microhardness measurements were performed by using a microhardness tester (Digital Micro Vickers Hardness Tester – TH-714) fitted with a Vickers indenter. Vickers hardness is measured according to ISO 14705:2008 [21]. The applied force 9.8 N, dwell time 30 sec ,and average of seven measurements were taken as an average value of HV on cylindrical samples. Fracture toughness of 5Y-TZP/nano Al_2O_3 composite was optioned by using Vickers indentation fracture test. This method is one of the most untraditional methods to measure fracture toughness and consider a non-destructive test. The cracks lengths were measured by using an optical microscope. The fracture toughness was calculated according to reference [22].

$$K_{IC} = 0.016 \frac{F}{\sqrt[3]{C^2}} \times \left({}^{e}/_{H_V}\right)^{0.5} \quad (1)$$

where:

K_{CI} is the fracture toughness (MPa.$m^{1/2}$)

F is the applied load (N)

C is cracking length from the centre of the impression to the crack tip (mm), E is Young modulus(MPa).

3. Results and discussion

Figure 2 a typical scanning electron microscope image of the yttria tetragonal zirconia polycrystal (5Y-TZP) sample sintered at 1500 °C shows the morphology, porosity, grain boundaries grains size and tetragonal phase. The microstructure consists of the solid solution of ZrO_2 and a stabilizing oxide Y_2O_3. The yttria dopant is added to stabilize the high-temperature tetragonal in the sintered microstructure. As can be the image reveals that the sample has low densification and is composed of large grains with some finer grains ranging between 105.08 nm and 205.21 nm (on the average of 141.87 nm) at higher magnification power. This result is agreed with Garcia, R. H. L., et al.[23].

Figure 3 presents SEM micrographs of the micro-structure sintered nanocomposite of the yttria tetragonal zirconia polycrystal (5Y-TZP – 1% nano Al_2O_3), it can be notice high densification, homogeneous zirconia distribution ,and grain growth destruction were obtained. In addition to it was found at higher magnification power significant differences in particle size and the reduction of some crystallite size to the nanometre regime can result in the stabilization of high-temperature phases ranging between 221.99 nm and 72.39 nm. With an increasing percentage of nano Al_2O_3 in the yttria tetragonal zirconia polycrystal samples(Figure 4 and Figure 5) it can be seen a decrease in porosity and enhancement in the densification of zirconia matrix (5Y-TZP – 2 % nano Al_2O_3 and 5Y-TZP – 4% nano Al_2O_3). Moreover, continues decreasing the size of grains ranging between 74.22 nm and 124.28 nm (on the average of 103.51 nm) at higher magnification power of 5Y-TZP – 2 % nano Al_2O_3 sample as shown in Figure 4(b,c) and all crystallite size to the nanometre ranging between 72.08 nm and 95.15 nm (on the average of 83.77 nm) at higher magnification power of 5Y-TZP – 2 % nano Al_2O_3 sample as shown in Figure 5(b and c). This observation in agreement with other researchers' results [24].

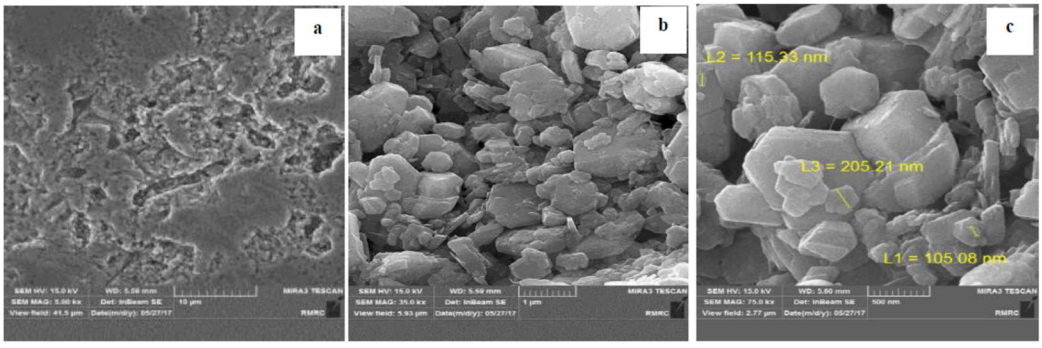

Fig.2. Scanning electron microscope image for 5Y-TZP ceramic at different magnification power

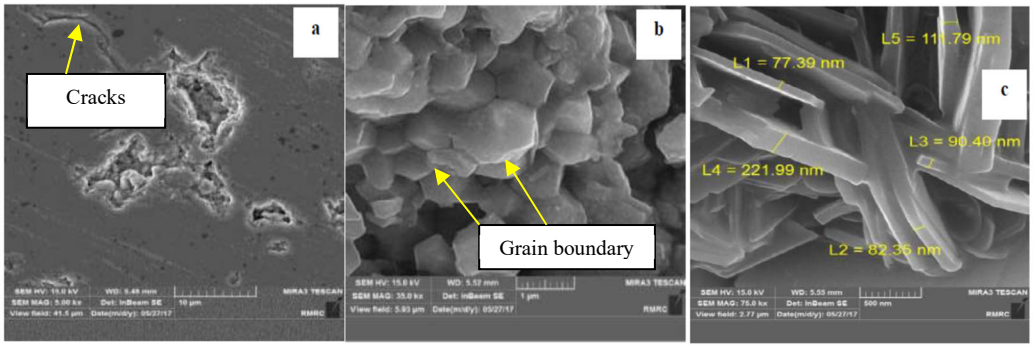

Fig. 3. Scanning electron microscope image for 5Y-TZP – 1 wt.% nano Al_2O_3 at different magnification power

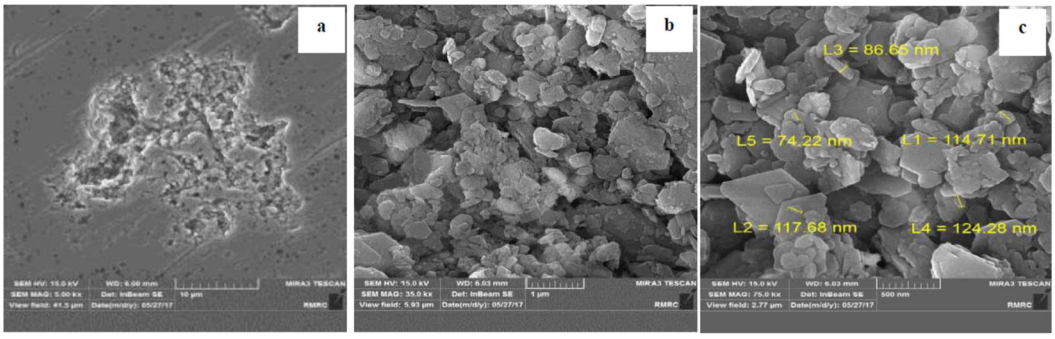

Fig. 4. Scanning electron microscope image for 5Y-TZP – 2 wt.% nano Al_2O_3 at different magnification power

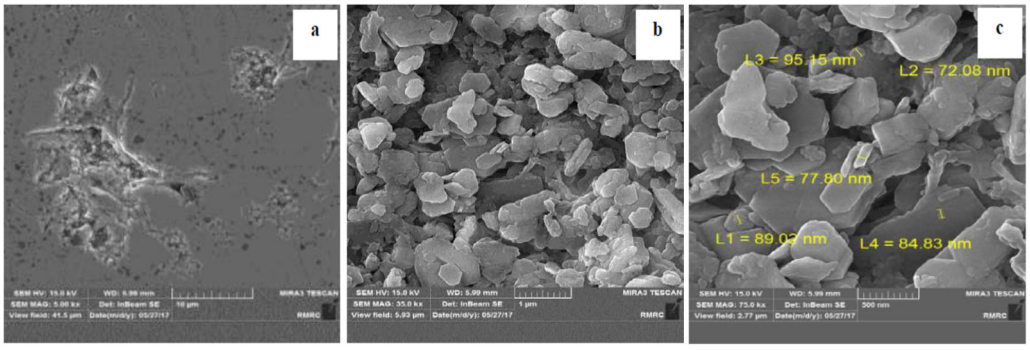

Fig. 5. Scanning electron microscope image for 5Y-TZP – 4. wt.% nano Al_2O_3 at different magnification power

Figure 6 shows the relationship between the Vickers microhardness and the addition of nano Al_2O_3 of 5Y-TZP/nano Al_2O_3 composite. It is observed that with an increase in the amount of nano Al_2O_3 the hardness of 5Y-TZP/nano Al_2O_3 composite will increase up to 1550 HV because disappear the porosity in the microstructure of samples with increasing of nano Al_2O_3 and the smaller grain size may be the major reason for the improved hardness values observed, this agreement with reference [25]. Another reason to explain the mechanism of increased hardness of 5Y-TZP nano Al_2O_3, the dispersed phase (nano alumina) opposes the movements of dislocation either by creating a stress field in the matrix or by requiring atomic disordering if a dislocation in to cut through a particle [26].

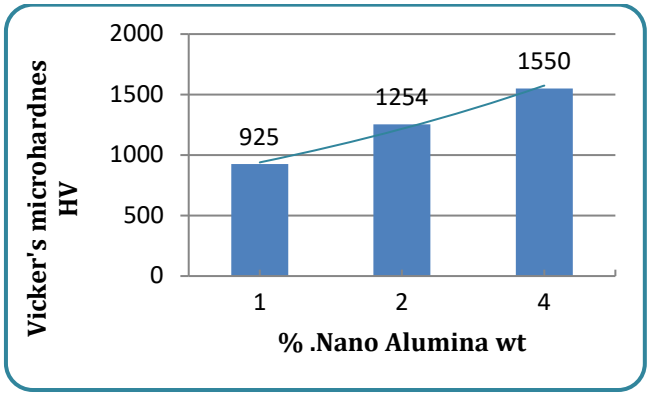

Fig. 6. Vicker's microhardness of 5Y-TZP/nano Al_2O_3 samples

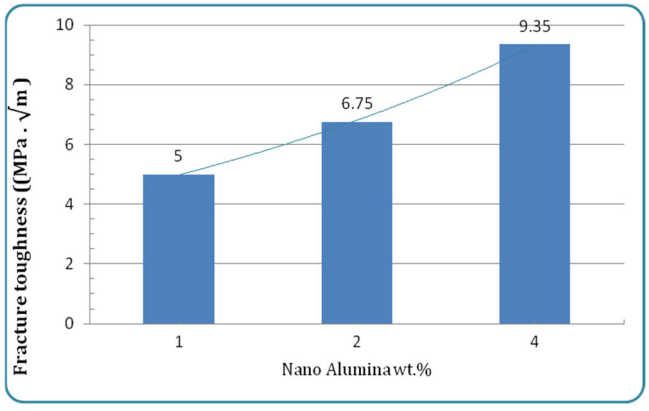

Fig. 7. Fracture toughness of 5Y-TZP/nano Al_2O_3 samples

The fracture toughness of the 5Y-TZP/nano Al_2O_3 composite is shown in Figure 7. The fracture toughness increases from 5 MPa.√m to 9.35MPa.√m with increasing Al_2O_3 content. The increment in fracture toughness can be elucidated as the reduction in porosity and grain size due to the densification process. The reduction in grain size leads to a reduction in the flaw size and according to the principle of fracture toughness[27].

4. Conclusions

- The microstructure of 5Y-TZP ceramic consists matrix of solid solution zirconia and stabilizing yttria.
- The 5Y-TZP sample has low densification and is composed of large grains with some finer grains ranging between 105.08 nm and 205.21 nm (on the average of 141.87 nm).
- Decrease the size of grains with an increasing percentage of nano Al_2O_3.
- Homogeneous zirconia distribution and grain growth destruction with an increasing percentage of nano Al_2O_3.
- Decreasing in porosity with increased percentage of nano Al_2O_3 and enhancement in the densification of 5Y-TZP /nano Al_2O_3 composite samples.
- Increasing Vicker's microhardness of 5Y-TZP /nano Al_2O_3 composite with the increased nano Al_2O_3 wt.% content.

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