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### Field-assisted sintering of multiphase toughening zirconia ceramics

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Field-assisted sintering technology was used to fabricate multiphase zirconia ceramics composed of tetragonal and cubic phases. The composites were based on 3 mol. % yttria tetragonal zirconia polycrystals (3Y-TZP) with the addition of 8.5 mol. % yttria-stabilized zirconia (8.5Y-YSZ). The phase composition of the ceramics was characterized by X-ray diffraction, and the fracture surface of the bulks was examined by field emission scanning electron microscopy. The phase volume fractions of the tetragonal and cubic phases in the sintered ceramics were estimated and the mechanical properties of the ceramics were analyzed. The multiphase structure and mechanical properties of the ceramics were investigated. The results showed that 10 wt. % 8.5YSZ with a yttria content of 3.55 mol. % exhibited the optimal mechanical properties. The maximum fracture toughness and Vickers hardness were obtained in the ceramic with a tetragonal composition of 99.7 vol. % that was sintered at 1400°C. These results suggest that the improved mechanical properties of the composites are due to a multiphase effect.

Key words: Multiphase zirconia ceramics, Field assisted sintering technology, Microstructure, Phase transformation, Mechanical properties.

### Introduction

Ceramics have many applications owing to their excellent properties such as density, hardness, wear, and heat resistivity. One of the most important ceramics is zirconia (ZrO<sub>2</sub>), which possesses excellent mechanical, physical, electrical, and chemical properties, including high strength, hardness, impact toughness, wear resistance, low friction ratio, high melting point, low heat conductivity, chemical inertness, good biocompatibility, and potentially, superior esthetics [1-5]. Therefore, it has been an attractive candidate for several applications in different fields for a long time [6]. Pure ZrO<sub>2</sub> is a polymorphic material and can exist in three crystals structures under atmospheric pressure: monoclinic phase (*m*), which is stable from room temperature up to  $1170 \,^{\circ}\text{C}$ ; tetragonal form (t), which is stable between  $1170 \,^{\circ}\text{C}$  and 2370 °C; and cubic structure (c), which is stable at temperatures from 2370 °C to the melting point (2680 °C) [7, 8]. Unalloyed  $ZrO_2$  cannot be used or produced due to cracks induced by a phase transformation that occurs during cooling from high temperatures [9]. To prevent such transformations, many metal oxides, such as CaO, MgO, CeO, or  $Y_2O_3$ , which are termed stabilizing agents, can be added to ZrO<sub>2</sub>; hence, the high temperature phases of  $ZrO_2$  can be maintained at room temperature [10]. Among the many metal oxides used to stabilize  $ZrO_2$ , yttria  $(Y_2O_3)$  is the most common stabilizer that has been used widely in most applications of zirconia [11, 12]. On the other hand, a Y<sub>2</sub>O<sub>3</sub> concentration in ZrO<sub>2</sub> below 2 mol. % is insufficient to stabilize the tetragonal phase at room temperature [13, 14]. Therefore, 3 mol. % Y<sub>2</sub>O<sub>3</sub> or more is required to obtain a tetragonal crystal structure of ZrO<sub>2</sub> at room temperature, whereas the cubic form can be maintained at room temperature with the addition of approximately 8 mol. % Y<sub>2</sub>O<sub>3</sub> [15]. Moreover, there are two types of tetragonal zirconia, one is the transformable tetragonal phase (t), which can be transformed to monoclinic phase under various stresses, and the other is called a metastable tetragonal phase (t'), which cannot be transformed to a monoclinic phase when exposed to a range of stresses [16]. The major difference between these two phases is the amount of  $Y_2O_3$  in the ZrO<sub>2</sub> matrix, which is estimated to be 4 mol. % or more for the metastable tetragonal phase. In addition, the applications of zirconia are reliant on their crystal structure and phase transformations [16-18]. On the other hand, the stability of zirconia depends strongly on both the amount of stabilizer and grain size, which can affect the mechanical properties of  $ZrO_2$  [19]. YSZ ceramics with the optimal mechanical properties can be obtained with tetragonal zirconia containing approximately 3 mol. % Y<sub>2</sub>O<sub>3</sub>due to a toughening mechanism that occurs as a result of a martensitic transformation from the t phase to the mphase when subjected to various stresses [16, 20, 21].

The mechanical properties of ceramic materials are influenced strongly by their microstructure, microcracks,

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Composites	D	Sintering	Fracture Tough	ness [MPa $\cdot$ m <sup>1/2</sup> ]	Hardness	Reference
	Process		N <sup>a</sup>	A <sup>b</sup>	[GPa]	
3Y-TZP- 5 wt.%Al <sub>2</sub> O <sub>3</sub>	CS	1450 °C-2 h	_	$4.20\pm0.20$	$13.76\pm0.17$	Ref. [27]
3Y-TZP- 1.5 wt. %SWCNTs	SPS	1250 °C-5 min	_	$4.60\pm0.30$	$12.90\pm0.30$	Ref. [29]
3Y-TZP- 2 vol. % MWCNTs	SPS	1350 °C-5 min	_	$4.50\pm0.10$	$13.30\pm0.30$	Ref. [30]
3Y-TZP- 3.5 wt. % WCNTs	SPS	1350 °C-1 0min	$5.04\pm0.59$	_	$12.55\pm0.50$	Ref. [31]
3Y-TZP- 2 wt. % CNT	SPS	1350 °C-5 min	_	$4.97\pm0.06$	$9.52\pm0.05$	Ref. [32]
3Y-TZP	MS	1400 °C-5 min	$4.48\pm0.20$	_	15.09	Ref. [39]

Table 1. Mechanical properties of YSZ ceramics obtained in previous studies.

<sup>a</sup>Fracture toughness calculated using the expression reported by Niihara [53].

<sup>b</sup>Fracture toughness calculated using the expression reported by Anstis et al.[54].

and residual stress [22]. Moreover, the applications of ceramics materials as engineering components are restricted by their brittleness. To produce tough ceramics with high reliability and enhanced toughness, several studies have evaluated the addition of a second phase with high strength, high modulus, and high ductility [23-25]. Many substances in different forms have been added to the zirconia matrix to enhance its mechanical properties, such as flexural strength, fracture toughness, and hardness. Al<sub>2</sub>O<sub>3</sub> is one of the most commonly used additives for such purpose [26, 27]. Other materials, such as boron nitride nanotubes (BNNTs) [28], single wall carbon nanotubes (SWCNT) [29], or multiwall carbon nanotubes (MWCNTs) [30-32], have also been investigated. Unfortunately the addition of a second phase is often restricted by the effects of the second phase on the other properties, such as electrical and thermal properties. To overcome this limitation, different methods have been used to produce monolithic YSZ, focusing on the grain size and / or  $Y_2O_3$  content in  $ZrO_2$ . A suitable grain size can be maintained using either nanocrystalline powders of YSZ with a low Y2O3 content [33, 34], or employing a range of sintering processes using different sintering conditions, such as conventional sintering (CS) [35], spark plasma sintering (SPS) [22, 36], two-step sintering (TSS) [37, 38], microwave sintering (MS) [35, 39-41], or pulsed electric currentassisted-sintering (PECS) [42]. On the other hand, the concentration of  $Y_2O_3$  in  $ZrO_2$  can be controlled using a range of methods, such as doping Y<sub>2</sub>O<sub>3</sub> into ZrO<sub>2</sub>, which is the most common process that involves several chemical methods using different precursors of both oxides [15, 43, 44]. Another method is to mix different amounts of Y2O3 powder and ZrO2 powder by ball milling [45]. The mixing of different powders of YSZ with different yttria contents to fabricate multiphase zirconia ceramics is rare. Nevertheless, new ideas, methods, and approaches are still needed to further enhance the performance of YSZ ceramics to expand its

applications. Table 1 lists the results of previous studies.

Field assisted sintering technology (FAST) is considered a non-conventional sintering process, and includes spark plasma sintering (SPS) and plasma activated sintering (PAS). In the latter, a pulse voltage is employed before sintering, which shock vaporizes impurities on the particles surfaces [46, 47]. In addition, during sintering a combination of pressure application and plasma generation with resistance heating can be established [48]. Moreover, the plasma environment activates the particle surface and promotes the sinterability. Therefore, the sintering process can be performed at lower sintering temperatures within a very short time.

In the current study, multiphase YSZ powders were prepared by mixing two powders of YSZ containing different amounts of  $Y_2O_3$  (3 and 8.5 mol %) using a planetary ball milling machine followed by sintering using FAST. Multiphase zirconia ceramics with a tetragonal phase and cubic phase were produced and the phase compositions, microstructure, and mechanical properties were characterized. The effects of the multiphase on the mechanical properties of the zirconia ceramics were investigated.

### **Experimental Procedure**

### Raw material and processing

Two commercially available YSZ powders (Ausmasco Pharma Co., Ltd., Japan) with high purity (99.9%) were selected as the starting materials. The  $Y_2O_3$  concentrations in the as-received powders were 3 and 8.5 mol. % (referred to as 3Y and 8.5Y, respectively). Both powders had a specific surface area of 10 m<sup>2</sup>/g and a mean particle size of 500 nm. Different amounts of 8.5Y powder (10, 30, 50, 70, and 90 wt. %) were added to the 3Y powder to produce the multiphase YSZ powders. To obtain a more homogeneous particle distribution, the powders were first dissolved in ethanol using a porcelain jar and then ball milled using a planetary ball milling machine (QM-

Table 2 Amount of 8.5Y wt. %, nomenclature, and estimated Y<sub>2</sub>O<sub>3</sub> content in the composites.

8.5Y [wt.%]	0	10	30	50	70	90	100
Nomenclature	3Y	3Y/10-8.5Y	3Y/30-8.5Y	3Y/50-8.5Y	3Y/70-8.5Y	3Y/90-8.5Y	8.5Y
Y <sub>2</sub> O <sub>3</sub> [mol. %]	3	3.55	4.65	5.75	6.85	7.95	8.5

3SP2, Nanjing) at a speed and holding time of 300 rpm and 24 h, respectively, using zirconia balls as the milling media. The resulting mixtures were dried at 80 °C in air for three days. Finally, the dried mixtures were ground and sieved through an 80 mesh screen. The pure powders, 3Y and 8.5Y, were also subjected separately to such processes to ensure the same preparation conditions. The final concentrations of  $Y_2O_3$  in the mixed powders, which are listed in Table 2, were estimated using a simple mixing rule.

### **Ceramic sintering**

FAST was used to perform the sintering process by applying a plasma activated sintering system (ED-PAS III, Elenix Ltd., Japan). Initially, a suitable amount of the as-prepared powders was poured into a graphite die, 32 mm in diameter, and graphite papers were used to separate the powders from the die and punch surfaces. Secondly, the samples were sintered at 1200 °C, 1300 °C, and 1400 °C under a uniaxial pressure of 20 MPa for 3 min as the soaking time in the presence of  $N_2$ . The heating rate from room temperature was 100 °C/min and an infrared apparatus was employed to observe the temperature throughout the sintering process. The sintered samples were ground with a precision surface grinder (BLOHM Orbit 25EP, Germany) and polished to 0.05 µm using a precision automatic polishing machine (Simplimet xpsl, Ecomet 250/Automet 250).

### Characterization

The phases of the as-received and as-prepared powders as well as the sintered ceramics were examined by X-ray diffraction (XRD, Ultime III, Rigaku, Japan) using Cu-Ka radiation (wavelength,  $\lambda = 1.5406$  Å). The XRD patterns were obtained first from 10° to 120° 2 $\theta$  with a scan speed of 2 °/min and 4 °/ min for the powders and sintered ceramics, respectively. Owing to the structural similarity of the tetragonal phase and cubic phase, the XRD diffraction peaks of these two forms cannot be separated at a small diffraction angle. Therefore, XRD of the sintered ceramics was also carried out over a narrow 20 range from 72  $^{\rm o}$  to 76  $^{\rm o}$  20 with a step width of 0.02 ° and a scan time of 15 s per step to distinguish between the peaks of the tetragonal and cubic phases and to calculate the volume fraction of each phase. The integrated intensity ratio for the tetragonal phase,  $X_t$ , was estimated using equation (1) [11, 49, 50]

$$X_{t} = \frac{I(004)_{t} + I(220)_{t}}{I(004)_{t} + I(220)_{t} + I(400)_{c}}$$
(1)

where  $I_t$  and  $I_c$  represent the integrated intensity of the tetragonal (004) and (220) and cubic (400) peaks after a background correction

Tetragonal phase volume fraction,  $V_t$  was determined using equation (2) [50]

$$V_t = \frac{PX_t}{1 + (P-1)X_t} \tag{2}$$

where P is a correction factor that depends on the  $Y_2O_3$  content in the  $ZrO_2$  matrix, which was calculated from the nonlinear calibration curve of the volume fraction vs. integrated intensity ratio [50].

The densities of the sintered bodies were measured using the Archimedes procedure in distilled water and the theoretical densities of pure 3Y and 8.5Y were assumed to be 6.074 and 5.96 g/cm<sup>3</sup>, respectively. Field emission scanning electron microscopy (FE-SEM, Quanta-250) was performed to characterize the microstructure of the fracture surface of the obtained ceramics. The mean grain size was measured using the linear intercept length method.

The fracture toughness (K<sub>IC</sub>) and Vickers hardness (Hv) were measured by a Vickers microhardness tester (Wolpert430 SVD, USA). A total of 10 indentations were made for each sample using a Vickers indenter by applying a load of 98.1 N on the polished surface with a dwell time of 15 s. The fracture toughness was calculated using the indentation fracture (IF) method. Two types of crack patterns are produced using a Vickers indenter in the indented specimens: the Palmqvist crack mode, which is comprised of four cracks with a semi-elliptical shape; [51] and the median/radial crack form, which is composed of two half penny-shaped cracks [52]. For the Palmqvist indentation crack type, the formula proposed by Niihara [53] is most suitable for calculating the indentation, K<sub>IC</sub>, whereas the expression of Anstis et al. [54] is commonly used to calculate K<sub>IC</sub> for the median/radial crack type. In this study, for comparison, both formulae [53] and [54] were used to calculate  $K_{IC}$ . FE-SEM (Quanta-250) was used to measure the diagonals and cracks of the indentation. The Young's modulus, E, was calculated using the following equations (3):

$$E = (L^{3} F)/(4bd^{3} y)$$
(3)



Fig. 1. XRD patterns of the (a) as-received powders and (b) as-prepared powders.

where L, b, and d is the span length, width, and thickness of the sample, respectively, F is the maximum applied load, and y is the deflection corresponding to the maximum load.

### Results

## Phase compositions of the as-received and as-prepared powders

Fig. 1 (a) and (b) presents XRD patterns of the asreceived and as-prepared powders, respectively. As shown in Fig. 1 (a), XRD of the 3Y powder revealed a tetragonal phase with the presence of a monoclinic phase, whereas only a cubic phase was observed in the 8.5Y powder. In addition, the as-prepared powders were composed of monoclinic, tetragonal, and cubic phases; the amount of monoclinic phase decreased with increasing amount of 8.5Y wt. % powder, as shown in Fig. 1 (b).

## Phase compositions of the sintered multiphase zirconia ceramics

Although the monoclinic phase was observed in the as-received and as-prepared powder, XRD of the bulk only reveled tetragonal and cubic phases, as shown in Fig. 2(a-c), which can confirm that the monoclinic phase had been transformed completely to a high temperature phases with no martensitic transformation from the t phase to the m phase during sintering process. Fig. 3(a-e) presents XRD patterns from 72 ° to 76° 20 of the various ceramics sintered at different temperatures. As shown in Fig. 3(b-d), the peaks of the tetragonal phase (400)t and (220)t and a peak of the cubic phase (400)c were observed in XRD patterns of the ceramics sintered at different temperatures, which were produced from the as-prepared powders. These peaks were used to distinguish between the tetragonal phase and cubic phase and to calculate the volume fractions of these two phases. The volume fractions of the tetragonal and cubic phases were strongly influenced by the amount of 8.5Y and temperature. At the same sintering temperature, the tetragonal volume fraction decreased with increasing amount of 8.5Y, and



Fig. 2 XRD patterns between  $10^{\circ}$ - $120^{\circ} 2\theta$  of the ceramics sintered at (a)  $1200^{\circ}$ C (b)  $1300^{\circ}$ C and (c)  $1400^{\circ}$ C.

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**Fig. 3.** XRD patterns between 72 °-76 ° 20 of various ceramics sintered at different temperatures (a) 3Y (b) 3Y/10-8.5Y (c) 3Y/50-8.5Y (d) 3Y/90-8.5Y and (e) 8.5Y.

Table 3. Density, theoretical density, relative density, vol. % of tetragonal and cubic phases, mean grain size, and Vickers hardness of some ceramics sintered at different temperatures.

Nomenclature	Temperature [°C]	Density [g/cm <sup>3</sup> ]	Theoretical density [g/cm <sup>3</sup> ]	Relative density [%]	T phase [vol.%]	C phase [vol.%]	Mean grain size [µm]	Vickers hardness [GPa]
3Y	1300	6.05	6.07	99.67	100.0	00.00	0.59	$13.96\pm0.27$
3Y	1400	6.06	6.07	99.84	100.0	00.00	0.71	$13.72\pm0.10$
3Y/10-8.5Y	1200	6.06	6.07	99.84	99.33	00.67	0.54	$13.43\pm0.33$
3Y/10-8.5Y	1300	6.07	6.07	100.0	99.52	00.48	0.65	$13.65\pm0.39$
3Y/10-8.5Y	1400	6.07	6.07	100.0	99.70	00.30	0.80	$14.01\pm0.22$
3Y/50-8.5Y	1300	6.02	6.04	99.67	70.53	29.47	0.69	$13.91\pm0.24$
3Y/50-8.5Y	1400	6.04	6.06	99.67	95.70	04.30	0.88	$13.70\pm0.22$
3Y/90-8.5Y	1300	5.96	5.99	99.50	18.1	81.90	0.83	$13.83\pm0.51$
3Y/90-8.5Y	1400	5.97	5.99	99.67	21.11	78.89	0.99	$13.12\pm0.88$
8.5Y	1300	5.93	5.98	99.16	0.8	99.20	0.86	$13.31\pm0.49$
8.5Y	1400	5.95	5.99	99.33	2.16	97.84	1.05	$13.70\pm0.51$

hence  $Y_2O_3$  content. The tetragonal volume fraction increased with increasing sintering temperature for the same 8.5Y content. In addition, the (004)t peak of the tetragonal phase was detected in the composites fabricated from the pure 8.5Y powder sintered at 1300 °C and 1400 °C (Fig. 3(e)). Table 3 lists the vol. % of the tetragonal and cubic phases for some ceramics sintered at different temperatures.

# Relative density of the sintered multiphase zirconia ceramics

The relative density of the bulks was affected strongly by both the temperature and amount of 8.5Y added to 3Y. As expected, the relative density of all ceramics increased with increasing sintering temperature. On the other hand, the relative density decreased with increasing amount of 8.5Y, which would increase the



**Fig. 4.** SEM images of the fracture surfaces of (a) 3Y/10-8.5Y sintered at  $1200 \,^{\circ}$ C, (b) 3Y/10-8.5Y sintered at  $1300 \,^{\circ}$ C, (c) 3Y/10-8.5Y sintered at  $1400 \,^{\circ}$ C, (d) 3Y sintered at  $1300 \,^{\circ}$ C, (e) 3Y/50-8.5Y sintered at  $1300 \,^{\circ}$ C, and (f) 8.5Y sintered at  $1300 \,^{\circ}$ C.

 $Y_2O_3$  concentration, and increase the ability to form oxygen vacancies, resulting in higher porosity. The fluctuations of the relative density are due to the variations of the estimated theoretical density, which is affected significantly by the volume fraction of both the tetragonal and cubic phases that had been calculated previously from the XRD patterns. On the other hand, most of the ceramics showed a relative density of 90% or more of the theoretical density, whereas full density was achieved for the ceramics containing 10 wt. % 8.5Y and 3.55 mol. %  $Y_2O_3$  which were sintered at 1300 °C and 1400 °C. Table 3 lists the density, theoretical density, and relative density of some zirconia ceramics sintered at different temperatures.

### Microstructure of the sintered ceramics

The grain size has significant effects on the mechanical properties. Fig. 4 presents SEM images of the fracture surfaces of various ceramics sintered at different temperatures. The grain size increased significantly with increasing  $Y_2O_3$  content due to the increasing amount of 8.5Y wt. %. In addition, the grain size increased with increasing sintering temperature. Table 3 lists the mean grain size of some ceramics sintered at different temperatures.

## Mechanical properties of the multiphase zirconia ceramics

For the fracture toughness measurements, G. D. Quinn et al. [55] used one of the standardized methods [56-58] to obtain more reliable and comparable fracture toughness values instead of micro-indentation methods, because they claimed that the latter does not provide the real fracture toughness. On the other hand, micro-indentation methods are used widely for measuring the



**Fig. 5.** Fracture toughness of the ceramics as a function of the T phase vol. % calculated using the expressions proposed by (a) Niihara [53] and (b) Anstis et al. [54]. The zone congested by the data is clarified in the rectangles attached.

fracture toughness owing to their simplicity and they can be employed for specimens with a smaller size [54]. Therefore, in the current study, the microindentation method was used to measure the fracture toughness of the obtained ceramics. Fig. 5(a) and (b) presents the fracture toughness of all the composites sintered at different temperatures as a function of the tetragonal phase vol.% for the Palmqvist crack mode and median/radial crack type, respectively. Multiphase zirconia ceramic with 99.7 tetragonal phase vol. % and 10 wt. % 8.5Y (3.55 mol. %  $Y_2O_3$ ), which was sintered at 1400°C, possessed a maximum fracture toughness of  $5.74 \pm 0.3$  MPa  $\cdot$  m<sup>1/2</sup> and  $4.89 \pm 0.25$  MPa  $\cdot$  m<sup>1/2</sup> for a Palmqvist crack and a median/radial crack type, respectively. Compared to the pure 3Y zirconia ceramic sintered under the same conditions, 10.38% and 7% increases in fracture toughness were calculated using the expressions proposed by Niihara [53] and Anstis et al. [54], respectively. On the other hand, the fracture toughness decreased with more 8.5Y addition, which led to an increase in the concentration of Y2O3 and caused a decrease in the volume fraction of the tetragonal phase.

The highest micro-hardness of  $14.01 \pm 0.22$  GPa was obtained in the samples sintered at 1400 °C with a tetragonal phase vol. % of 99.7 and a 8.5Y content and Y<sub>2</sub>O<sub>3</sub> concentration of 10 wt. % and 3.55 mol. %, respectively. The hardness decreased with decreasing density. Table 3 lists the Vickers hardness for some ceramics sintered at different temperatures.

### Discussion

### Microstructure of the multiphase zirconia ceramics

The 8.5Y/3Y ceramics were composed of both tetragonal and cubic phases, which is in good agreement with the phase diagram of the  $ZrO_2$ - $Y_2O_3$  system [59, 60]. Therefore, a multiphase structure was obtained by adding full cubic zirconia powder (8.5Y) to the tetragonal zirconia polycrystal powder (3Y-TZP). In other words, the concentration of Y2O3, and the phase composition was controlled by adding 8.5Y. As mentioned before, Sheu et al. [61] reported a decrease in the tetragonal phase volume fraction with increasing  $Y_2O_3$ . On the other hand, the volume fraction of the tetragonal phase increased with increasing temperature for the same amount of 8.5Y, which is in line with previous reports [61] and [62]; this can be related to anisotropic thermal expansion and the transformation from a cubic to a tetragonal phase during cooling from the sintering temperature, respectively. The grain size increased with increasing sintering temperature and Y<sub>2</sub>O<sub>3</sub> concentration. The latter is consistent with previous results [63-65], which stated that the grain growth of YSZ is affected by the phase content, and suggested that grain growth of the cubic phase of YSZ is approximately 30-250 times faster than that of the tetragonal phase.



**Fig. 6.** SEM images of (a) micro-hardness indent, (b) crack deflection, (c) crack bridging, and (d) crack mode.

## Effects of a multiphase on the mechanical properties of zirconia ceramics

The improved mechanical properties, particularly the fracture toughness, are due to the stress-induced martensitic transformation of the transformable tetragonal phase t to the monoclinic phase m [16, 20, 21], which is associated with volume expansion. This expansion can absorb energy and decrease the elastic strain at the crack tip [66, 67], resulting in an increase in the fracture toughness. Transformation toughening increases with increasing grain size [33, 68], particularly at a relatively high concentrations of  $Y_2O_3$  (~ 3 mol. %), which is in good agreement with the present results; the highest fracture toughness was achieved with a relatively coarse grain size of approximately 0.8 µm within the sample sintered at the highest temperature of 1400 °C. On the other hand, although the grain size increased, the fracture toughness decreased with increasing amount of Y<sub>2</sub>O<sub>3</sub>. This was attributed to the presence of a nontransformable phase (t'), which is usually formed at a Y<sub>2</sub>O<sub>3</sub> concentration of more than 4 mol. %; this phase has no ability to transform to the monoclinic phase, as mentioned before. Moreover, strong interfacial bonding between the YSZ grains would be achieved due to the excellent homogeneity of the composite. Such powerful bonding forces the crack to change its direction to weak interfacial bonding, causing crack deflection (Fig. 6(b)), which forms a longer sinuous path that acts to release the stress and energy and increase the fracture toughness. In addition, a crack bridge (Fig. 6(c)) will be formed when crack deflections cannot take place due to sufficient interfacial bonding, which will effectively dissipate the energy and enhance the fracture toughness. Furthermore, the strong interfacial bonding with highly dense and compact grains are helpful for increasing the

fracture toughness, because they provide very high resistance to crack propagation via crack mode, which involves both inter-granular and trans-granular fracture, the latter being more dominant (Fig. 6(d)).

The fracture toughness obtained in the current study was larger than those obtained elsewhere [27, 29, 30], which applied the expressions proposed by [54], and also higher than those obtained in [31, 39], who used the formula proposed by [53]. Note that the same method and formulae were used to calculate the fracture toughness. In the present study, the modulus of elasticity was calculated. In contrast, Borrell et al. [39] used a given value of 220 MPa for the modulus of elasticity, which resulted in a larger fracture toughness. The results showed that there is a strong dependence of the micro-hardness on the density rather than the grain size or phase content.

### Conclusions

Multiphase zirconia powders composed of three phases, monoclinic, tetragonal, and cubic, were prepared by adding different amounts of fully cubic 8.5Y-YSZ powder to tetragonal zirconia polycrystal powder 3Y-TZP using a ball milling machine. The as-prepared powders were sintered at different sintering temperatures using FAST, and multiphase zirconia ceramics with different Y2O3 contents and different volume fractions of tetragonal and cubic phases were fabricated. The relationship among the multiphase, microstructure, and mechanical properties was investigated systematically. The addition of cubic YSZ powder to 3Y-TZP powder could improve the mechanical properties at a low  $Y_2O_3$ content via the multiphase structure. Full density and excellent mechanical properties were achieved compared to the samples obtained from monolithic 3Y-TZP. In addition, the enhanced fracture toughness was attributed to transformation toughening, crack bridging, crack deflection, and fracture mode, which was dominantly trans-angular. The increased hardness is related to the high density. On the other hand, an inappropriate amount of cubic YSZ powder leads to a significant decrease in mechanical properties due to either the low volume fraction of the tetragonal phase or a decrease in the relative density.

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