Fracture Toughness of 3Y-TZP Dental Ceramics by Using Vickers Indentation Fracture and SELNB Methods

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ABSTRACT

The objective of this research is to analyze the fracture toughness of pure and silica co-doped yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) bioceramics containing 0.1 and 0.2 wt.% of alumina, and sintered at a temperature of 1500 °C. Because of the relatively easy preparation of the test specimens and the high speed of testing, the Vickers indentation fracture (VIF) technique is more frequently used to evaluate the fracture toughness of biomaterials and hard biological tissues. The Young's modulus and hardness values were obtained by means of nanoindentation and indentation methods. The fracture toughness values of 3Y-TZP bioceramics were calculated and analyzed using 15 equations related to the VIF technique, and loadings of 49.03 and 196.13 N with a Vickers diamond. For validation, the results were compared with fracture toughness values obtained by the single-edge laser-notch beam (SELNB) method with an almost atomically sharp laser-machined initial notch.

Key words : Zirconia, Fracture toughness, Dental materials, Bioceramics

1. Introduction

Stabilized zirconia, a polycrystalline ceramic material, is an important structural ceramic that is used as a biomaterial for dental and bone implants.^{1,2)} Yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is known to be an important structural ceramic with excellent mechanical properties, such as high fracture toughness, strength, and hardness.^{3–5)} In clinical applications, the performance of dental ceramics is influenced by several parameters, including fracture toughness.⁶⁾ 3Y-TZP with 3 mol% Y_2O_3 .^{7–11)} as a phase stabilizer is well-known for dental application by machining pre-sintered blocks.⁷⁾

The ability to preserve the tetragonal phase in PSZ at room temperature leads to favorable mechanical properties, especially a high K_{IC} value.¹¹⁾ When stress in Y-TZP ceramic causes a crack, (t) → (m) phase transformation occurs at the crack tip; as a result, an expansion in the ceramic causes compressive stresses around the crack and prevents its propagation, and thus, K_{IC} increases. This reinforcing mechanism is known as the transformation toughening mechanism, and it makes zirconia much tougher than other ceramic materials.^{12–14)} For example, the fracture toughness of Y-TZP, which is used as a core material in three- or four-unit fixed dental prostheses (FDPs), has been reported up to 10 MPa.m^{1/2}.^{7,15)} Another important factor that should be noted regarding Y-TZP ceramics is their susceptibility to

aging or low-temperature degradation (LTD). The LTD process occurs by itself and is time-dependent. It also affects the (t) → (m) phase transformation mechanism¹⁶⁾ and may also reduce the physical and mechanical properties, such as density and fracture toughness.^{17,18)}

The fracture resistance of brittle materials such as bioceramics is evaluated by their K_{IC} value. In fact, K_{IC} is the critical value of stress intensity factor in the opening mode where there is the occurrence of fracture initiation and unstable crack propagation.^{14,19,20)}

There are different techniques for measuring the K_{IC} values of materials, which can be divided into two groups. One group includes conventional fracture mechanics and allows the determination of K_{IC} values by using notches and induced precracks. This group of techniques, which has been employed for many years to determine the K_{IC} values of different parameters, includes methods such as Single Edge Notch Beam (SENB), Single Edge Precracked Beam (SEPB), and Chevron Notched Beam (CNB). Some of these methods have also been used for obtaining the K_{IC} values of biomaterials.^{21,22)} The second group, which is known as indentation fracture (IF) methods, is based on the use of a sharp indenter such as a Vickers/Knoop indenter. In general, depending on the type of material and the required degree of accuracy, each one of the abovementioned methods is used differently.^{21,23)} However, according to the existing reports, IF techniques are more commonly employed in research works for determining the fracture toughness of ceramic materials, biomaterials, hard biological tissues, etc..^{24–29)}

The IF method for biomaterials and hard biological tis-

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sues can be classified into various groups, such as Vickers indentation fracture (VIF) test, cube corner indentation fracture (CCIF) test, Vickers crack opening displacement (VCOD) test, and interface indentation fracture (IIF) test.²⁹⁾ Some of the advantages of the IF method include the requirement of less equipment and raw materials, and the easier and faster preparation of test specimens.^{24,28,30)} In the VIF technique, the K_{IC} value of the specimens is often determined by directly measuring the length of cracks created by the Vickers diamond tip around the indented zone of a sample, and using the relevant governing equations.²⁹⁾ A variety of equations exists in this method; in some papers the high accuracy of this technique for some ceramics³¹⁾ has been reported and in some other papers the existence of large errors, even up to 48%²⁶⁾ has been reported.

In the present research, nanoindentation and indentation tests have been used to determine the hardness and Young's modulus values of various specimens of pure and silica-doped TZ-3YB, TZ-PX-242A, and TZ-3YSB-E bio-ceramics containing 0.1 and 0.2 wt.% of α -alumina and sintered at 1500°C. The K_{IC} values of samples have been evaluated and analyzed by using 15 equations from the VIF method. Moreover, the values obtained by the VIF method have also been compared with those obtained by the single-edge laser-notch beam (SELNB) technique. Although better results can be obtained by adding other additives or changing other parameters (e.g. using the fabrication method or sintering temperature for mechanical properties of 3Y-TZP), the specimens in this research have been investigated because of their extensive applications as dental materials and because of other favorable characteristics, such as improved phase composition (because the tetragonal phases are suitably obtained in the 3Y composition) and aging properties and the positive effect of alumina and silica addition on LTD.³²⁾

2. Experimental Procedure

2.1. Samples preparation

In order to compute and analyze the values of K_{IC} , three types of commercially-available 3Y-TZP powders (made by Tosoh Co., Japan), including TZ-3YB, TZ-PX-242A and TZ-3YSB-E, were utilized to prepare the test specimens. The chemical compositions of these powders and their abbreviated designations used in the present research are listed in Table 1. All these powders contain 3–4 wt.% of acrylic binder; although, to a larger extent, their chemical compositions are slightly different. The average size of powder grains is about 29 nm.³²⁾ The specific surface areas for stan-

dard-grade (pure) TZ-3YB, slightly higher Y_2O_3 -containing biomedical grade TZ-PX-242A along with small quantities of α -Alumina (~ 0.1 wt.%), and also for slightly lower Y_2O_3 -containing biomedical grade TZ-3YSB-E along with large quantities of α -Alumina (~ 0.2 wt.%) are about 16, 10, and 7 m²/g, respectively. Because of its higher translucency, TZ-PX-242A grade is used nowadays for the preparation of porcelain-free, full-contour fixed partial dentures (FPDs), while TZ-3YSB-E grade needs veneering because of its considerable opaqueness. The powders were dry-pressed for 1 min at a pressure of 150 MPa and then pressed using a CIP machine (ABRA Fluid CO.) at a pressure of 250 MPa. The pressed samples in stainless steel mold of 3.34" size (in the form of 25 × 5 × 2 mm³ plates) were pre-sintered in air for 2 h. The pre-sintering temperature was 1000°C for 0.1A-TZ and TZ specimens and about 1100°C for the 0.2A-TZ specimen.

The pre-sintered samples were divided into two groups. One group was left untreated and used as the control in the analysis of the K_{IC} variations. The samples of the second group were infiltrated with a silica sol, synthesized in situ using the sol-gel method through the hydrolysis of a silan-based solution (Dynasylan® 6490 made by Evonik, Germany). In this procedure, the specimens were immersed in a combination of ethanol (C₂H₅OH) and silan-based solution. Hydrolysis was performed at room temperature by adding aqueous ammonia (20 wt.%) to the samples drop-by-drop. The samples were infiltrated for 30 min. They were then washed with ethanol, dried at room temperature for 24 h, and pyrolyzed at 700°C for 2 h, at a heating rate of 100°C/h. By weighing the specimens before and after the infiltration-drying-pyrolysis cycle, the amount of silica introduced during the infiltration process was determined. Finally, all the specimens of both groups were sintered for 5 h at 1500°C, and the heating/cooling rate was about 5°C/min.

2.2. Mechanical properties measurement

The densities of the samples were obtained by the Archimedes method and based on the European Standard EN 1389:2003.³³⁾ These densities were obtained by considering the theoretical density of $\rho_T = 6.08$ g/cm³,³²⁾ and by averaging five measurements, the relevant SD was also determined.

For analyzing the diagrams obtained from the nanoindentation test, the graphs of force (P) versus penetration depth (h), during sample loading/unloading, were plotted using Equations (1), (2), and (3).³⁴⁾

$$Hv = \frac{P_{\max}}{A_c} \quad (1)$$

Table 1. Chemical Composition of the 3Y-TZP Powders (wt.%)

Powder grade	Abbreviations	Y ₂ O ₃	α -Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	Na ₂ O	Specific surface area (m ² /g)
TZ-3YB	TZ	5.27	< 0.01	< 0.005	< 0.005	< 0.02	~ 16
TZ-PX-242A	0.1A-TZ	5.34	0.1	< 0.005	< 0.005	< 0.01	~ 10
TZ-3YSB-E	0.2A-TZ	5.18	0.2	< 0.005	< 0.005	< 0.01	~ 7

$$E = \frac{\sqrt{\pi}}{2\sqrt{A}} \times \frac{dp}{dh} \quad (2)$$

$$\frac{1}{Er} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i} \quad (3)$$

In these equations, $P_{\max}$, A_c and Hv indicate the maximum loading, contact area, and Vickers hardness, respectively. Also, dp/dh represents the material stiffness, and Er denotes the calculated modulus. A is the area of all formed micro-cracks around the indented zone, ν is Poisson's ratio, and index i specifies the indenter.

Young's modulus and Poisson's ratio of the diamond indenter are 1141 and 0.07 GPa, respectively; the same values are used in the nanohardness measurement device.³⁴⁾ The maximum load applied during the nanohardness test was 800 mN. The nanoindentation tests were performed by using a nanoindentation XP instrument (Nanoindentation Innovation Center, MTS systems, TN, USA) and a Berkovich diamond indenter with a tip radius of 50 nm. The instrument was operated by applying the continuous stiffness measurement methodology.³⁵⁾ The maximum depth of penetration into the material was 1300 nm. Before performing the nanoindentation tests, the system was calibrated by using the fused silica standard; all the tests were performed at room temperature. Based on the graphs obtained from the nanoindentation tests, the hardness and Young's modulus values of the samples were determined as a function of the penetration depth. In addition, the Vickers indentation test was used to measure and compare the hardness values. For this purpose, the samples were polished to 1 μm , and the Vickers hardness tests were carried out by applying a Vickers diamond loading of 100 N for a duration of 15 s. The average hardness values were obtained through 7 measurements. The Vickers microhardness test was carried out under laboratory conditions and by using a Tukon 2100B hardness tester.

The VIF test method were used to measure the K_{IC} values of samples polished to 1 μm . Loadings of 29.42, 49.03, 147.09, 196.13, and 245.15 N were applied in order to find the c/a ratios. After establishing the ranges of $c/a < 2.5$ and $c/a \geq 2.5$, for using the equations of Palmqvist and median crack models, two loads of 49.03 N and 196.13 N were respectively applied for 10 s. The K_{IC} values were computed by using the equations of the curve fitting technique and the loading of 196.13 N. By using an optical microscope (Olympus Imaging Corp., Tokyo, Japan), the diagonal and the length of cracks were measured immediately after unloading. To determine the K_{IC} value of each specimen, the test was repeated 10 times and the average value was obtained from the measurements.

In order to compare the K_{IC} values of the samples obtained by the VIF method, these values are also obtained through other approaches, such as the single-edge laser-notch beam (SELNB) method.³⁶⁻³⁹⁾ In the SELNB method, an ultra-sharp V-notch is made on a specimen by laser pulses, and

Fig. 1. View of a laser-produced V-notch in 0.2A-TZ/SiO₂ specimen sintered for 5 h at 1500°C.

the specimen is then subjected to a three-point bending (TPB) test. The radius of the notch tip can affect the resulting K_{IC} values.^{40,41)} In order to minimize the effect of the notch radius on the K_{IC} , its sharpness must be less than 2 μm .²¹⁾ Because of the difficulties involved in machining a notch of the desired length, and to avoid unexpected cracks in brittle materials, infrared ultra-short laser pulses are used to create ultra-sharp V-notches on test samples. The notch depth in all the specimens was between 90 and 130 μm . The most important drawback of the SELNB technique is that, due to the saturation effect of the plasma, longer notches cannot be created by this process. Fig. 1 shows a laser notching process for producing a notch of proper geometry and for avoiding unwanted major damage around the created notch. After notching, the laser-notched samples were tested at room temperature by a TPB machine with an opening of 10 mm and constant displacement velocity of 0.15 mm/min. Fig. 2 shows a schematic of a laser-notched specimen during testing. The test was repeated 6 times for each group of samples, and the average K_{IC} values of these samples were obtained. Equations (4) and (5) were used to calculate the K_{IC} values.⁴²⁾

$$K_{IC} = \frac{3L_s P_{\max}}{2BW^{\frac{3}{2}}} \cdot \alpha^{\frac{1}{2}} \cdot Y^* \quad (4)$$

Fig. 2. Schematic of a laser-notched sample in the SELNB test.

$$Y^* = \frac{(1.989 - 0.356\beta) - (1.217 + 0.315\beta)\alpha + (3.212 + 0.705\beta)\alpha^2 - (3.222 + 0.02\beta)\alpha^3 + (1.226 - 0.015\beta)\alpha^4}{(1 - 2\alpha)(1 - \alpha)^{\frac{3}{2}}} \quad (5)$$

P_{max} is the applied load at fracture, B is the width of the specimens bar, W is the thickness of the specimen bar, α is the notched depth ($\frac{a}{W}$), Y^* is the stress intensity shape factor, and $\beta = \frac{W}{L_s}$.

3. Results and Discussion

3.1. Relative density and microstructure

Table 2 shows the relative density percentages of various samples (TZ, 0.1A-TZ and 0.2A-TZ) under different conditions. As is observed, the relative density of the 0.1A-TZ specimen in all three pre-sintered, sintered (at 1500°C for 5 h), and silica-doped states is higher than that of the other two specimens. The gravimetrically determined amounts of silica in the infiltrated series of specimens are 0.15, 0.10, and 0.15 wt.% for the TZ, 0.1A-TZ, and 0.2A-TZ samples, respectively. The smallest amount of infiltrated silica is found in the 0.1A-TZ sample, which can be attributed to its higher density relative to the density of other specimens. Fig. 3 shows the SEM images taken from the thermally etched and polished surfaces of the pure and silica-doped 0.1A-TZ samples. As is observed, the size of the grains in both ceramic grades, under similar sintering conditions, is almost the same, and there is no distinct secondary phase. In Fig. 3(b), which shows the grain pattern of the silica-doped 0.1A-TZ specimen, the shapes of grains have changed slightly and their jagged edges have become rounder; this can be attributed to the silica enrichment of the sample. Silica also plays a major role in reducing the amount of strain

Fig. 3. SEM images taken from thermally-etched and polished surface of (a) pure 0.1A-TZ sample and (b) silica-doped 0.1A-TZ sample.

within the grains.⁴³⁾ Other research works have reported the formation of amorphous grains in alumina/3Y-TZP and silica-enriched materials through TEM analysis and energy-dispersive X-ray spectroscopy (EDS).^{32, 44)}

3.2. Nanoindentation and indentation test

Figure 4 shows the load–displacement (p – h) curves obtained from the nanoindentation test performed on the specimens. A lack of discontinuity in these graphs indicates that fracture has not occurred during the loading/unloading of test samples and signifies the fact that the mechanical integrity of the specimens has been maintained throughout the nanoindentation test. Thus, the obtained empirical results can be reliably used for the evaluation of the samples' mechanical properties. Also, the elastoplastic behavior of specimens, which have slightly entered the plastic zone due to the application of mN size loads, is significant. This phenomenon may indicate that samples are neither fractured due to the applied milli-Newton loads, nor are they deformed in a ductile manner. Figs. 5 and 6 respectively show the hardness (H) and Young's modulus (E) values of pure and silica-doped TZ, 0.1A-TZ, and 0.2A-TZ specimens versus the penetration depth. At a penetration depth of more than 50 nm, the E and H values of all the specimens are almost independent of the penetration depth. As is observed, the E and H values of the 0.1A-TZ samples are greater than those of the other specimens, and the E and H values of all silica-doped samples are higher than those of

Table 2. Relative Densities Values in Percentages of Pre-sintered, Sintered (1500°C, 5 h) Pure and Silica-doped Specimens (mean $\pm$ SD)

	TZ	0.1A-TZ	0.2A-TZ
Pre-sintered	52.3 $\pm$ 0.3	55.6 $\pm$ 0.4	53.1 $\pm$ 0.5
As sintered	99.3 $\pm$ 0.2	99.6 $\pm$ 0.1	99.0 $\pm$ 0.2
Silica-doped/sintered	99.4 $\pm$ 0.2	99.8 $\pm$ 0.1	98.9 $\pm$ 0.1

Fig. 4. Load–displacement ($P-h$) curves obtained from nanoindentation tests for (a) pure TZ, 0.1A-TZ, and 0.2A-TZ samples and (b) silica-doped TZ, 0.1A-TZ, and 0.2A-TZ samples.

Fig. 5. Hardness–displacement ($H-h$) curves obtained from nanoindentation tests for (a) pure TZ, 0.1A-TZ, and 0.2A-TZ samples and (b) silica-doped TZ, 0.1A-TZ, and 0.2A-TZ samples.

Fig. 6. Young's modulus–displacement ($E-h$) curves obtained from nanoindentation tests for (a) pure TZ, 0.1A-TZ, and 0.2A-TZ samples and (b) silica-doped TZ, 0.1A-TZ, and 0.2A-TZ samples.

pure samples. The increase or reduction in the hardness can be attributed to the increase or reduction in the density. Fan *et al.*⁴⁵⁾ obtained the hardness and Young's modulus values of a sample made by slip casting technique and consisting of 60 vol.% alumina and 40 vol.% Y-TZP as 15.6 and 340 GPa, respectively. Samodurova *et al.*³²⁾ determined the hardness values of pure and silica-doped 3Y-TZP and 0.05/0.25 wt.% Al₂O₃-3Y-TZP specimens in the range of 13.5–

15.7 GPa. By examining and comparing the Young's modulus values of samples, it can be realized that Young's modulus is a function of the relative density. Since Young's modulus has a direct relationship to density according to the ASTM C769 standard⁴⁶⁾ and Eq. (6), by increasing the density of samples, which reduces their porosity and the speed of sound in the samples, Young's modulus increases as well, and vice versa.

Fig. 7. Image of the trace left from Vickers microhardness in pure 0.1A-TZ samples in loading of 100 N at 0.01 s after unloading.

$$E = \rho v^2 \quad (6)$$

In the above equation, E is a sample's Young's modulus, ρ is its density, and v is the velocity of sound passing through the sample. The average E and H values obtained from 20 nanoindentation tests for each specimen are listed in Table 3.

In order to compare nanoindentation hardness test results, the Vickers microhardness values of samples are listed in Table 3. As is observed, the Vickers microhardness of each sample is less than the amount of hardness obtained from the nanoindentation test. This discrepancy is justified considering the different ways of measuring hardness in the nanoindentation and Vickers indentation tests, because contrary to the nanoindentation hardness test values, the Vickers microhardness values are measured with the help of residual imprint upon unloading, which results in errors

Table 3. Obtained Hardness and Young's Modulus of TZ, 0.1A-TZ, and 0.2A-TZ: Average Elastic Modulus (E) and Hardness (H) Obtained by Nanoindentation and Vickers Hardness (H_v)

Materials		Nanoindentation		Vickers indentation
		H (GPa)	E (GPa)	H_v (GPa)
Pure	TZ	14.3±0.6	215±7	11.7±0.1
	0.1A-TZ	15.2±0.4	272±6	12.9±0.2
	0.2A-TZ	13.4±0.4	193±4	11.5±0.3
Silica-doped	TZ	15.4±0.7	250±5	12.5±0.3
	0.1A-TZ	17.3±0.6	282±5	14.1±0.3
	0.2A-TZ	14.5±0.5	235±8	12 ±0.2

that are mostly caused by elastic relaxation occurring during or after unloading. This phenomenon is important in ceramic materials, and it can explain the existing difference between the results of the two tests. However, the Vickers microhardness of the samples were determined immediately after their unloading so that the difference between the nanoindentation hardness test and the Vickers microhardness test is somewhat reduced. Fig. 7 shows an image of the trace left from the Vickers microhardness in pure 0.1A-TZ samples in loading of 100 N at 0.01 s after unloading.

3.3. Fracture toughness assessment

3.3.1. Overview of models for the determination of fracture toughness by VIF method

In general, two models of Palmqvist and median/half penny cracks are considered in the VIF approach.⁴⁷⁾ In the Palmqvist crack model, the length of the formed cracks is assumed to extend only from the tip end of the indented

Fig. 8. Crack traces left in the sample by the Vickers indenter: (a) Palmqvist crack model and (b) median/half penny crack model.

zone,⁴⁸⁾ while in the median/half penny crack model, the crack length is assumed to extend radially from the center of the indented zone.⁴⁹⁾ Also, the Palmqvist model and the median crack model mostly occur at low and high loads, respectively. A simple way to make a distinction between these two models is that in the Palmqvist crack model, after polishing the surface of a tested specimen, the formed crack is usually seen separate from the diamond-shaped surface trace left by the Vickers indenter; however, in the median crack model, the crack is completely attached to the surface trace left by the indenter.^{50,51)} Another way of recognizing these two models is that if $c/a < 2.5$, the model will be assumed as the Palmqvist crack model, and if $c/a \geq 2.5$, the model will be considered as the median crack model.^{52,53)} Fig. 8 shows these two models and their differences.

Lawn *et al.*⁵⁴⁾ analyzed the elastoplastic behavior of the indented zone in various specimens. They assumed the Palmqvist and median crack models due to the presence of tensile stresses during loading, and obtained Eq. (7).

$$K_{IC} = \alpha \sqrt{\frac{E}{Hv}} \frac{P}{C^{3/2}} \quad (7)$$

In the above equation, K_{IC} is the fracture toughness of the material, P is the amount of loading by Vickers diamond, C is the length of the surface trace of the median crack measured from the center of the indent, Hv is the Vickers hardness of the sample, E is the Young's modulus, and α is a numerical constant, which is obtained empirically.

After Evans and Charles, other researchers presented numerous equations for calculating the K_{IC} value. More

Table 4. Equations Used in Calculation of Fracture Toughness of Samples

No.	Equation	Crack type	Author	Ref.
1	$K_{IC} = 0.018Hva^{1/2} \left(\frac{E}{Hv} \right)^{0.4} \left(\frac{c}{a-l} \right)^{-0.05}$	Palmqvist	Niihara <i>et al.</i>	[56]
2	$K_{IC} = 0.0515 \left(\frac{P}{c^{3/2}} \right)$	Palmqvist	Lawn and Fuller	[57]
3	$K_{IC} = 0.079 \left(\frac{P}{a^{3/2}} \right) \text{Log} \left(4.5 \frac{a}{c} \right)$	Palmqvist	Evans and Wilshaw	[58]
4	$K_{IC} = 0.015 \left(\frac{E}{Hv} \right)^{2/3} \left(\frac{P}{c^{3/2}} \right) \left(\frac{l}{a} \right)^{-0.5}$	Palmqvist	Laugier	[59]
5	$K_{IC} = 0.016 \left(\frac{E}{Hv} \right)^{1/2} \frac{P}{C^{3/2}}$	Median	Anstis <i>et al.</i>	[49]
6	$K_{IC} = 0.0752 \frac{P}{c^{3/2}}$	Median	Evans and Charles	[60]
7	$K_{IC} = 0.0726 \frac{P}{c^{3/2}}$	Median	Lawn and Fuller	[57]
8	$K_{IC} = 0.014 \left(\frac{E}{Hv} \right)^{1/2} \frac{P}{C^{3/2}}$	Median	Lawn and Evans	[54]
9	$K_{IC} = 0.16Hva^{1/2} \left(\frac{c}{a} \right)^{3/2}$	Median	Evans and Charles	[60]
10	$K_{IC} = 0.0889 \left(\frac{Hv.P}{\sum_{i=1}^4 c_i} \right)^{1/2}$	Curve fitting technique	Shetty <i>et al.</i>	[61]
11	$K_{IC} = 0.4636 \left(\frac{E}{Hv} \right)^{2/5} \frac{P}{a^{3/2}} 10^F$	Curve fitting technique	Evans	[62]
12	$K_{IC} = 0.018 \left(\frac{E}{Hv} \right)^{1/2} \frac{P}{C^{3/2}}$	Curve fitting technique	Japanese Standards Association	[63]
13	$K_{IC} = (Hva^{1/2}) \left(\frac{E}{Hv} \right)^{2/5} 10^y$	Curve fitting technique	Evans	[62]
14	$K_{IC} = 0.0782(Hv a^{1/2}) \left(\frac{E}{Hv} \right)^{2/5} \left(\frac{C}{a} \right)^{-1.56}$	Curve fitting technique	Lankford	[64]
15	$K_{IC} = 0.00366 \left(\frac{E}{Hv} \right)^{1/2} \frac{P}{\ell_{Ave}^{3/2} A^{3/2}}$	Curve fitting technique	Moradkhani <i>et al.</i>	[31]

than 30 of these equations have been presented,⁵⁵⁾ and some of them have been listed in Table 4. Several equations in this table use the curve fitting technique for the acquired data. This group of equations can be used for both the Palmqvist and median crack models. However, the formulation of this group of equations is also based on the two mentioned models, which still make the calculation of K_{IC} dependent on crack morphology.²⁹⁾

In Table 4, K_{IC} denotes fracture toughness ($\text{MPa}\cdot\text{m}^{1/2}$), P is the amount of loading by Vickers diamond (N), c is the average crack length (m), a is the diagonal half-length of Vickers impression (m), $l = c - a$ (m), Hv is the Vickers hardness (GPa), and E is the Young's modulus of the specimen (GPa), t_{Ave} is the average thickness of microcracks formed around the indented zone (mm), and A is the total area of crack traces formed around the indent (mm^2). F and y in Equations (11) and (13) from Table 4 are determined as:⁶²⁾

$$F = -1.59 - 0.34x - 2.02x^2 + 11.24x^3 - 24.97x^4 + 16.32x^5 \quad (8)$$

$$y = -1.59 - 0.34x - 2.02x^2 + 11.24x^3 - 24.97x^4 + 15.32x^5 \quad (9)$$

The value of x in Eqs. (8) and (9) is obtained from:

$$x = \text{Log}\left(\frac{c}{a}\right) \quad (10)$$

3.3.2. Fracture toughness of samples

Figure 9 shows the indented surface views of pure and silica-doped TZ, 0.1A-TZ, and 0.2 A-TZ specimens, with crack traces at the corners of the indented zone left by the Vickers

indenter. Fig. 10 illustrates the c/a ratios with respect to the loadings applied on the test samples. As is observed, for loads of 29.42 and 49.03 N, this ratio indicates a Palmqvist crack system, and for loads of 147.09, 196.13, and 245.15 N, it signifies a median crack system. Hence, by using a loading of 49.03 N in the Palmqvist crack system and a loading of 196.13 N in the median crack system, more reliable K_{IC} results can be obtained. A loading of 196.13 N has also been considered in calculating the K_{IC} values by the curve fitting technique. Although, the Palmqvist crack pattern is normally created in a hard material, the K_{IC} values of many brittle materials can be computed by employing both models, and the choice of which of these two models to use

Fig. 10. c/a ratios versus applied loads in the Vickers indentation test for various pure and silica-doped TZ, 0.1A-TZ, and 0.2A-TZ specimens.

Fig. 9. Indented surface views of 3Y-TZP composites: (a) Pure TZ at 49.03 N, (b) Pure 0.1A-TZ at 49.03 N, (c) Pure 0.2A-TZ at 49.03 N, (d) Silica-doped TZ at 196.13 N, (e) Silica-doped 0.1A-TZ at 196.13 N, and (f) Silica-doped 0.2A-TZ at 196.13 N.

Fig. 11. Fracture toughness values obtained from 15 different equations in Table 4 (pertaining to VIF method) and from SELNB test for (a) pure TZ, 0.1A-TZ, and 0.2A-TZ samples and (b) silica-doped TZ, 0.1A-TZ, and 0.2A-TZ samples.

depends on the amount of loading applied by Vickers indenter.^{50,51} In many brittle materials, Palmqvist cracks only occur at low magnitudes of loading.⁶⁵ Also, at high loading magnitudes, Palmqvist cracks only form in fairly tough materials such as WC-Co composites containing more than 6 wt.% CO.⁵³

Figure 11 shows the fracture toughness values obtained by the VIF method (using the 15 equations listed in Table 4) and also by the SELNB approach. It can be seen in Fig. 11 that the variations in K_{IC} obtained from the VIF equations for each specimen are close to each other in many cases, and the difference between them is less than 5%; this difference, in comparison with the results of the SELNB method, is less than 10%. Other research works have maintained that, for brittle materials, the K_{IC} results obtained by the SENB approach are more reliable than those obtained by the VIF method,⁶⁶ and that the values reported by the SELNB method are more accurate than the values obtained by the SENB approach.^{67,68} Therefore, in the present work, the results of the SELNB method are considered as the K_{IC} values of samples. Eq. (15) in Table 4 is the most recent of these equations, and by comparing the results obtained from this and other equations and also by the SELNB approach, it is found that Eq. (15) yields fairly accurate results. The accuracy of this equation in determining the K_{IC} values of some other brittle materials have also verified the findings of this research.⁶⁹⁻⁷⁴ By analyzing the obtained results, it is realized that by adding 0.1 wt.% alumina to pure TZ, the K_{IC} values increase by about 10%, and that the addition of 0.2 wt.% alumina diminishes the K_{IC} values by about 6%. This trend is also seen in silica-doped samples, with an increase and reduction of K_{IC} values by 12 and 4%, respectively, as the mentioned weight fractions of alumina are added. The maximum and minimum K_{IC} values (6.36 and 5.2 MPa.m^{1/2}) are observed in silica co-doped 0.1A-TZ and pure 0.2A-TZ samples, respectively. These results somewhat match the results of other research works on pure and silica-doped 3Y-TZP and 0.05/0.25 wt.% alumina-

3Y-TZP samples.³² According to Eq. (11), presented by Griffith, the K_{IC} and Young's modulus values have a direct relationship.

$$K_{IC} = \sqrt{2E\gamma} \quad (11)$$

In the above equation, E is the Young's modulus and γ denotes the surface energy. Also, by examining the Young's modulus and K_{IC} values of specimens, it is observed that the trends of these parameters match and overlap each other. Therefore, the increase in K_{IC} can be attributed to the increase in density and, consequently, the increase in Young's modulus.

4. Conclusions

The relative density, hardness, and Young's modulus values of pure and silica-doped TZ, 0.1A-TZ, and 0.2A-TZ biomaterials have been investigated in this research through nanoindentation and indentation tests. The fracture toughness values of these materials have been computed and analyzed by means of 15 equations related to the VIF method and compared with those obtained by the SELNB approach. The findings of this research can be summarized as follows:

- The relative density, hardness, Young's modulus, and fracture toughness of 3Y-TZP biomaterials are improved with the addition of 0.1 wt.% of alumina. These properties diminish by adding an extra amount of alumina (up to 0.2 wt.%). Also, the mentioned properties are more improved in silica-doped samples than in pure undoped samples. The SEM images of some specimens indicate that there is no distinct secondary phase of alumina present and that the grains of silica-doped samples are often rounder, which can be attributed to the silica enrichment of the samples.
- The maximum relative density, hardness, Young's modulus, and fracture toughness values of the samples are 99.8%, 17.3 GPa, 282 GPa, and 6.36 MPa.m^{1/2}, respectively, which have been obtained for the silica-doped

0.1A-TZ. Also, by adding 0.1 wt.% of alumina, the hardness and the Young's modulus of pure 3Y-TZP specimen increased by 6% and 26%, respectively. Moreover, those of the silica-doped 3Y-TZP specimen increased by 12% and 13%, respectively, at the sintering temperature of 1500°C.

- In computing the fracture toughness values of all the pure and silica-doped Alumina/3Y-TZP samples by the VIF method, $c/a < 2.5$ for loadings of 29.42 and 49.03 N. Therefore, it is more appropriate to use the Palmqvist crack model equations to calculate the fracture toughness. For loadings of 147.09, 196.13, and 245.15 N, $c/a \geq 2.5$, and therefore, it is more appropriate to use the median crack model equations to obtain the fracture toughness values.
- By comparing the fracture toughness results obtained using the SELNB and VIF methods, it is found that the values obtained by VIF method, by taking proper loadings into consideration, have a discrepancy of less than 10% relative to the results using SELNB approach. Also, the difference between the results of various VIF equations for each sample is less than 5%.
- By adding 0.1 wt.% alumina to pure 3Y-TZP material, its fracture toughness is increased by about 10%. Also, the addition of 0.2 wt.% alumina reduced the fracture toughness by about 6%. This trend is also observed more prominently in silica-doped samples, which showed the increase and reduction of fracture toughness by 12 and 4%, respectively, as the mentioned weight fractions of alumina were added.

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