

Mechanical Properties of Alumina-Zirconia Composites for Ceramic Abutments

Maria Cecilia Corrêa de Sá e Benevides de Moraes^{a*}, Carlos Nelson Elias^a,
Jamil Duailibi Filho^b, Leandra Guimarães de Oliveira^b

^aDepartment of Mechanical and Metallurgical Engineering (DE-4), IME,
Praça General Tibúrcio, 80, Praia Vermelha, 22290-270, Rio de Janeiro-RJ, Brazil

^bINT, Av. Venezuela 82, sala 604, Saúde, 20081-310, Rio de Janeiro-RJ, Brazil

Received: October 29, 2003; Revised: May 20, 2004

The need for new materials to substitute injured or damaged parts of the human body has led scientists of different areas to investigate bioceramics since the 70's, when other materials in use started to show rejection problems. Ceramics show some advantages like being the material that best mimics the bone tissue, although present low toughness when compared with the metallic materials. As patients have become more and more demanding regarding esthetic and biocompatibility aspects of their dental restorations, ceramic material has become a main object of scientific interest especially from the material point of view. The alumina-zirconia composites are one of the relatively good and promising candidate for biomaterials application, due to biocompatibility and their mechanical properties that combines high flexural strength with a high toughness. The aim of the present work is to analyze the mechanical properties of these composites, where Y-TZP zirconia content was varied from 5 to 80 wt.%. These systems can achieve a flexural strength 93 % and fracture toughness 29 % superior when compared to the pure alumina ceramics. These results showed that ceramic abutments components can be prepared for prosthetic rehabilitations with dental implants.

Keywords: ceramic abutments, alumina-zirconia composites, bioceramics and mechanical properties

1. Introduction

A great progress in dental restorations technique has been established by the use of ceramic materials since the 70's. In some field application, the tendency is the elimination of the metallic substructure, including implant restorations. These materials show relative advantages, like esthetic, biocompatibility and chemical resistance. One problematic aspects of ceramic materials and of dental ceramics in particular is their low mechanical resistance and fracture toughness (K_{IC})¹.

Osseointegrated dental implants are been used since 80's in the rehabilitation of partially and totally edentulous patients². The metallic abutments used in prosthetic restorations with implants may compromise the esthetic in some cases³. To minimize this problem, some implant systems developed ceramic abutments. The use of alumina and zirconia with high density like biomaterial have been proposed. Alumina has shown excellent biocompatibility and wear resistance

however exhibits low flexural strength and toughness⁴. The pure zirconia cannot be used in the manufacture of parts without the addition of stabilizers⁵. The yttria-tetragonal zirconia polycrystal (Y-TZP) has become a popular alternative to alumina as biomaterial and is used in dental applications such as endodontic posts⁶, orthodontic brackets⁷, crowns and bridges^{8,9} and in ceramic abutments¹⁰, beside the extensive experience of using the ceramic in orthopedics implants^{11,13}. Zirconia presents good esthetic aspects after polishing, is inert in physiological environment, presents greater flexural resistance and toughness and lower Young's modulus when compared with pure alumina¹⁴. The ability of Y-TZP to transform from tetragonal crystalline structure to a more voluminous monoclinic structure, and thus obstruct crack propagation, gives the material its strength and toughness⁹.

The addition of zirconia in the alumina as sintering additive have been used for long time with the objective of

*e-mail: edujm@uol.com.br

alumina based ceramics densification. However, the concept of toughening alumina ceramics with dispersion of zirconia particles in a matrix was only recognized in the last 20 years. The introduction of small amounts of zirconia in alumina as additive of sintering causes the formation of solid solution which promotes the densification process by the introduction of defects¹⁵.

On the other hand, the microstructure of a composite material is formed from the addition of one second phase. A composite material is the way to improve reliability and lifetime of ceramic abutments by providing higher fracture toughness and mechanical strength. Material scientists are already familiar with composites like alumina-zirconia¹⁵⁻²¹. Since both materials are biocompatible, this could prove to be a new approach to dental abutments. Despite the community of biomaterials being already familiar with alumina and zirconia separately, by being both biocompatible, little was published in literature on the use of alumina-zirconia composites as biomaterial¹⁴.

Two types of composites can be prepared from this system: a matrix of zirconia stabilized and strengthened with particles of alumina (Y-ATZ) or an alumina matrix strengthened with particles of zirconia (Y-ZTA)^{4,21-23}. With both materials higher toughness values when compared with the monophase ceramics can be reached²⁴.

In the current study, the hardness and fracture toughness of alumina-zirconia composites with respect to zirconia content are explored in details by utilization of Vickers indentation technique. The mechanical strength is also analysed by four point flexural tests.

2. Experimental procedure

The commercially available powders used in this work were the oxides alumina, and yttria-stabilized zirconia (3 mol%) from two different manufacturers. Alumina SG-1000 (Alcoa-USA) presented specific surface area of 10,47 m²/g(BET) and average particle size determined by laser scattering ($d_{50} = 0,44 \mu\text{m}$). The yttria-stabilized zirconia (TZ3YSB - Tosoh Corporation - Japan) containing binder deposited by spray-drier, presented specific surface area of 6,21 m²/g(BET) and average particle size by laser scattering ($d_{50} = 0,35 \mu\text{m}$). The yttria-stabilized zirconia (3Y-TZP - Mel Chemicals - England), presented specific surface area of 11,10 m²/g(BET) and average particle size by laser scattering ($d_{50} = 0,19 \mu\text{m}$).

Different compositions were prepared with mixture of oxides varying the zirconia amount from 5 to 80 wt.%. To attain the compositions, mechanical milling was performed in ethanol with 50 wt.% of solids content for 1 h and a half, in ball milling, with zirconia grinding medias ($\varnothing = 2 \text{ mm}$). Citric acid (0.6% wt.%) was used as dispersant and polyethylene glycol (2 wt.%) as the binder. The slurry of

the powder mixtures was dried in rotatory evaporator, and the dried lumps were crushed and passed through a sieve. (65 Tyler mesh(0,212 mm)). Powder compacts with dimensions of 5 × 6 × 80 mm were conformed by uniaxially pressing at 55 MPa. The green bodies density was calculated from the ratio weight/volume. The withdrawal of the binder was carried through in a furnace type muffle with heating rate of 6 °C/min at 600 °C per 180 min. The sintering was carried out in air in a box furnace and in the temperature range of 1500 - 1600 °C for two hours, as the composition. The final density was measured by Archimedes method.

After the inlaying of the samples in bakelite, they were ground by silicon carbide (SiC) paper in sequence of #150, #180, #220, #320, #400, #600, for the complete removal of the material of inlaying and attainment of a plain surface. After, the samples had been polished with diamond pastes in the sequence of 15, 9, 6, 3, 1 and 0,25 μm to produce optical reflective surface. During this stage, the samples were observed in optical microscope OLYMPUS LECO PME-3, with the aid of the image software IMAGE IA-3001, for the evaluation of the burnishing process. The burnishing time was not fixed, it was a function of the evolution of the process, the paste was only changed when the average size of the defect in the polishing surface, which was quantified through the optical microscope, was in the order of magnitude of the abrasive.

For all tested materials, the average grain size of the alumina and zirconia grains was determined from SEM images of randomly selected areas of the polished and thermally etched specimens using the linear intercept method. The average value was obtained based on the analysis of at least 150 grains for each material.

The methodology used for the determination of hardness, was in accordance with ASTM C 1327-99²⁵. Thirty Vickers impressions had been carried through in the surfaces of each one of the samples, which already were polishing, using an applied load of 10 kgf (98.1 N) during ten seconds. After the diagonal length measurement, the values of the Vickers hardness (GPa) were calculated, by the following equation:

$$H_v = 0.0018544 (P/d^2) \quad (1)$$

Where:

H_v = Vickers hardness (GPa);

P = applied load (N);

d = arithmetic mean of the two diagonal length(mm).

The determination of fracture toughness at ambient room temperature followed the pattern ASTM C 1421-99²⁶. Fracture toughness is given by the values of K_{IC} , the mode I critical stress intensity factor, under which failure does not occur. The factor K_{IC} was determined using the direct crack measurement method. In this procedure, the crack measurement was carried out soon after indentation, searching to prevent the slow crack growth after the impression, initiated by the

stress field that acts after removal of indenter. Each Vickers impression presented two pairs of radial cracks emerging from the corners. A total of sixty pairs of cracks were obtained. For calculations, at least thirty perfect pairs of cracks were used for each composition, those that did not present interactions with burnishing imperfections and deviations from crack. Three equations were used to determine the K_{IC} values as follows: Eq. 2 - Anstis²⁷, Eq. 3 - Casellas²⁸ and Eq. 4a and 4b - Niihara²⁹.

$$K_{IC} = 0.016 \left(\frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \quad (2)$$

$$K_{IC} = 0.024 \left(\frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}} \quad (3)$$

$$K_{IC} = 0.129 \left(\frac{c}{a} \right)^{-3/2} \left(\frac{E\phi}{H} \right)^{2/5} \left(\frac{Ha^{0.5}}{\phi} \right) \text{ for } \frac{c}{a} \geq 2.5 \quad (4a)$$

$$K_{IC} = 0.035 \left(\frac{l}{a} \right)^{-1/2} \left(\frac{E\phi}{H} \right)^{2/5} \alpha \left(\frac{Ha^{0.5}}{\phi} \right) \text{ for } 0.25 \leq \frac{l}{a} \leq 2.5 \quad (4b)$$

Where:

K_{IC} = fracture toughness (MPa.m^{1/2});

P = load applied(N);

E = Young's modulus(GPa);

H = hardness (GPa);

a = diagonal half-length of Vickers impression(m);

l = crack length(m);

c = l + a (m).

In this work, the Young's modulus were obtained by rule of mixtures starting from 210 GPa for zirconia^{14,30,31} and 380 GPa for alumina^{14,30}. Tuan, W.H. *et al.*³² measured the Young's modulus by ultrasonic wave and for comparison plotted the line predicted by the rule of mixtures. Only slightly lower values of Young's modulus than that predicted by the rule of mixtures were found for the analysed compositions. We can justify the use of this method because we have had samples with high density and a low defects number.

The presence of porosity can reduce fracture toughness by the reduction of the resistant area and by the effect of stress concentration in the pores. The correction was carried out by using the following equation²⁷:

$$K_{IC} = K_{ICd} \cdot e^{-bp} \quad (5)$$

Where:

K_{IC} = fracture toughness considering the porosity(MPa.m^{1/2});

K_{ICd} = fracture toughness for dense material.(MPa.m^{1/2});

p = apparent porosity of the material;

b = constant for material 5.3 ± 0.4. (In the calculations, b was assumed to be 5.3)³³.

The sintered specimens were machined longitudinally with a 400 grit resin-bonded diamond wheel at a depth of

5 µm/pass with ample water coolant. The final dimensions of the specimens were 3 × 4 × 60 mm. The strength of the specimens was determined according with ASTM-1161-90³⁴ by four-point flexural test(EMIC-DL 10000 - universal test machine), at ambient room temperature conditions. The upper and lower spans were 15 and 30 mm, respectively. The cross-head speed was 0.5 mm/min. At least thirty specimens for each composition were tested.

Throughout this paper, the following nomenclature has been used for pure oxides and composites: "A" indicates alumina, "XX ZT" represents zirconia Tosoh, "XX ZM" represents zirconia Mel Chemicals and XX represents the zirconia content (wt.%) in the composite. For instance, 80 ZT means composite with zirconia from Tosoh with 80 wt.% of zirconia.

3. Results and discussion

3.1. Density

The sintered pure oxides and composites showed relative density in the range of 99.13% to 99.86%(Table 1). The total porosity less than 1%vol is required to high pure ceramics of alumina and zirconia for surgical implantations.(ASTM F-603-00³⁵ and ASTM F-1873-98³⁶, respectively). In general, sintered samples with higher relative density exhibit greater mechanical properties. However, there is also an influence of microstructure uniformity on mechanical properties. The highest density were obtained for 80 ZT e 80 ZM (Table1). From pure alumina (A) until 21 wt.% zirconia Tosoh (21 ZT), the density of the composites increases with the increase of zirconia content, indicating that the addition of zirconia particles allowed the densification of alumina matrix. In the same way, starting from pure zirconia to 80 ZT, the addition of alumina enhanced the composite densification. Observations of the sintered materials by scanning electron microscopy (SEM, JSM-5200-LV, Jeol, Japan) showed highly homogeneous microstructures without agglomerates, pores or abnormally grown alumina grains. X-ray diffraction analyses of the samples indicated that only α-alumina, tetragonal and monoclinic zirconia are the crystalline phases present in both the pure and in the composites. A fine and a bimodal distribution of particles size allowed the most efficient fulfilling of the matrix socket for uniaxially pressing, promoting better densification of the powders. The use of 0.6 wt.%^{37,38} of citric acid as dispersant was effective to prevent agglomerates which are sources of imperfections during sintering³⁹.

3.2. Indentation load and Vickers hardness

The hardness Vickers of the composites obeys a linear mixture rule⁴⁰. It can be observed that with the increase of zirconia content, the hardness of the composites diminishes (Table 1). Higher hardness values imply in good wear and scratch resistance. However, the machining can be very hard

to be done. The indentation load is critical in the measure of hardness and fracture toughness using the indentation method. The amount of load could affect the indentation size and the crack length, being dependent of the sample dimension and the microstructure of the material. Many ceramic that show a decrease of hardness with the load increase can have this problem solved⁴¹. The solution is that the measure of the Vickers hardness be carried out in raised loads⁴², above of 50 N, where the influence of the load in the measure of the hardness decreases and so the standard hardness can be obtained.

3.3. Flexural strength measures

The ceramic strength values usually exhibit a large scattering (up to 100%) even for high performance ceramics⁴³. This well known phenomenon, which arises from the scattering of the initial sizes of the defects responsible for failure, is generally studied statistically by means of Weibull's analysis. The first and most important step for improving the strength and reliability of ceramics is to reduce the size of cracks and defects⁴⁴. This can be achieved by obtaining a fine-grained more uniform and more dense structure by use of improved powders (higher purity and a fine and narrow initial particle size distribution) as well as by adopting better processing techniques. The composites 80 ZT and 80 ZM presented flexural strength 90% and 93% higher when compared with pure alumina, respectively, showing that the second phase addition provided a composite with a more dense and refined microstructure, a reduced size of the crack population, and as expected that the strength of ceramics is inversely proportional to the square root of the grain size⁴⁵.

3.4. Measure of fracture toughness

The zirconia addition had a large effect in increasing the fracture strength²⁸, but its influence on fracture toughness was much smaller (Table 1). The reason for this behavior

can be the absence of two of the main toughening mechanisms of ZTA: crack bridging and the transformation toughening. The crack bridging is negligible because of the small grain size of alumina (only small bridges were detected in pure alumina). The transformation toughening is also absent because only 5% of ZrO_2 transforms on fracture surfaces. The reason for this can be the fact of the ZrO_2 average particle size is too small that it can not activate the stress-induced phase transformation. In fact, the authors had found greater values of fracture toughness when the grain size of alumina matrix and zirconia particles had been increased with thermal treatments of grain growth. The size of the tetragonal particle and the stabilizer content, have great influence in the tension required for the transformation of tetragonal particle⁴⁶. The critical grain sizes increase with the Young's modulus of the composite, which is related with the restrictions imposed to the matrix. A time that the Young's modulus decreases with the zirconia content in accordance with the rule of mixtures, the critical grain sizes will be lesser in composites with higher zirconia.

In the composites with alumina matrix or either with a high percentage of alumina, the increase in the amount of tetragonal zirconia generally involves a considerable increase of K_{IC} ^{20,42,47}. Starting from pure alumina up to 21 ZT or 15 ZM, an increase of K_{IC} can be observed. Hardness and fracture toughness values from Table 1 have shown that as the hardness values reduce with the increase of zirconia content in the composite, the K_{IC} values had increased. This result suggests that the zirconia addition change the composite behaviour. Possibly a phase transformation occurs⁴⁸, which is an important mechanism in the alumina-zirconia composites. Other mechanisms such as microcrack formation or crack ramification can even be present⁴⁹. The presence of the tetragonal phase has two beneficial effect: one of the factors is that there is no formation of microcracks in the material due the volumetric expansion (3 to 5%) under martensitic phase

Table 1. Material properties

Material	Relative Density (%)	H_v (GPa)	c/a	c (μ m)	E (GPa)	K_{IC} (MPa.m ^{1/2}) Anstis, G.R. <i>et al.</i> 1981	K_{IC} (MPa.m ^{1/2}) Casellas, D. <i>et al.</i> 1997	K_{IC} (MPa.m ^{1/2}) Niihara, K. 1983	Flexural strength σ_{4p} (MPa)
100 ZT	99.46 ± 0.08	13.20 ± 0.48	2.00 ± 0.07	117.32 ± 3.80	210.00	4.77 ± 0.24	7.16 ± 0.36	6.82 ± 0.19	736.55
80 ZT	99.60 ± 0.46	15.44 ± 0.46	2.06 ± 0.09	112.22 ± 4.92	230.64	5.00 ± 0.32	7.49 ± 0.48	7.15 ± 0.27	755.35
21 ZT	99.55 ± 0.04	17.14 ± 0.38	2.53 ± 0.21	130.25 ± 10.78	324.79	4.57 ± 0.69	6.85 ± 1.04	6.54 ± 0.96	510.79
15 ZT	99.52 ± 0.35	17.38 ± 0.23	2.68 ± 0.21	137.30 ± 10.82	338.85	4.43 ± 0.54	6.65 ± 0.81	6.34 ± 0.74	491.14
10 ZT	99.35 ± 0.20	17.41 ± 0.21	2.69 ± 0.15	137.88 ± 8.09	351.54	4.29 ± 0.38	6.43 ± 0.57	6.14 ± 0.53	473.13
5 ZT	99.29 ± 0.10	17.48 ± 0.76	2.75 ± 0.27	140.11 ± 13.47	365.22	4.25 ± 0.60	6.38 ± 0.89	6.09 ± 0.89	441.40
A	99.13 ± 0.13	17.53 ± 0.52	2.90 ± 0.17	146.31 ± 8.86	380.00	4.06 ± 0.36	6.10 ± 0.54	5.81 ± 0.54	396.71
100 ZM	99.35 ± 0.92	12.78 ± 0.34	1.97 ± 0.11	117.18 ± 6.30	210.00	5.08 ± 0.43	7.61 ± 0.65	7.26 ± 0.35	635.02
80 ZM	99.86 ± 0.06	14.32 ± 0.77	2.01 ± 0.08	110.98 ± 6.81	230.64	5.22 ± 0.40	7.82 ± 0.65	7.46 ± 0.29	763.70
15 ZM	99.41 ± 0.38	16.98 ± 0.38	2.58 ± 0.21	134.10 ± 10.89	338.85	4.63 ± 0.59	6.94 ± 0.88	6.62 ± 0.81	471.38

transformation, when the tetragonal zirconia particles transform into monoclinic zirconia. The formation in excess of microcracks can degrade its mechanics resistance. Another effect is that when the transformation tetragonal to monoclinic occurs, the resultant expansion creates compression zones that make it difficult the crack propagation from the defects of the material. As the second mechanism that acts in these composites, the microcracking that occurs around zirconia particles transformed causes energy dispersion and toughening the material. This mechanism operates in the same direction of the phase transformation and it is more important in the composites with higher zirconia content⁵⁰. It can also be verified leaving from the pure zirconia that the 20 wt.% of alumina addition in the matrix of zirconia (80 ZT or 80 ZM), promoted the highest value of K_{IC} , been 23% and 29% superior to the K_{IC} value found for pure alumina, respectively. Superior K_{IC} values for 80 ZM can be explained by the better densification of the composite and by a finer and narrow particle size distribution, a greater specific surface area and a higher retention of the tetragonal phase⁵¹ when compared with 80 ZT. In addition, other mechanisms that promotes the increase of fracture toughness such as crack bridging and crack deflection could be activated when alumina is added the zirconia stabilized with yttria.

3.5. Use of equations for indentation fracture toughness calculations

It is well known that toughness measurement using microindentation methods are readily influenced by the surface condition of sample as compared to that using the bulk techniques⁵². The employment of various techniques to measure fracture toughness would yield different results possibly due to difference of crack mechanisms induced, although, arguments may be just due to the different toughness equations used (Table 1). The Eq. 3 and Eqs. 4a and 4b supply K_{IC} values that are 50% and 43% superior for the same material when compared to the values obtained from Eq. 2, respectively. The low K_{IC} obtained in samples with residual stress produced by indentation can be attributed to the fact that the equation proposed by Anstis with the parameters of adjustment $A = 0.016$ and $n = 0.5$ underestimates the residual stress field. Some authors have proposed correction for the parameter A (more than 20%) to be used in ceramics containing zirconia^{20,28}. Despite the Anstis equation be widely applied for estimates the fracture toughness, its results are only average numbers calculated to adjust the results for a high range of materials. In the case of alumina, these values are used and present good results.

3.6. Crack system

Two types of crack system are at least produced by a Vickers indenter, i.e., a median crack system and a Palmqvist crack system. Generally, the high toughness material shows

Palmqvist crack system. However, most materials will present both crack systems dependent on the amount of load^{31,52}. One way to distinguish the crack system present in the analysed material consists on verification of c/a ratio²⁹. If it is less than 2.5, than the material shows Palmqvist crack system as for the samples 100 ZT, 80 ZT, 100 ZM e 80 ZM in Table 1. If it is greater or equal 2.5, the material exhibits median crack system. The crack system mode for Y-TZP and composite with 80 wt.% of Y-ZrO₂ under loads of < 98 N, presents the Palmqvist crack system regardless of fracture toughness. All the other samples had presented median crack system⁵³. For each sample, the value of a (semi-diagonal length) is approximately constant and therefore, the ratio c/a increases with the increase of the crack length⁵⁴ and lower is the fracture toughness of the analyzed sample. It is possible to verify in Table 1 that lesser crack length was presented for the samples 80 ZT and 80 ZM and that higher were the K_{IC} values, independently of the equation used for its calculation, which allows to conclude that the trend observed for the K_{IC} values for the samples of this work is not related with R-curve effects, where the resistance to crack propagation or the toughness would increase with the increase of crack length³¹.

4. Conclusions

1. The analysis of the zirconia-alumina composites have shown that the zirconia addition promotes composites with higher densities, higher flexural strength and fracture toughness. It was shown that there is an inverse dependence of K_{IC} on the hardness.
2. These composites can achieve a flexural strength 93% and fracture toughness 29% superior when compared to the pure alumina ceramics.
3. The pure zirconia and the composite with 80% of zirconia addition have shown Palmqvist crack system under indentation load of 98,1 N. The other compositions have shown median crack system.
4. The use of different equations for indentation fracture toughness calculations have shown differences of 50% for K_{IC} values for the same composition.
5. The composites with higher zirconia content studied in this work exhibited higher flexural strength and fracture toughness when compared with the pure alumina or even with the pure zirconia. These composites seem to be an adequate material to be used in the manufacture of implant abutments instead of the pure oxides actually in use. The relatively lower hardness values obtained for these composites when compared with pure alumina can be suitable for ceramic abutments in the attainment of their final form by machining. Although, other works including in vivo tests with alumina-zirconia composites with higher zirconia content should

be developed in order to obtain a more reliable result of its performance in this specific dental application.

References

- Fischer H.; Marx R. Fracture toughness of dental ceramics: comparison of bending and indentation method, *Dental Materials*, v. 18, n. 1, p. 12-19, 2002.
- Brånemark P.I.; Zarb G. A.; Albrektsson T. Tissue-integrated prostheses: osseointegration in clinical dentistry, *Quintessence Verlags - GmbH - Berlin*, 1987.
- Sadoun M.; Perelmuter S. Alumina-zirconia machinable abutments for implant-supported single-tooth anterior crowns, *Practical Periodontics and aesthetic dentistry*, v. 9, n. 9, p. 1047-1053, 1997.
- De Aza, A.H.; Chevalier, J.; Fantozzi, G. et al., Crack growth resistance of alumina, zirconia and zirconia toughened alumina ceramics for joint prostheses, *Biomaterials*, v. 23, p.937-945, 2002.
- Hannink, R.H.J. Transformation toughening in Zirconia-Containing Ceramics, *J. Am. Ceram. Soc.*, v. 83, n. 3, p. 461- 487, 2000.
- Fischer H.; Rentzsch, W.; Marx, R. Elimination of low-quality ceramic posts by proof testing, *Dental Materials*, v. 18, n. 8, p. 570 - 575, 2002.
- Keith, O.; Kusy, R.P.; Whitey J.Q. Zirconia brackets: an evaluation of morphology and coefficients of friction *Am. J. Orthod. Dentofacial Orthop*, v. 106, n. 6, p. 605 - 614, 1994.
- Cando, D.C. Zirconia: una alternativa fiable, *Labor Dental*, v. 4, n. 2, p.1-7, 2001.
- Ardlin, B.I. Transformation-toughened zirconia for dental inlays, crowns and bridges: chemical stability and effect of low-temperature aging on flexural strength and surface structure, *Dental Materials* v. 18, n. 8, 590-595, 2002.
- Prestipino, V. and Ingber, A. All-ceramic implant abutments: Esthetic indications, *Journal of Esthetic Dentistry* v. 8, p. 255-262, 1996.
- Christel, P.; Meunier, A.; Heller, M; Torre, J.P.; Peille, C.N. Mechanical properties and short-term in vivo evaluation of yttrium-oxide-partially-stabilized zirconia, *J. Biomed. Mater. Res*, v. 23, p. 45-61, 1989.
- Affatato, S.; Goldoni, M.; Testoni, M.; Toni, A. Mixed-oxides prosthetic ceramic ball heads, Part 3: effect of the ZrO₂ fraction on the wear of ceramic on ceramic hip joint prostheses, A long term in vitro wear study, *Biomaterials*, v. 22, p. 717-723, 2001.
- Serra, E.; Tucci, A.; Esposito, L.; Piconi, C. Volumetric determination of the wear of ceramics for hip joints, *Biomaterials*, v. 23, p. 1131-1137, 2002.
- Piconi, C.; Maccauro, G. Zirconia as a ceramic biomaterial, *Biomaterials* v. 20, p. 1-25, 1999.
- Wang, J.; Stevens, R. Review zirconia-toughened alumina(ZTA) ceramics, *J. Mat. Science*, v. 24, p. 3421-3440, 1989.
- Claussen, N.J.; Fracture toughness of Al₂O₃ with an unstable ZrO₂ dispersed second phase, *J. American Ceram. Soc.*, v. 61, p. 49-51, 1976.
- Lange, F.F. Transformation toughening: Part 4 - Fabrication fracture toughness and strength of Al₂O₃-ZrO₂ composites, *J. Mat. Sci.*, v. 17, p. 247-254, 1982.
- Hori, S.; Yoshimura, M.; Somiya, S. Strength-toughness relation in sintered and isostatically hot-pressed ZrO₂- toughened Al₂O₃, *J. Am. Ceram. Soc.*, v. 69, p. 169-172, 1986.
- Evans, A.G.; Perspective on the development of high-toughness ceramics, *J. Am. Ceram. Soc*, v. 73, p. 187-206, 1990.
- Casellas, D.; Ràfols, I.; Llanes, L.; Anglada, M. Fracture toughness of zirconia-alumina composites, *Int. J. of Refractory Metals & Hard Materials*, v. 17, p. 11-20, 1999.
- Gregori, G.; Burger, W.; Sergo, V. Piezo-spectroscopic analysis of the residual stress in zirconia-toughened alumina ceramics: the influence of the tetragonal-to-monoclinic transformation, *Materials Science and Engineering*, v. A271, p. 401-406, 1999.
- Karihaloo, B.L. Contributions of t-m phase transformation to the toughening of ZTA, *J. Am. Ceram. Soc.* v. 74, p. 1703-1706, 1991.
- Becher, P.F.; Alexander, K.B.; Warmick, W. Influence of ZrO₂ grain size and content on the transformation response in the Al₂O₃- ZrO₂(12% mol CeO₂) system, *J. Am. Ceram. Soc.* v. 76, p. 657-663, 1993.
- Fantozzi, G.; Orange, G. Thermomechanical properties of zirconia toughened alumina materials, In: eds. Moya, J.S.; De Aza, S. editors, *Processing of advanced ceramics*, Soc. Esp. Ceram. Vidr. Arganda Del Rey Madrid Spain, p. 187-215, 1986.
- ASTM: C1327-99, *Standard test method for Vickers indentation hardness of advanced ceramics*, p.1-8, 1999.
- ASTM: C-1421-99, *Standard test method for determination of fracture toughness of advanced ceramics at ambient temperature*, p. 1-32, 1999.
- Anstis, G.R.; Chantikul, P.; Lawn, B.R.; Marshall, D.B. A critical evaluation of indentation techniques for measuring fracture toughness: I direct crack measurements, *J. of American Ceramic Society*, v. 64, n. 9, p. 533-538, 1981.
- Casellas, D.; Nagl, M.M.; et al., Growth of Surface Indentation Cracks in Alumina and Zirconia Toughened Alumina, *Key Engineering Materials*, v. 127-131, p. 895-902, 1997.
- Niihara, K.; Morena, R.; Hasselman, D.P.H. In: Bradt R.C.; Evans. A.G.; Hasselman D.P.H.; Lange. F.F. editors, *Fracture mechanics of ceramics*, New York, Plenum: v. 5, p. 97, 1983.
- Hannink, R.H.J. Transformation toughening in Zirconia-Containing Ceramics, *J. Am. Ceram. Soc.*, v. 83, n. 3, p. 461-487, 2000.
- Bravo-Leon, A.; Morikawa, Y.; Kawahara, M.; Mayo, M.J. Fracture toughness of nanocrystalline tetragonal zirconia with

- low yttria content, *Acta Materialia*, v. 50, p. 4555-4562, 2002.
32. Tuan, W.H.; Chen, R.Z.; Wang, C.H.; Cheng, P. S.; Kuo, P.S. Mechanical properties of $Al_2O_3//ZrO_2$ composites, *J. European Ceram Soc.*, v. 22, p. 2827-2833, 2002.
 33. Silva, F.R. *Cinética de sinterização e desgaste de pastilhas de corte do sistema $Si_3N_4-CeO_2-AIN$* , Tese de Doutorado, Universidade de Aveiro, 1992.
 34. ASTM: C1161-90, *Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature*, p. 329-335, 1990.
 35. ASTM: F-603,00, *Standard specification for high-purity aluminium oxide for medical application*, p. 103-105, 2000.
 36. ASTM F-1873-98, *Standard specification for high-purity dense yttria tetragonal zirconium oxide polycrystal(Y-TZP) for surgical implant applications*, p. 1349-1351, 1998.
 37. Kunes, K.; Havrda, J.; et al. Stabilization of Bioceramic suspensions prepared from alumina-containing zirconia powders, *Ceramics*, v. 44, n. 1, p.1-8, 2000.
 38. Oliveira, L.G. *Efeito do processamento nas propriedades mecânicas da alumina tenacificada por zircônia parcialmente estabilizada com cálcia ou ítria*, M. Sc. Thesis, COPPE-UFRJ, 2002.
 39. Mukherjee, A.; Maiti, B.; Shara, A.D.; Basu, R.N.; Maiti, H.S. Correlation between slurry. rheology. green density and sintered density of tape cast yttria stabilised zirconia. *Ceramics Int.*, v. 27, p. 731-739, 2001.
 40. Lange, F.F. Transformation toughening: Part 4 - Fabrication fracture toughness and strength of $Al_2O_3-ZrO_2$ composites, *J. Mat. Sci.*, v. 17, p. 247-254, 1982.
 41. Krell, A. Load dependence of hardness in sintered submicrometer Al_2O_3 e ZrO_2 , *J. Am. Ceram. Soc.*, v. 78, n. 5, p. 1417-1419, 1995.
 42. Celli, A.; Tucci, A.; Esposito, L.; et al., Fractal analysis of cracks in alumina-zirconia composites, *J. Eur. Ceram. Soc.*, v. 23, p. 469-479, 2003.
 43. Absi, J.; Glandus, J.C. Numerical separation of bi-modal strength distributions, *Journal of the European Ceramic Society*, v. 22, p. 593-601, 2002.
 44. Tuan, W.H.; Kuo, J.C. Contribution of residual stress to the strength of abrasive ground alumina, *J. Eur. Ceram. Soc.* v. 19, p. 1593-1597, 1999.
 45. Spriggs, R.M.; Vasilos, T. Effect of grain size on transverse bend strength of alumina and magnesia, *J. Am. Ceram. Soc.* v. 46, p. 224-228, 1963.
 46. Gupta, T.K.; Lange, F.F.; Bechtold, J.H. Effect of stress-induced phase transformation on properties of polycrystalline zirconia containing metastable tetragonal phase, *J. Mater. Sci.*, v. 13, p. 1464-1470, 1978.
 47. Huang, X.W.; Wang, S.W.; Huang, X.X. Microstructure and mechanical properties of ZTA fabricated by liquid phase sintering, *Ceramics International*, article in press.
 48. Tomaszewski, H. Toughening effects in $Al_2O_3-ZrO_2$ system, *Ceram. Intern.*, v. 14, p. 117-125, 1988.
 49. Hori, S.; Yoshimura, M.; Somiya, S. Strength-toughness relation in sintered and isostatically hot-pressed ZrO_2 - toughened Al_2O_3 , *J. Am. Ceram. Soc.*, v. 69, p. 169-172, 1986.
 50. Lange, F.F. Transformation toughening: Part 1- Size effects associated with the thermodynamics of constrained transformations, *J. Mat. Sci.*, v. 17, p. 225-234, 1982.
 51. Srdic, V.; Radonjic, L. Processing of the alumina-zircônia composite and its mechanical properties. In: *Third Euro-Ceramics*, ed. Urán, P.; Fernández, J.F., v. 3, p. 701-706, 1993.
 52. Lin, J.D.; Duh, J.G. Fracture toughness and hardness of ceria and yttria-doped tetragonal zirconia ceramics, *Materials Chemistry and Physics*, v. 78, p. 253-261, 2002.
 53. Basu, D.; Sarkar, B.K. Toughness determination of zirconia toughened alumina ceramics from growth of indentation-induced cracks, *J. Mater. Res.*, v. 11, n. 12, p. 3057-3062, 1996.
 54. Wang, J.; Gong, J.; Guan, Z. Variation in the indentation toughness of silicon nitride, *Materials letters*, Article in press, 2002.