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Fracture toughness improvements of dental ceramic through use of yttria-stabilized zirconia (YSZ) thin-film coatings

Ryan N. Chan^{a,b}, Brian R. Stoner^b, Jeffrey Y. Thompson^c, Ronald O. Scattergood^a, Jeffrey R. Piascik^{b,a,*}

^a North Carolina State University, Department of Materials Science and Engineering, 911 Partner's Way, EB1 Room 3002, Raleigh, NC 27695, United States

^b RTI International, Center for Materials and Electronic Technologies, PO Box 12194, 3040 Cornwallis Road RTP, NC 27709, United States

^c Nova Southeastern University, Section of Prosthodontics, 3301 College Avenue, Fort Lauderdale, FL 33328, United States

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ABSTRACT

Objectives. The aim of this study was to evaluate strengthening mechanisms of yttria-stabilized zirconia (YSZ) thin film coatings as a viable method for improving fracture toughness of all-ceramic dental restorations.

Methods. Bars (2 mm × 2 mm × 15 mm, $n=12$) were cut from porcelain (ProCAD, Ivoclar-Vivadent) blocks and wet-polished through 1200-grit using SiC abrasive. A Vickers indenter was used to induce flaws with controlled size and geometry. Depositions were performed via radio frequency magnetron sputtering (5 mT, 25 °C, 30:1 Ar/O₂ gas ratio) with varying powers of substrate bias. Film and flaw properties were characterized by optical microscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD). Flexural strength was determined by three-point bending. Fracture toughness values were calculated from flaw size and fracture strength.

Results. Data show improvements in fracture strength of up to 57% over unmodified specimens. XRD analysis shows that films deposited with higher substrate bias displayed a high %monoclinic volume fraction (19%) compared to non-biased deposited films (87%), and resulted in increased film stresses and modified YSZ microstructures. SEM analysis shows critical flaw sizes of $67 \pm 1 \mu\text{m}$ leading to fracture toughness improvements of 55% over unmodified specimens.

Significance. Data support surface modification of dental ceramics with YSZ thin film coatings to improve fracture toughness. Increase in construct strength was attributed to increase in compressive film stresses and modified YSZ thin film microstructures. It is believed that this surface modification may lead to significant improvements and overall reliability of all-ceramic dental restorations.

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* Corresponding author at: RTI International, Center for Materials and Electronic Technologies, PO Box 12194, 3040 Cornwallis Road RTP, NC 27709, United States. Tel.: +1 919 931 5622.

E-mail address: jpiascik@rti.org (J.R. Piascik).

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1. Introduction

All-ceramic dental restorations have become extremely popular due to their outstanding esthetic characteristics and biocompatibility [1–4]. However, these materials, when placed in load bearing applications, often display an inability to resist stress-induced crack propagation [5–7]. Analysis of clinically failed all-ceramic restorations revealed that the majority of failures were initiated at the surface, where tensile stresses and flaws were found to accelerate construct failure [2,8–12]. Additionally, clinicians typically etch or particle air-abrade the inside of the restoration to roughen surfaces for enhanced adhesion, inducing large surface flaws and further promoting premature failure [11,13–16]. This study seeks to modify surface stress states and facilitate flaw modification to increase the fracture toughness of dental porcelain.

Fracture toughness improvements have been demonstrated through various surface modification methods; such as, flaw modification, heat treatments, and coatings. Fundamentally, by controlling surface finishing techniques, fracture toughness can be enhanced through modification of flaw size or geometry, resulting in reduced stress concentration in the local area of a critical flaw and therefore increasing overall strength of a specimen [11,17]. Compressive stress gradients can also be introduced to a specimen through thermal processing, where a surface is cooled at a faster rate than the interior of a specimen, resulting in a compressive surface [18,19]. Composition gradients and ion implantation techniques have also been used to induce strain into the crystal lattice, resulting in compressive stresses on a specimen surface [20]. These stress-modification mechanisms require crack expansion (or increased tensile stresses), to overcome the induced compressive stresses on the surface of a specimen. As a result, crack propagation is inhibited, resulting in increased fracture toughness.

Thin film coatings have been employed to strengthen substrates; first through critical flaw modification and secondly through inducing compressive surface stresses [21,22]. Rudell et al. have shown improvements in fracture strength of up to 19% by coating ceramic substrates with less than 10 μm of sputtered metallic thin films [21,23]. Earlier work reported increases in strength of up to 32% by using a multilayer film (10 μm) structure consisting of alternating layers of yttria-stabilized zirconia (YSZ) (1 μm) and parylene (1 μm) to induce crack deflection [24]. Additionally, it was determined that a 2–3 μm thick YSZ thin film sputtered on porcelain substrates provide the maximum benefit for increased construct fracture strength [22].

YSZ is a material of particular interest not only due to its biocompatibility, but also for its excellent wear resistance properties [25–27]. Reports have shown that microstructure and film stress properties of YSZ thin films can be tailored through the control of deposition parameters [28,29]. By applying a substrate bias during deposition, an interrupted microstructure consisting of lateral microcracks and increased compressive film stresses can be obtained. It is therefore hypothesized that these structures may provide an ideal thin film for increasing the fracture toughness of ceramic substrates.

This study examines the benefits of varying YSZ thin film microstructures and magnitudes of compressive thin film states on substrates that have been fabricated with a controlled flaw size of a given geometry. Analysis of construct fracture strength and measurements of critical flaw size allows for the determination of overall construct fracture toughness. Here we discuss the impact of thin film induced compressive stresses, flaw modification, and crack deflection on increased fracture toughness of modified dental porcelain.

2. Materials and methods

Porcelain substrates were fabricated by cutting $18 \times 2 \times 2$ mm bars from leucite-reinforced feldspathic porcelain blocks (ProCAD, Ivoclar-Vivadent, Schaan, Liechtenstein). All four surfaces of porcelain substrates were polished through 1200 grit (CAMI Grit Designation) SiC abrasive and edges rounded to limit corner/edge failures. Substrates were ultrasonically cleaned in acetone to remove surface debris prior to deposition, then randomly divided into four groups ($n = 30$). A Vickers indenter (Mitutoyo Model AAV-500, Aurora, IL) was used to apply a single, controlled flaw into the center of a substrate surface with indent corners perpendicular to substrate edges, in order to normalize failures through control of critical flaw size and geometry. A 0.5 N load was applied with a load time of 15 s, resulting in an indent size of 34–37 μm to simulate critical flaw sizes in clinical surface preparation methods [8].

Thin film depositions were performed in an r.f. magnetron sputter reactor (CVC Model SC-400, Rochester, NY). A second r.f. power source was capacitively coupled to the substrate and used for depositions requiring a substrate bias. Target material used for sputtering was 99.99% pure zirconia doped with 3 mol% yttria (Plasmaterials, Livermore, CA). Substrates were mounted directly above the sputtering gun (US Gun, San Jose, CA) in a custom fixture at a working distance of 75 mm. At the start of each deposition, a 50 W substrate bias in an argon atmosphere was applied for 5 min to clean organic debris from specimen surfaces and to promote film adhesion. All depositions were performed with a target power of 350 W, atmosphere of 30:1 argon to oxygen, and substrate bias varied between 0 and 100 W. Films were grown to a thickness of 2–3 μm to maximize fracture strength [24].

X-ray diffraction (XRD) (PANalytical X'Pert PRO MRD HR, Westborough, MA) was used to determine the percentage of tetragonal and monoclinic phases [30] in the YSZ films and thus qualitatively determine trends in film stress [31]. Fracture strength values were determined using a three-point bending fixture, with specimens oriented with films in tension per ASTM Standard C1161 [32]. Flexural testing (Instron Model 5542, Norwood, MA) was conducted with a 10 mm span at a crosshead speed of 0.5 mm/min. Bar dimensions were measured prior to fracture for fracture stress calculations. Peak loads and fracture stresses for each specimen were recorded and single-factor analysis of variance (ANOVA) at a 5% confidence level was performed to evaluate for statistical similarities. For testing controls, a polished sample set with a Vickers indentation was left unmodified by films in order to determine inert strengths of substrates at a given flaw size. Post fracture, scanning electron

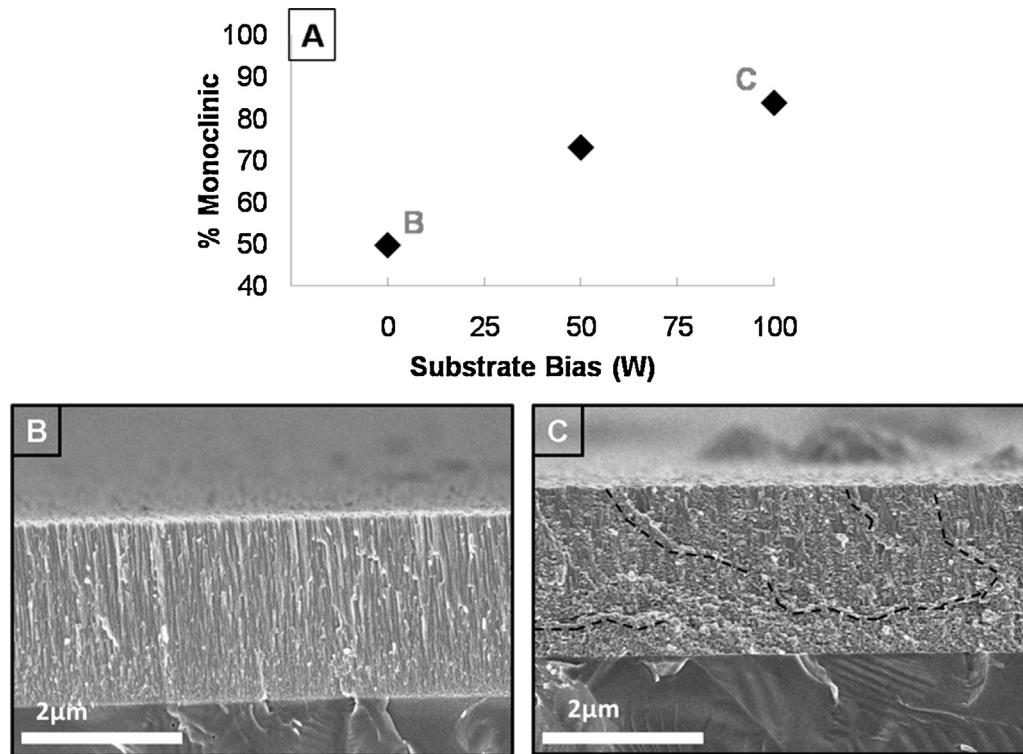


Fig. 1 – (a) X-ray diffraction data show an increase in %monoclinic phase as a function of substrate bias, indicating an associated increase in compressive film stresses. SEM images of fracture surfaces showing microstructure differences between films deposited (b) without substrate bias (c) with 100 W substrate bias. Changes in microstructure suggest a potential crack deflection strengthening mechanism, highlighted by dotted lines.

microscopy (SEM) (Hitachi, S-4700 FE, Tokyo, Japan) and optical microscopy (Sony DXC-390, Exwave HAD, Irvine, California) was used to image specimen fracture surfaces to characterize critical flaws. Fracture strength and critical flaw size measurements then were used to calculate apparent fracture toughness [5].

3. Results

%Monoclinic values were found to increase with substrate bias (Fig. 1A), ranging from 50% (no substrate bias) to 84% (100 W substrate bias). Observable changes in film microstructure were associated with increasing volume fraction of %monoclinic. A representative SEM image of a non-biased film structure (Fig. 1B) was shown to have a columnar morphology; whereas, 100 W film (Fig. 1C) shows interrupted structure, characterized by the presence of inter-granular ledges.

In order to standardize critical flaw size, a Vickers indent was applied to each surface prior to thin film deposition and characterized. Fig. 2A is a representative optical image of an indent that shows the damage zone ($35 \pm 2 \mu\text{m}$) and critical flaw size ($67 \pm 1 \mu\text{m}$) characterized by the radial cracking from the indent corners. Fig. 2B shows a critical flaw on the fracture surface of a fractured specimen. A damage zone can be seen in the area immediately under the indent (indicated by small arrows), from which the radial cracks identify the size of critical flaw (indicated by large

arrows). Radial crack traces, perpendicular to the fracture surface (beyond the borders of the micrograph), can be observed (indicated by black arrow). Additionally, Fig. 2C shows a post-fracture specimen indicating failure at the indent (critical flaw) and excellent adhesion between the thin film and porcelain.

Fig. 3 shows fracture strength values for control specimens and specimens modified by YSZ thin films. Unmodified specimens showed fracture strengths of $107 \pm 6 \text{ MPa}$. Specimens coated with unbiased thin films displayed a fracture strength of $119 \pm 5 \text{ MPa}$; whereas, specimens coated with thin films deposited at 100 W substrate bias showed fracture strength of $166 \pm 11 \text{ MPa}$. Fracture stress increased over unmodified specimens ranging from 14% to 55% and ANOVA analysis showed that all groups were statistically different (Table 1).

Fracture strength and flaw size measurements were used to calculate apparent fracture toughness values for each specimen (Fig. 4). Apparent fracture toughness calculations used the equation

$$K_{IC} = Y\sigma_f \cdot a^{1/2} \quad (1)$$

where K_{IC} is the mode I critical stress intensity factor (fracture toughness) [$\text{MPa m}^{1/2}$], Y is the shape factor (1.29), σ_f is fracture stress [MPa], and a is the radius of the critical flaw [m] [36]. Apparent fracture toughness shows similar trends to

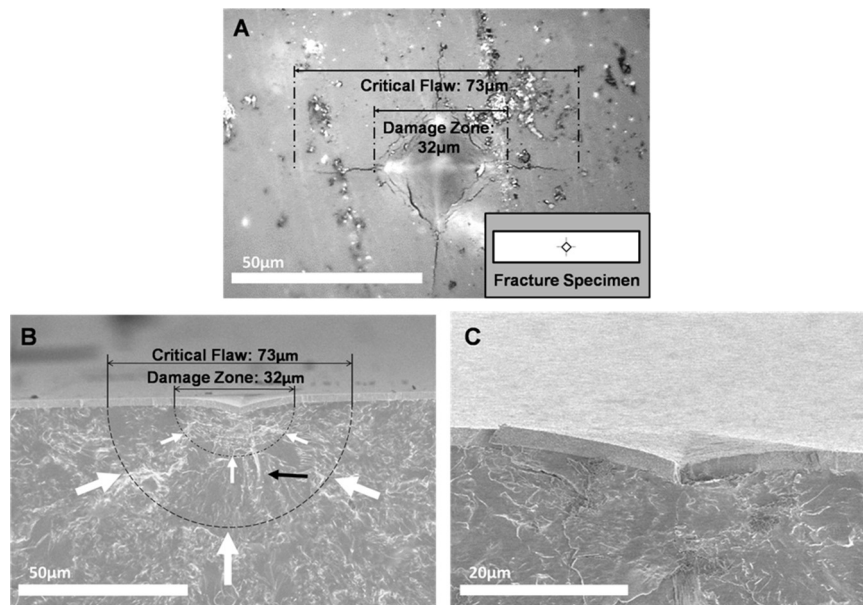


Fig. 2 – (a) Optical micrograph showing a Vickers indent on the surface of a porcelain substrate. Radial cracks denote size of critical flaw. Inset shows flaw orientation on an example fracture specimen. (b) SEM micrograph of a fracture surface showing characteristics of a critical flaw as a result of Vickers indentation. Critical flaw diameters are measured to range between 62 and 64 μm . (c) After sputter depositing an YSZ thin film, SEM micrograph demonstrates good adhesion to a Vickers indent and the substrate surface.

Table 1 – ANOVA comparisons of fracture strength.

Source of variation	SS	df	MS	F	P-value	F crit
Between groups	59,351	3	19,784	39	2.0 E–17	2.7
Within groups	59,246	116	511			
Total	118,597	119				

fracture strength (increases of 11–51%). Literature values show porcelain fracture toughness at approximately $1.0 \text{ MPa m}^{1/2}$ [33–37] with unmodified specimens in this study having apparent fracture toughness values of $0.83 \pm 0.04 \text{ MPa m}^{1/2}$. For 100 W thin films, apparent fracture toughness increases to $1.21 \pm 0.1 \text{ MPa m}^{1/2}$ (51% increase).

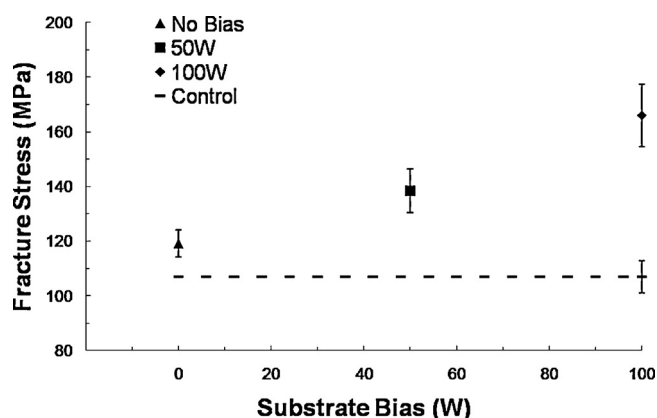


Fig. 3 – Fracture stress values measured from 3pt bend flexure testing. The dotted line indicates fracture strength for control specimens. Data show an improvement in fracture strength of 55% over unmodified substrates. Error bars represent 95% confidence levels.

4. Discussion

Fracture toughness is a material property that describes a material's innate ability to resist crack propagation. Experimentally, fracture toughness is determined by imparting a

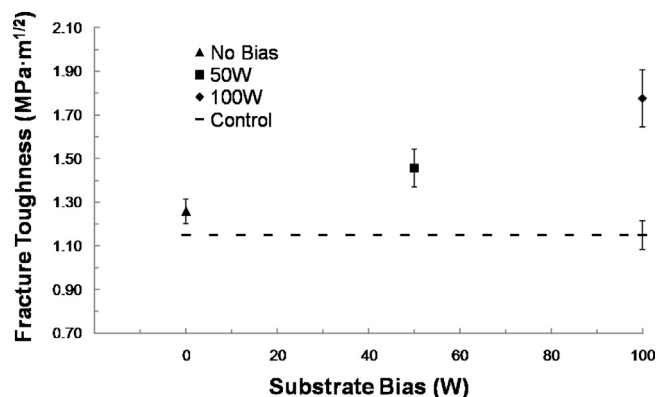


Fig. 4 – Apparent fracture toughness values calculated from 3pt bend fracture strength data and flaw size measurements. By depositing an YSZ thin film with 100 W substrate bias, an improvement in apparent fracture toughness of 55% can be attained over unmodified substrates. Error bars represent 95% confidence levels.

flaw in a specimen, determining the location of crack initiation. In this study, flaws were imparted using a Vickers indenter in order to control flaw sizes and geometry. Observed critical flaw sizes are on the order of 73 μm and have a semi-circular geometry, comparable to studies of fractured porcelain showing critical flaw sizes of 60–70 μm for air-abraded porcelain specimens [38]. Measured fracture toughness values in this study were $0.80 \pm 0.04 \text{ MPa m}^{1/2}$ whereas literature values show porcelain fracture toughness to be approximately $1.0 \text{ MPa m}^{1/2}$ [33–37]. By applying thin film YSZ coatings over the substrate surface, we achieved a 51% increase in fracture toughness over unmodified specimens.

Increases in fracture toughness may be attributed to three strengthening mechanisms; compressive film stresses, crack deflection, and flaw modification. Compressive film stresses arise as particle bombardment occurs during film growth [39]. During this process, kinetic energy of impinging atoms causes agglomeration of deposited atoms parallel to a substrate surface. As film growth continues, proximity of atoms induces lattice expansion; however, expansion is inhibited by neighboring atomic clusters. As a result, compressive stresses along this plane are generated, while expansion in the direction perpendicular to the substrate remains uninhibited. This mechanism has been used to describe typical columnar film morphology in physical vapor deposited (PVD) films [40,41]. In sputtered, YSZ thin films, film stresses induce a tetragonal to monoclinic phase transformation, allowing for a correlation between film stress and %monoclinic volume fraction [29]. Monoclinic volume fraction was then measured by comparing relative intensities of the Tetragonal (IT(1 1 1)) and Monoclinic (IM(1 1 1)) peaks [30]. With the application of a substrate bias during deposition, kinetic energy of impinging atoms are increased, resulting in increased film stresses and increased %monoclinic phase (Fig. 1A).

As %monoclinic volume fraction is increased, film morphology shifts from a columnar structure (Fig. 1B) toward an interrupted structure (Fig. 1C), (characterized by lateral cracks) as a mechanism for film stress relief [29]. With a columnar structure, crack paths are expected to propagate through the straight, inter-columnar grain boundaries. However, presence of lateral defects may provide a crack deflection mechanism during failure. As cracks lengths are increased, fracture energy is increased, exhibited by an increase in fracture strength. Fracture surfaces of these interrupted microstructures show ledges (Fig. 1C), indicating evidence for crack deflection during fracture.

Impinging atoms can preferentially fill cracks as they migrate across a surface, leading to an increase in local radius of curvature [21]. In this case, crack tips are blunted, increasing force required for crack propagation. Fig. 2C shows adhesion of a deposited YSZ film over a Vickers indent. Crack tip blunting effects are inconclusive from SEM analysis. However, critical flaw characteristics can be determined, leading to insight regarding failure mechanisms. These flaws are shown to exhibit a standard, semi-circular shape, $67 \pm 1 \mu\text{m}$ in diameter (Fig. 2B). Geometry of the Vickers indent can be seen with a corresponding damage zone immediately under the indent, in the substrate. It is expected that under flexure loading, fracture initiation occurs at the interface between this damage zone and the rest of the substrate. During fracture initiation,

compressive stresses in the coatings resist crack propagation. Once this compressive stress is overcome by tensile loading conditions, crack propagation begins, resulting in critical flaw characteristics. Also during crack propagation, lateral cracking in interrupted microstructures will provide additional strengthening to the construct. Therefore compressive film stresses likely act as a primary strengthening mechanism whereas crack deflection acts as a secondary strengthening mechanism in this system.

Imparting flaws into a surface with an indenter allows for a method that produces consistent flaws, resulting in consistent fracture characteristics. As a result, fracture data is normalized for flaw size and geometry, leading to reduced error. As expected, fracture strength of specimens modified by films are shown to be improved ($P \leq 0.05$) over substrates unmodified by a film (Fig. 3). Due to film strengthening effects, increases in fracture strength of up to 55% can be observed with increasing substrate bias. While increasing substrate bias is expected to lead to further improvements in fracture strength, bias plasma becomes unstable and increased film stresses leads to delamination, reducing the effectiveness of films.

Combining critical flaw analysis and fracture strength data allows for calculation of apparent fracture toughness values. Similar to fracture strength improvements, increases in apparent fracture toughness of up to 51% can be observed with increasing substrate bias. In order to more accurately characterize the contributions of each mechanism to overall strength of the construct, the system must be modeled by analyzing stresses throughout the construct. It has been shown that stresses on a surface of cracks of this geometry can be effectively modeled [42]. By modifying models to consider a compressive coating over a semi-circular crack, contributions of strengthening of from film stress, crack deflection, and flaw modification of YSZ films on substrate can be quantified. This model may offer insight to the location of failure initiation, leading to more effective design and further strength improvements.

Data show support for the hypothesis for biased-YSZ coatings as an effective method for improving apparent fracture toughness of dental porcelain by up to 51% over unmodified specimens. Improvements are attributed to compressive stresses and modified film microstructures, which upon loading, act to resist crack propagation. Introduction of controlled flaws allows for future work to model and extrapolate strengthening effects on varying flaw sizes, geometries, and substrates. Increases in apparent fracture toughness demonstrate YSZ coatings as a viable method for improving reliability of all-ceramic dental restorations.

5. Conclusion

Surface modification of dental ceramics with YSZ thin film coatings is shown as a viable method for improved reliability of dental ceramic materials. Increases in strength are correlated to increased compressive film stresses and modified YSZ microstructures. Future studies will evaluate clinically relevant constructs and a development of a thin film strengthening model. A 55% increase in overall apparent fracture

strength can have significant ramifications on the longevity and reliability of all-ceramic restorations.

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REFERENCES

- [1] Kelly JR. Ceramics in restorative and prosthetic dentistry. *Annual Review of Materials Science* 1997;27:443.
- [2] Anusavice KJ. Degradability of dental ceramics. *Advances in Dental Research* 1992;6:82.
- [3] Albakry M, Guazzato M, Swain MV. Effect of sandblasting grinding polishing and glazing on the flexural strength of two pressable all-ceramic dental materials. *Journal of Dentistry* 2004;31:91.
- [4] Cattell MJ, Clarke RL, Lynch EJR. The biaxial flexural strength and reliability of four dental ceramics – Part II. *Journal of Dentistry* 1997;25(5):409.
- [5] Barsoum MW, Cantor B, Goringe MJ, editors. *Fund ceram*. New York, NY: Taylor & Francis Group; 2003. p. 356–99.
- [6] Sakaguchi RL, Cross M, Douglas WH. A simple model of crack propagation in dental materials. *Dental Materials* 1992;8:131.
- [7] Wiederhorn SM. Brittle fracture and toughening mechanisms in ceramics. *Annual Review of Materials Science* 1984;14:373.
- [8] Thompson JY, Anusavice KJ, Naman A, Morris HF. Fracture surface characterization of clinically failed all-ceramic crowns. *Journal of Dental Research* 1994;73(12):1824.
- [9] Kelly JR, Tesk JA, Sorensen JA. Perspectives on strength. *Journal of Dental Research* 1995;74(6):1253.
- [10] Taskonak B, Mecholsky JJ, Anusavice KJ. Residual stresses in bilayer dental ceramics. *Biomaterials* 2005;26:3235.
- [11] De Jager N, Feilzer AJ, Davidson CL. The influence of surface roughness on porcelain strength. *Dental Materials* 2000;16:381.
- [12] Rekow ED, Silva NRFA, Coelho PG, Zhang Y, Guess P, Thompson VP. Performance of dental ceramics-challenges for improvements. *Journal of Dental Research* 2011;90:937.
- [13] Stangel I, Nathanson D, Hsu CS. Shear strength of the composite bond to etched porcelain. *Journal of Dental Research* 1987;66:1460.
- [14] Aboushelib MN, Wang H. Effect of surface treatment on flexural strength of zirconia bars. *Journal of Prosthetic Dentistry* 2010;104(2):98.
- [15] Curtis AR, Wright AJ, Fleming GJP. The influence of surface modification techniques on the performance of a Y-TZP dental ceramic. *Journal of Dentistry* 2006;34:195.
- [16] Lawn BR, Hockey BJ, Wiederhorn SM. Atomically sharp cracks in brittle solids-an electron microscopy study. *Journal of Materials Science* 1980;15:1207.
- [17] Mecholsky JJ, Rice RW, Freiman SW. Prediction of fracture energy and flaw size in glasses from measurements of mirror size. *Journal of the American Ceramic Society* 1974;57(10):440.
- [18] Rekhson SM. Thermal stresses relaxation and hysteresis in glass. *Glass Science and Technology* 1993;76(5):1113.
- [19] Narayanaswamy OS. Stress and structural relaxation in tempering glass. *Journal of the American Ceramic Society* 1978;61(3):146.
- [20] Xing A, Jun Z, Chuazhen H, Jianhua Z. Development of an advanced ceramic tool material – functionally gradient cutting ceramics. *Materials Science and Engineering A* 1998;248(1):125.
- [21] Ruddell D, Thompson JY, Stoner BR. Mechanical properties of dental ceramic coated by RF magnetron sputtering. *Journal of Biomedical Materials Research* 1999; 51:316.
- [22] Teixeira EC, Piascik JR, Stoner BR, Thompson JY. *Journal of Biomedical Materials Research Part B* 2007; 83:459.
- [23] Ruddell D, Stoner BR, Thompson JY. Effect of YSZ thin film coating thickness on the strength of a ceramic substrate. *Thin Solid Films* 2003;445:14.
- [24] Teixeira EC, Piascik JR, Stoner BR, Thompson JY. Zirconia-parylene multilayer thin films for enhanced fracture resistance of dental ceramics. *Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science* 2009; 223:897.
- [25] Piconi C, Maccauro G. Zirconia as a biomaterial. *Biomaterials* 1999;20:1.
- [26] Chevalier J. What future for zirconia as a biomaterial. *Biomaterials* 2006;27:535.
- [27] Zhou J, Mah J, Shrotriya P, Mercer C, Soboyejo WO. Contact damage in an yttria stabilized zirconia-implications for biomedical applications. *Journal of Materials Science: Materials in Medicine* 2007;18:71.
- [28] Amor SB, Rogier B, Baud G, Jacquet M, Nardin M. Characterization of zirconia films deposited by r.f. magnetron sputtering. *Materials Science and Engineering B* 1998; 57:28.
- [29] Piascik JR, Zhang Q, Bower CA, Thompson JY, Stoner BR. Evidence of stress-induced tetragonal-to-monoclinic phase transformation during sputter deposition of YSZ. *Journal of Materials Research* 2007;22:1105.
- [30] Wang JH, Li XP. Phase structure characteristics of r.f. reactively sputtered zirconia thin film. *Thin Solid Films* 1994;250:132.
- [31] Piascik JR, Thompson JY, Bower CA, Stoner BR. Stress evolution as a function of substrate bias in r.f. magnetron sputtered yttria-stabilized zirconia films. *Journal of Vacuum Science & Technology* 2006;24:4.
- [32] ASTM C1161 – 02c(2008)e1. Standard test method for flexural strength of advanced ceramics at ambient temperature. *American Society for Testing and Materials* 2008;15.01: 1–16.
- [33] Taira M, Nomura Y, Wakasa K, Yamaki M, Matsui A. Studies on fracture toughness of dental ceramics. *Journal of Oral Rehabilitation* 1990;17(6):551.
- [34] Scherrer SS, Kelly JR, Quinn GD, Xu K. Fracture toughness of a dental porcelain determined by fractographic analysis. *Dental Materials* 1999;15:342.
- [35] Seghi RR, Denry IL, Rosenstiel SF. Relative fracture toughness and hardness of new dental ceramics. *Journal of Prosthetic Dentistry* 1995;74(2):145.
- [36] ASTM C1322 – 05b(2010). Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics 2010;15.01:1–51.
- [37] Yilmaz H, Aydin C, Gul BE. Flexural strength and fracture toughness of dental core ceramics. *Journal of Prosthetic Dentistry* 2007;98(2):120.
- [38] Smith RL, Thompson JY, Chan RN, Stoner BR, Piascik JR, Mecholsky JJ. Triple blind fractography study of yttria-stabilized zirconia thin-film-modified porcelain. *Fractography of Glasses and Ceramics VI: Ceramic Transactions* 2012;230:265.

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- [39] Knotek O, Elsing R, Kramer G, Jungblut F. On the origin of compressive stress in PVD coatings – an explicative model. *Surface and Coatings Technology* 1991;46:265.
 - [40] Thornton JA. The microstructure of sputter-deposited coatings. *Journal of Vacuum Science and Technology A* 1986;6:3059.
 - [41] Thompson CV. Structure evolution during processing of polycrystalline films. *Annual Review of Materials Science* 2000;30:159.
 - [42] Lawn BR, Fuller ER. Equilibrium penny-like cracks in indentation fracture. *Journal of Materials Science* 1975;10:2016.