

Enamel Wear Opposing Polished and Aged Zirconia

JO Burgess • S Janyavula • NC Lawson
TJ Lucas • D Cakir

Clinical Relevance

Zirconia is an esthetic material with mechanical properties similar to steel. However, when subjected to loading in the oral cavity, zirconia exhibits phase transformation that increases the surface roughness. The roughness, however, does not increase opposing enamel loss.

SUMMARY

Aging of dental zirconia roughens its surface through low temperature degradation. We hypothesized that age-related roughening of zirconia crowns may cause detrimental wear to the enamel of an opposing tooth. To test our hypothesis, we subjected artificially aged zirconia and reference specimens to simulated mastication in a wear device and measured the wear of an opposing enamel cusp. Additionally,

the roughness of the pretest surfaces was measured. The zirconia specimens, artificially aged by autoclave, showed no significant increase in roughness compared to the nonaged specimens. Furthermore, no significant difference in material or opposing enamel wear between the aged and nonaged zirconia was seen. All zirconia specimens showed less material and opposing enamel wear than the enamel to enamel control or veneering porcelain specimens. Scanning electron micrographs showed relatively smooth surfaces of aged and nonaged zirconia following wear testing. The micrographs of the veneering ceramic showed sharp fractured edges and fragments of wear debris. Zirconia may be considered a wear-friendly material for restorations opposing enamel, even after simulated aging.

*John O Burgess, DDS, University of Alabama at Birmingham, Prosthodontics and Biomaterials, AL, USA

Sridhar Janyavula, BDS, MS, University of Alabama at Birmingham, School of Dentistry, Clinical and Community Sciences, Birmingham, AL, USA

Nathaniel C Lawson, DMD, MS, University of Alabama at Birmingham, Clinical and Community Sciences, Birmingham, AL, USA

Thomas J Lucas, MS, University of Alabama at Birmingham, School of Dentistry, Birmingham, AL, USA

Deniz Cakir, DDS, MS, University of Alabama at Birmingham, School of Dentistry, Clinical and Community Sciences, Birmingham, AL, USA

*Corresponding author: University of Alabama at Birmingham, Clinical and Community Sciences, Division of Biomaterials, 1919 7th Avenue South, Birmingham, AL 35294, USA; e-mail: jburgress@uab.edu

DOI: 10.2341/12-345-L

INTRODUCTION

As a result of its unique microstructure, zirconia has mechanical properties superior to those of other dental ceramics.¹ It is a multiphasic ceramic, existing in three temperature-dependent crystallographic structures: monoclinic (room temperature-1170°C), tetragonal (1170°C-2370°C), and cubic (above 2370°C).² The tetragonal phase of zirconia

can be stabilized at oral temperatures by alloying the zirconia with an oxidizer, such as yttria.³ Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is metastable at oral temperatures, meaning that mechanical and thermal stimuli induce a tetragonal to monoclinic (t-m) transformation.⁴ This phase transformation causes a 4.5% volume expansion in the material and presents both advantages and disadvantages for Y-TZP.⁵ Following crack initiation in Y-TZP, the t-m phase-induced expansion compresses the growing crack tip in a phenomenon known as transformation toughening.⁴ The t-m phase transformation can also spontaneously occur over time in a process known as low temperature degradation.⁶

Zirconia is used in dental applications for its dual esthetic and mechanical advantages. As a result of its opacity, early uses of dental zirconia were for crown cores and frameworks over which a more translucent veneering porcelain was applied. As a result of a mismatch in the coefficient of thermal expansion of the core zirconia and the veneering porcelain,⁷ however, chipping of the veneering porcelain was observed. In response, monolithic, full-contour zirconia restorations were introduced.⁸ An initial concern with nonveneered zirconia was wear of opposing enamel, as zirconia is over twice as hard as veneering porcelain.^{9,10} Recent studies,^{11,12} however, have shown that polished zirconia appears to be wear-friendly with regard to opposing tooth structure.

Although the *in vivo* wear of zirconia is a relatively new problem in dentistry, zirconia has been used in artificial hip joints since the late 1980s, and orthopedic investigators^{13,14} have noted increased roughening and wear of zirconia after prolonged *in vivo* service. As zirconia ages, it undergoes low temperature degradation. This degradation has been associated with surface roughening and particle release,¹⁵ which increase its wearing potential. The wear associated with zirconia-based artificial hip joints led to manufacturer recalls¹⁶; therefore, it is critical to examine the effects of zirconia aging for dental applications. Since full-contoured zirconia crowns have only been commercially available for a short period of time, it is necessary to artificially age zirconia in order to predict its long-term wear performance. Several methods exist to artificially age zirconia, including autoclaving and boiling.¹⁶⁻¹⁸ In this project, zirconia was artificially aged by autoclave.

In this study, we examined polished and aged zirconia. Roughness, wear, and opposing tooth wear

were compared. Enamel and veneering porcelain were used as controls. The null hypothesis was that there would be no difference in wear or opposing enamel wear between any of the tested materials.

MATERIALS AND METHODS

Specimen Preparation

Sixteen Y-TZP specimens (LAVA, 3M ESPE, St Paul, MN, USA) were surface polished in the pre-sintered (green) state with 2500-grit silicon carbide sandpaper and then sintered at 1500°C. Eight of the specimens were then polished with a 3- μ m diamond disk and polishing compound as the final step and assigned to the polished group. The remaining eight specimens were heated in a dental autoclave (biomedis FVS 2 Steam Autoclave, biomedis GmbH) for five hours at 135°C and two-bar pressure. These specimens were assigned to the aged group. Specimens (n=8) of a veneering porcelain (Lava Ceram; 3M ESPE) were prepared by the manufacturer. Flat human maxillary incisors (n=8) were collected for reference enamel and mounted to expose their facial surfaces. Opposing enamel cusps (antagonists) were prepared from extracted caries-free mandibular molars. Their mesiobuccal cusps were standardized to a cone (diameter=5 mm, height=2 mm) with a diamond bur (Sintered diamond part #5014006OU; Brasseler, Savannah, GA, USA). The cusp tips were not abraded by the standardizing bur and therefore represent uncut enamel.

Wear Testing and Measurement

Wear testing was performed in the modified Alabama wear testing device. A vertical force of 10 N was applied by enamel antagonists followed by a 2-mm horizontal slide. The test was cycled at 0.66 Hz, stopping at 100,000 and 200,000 cycles. A 1:3 glycerine:distilled water solution continuously lubricated the specimens. Impressions of the enamel styli were taken with siloxane impression material (Imprint 3 Light Body; 3M ESPE) at baseline and at 200,000 and 400,000 cycles and poured with gypsum die stone (Silky-Rock; Whip Mix Corporation, Louisville, KY, USA). The stone models and wear specimens were scanned with a noncontact light profilometer (Proscan 2000; Scantron Industrial Products Ltd, Tauton, UK) at a 20 μ m \times 20 μ m resolution. The profilometer scans at baseline-100,000 cycles and baseline-200,000 cycles were superimposed (ProForm Software; Scantron Industrial Products Ltd) and aligned to measure the volumetric loss of enamel and ceramic. Representative samples were then examined by light microscop-

Table 1: Volumetric Wear of Enamel Antagonists and Ceramic/Enamel Substrates and Roughness of Pretest Substrates^a

	Volumetric Enamel Antagonist Wear, mm ³		Volumetric Ceramic/Enamel Substrate Wear, mm ³		Ceramic/Enamel Substrate Roughness (Ra), $\mu\text{m Ra}$
	100,000 Cycles	200,000 Cycles	100,000 Cycles	200,000 Cycles	Pretest
Polished zirconia	0.099 \pm 0.027 A	0.177 \pm 0.049 A	—	—	0.04 \pm 0.01 A
Aged zirconia	0.139 \pm 0.023 A	0.202 \pm 0.032 A	—	—	0.10 \pm 0.05 A
Veneering porcelain	0.359 \pm 0.053 c	0.512 \pm 0.051 c	0.28 \pm 0.05 B	0.36 \pm 0.06 B	0.35 \pm 0.05 A
Enamel	0.237 \pm 0.045 B	0.358 \pm 0.075 B	0.17 \pm 0.03 A	0.27 \pm 0.06 A	2.37 \pm 0.74 B

^a Similar letters represent statistically similar groups within each column.

py (VHX600; Keyence, Itasca, IL, USA) and scanning electron microscopy (SEM) (Quanta FEG 650; FEI, Hillsboro, OR, USA). Specimens were gold-palladium sputter-coated prior to SEM. Enamel specimens became desiccated during sputter-coating and therefore could not be imaged with SEM.

Roughness Measurement

Surface roughness (Ra) of all the specimens was determined using a noncontact light profilometer (Proscan 2000). As pretest surfaces were assumed to be homogeneous, an area in the middle of each specimen was selected for testing. A 0.7- μm length was measured with a 0.8-mm cutoff length and a 40 surface filter number selected for polished and aged zirconia and a 2.5-mm cutoff length and a 125 surface filter number selected for all other groups (based on ISO 4288-1996).

Statistical Analysis

A repeated-measures general linear model was used to determine significant differences between the paired groups of 100,000 and 200,000 cycles ($\alpha=0.05$) and the differences between material groups ($\alpha=0.05$). Post hoc analyses among group means were conducted using a Tukey test ($\alpha=0.05$).

RESULTS

The volumetric wear of the opposing enamel specimens, as well as the ceramic and enamel specimens, are both presented in Table 1. The samples and opposing enamel had significantly more wear after 200,000 cycles than after 100,000 cycles ($p<0.01$). The enamel opposing aged and nonaged zirconia showed statistically similar wear. The enamel-enamel group showed significantly more wear than both of the zirconia groups, but the greatest wear was seen on enamel opposing veneering porcelain ($p<0.05$).

At both 100,000 and 200,000 cycles, no detectable wear was measured on either the polished or aged zirconia. The veneering porcelain showed significantly more wear than the enamel control ($p<0.05$). Light micrographs of the ceramic and enamel specimens can be seen in Figure 1. No detectable scratches or cracks can be seen on the surface of the polished or aged zirconia samples. The veneering porcelain and enamel exhibited a rough wear scar.

SEM of the polished and aged zirconia showed relatively smooth surfaces of ceramic at an original magnification of 5000 \times (Figure 2) and only fine scratches from polishing could be observed. SEM images of the veneering porcelain showed sharp fractured edges of the porcelain in the wear scar (Figure 2). Additionally, fragments of the worn material could be seen on the specimen surface.

Roughness values are presented in Table 1. Both zirconia groups and the veneering porcelain had statistically similar roughness values. The enamel group was significantly rougher than all other groups ($p<0.05$).

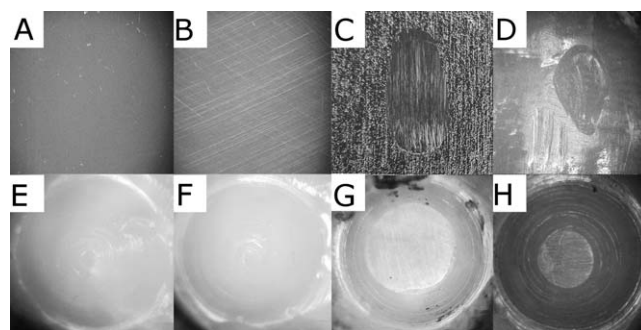


Figure 1. Light micrographs of (A) polished zirconia, (B) aged zirconia, (C) veneering porcelain, (D) incisor enamel, (E) enamel opposing polished zirconia, (F) enamel opposing aged zirconia, (G) enamel opposing veneering porcelain, and (H) enamel opposing enamel.

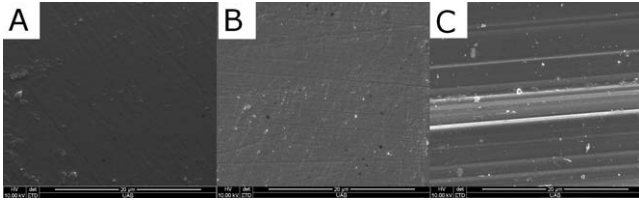


Figure 2. SEM micrographs of (A) polished zirconia, (B) aged zirconia, and (C) veneering porcelain.

DISCUSSION

Aged zirconia demonstrated statistically similar amounts of wear and produced similar amounts of opposing enamel wear when compared to nonaged, polished zirconia. Both zirconia groups showed less wear and opposing enamel wear than the control enamel and veneering porcelain groups. Therefore, the null hypothesis is rejected. Within the limitations of this study, the long-term wear of zirconia and its opposing enamel should be as clinically acceptable as that of veneering porcelain or natural enamel.

The three basic substrates in this study (veneering porcelain, zirconia, and enamel) all have different microstructures and methods of wear. The veneering porcelain used in this study, Lava Ceram (3M ESPE), is a feldspathic porcelain designed specifically for veneering zirconia frameworks. It is composed of a crystalline leucite phase surrounded by an amorphous glassy matrix.²⁰ Presumably, Lava Ceram would have a low concentration of leucite phase (5%-10%) to lower its coefficient of thermal expansion to match zirconia.²¹ This crystalline leucite phase contributes to its mechanism of wear in two ways. The hard leucite crystals are more abrasive than the surrounding glass and can more easily damage opposing enamel. The leucite crystals also strengthen the porcelain through dispersion strengthening and help prevent fracture.^{20,22} The porcelain material used in this study is predominantly composed of glass, and therefore it is susceptible to fracture. The SEM image of the veneering porcelain (Figure 2) shows the sharp edges of porcelain that have been fractured through the wear process. Fragments of wear debris are observed on the surface of the specimen. We theorize that the sharp fractured edges of the worn porcelain and the hard fragments of wear debris caused abrasive damage to the opposing tooth structure. The wear process may have been initiated by the initial roughness of the porcelain ($Ra=0.35\pm 0.05\ \mu\text{m}$). A previous study showed that hydroxyapatite (HA) wear increased by a 20-fold measure as a

counter-surface roughness was increased from 0.014 to $0.649\ \mu\text{m Ra}$.²³ Unlike veneering porcelain, zirconia is a polycrystalline ceramic with no glass content.²⁰ Its crystalline microstructure along with its transformation toughening ability make zirconia very resistant to fracture.⁹ The SEM image of polished zirconia (Figure 2) shows no signs of scratching or cracking after 200,000 cycles of wear. We theorize that the smooth surface of worn zirconia prevented abrasive wear of the opposing enamel. Aging zirconia roughens its surface through the growth of transforming monoclinic phases and the corresponding surface relief.^{19,24} This study is in agreement with other studies,^{24,25} that aging zirconia increases its surface roughness (although not by a statistically significant amount); however, no increase in opposing enamel wear was noted. The SEM image of the aged zirconia (Figure 2) demonstrates a similar surface smoothness to nonaged zirconia. A study by Liu and Xue²⁶ found that increasing the normal load applied to zirconia during sliding wear above 20 N altered the mechanism of wear from plastic deformation to microcracking. The shift in wear mechanism led to increased wear.

Enamel is composed of HA crystals embedded in an organic matrix. The orientation of these crystals divides enamel into structures known as rods.²⁷ Analysis of enamel subjected to sliding wear reveals that it fails by microcracking.²⁸ Pure fluorapatite (similar to HA) wears by brittle fracture.²⁹ The arrangement of HA into rod structures in enamel can hinder the propagation of cracks by redirecting them.²⁸ The light micrograph of the worn enamel demonstrates a rough wear scar. We speculate that wear of the contacting enamel surfaces was initiated by the relatively rough enamel surface ($Ra=2.37\pm 0.74\ \mu\text{m}$) and potentiated by fracturing of opposing enamel asperities.

Enamel is an anisotropic material, as its mechanical properties are dependent on the orientation of its rods.²⁸ In this study, the incisors used as enamel specimens for the enamel-enamel group were oriented with their facial surfaces in contact with the occlusal surface of the antagonist tooth. We chose this orientation with the assumption that enamel rods are aligned perpendicular to the surface of the tooth. A study by Fernandes and Chevitarrese,³⁰ however, indicated that rods are oriented at different directions in different parts of the tooth. Wear of tooth structure is also significantly increased when the layer of enamel is worn away to expose dentin.²⁸ The depth of the wear did not exceed 0.1 mm on any of the incisor specimens,

however, so we can assume that all wear occurred in the enamel.

The zirconia in this study was aged in an autoclave for five hours. Although there has not been a study to show the direct correlation between the time of aging *in vitro* and *in vivo*, a previous study¹⁸ has shown that autoclaving will transform surface tetragonal zirconia to its monoclinic phase and roughen its surface. Additionally, retrieval studies^{13,14} have shown that zirconia undergoes monoclinic transformation *in vivo* over time, leading to increased roughness. Therefore, autoclaving zirconia appears to be a reasonable method for simulating accelerated aging.

CONCLUSIONS

The results of this study are in agreement with those of earlier studies that found that zirconia causes less wear to opposing teeth and experiences less surface wear than enamel or a veneering porcelain.¹² Additionally, the results of this study indicate that age-related degradation of zirconia does not make this material more likely to induce or undergo wear. Future studies should examine wear of zirconia at forces beyond 20 N to determine if microcracking occurs.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 4 March 2013)

REFERENCES

- Guazzato M, Albakry M, Ringer SP, & Swain MV (2004) Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II: Zirconia-based dental ceramics *Dental Materials* **20(5)** 449-456.
- Garvie RC, Hannink RH, & Pascoe RT (1975) Ceramic steel? *Nature* **258(5537)** 703-704.
- Garvie RG, & Nicholson PS (1972) Structure and thermomechanical properties of partially stabilized zirconia in the CaO-ZrO₂ system *Journal of the American Ceramic Society* **55(5)** 152-157
- Kelly JR, & Denry I (2008) Stabilized zirconia as a structural ceramic: An overview *Dental Materials* **24(3)** 289-298.
- Evans AG, & Heuer AH (1980) Review—Transformation toughening in ceramics: Martensitic transformations in crack-tip stress fields *Journal of the American Ceramic Society* **63(5)** 241-248.
- Lughi V, & Sergio V (2010) Low temperature degradation—Aging—of zirconia: A critical review of the relevant aspects in dentistry *Dental Materials* **26(8)** 807-820.
- Fischer J, Stawarczyk B, Trottmann A, & Hammerle CH (2009) Impact of thermal misfit on shear strength of veneering ceramic/zirconia composites *Dental Materials* **25(4)** 419-423.
- Guess P, Bonfante EA, Coelho P, Ferencz JL, & Silva NR (2011) All ceramic systems: Laboratory and clinical performance *Dental Clinics of North America* **55(2)** 333-352.
- Pittayachawan P, McDonald A, Young A, & Knowles JC (2009) Flexural strength, fatigue life, and stress-induced phase transformation study of Y-TZP dental ceramic *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **88(2)** 366-377.
- Shijo Y, Shinya A, Gomi H, Lassila LV, Vallittu PK, & Shinya A (2009) Studies on mechanical strength, thermal expansion of layering porcelains to alumina and zirconia ceramic core materials *Dental Materials* **28(3)** 352-361.
- Preis V, Behr M, Kolbeck C, Hahnel S, Handel G, & Rosentritt M (2011) Wear performance of substructure ceramics and veneering porcelains *Dental Materials* **27(8)** 796-804.
- Rosentritt M, Preis V, Behr M, Hahnel S, Handel G, & Kolbeck C (2012) Two-body wear of dental porcelain and substructure oxide ceramics *Clinical Oral Investigations* **16(3)** 935-943.
- Fernández-Fairén M, Sala P, & Gil FJ (2006) Failures of yttria-stabilised tetragonal zirconia: 52 retrieved ceramic femoral heads of total hip prostheses *Biomedical Material Engineering* **16(6)** 415-422.
- Fernandez-Fairen M, Blanco A, Murcia A, Sevilla P, & Gil FJ (2007) Aging of retrieved zirconia femoral heads *Clinical Orthopaedics and Related Research* **462** 122-129.
- Deville S, Chevalier J, & Gremillard L (2006) Influence of surface finish and residual stresses on the ageing sensitivity of biomedical grade zirconia *Biomaterials* **27(10)** 2186-2192.
- Kraay MJ, Thomas RD, Rinnac CM, Fitzgerald SJ, & Goldberg VM (2006) Zirconia versus Co-Cr femoral heads in total hip arthroplasty: Early assessment of wear *Clinical Orthopaedics and Related Research* **453** 86-90.
- Papanagiotou HP, Morgano SM, Giordano RA, & Pober R (2006) In vitro evaluation of low- temperature aging effects and finishing procedures on the flexural strength and structural stability of Y-TZP dental ceramics *Journal of Prosthetic Dentistry* **96(3)** 154-164.
- Roy ME, Whiteside LA, Katerberg BJ, & Steiger JA (2007) Phase transformation, roughness, and microhardness of artificially aged yttria- and magnesia-stabilized zirconia femoral heads *Journal of Biomedical Materials Research Part A* **83(4)** 1096-1102.
- Deville S, & Chevalier J (2003) Martensitic relief observation by atomic force microscopy in yttria-stabilized zirconia *Journal of the American Ceramic Society* **86(12)** 2225-2227.
- Kelly JR, & Benetti P (2011) Ceramic materials in dentistry: Historical evolution and current practice *Australian Dental Journal* **56(Supplement 1)** 84-96.

21. Jamaluddin O, & Zainal AA (2004) Study on changing the coefficient of thermal expansion (CTE) of dental porcelain *Malaysian Journal of Medical Sciences* **59(Supplement B)** 143-144.
22. Oh WS, DeLong R, & Anusavice KJ (2002) Factors affecting enamel and ceramic wear: A literature review *Journal of Prosthetic Dentistry* **87(4)** 451-459.
23. Kalin M, Jahanmir S, & Ives LK (2002) Effect of counterface roughness on abrasive wear of hydroxyapatite *Wear* **252(9-10)** 679-685.
24. Denry IL, Peacock JJ, & Holloway JA (2010) Effect of heat treatment after accelerated aging on phase transformation in 3Y-TZP *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **93(1)** 236-243.
25. Chowdhury S, Vohra YK, Lemons JE, Ueno M, & Ikeda J (2007) Accelerating aging of zirconia femoral head implants: Change of surface structure and mechanical properties *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **81(2)** 486-492.
26. Liu H, & Xue Q (1996) Wear mechanisms of zirconia/steel reciprocating sliding couple under water lubrication *Wear* **201(1-2)** 51-57.
27. Boyde A (1967) The development of enamel structure *Journal of the Royal Society of Medicine* **60(9)** 923-928.
28. Zheng J, Zhou ZR, Zhang J, Li H, & Yu HY (2003) On the friction and wear behavior of human tooth enamel and dentin *Wear* **255(8)** 967-974.
29. Powers JM, & Craig G (1973) Wear of dental enamel *Wear* **23(2)** 141-152.
30. Fernandes CP, & Chevotarese O (1991) The orientation and direction of rods in dental enamel *Journal of Prosthetic Dentistry* **65(6)** 793-800.