EFFECT OF FULL-CONTOUR Y-TZP ZIRCONIA SURFACE ROUGHNESS ON WEAR OF GLASS-BASED CERAMICS

by

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DEDICATION

This thesis is dedicated to my beloved family, especially my parents, who have been great support, inspiration and motivation.

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TABLE OF CONTENTS

Introduction 1

Review of Literature.......... 5

Methods and Materials 18

Results.......... 24

Tables and Firgures.......... 28

Discussion 53

Summary and Conclusions 59

References 61

Abstract 67

Curriculum Vitae

LIST OF ILLUSTRATIONS

TABLE I Roughness summary……………………………………………...…… 29

TABLE II Summary of vertical loss and volume loss of tested materials…..…… 30

FIGURE 1 The full-contour Y-TZP ceramic slider (side view)…………..………. 31

FIGURE 2 The full-contour Y-TZP ceramic slider (top view)…………...……….. 31

FIGURE 3 Glass-based ceramic tabs…………………………………………..... 32

FIGURE 4 Non-contact optical profilometer……………………………………... 33

FIGURE 5 Ceramic tab embedded in brass holder……………………………...... 34

FIGURE 6 Schematic representation of roughness measurement on ceramic samples……………………………………………………...……. 34

FIGURE 7 Two-body pin-on-disc wear testing machine…………………..…...... 35

FIGURE 8 Schematic representation of the study design……………...……….. 36

FIGURE 9 2D vertical digital measurement device……………………….......... 37

FIGURE 10 Height loss measurement on Y-TZP sliders at baseline……..…...... 38

FIGURE 11 Height loss measurement on ceramic tabs………………………..... 39

FIGURE 12 Height loss measurement on ceramic tabs using Proscan software…… 39

FIGURE 13 Volume loss measurement on ceramic tabs…………………...…… 40

FIGURE 14 Volume loss measurement on ceramic tabs using Proscan software..… 40

FIGURE 15 3D surface topography by Proscan…………………………….…. 41

FIGURE 16 Wear track of IPS Empress CAD from Proscan…………….……... 42

FIGURE 17 Summary chart of roughness measurements……………………..….… 43

FIGURE 18 Representative SEM micrograph of as-machined Y-TZP slider (75 ×). 44

FIGURE 19 Representative SEM micrograph of as-machined Y-TZP slider at 500 × and 1,500 ×……………….. 44

FIGURE 20 Representative SEM micrograph of glazed Y-TZP slider (75 ×)………45

FIGURE 21 Representative SEM micrograph of glazed Y-TZP slider at 500 × and 1,500 ×……………………………………………………………… 45

FIGURE 22 Height loss summary chart for Zirconia sliders…………….………… 46

FIGURE 23 Height loss summary chart for ceramic antagonists………………..… 47

FIGURE 24 Volume loss summary chart for ceramic antagonists…………………… 48

FIGURE 25 SEM images of the worn surfaces of Empress at 75 ×………………... 49

FIGURE 26 SEM images of the worn surfaces of Empress at 500 × and 1,500 ×…. 50

FIGURE 27 SEM images of the worn surfaces of e.max at 75 ×…………………... 51

FIGURE 28 SEM images of the worn surfaces of e.max at 500 × and 1,500 ×……. 52

INTRODUCTION

The popularity of all-ceramic restorations has increased over the past two decades. Continuous patient demand for 'tooth-colored' restorations has taken esthetic dentistry to the next level. Ceramic materials are well known for their esthetic and biocompatible properties. Although they are very hard, they are also brittle in nature[1] and must be supported by a stronger framework to withstand functional occlusal loads, particularly in posterior regions. To meet the great demand for all-ceramic restorations, higher-strength and tougher ceramics are constantly being developed.[2]

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), hereafter zirconia, has been considered one of the strongest and toughest materials among the many commercially available dental ceramic systems. Unfortunately, current processing technologies cannot make zirconia as translucent as the natural tooth structure, nor does it allow internal shade characterization or facilitate customized shading.[3] Therefore, zirconia is commonly used to fabricate the cores or frameworks which are veneered with dental porcelains to achieve more favorable, close to natural tooth esthetics.

Two major concerns need to be considered when restoring posterior teeth. First, when the occlusal height of the tooth being restored is short, adequate occlusal tooth reduction to accommodate porcelain may not be possible. Second, the increased occlusal loading in patients with parafunctional activities (e.g., bruxism) often leads to fracture or chipping of the veneering ceramic.[4] Typically cast metal restorations are recommended in these clinical situations, however, all-zirconia restorations without veneering ceramic have recently been advertised as a tooth-colored option.[5-7]

Glass ceramic materials composed of leucite or lithium disilicate as basic crystalline structure have increasingly become more popular due to their improved physical, chemical and mechanical properties. They can be fabricated either by a pressable technique or Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) into full-contour restorations for inlays, onlays, veneers or even crowns and bridges. Situations where ceramic materials oppose each other intra-orally are not uncommon and ceramic restorations, especially zirconia, have been assumed to be abrasive to antagonistic teeth/restorations. Moreover, rough restoration surfaces were shown in the literature to influence the wear process.[8] In clinical situations, all-ceramic restorations that are glazed to decrease their surface roughness are routinely cemented with the glazed surface intact. Accordingly, material wear involving glazing materials becomes one of the concerns that will result in long-term stability or clinical failures of the restorations.

Studies on wear resulting from different surface treatments of zirconia ceramics against substrates other than human enamel are very minimal. The aims of this study were: (1) to investigate the effects of glazing on Y-TZP surface roughness, (2) to evaluate the influence of Y-TZP surface roughness (i.e., as-machined and glazed) on the wear of two distinct glass-based ceramic antagonists, namely leucite-based and lithium disilicate glass ceramics.

HYPOTHESES

The null hypotheses of this study were: (1) the surface roughness of Y-TZP ceramic would not be reduced due to the glaze application; (2) glass-ceramic wear would not be affected by zirconia surface roughness, and (3) the wear of glazed and as-machined zirconia against the two glass-based ceramics would not be distinguishable in spite of differences in physical, chemical and mechanical properties of the antagonist ceramics.

The alternative hypotheses of this study were: (1) the surface of Y-TZP ceramic would be significantly minimized due to the glaze application; (2) glass-ceramic wear would be affected by zirconia surface roughness, and (3) the wear of glazed and as-machined zirconia would be different when opposed by the two glass-based ceramics.

REVIEW OF LITERATURE

GLASS-BASED CERAMICS FOR FULL-CONTOUR RESTORATIONS

A variety of materials for the fabrication of full-contour single-unit all-ceramic restorations (e.g., inlays, onlays, crowns, etc) have been introduced to meet the increased demand for all-ceramic restorations. Glass-based ceramics containing an amorphous glassy phase and a wide variety of crystalline constituents as reinforcement (e.g., lithium disilicate, leucite, among others) are mostly suited for these situations. The interaction of the crystals and glassy matrix along with crystal size and amount is responsible for improved mechanical and physical properties such as fracture resistance and thermal-shock resistance.[9] Heat-pressing and CAD/CAM techniques are two of the most popular methods employed in the fabrication of reliable and predictable restorations in terms of strength, marginal fit and esthetics.[2] In addition, conditioning with 9.5% hydrofluoric acid selectively removing part of the glassy phase, followed by the application of silane and an adhesive luting agent helps enhancing the strength of the tooth-restoration complex providing favorable function and longevity.[10] A brief description of the most commonly used and researched systems, including the ones selected for the current study is presented below.

A castable glass-ceramic, named Dicor MGC, comprised of tetrasilicic fluoromica ($K_2Mg_5Si_8O_{20}F_4$) to provide increased fracture resistance and strength was one of the earliest glass-ceramic systems.[9] High translucency and simple lost-wax casting technique were two of the major advantages of this ceramic system. However, its relatively low flexural strength (~120 MPa) and the development of better injection mold of other systems decreased the popularity of its use significantly. An 8-year clinical study by Pallesen et al[11] reported bulk as well as chip fractures as the primary type of failure for Dicor restorations.

Vitablocs Mark II (Vident, Germany) is a feldspar-based full-contour ceramic with fine grain sizes manufactured into blocks specifically for CAD/CAM processing. The machinable blocks were reported to have good clinical outcomes in restoring endodontically-treated molars over 2 years.[12] Nevertheless, low flexural strength (~95 MPa) makes them a less suitable candidate for full-contour restorations especially in posterior areas.[13]

Leucite-reinforced glass ceramics

The next system incorporates leucite as its reinforcing agent. Leucite-reinforced glass ceramics include products such as IPS Empress (Ivoclar Vivadent, Amherst, NY) and Optimal Pressable Ceramic (OPC, Jeneric Pentron, Wallingford, CT). For IPS Empress system, pressable (IPS Empress Esthetic) and CAD/CAM (IPS Empress CAD) versions are available. Leucite crystals of a few microns (1.5-2.6 μm)[14-16] grow evenly in a multi-stage process directly from the amorphous glass phase. Like IPS Empress, OPC consists of needle-like leucite crystals (1.9-6.6 μm)[17] that inhibit crack propagation resulting in twice the strength of the previous generation of pressable ceramics. According to the manufacturer, the flexural strength of IPS Empress can go up to 160 MPa when the recommendations are adequately followed. This allows a wider range of use for these materials compared with the aforementioned systems. In a comparative study between IPS Empress and OPC, Gorman and colleagues found that OPC presented higher (153.60 MPa) biaxial flexural strength when compared to Empress (134.40 MPa).[18] In contrast, Cattell et al[17] in 1999 did not find any significant difference between unshaded Empress, unshaded OPC and shaded OPC specimens ranging from 135.8-139.1 MPa. Microstructural investigations conducted by Gordon et al revealed larger glassy areas with no changes in leucite crystalline structure and amount when analyzed before and after heat-treatment for Empress. In contrast, the microstructure of OPC material was converted to a glass-ceramic after processing.[18] According to the authors, it may be assumed that Empress might reach its maximum crystallinity prior to processing.

However, improving physical and mechanical properties can lead to materials that are more abrasive to the opposing teeth and restorations.[19] An *in vitro* study by Krejci et al[20] in 1993 reported on the wear characteristics of ceramic inlays against enamel cusps. Three different ceramic materials were investigated Dicor (castable glass ceramic), Biodent (feldspathic porcelain) and IPS Empress (pressable ceramic). Ceramics surface were polished or glazed. A computer-controlled six-chambered chewing simulator in combination with a toothbrush machine and chemical degradation were used to carry out the wear experiments. The test simulated 5 years *in vivo* function. They found IPS Empress to be significantly less abrasive to the antagonist enamel than castable ceramics and feldspathic porcelain. Furthermore, the material wear of polished IPS Empress yielded the lowest values among all four groups, followed by glazed IPS Empress without statistical significance. Indeed, SEM images indicated a rougher surface for glazed group when compared with the polished group. Likewise, Imai et al[21] found Empress to be the least abrasive to opposing flattened enamel among other ceramics, including Finesse, Softspar and Ceramco II. This study was carried out using a UAB wear testing machine with and without polymethyl methacrylate beads to act as the third body. The machine was run in multiple cycles up to 50,000 cycles at 1.2 Hz under a maximum load of 75.6 N. Replicas in combination with a profilometer were used for wear measurements. Interestingly, they also concluded that different properties of glazing materials could have played a role in the wear behavior of underlying materials.

Elmaria et al[22] performed an *in vitro* study to evaluate the influence of two surface treatments (i.e., glazed and polished) on the wear characteristics of three ceramic substrates (Finesse, All-Ceram and IPS Empress) and type III gold alloy (control) against human molar cusps (n=10). All specimens were placed in a custom-constructed wear machine with a 6 mm track length prior to reversal of direction. The cusp tips were positioned above the restorative specimens under a constant load (180 g) and tap water immersion for 10,000 cycles. Profile tracing by a profile projector was used to measure the height loss of the cusp tips. In contrast to the previously mentioned studies, IPS Empress was found to be the most abrasive among all tested materials against enamel antagonists (with glazed IPS Empress demonstrating the highest values with statistically significant difference). Finesse and All-Ceram yielded comparable enamel height loss to gold substrate. Roughness average parameter (Ra) was found to have a significant correlation with enamel height loss along with substrate properties such as elastic modulus, surface hardness, grain size and polishing method. These findings were in agreement with a study by Ramp et al[19] when using cone-shaped styli of Dicor, Vita Mark II Block, IPS Empress ceramics and type III gold alloy (Midas) on a two-body UAB wear testing machine. The specimens run for 100,000 cycles at 1.2 Hz under a load of 75 N. The enamel wear facet depths were measured using mechanical profilometry while profile subtraction was used to measure the stylus height loss. They reported IPS Empress and Vita Mark II to be significantly more abrasive to flattened enamel surface than Dicor and Midas. In summary, they suggested that the leucite content in both IPS Empress and Vita Mark II could have contributed to the significantly greater enamel wear.

Lithium disilicate glass ceramics

In order to have stronger ceramics that can withstand posterior forces but still maintain the good properties of glass ceramics i.e. good esthetics and biocompatible, lithium disilicate glass ceramics were introduced under the name IPS Empress 2 and more recently as IPS e.max (Ivoclar Vivadent, Amherst, NY). IPS e.max Press ingots are available for pressing and machinable IPS e.max CAD blocks are designed specifically for CAD/CAM technology. In contrast to IPS Empress, partially-sintered IPS e.max ceramic blocks require heat-treatment to complete their crystallization resulting in needle-like lithium disilicate crystals (3 to 6 μm in length, mean diameter 0.8 μm)[16] to grow up to 70% within the glassy matrix in a controlled manner.[23] However, no shrinking needs to be accounted for because of low thermal expansion during processing in the same manner as IPS Empress. Comparing with the previous systems, higher biaxial flexural strength at 440 MPa for IPS e.max Press[24] and 416.1 MPa for e.max CAD[25] were reported in laboratory studies. Accordingly, IPS e.max offers a variety of restorative indications including posterior fixed partial dentures due to the improved mechanical properties.[23] An eight-year clinical study by Wolfart et al[26] showed good outcomes at 93% for three-unit lithium-disilicate bridges placed on both anterior and posterior regions. Likewise, another study by Silva et al[27] revealed 100% success rate for monolithic lithium disilicate glass ceramic restorations (IPS e.max Press) over a four-year period. Interestingly, it was also found IPS e.max Press to have good wear resistance and comparable abrasiveness to veneering ceramic materials such as IPS d.SIGN and IPS Eris for E2 against antagonistic enamel.[27]

Esquivel-Upshaw et al[24] performed an *in vivo* study on thirty IPS e.max Press fixed partial dentures (FPDs) placed on posterior teeth. At the one-year recall, the FPDs were evaluated using eleven clinical criteria such as tissue health, secondary caries, occlusion, etc. The wear measurement was carried out on the white gypsum casts comparing baseline and after-test data using a 3D laser scanner. The results suggested that the wear rate of enamel opposing IPS e.max Press (mean occlusal wear was 88.4 μm, ranging from 29-255 μm) was higher than the measured mature enamel wear rate (38 μm) over a one-year period.[28]

FULL-CONTOUR ZIRCONIA

Strength improvements of glass ceramics are limited due to the presence of a usually weak glassy matrix. Crack propagation through the glassy matrix caused by applied stress can lead to restoration failure overtime.[10] For this reason, materials that completely eliminate the glassy phase by directly sintering the crystals together have been developed. The most recent introduction to dental ceramics is zirconia or zirconium oxide. Without a glassy phase, zirconia utilizes a transformation toughening mechanism to enhance its strength and toughness and makes zirconia the toughest ceramic core material currently available.[29] This particular process involves the addition of stabilizing oxides such as magnesia, ceria, yttria and calcia to retain the tetragonal phase at room temperature. The increase in volume of approximately 4% generates compressive strength that limits crack propagation.[30, 31] The most popular form of zirconia ceramics is 3 mol% yttria-containing tetragonal zirconia polycrystalline (Y-TZP)[31] with flexural strength values ranging from 900 to 1200 MPa[32] and fracture toughness between 8 to 10 $\mathrm{MPa \cdot m^{1/2}}$.[33] Fracture toughness is a very important material property that denotes a material resistance to crack propagation.

Two CAD/CAM techniques available for material fabrication are soft milling and hard milling. The first involves milling partially-sintered Y-TZP blocks that are enlarged approximately 25% to compensate for sintering shrinkage while the latter technique mills fully-sintered zirconia blocks directly to the desired dimensions.[30]

Zirconia ceramics are suitable for use as frameworks for crowns and fixed dental prostheses on posterior teeth.[9, 10, 34] Generally, they are veneered with a veneering ceramic to mask their opaque nature.[34] However, fracture within the veneering ceramic materials has been reported as the most frequent failure for zirconia-based all-ceramic restorations and fixed partial prostheses.[27, 30, 35] In addition, full-contour zirconia restorations have recently been advocated in situations with insufficient occlusocervical space.[5-7] Without veneering ceramics on top, these restorations are expected to be able to withstand high occlusal load in patients with parafunctional activities. Despite a high influx of advertisements by many manufacturers promoting full-contour zirconia restorations, very few studies have been reported on this topic.[34-36] Recently, several clinical cases using full-contour zirconia crowns and fixed dental prostheses on posterior teeth were shown to function well clinically.[37] However, only acceptable, but not optimal, esthetics were achieved.

GLAZE

It has been demonstrated by numerous research studies that unglazed porcelain or improper ceramic surface polishing can cause a high rate of wear of antagonist teeth/restorations.[8, 38] Jagger and Harrison[8] suggested that rough restoration surfaces result in greater wear of antagonistic materials.[8] Surface glazing was introduced as a solution to these problems.

Dental glazes consist of colorless glass powder that provides a glossy surface on fired dental porcelain.[39] Porcelain glazing is aimed at sealing the porosities throughout the surface of sintered restorations. In addition, glazed porcelain can also mimic the characterization and surface luster of the natural tooth surface.[40] Aksoy et al[39] showed that glazed porcelain provides the smoothest surface and greatest wettability compared with other surface treatments on dental porcelain. Another benefit is less plaque retention on the surface of the restorations. Al-Wahadni and Martin[40] reported that glazed porcelain provides a smooth and dense surface making it preferred in the clinical settings. In contrast, Jagger and Harrison[8] found that glazing of Vitadur porcelain did not reduce enamel wear rates compared to unglazed Vitadur porcelain.

WEAR

Wear occurs when two materials slide against each other. Wear can be attributed as adhesive, abrasive, corrosive and fatigue wear.[41] For ceramic materials, the most occurring form of wear intra-orally is abrasive wear, with less incidents of adhesive and fatigue wear.[22, 36, 42, 43] Additionally, surface roughness, fracture resistance and surface treatments tend to determine wear characteristics rather than hardness alone.[44] Imai et al[21] suggested that the size, shape or quantity of the crystal phase on the material surface could also influence the wear behavior of ceramic restorations. Abrasive wear can be categorized as two-body and three-body wear. The first type is when two materials are in contact with each other without other substances in between like in clenching or bruxing. Three-body wear occurs when there are other substances involved such as food bolus in chewing.

Zirconia ceramics are known to possess high strength, and toughness; however, its abrasive characteristics[38] are not well-defined. It has been assumed that zirconia may cause significant abrasive wear of opposing human enamel. Since tooth wear is a multi-factorial process, several aspects regarding wear have been investigated.

Jung et al[35] compared the wear of human enamel premolar cusps against either a recently launched more transparent and directly stainable full-contour zirconia (Zirkonzahn prettau®) or feldspathic porcelain (Vita Omega 900®) using two-body wear. The tested materials were divided into three groups and included polished feldspathic porcelain, polished zirconia and polished/glazed zirconia. A dual-axis chewing simulator was used to perform the wear testing under a 5 kg load for 240,000 cycles simulating one year of chewing. Before and after volume loss of the opposing teeth was measured and calculated. The results revealed that the polished zirconia group caused the least antagonistic tooth wear among the three groups followed by the glazed zirconia with no statistical significance. Feldspathic porcelain was proven the most abrasive with significant differences when compared with polished zirconia. These findings were in agreement with a study done by Geis-Gerstorfer[6] who compared Bruxzir® full-contour zirconia with Ceramco 3 feldspathic porcelain against steatite balls of 6 mm in diameter. The study was performed using a two-body pin-on-disk apparatus that consisted of

1,200,000 cycles under a vertical load of 50 N and a horizontal movement of 0.2 mm. The depth of wear track of the tested materials and the height loss of the antagonists were measured with the use of a 3D profilometer. The Ceramco 3 group was shown to have more wear values on both of the tested material specimens and the antagonists when compared with Bruxzir group.

Similarly, Preis et al[45] investigated the two-body wear of five different zirconia ceramics and four veneering porcelains when opposing enamel cusps and steatite balls. Tooth enamel and Vita Omega 900, which is a veneering ceramic for metal-ceramic restorations, were used as controls. One of the zirconia groups (Zeno Zr Bridge zirconia system) was fabricated without any veneering ceramic. All specimens were placed in the pin-on-block wear testing machine with a vertical load of 50 N for 120,000 cycles at a frequency of 1.6 Hz. Thermocycling for 600 cycles was also performed during the wear test. The vertical substance loss of the antagonists was measured using 3D profilometry. Zirconia specimens promoted comparable wear to steatite and enamel. They were also demonstrated to be significantly less abrasive to both mentioned antagonists than veneering ceramics. The same trend concerning unveneered zirconia was found in a similar study[46] which included glass-infiltrated and lithium disilicate ceramics. It was demonstrated in this study that zirconia and glass-infiltrated groups caused comparable steatite antagonist wear to the enamel reference. On the other hand, veneering ceramics and lithium disilicate glass provided significantly higher antagonist wear values. Regarding these two studies, they concluded that unveneered zirconia may be used for the fabrication of FPDs with clinically acceptable wear characteristics.[45, 46]

As for different types of antagonist materials, Albashaireh et al[34] performed a two-body wear testing on five different ceramic materials against zirconia balls. The rationale of the study was to replicate situations where unveneered zirconia is used against all-ceramic restorations. Yttrium-stabilized zirconia (IPS e.max ZirCAD), lithium disilicate glass ceramic (IPS e.max Press), leucite-reinforced glass ceramic (IPS Empress Esthetic) and two veneering ceramics, namely a fluorapatite glass ceramic (IPS e.max ZirPress) and a nanofluorapatite glass ceramic (IPS e.max Ceram) were tested. All specimens were polished and loaded in a dual-axis chewing simulator for 300,000 cycles under a load of 5 kg. Both vertical and volumetric ceramic substance loss were measured with a laser scanner. Overall, yttrium-stabilized zirconia demonstrated the least material loss, followed by the two pressable glass ceramics (i.e., IPS e.max Press and IPS Empress Esthetic) without significant differences and the two veneering ceramics (IPS e.max ZirPress and IPS e.max ceram). Based on the findings, the authors suggested that the differences in the substance loss of ceramic materials may have resulted from their microstructure and the physical characteristics, specifically flexural strength and toughness.

On the subject of surface treatments and roughness, Ghazal and Kern[36] in 2009 investigated the wear characteristics of different surface roughness of zirconia balls on human enamel and nanocomposite resin by performing two-body wear in a chewing simulator. The results revealed that increased zirconia surface roughness significantly increased the wear of both testing materials. This was also confirmed by Jung et al[35] that the polished zirconia ceramics contributed to less antagonistic tooth wear than the glazed group. One of the reasons for these findings was explained by Ghazal and Kern[36] that greater wear was resulted from the increased friction coefficient of higher surface roughness.

Based on the current literature, much interest has focused on developing ceramic materials for full-contour restorations.[18, 27, 33, 47] Wear is one of the main issues regarding these materials that have been widely discussed. Accordingly, the present study was conducted to assess the wear behavior of zirconia with different surface roughness against two distinct machinable glass-based ceramics.

METHODS AND MATERIALS

EXPERIMENTAL DESIGN

Thirty-two zirconia sliders were randomly allocated into two groups (n=16) according to their surface treatments. The first group was left as as-machined while the other was glazed. Eight zirconia specimens from each group were tested by means of two-body wear against two different glass-based ceramic (n=8) antagonists for 25,000 cycles at 1.2 Hz under a 3 kg load. Surface roughness values were measured using R_a and R_q roughness parameters (µm) by 3D non-contact optical profilometer. Before and after-test zirconia slider height measurements were taken to compare the slider height loss (µm) whereas only after-test vertical height loss (µm) and volume loss (mm^3) were measured for ceramic antagonists. Additionally, scanning electron microscopy (SEM) was used to evaluate the morphological features of glazing influence on zirconia surfaces as well as wear topography of ceramic antagonists. Comparisons between groups for differences in surface roughness were performed using one-way ANOVA. The effects of zirconia slider surface treatment and ceramic antagonist on antagonist height loss, antagonist volume, and slider height loss were performed using two-way ANOVA.

PREPARATION OF FULL-CONTOUR ZIRCONIA (Y-TZP) SPECIMENS

Thirty-two partially-sintered yttrium-stabilized tetragonal zirconia polycrystal ceramic blocks for CAD/CAM technique (Y-TZP, Ardent, New York, USA) were machined (CEREC® inLab MC XL, Sirona, Charlotte, NC, USA) into a predetermined geometry, that is 2 mm diameter cylindrical shape and 1.5 mm in height, hereafter named zirconia sliders, for the two-body pin-on-disc wear testing[48] (Figure1). A chamfer measuring 0.25 mm in width was placed circumferentially around the end of the slider reducing the testing surface to 1.5 mm in diameter. The base portion was 6 mm in diameter. The dimensional shrinkage of the specimens after sintering (20-25%) was compensated using CAD software calculation before milling. Each specimen was sintered in a high-temperature furnace (Programat® S1, Ivoclar Vivadent, Amherst, NY USA) according to the manufacturer's instructions. Next, the zirconia sliders were randomly allocated into two groups (n=16) as follows: G1: as-machined and G2: glazed. The rationale for testing glazed zirconia is based on the clinical situation where a zirconia restoration is cemented with the glazed surface intact. Diazir FCZ stain and glaze paste (Ardent, New York, USA) was applied to the zirconia surface and then fired in a furnace (Programat CS, Ivoclar Vivadent, Amherst, NY USA) according to the manufacturer's instructions. The average thickness measurement of the glaze layer using a 2D vertical measuring device for all samples revealed the approximate thickness of 8 μm. As-machined sliders were included as a control. Zirconia sliders from both groups were then embedded in auto-polymerizing acrylic resin (Bosworth Fastray, Harry J. Bosworth Co, IL USA), which was mixed and poured in custom-made brass holders. A dental surveyor was utilized to ensure that the specimens were mounted in the proper orientation. The flat surface of the specimen was affixed with a thin layer of blue wax (Inlay Wax Hard Blue; Henry Schein, Inc, Melville, NY) to the flat end of the surveyor rod, and the rod gradually lowered until the specimen base was embedded in the resin of the brass holder. The surveyor rod was maintained in this position until the resin was polymerized to ensure that the surface of the specimen remained 1.5 mm above the resin.[49]

Roughness parameters R_a (i.e., the arithmetic average of the absolute values of the roughness profile ordinates) and R_q (i.e., the square root of the average of the square of the deviation of the scan from the mean line) values were generated for each specimen after surface digitization and the subsequent image analysis. Mean values for both R_a and R_q were obtained and then associated with each experimental group. Each scanning area was limited to 0.6 x 0.6 mm using S5/03 sensor at 10 µm step size in both x and y directions. Meanwhile, one additional specimen per group was fabricated, sputter-coated with gold and evaluated at different magnifications under a scanning electron microscope (SEM) to obtain representative qualitative images of the Y-TZP surfaces.

PREPARATION OF GLASS-BASED CERAMIC ANTAGONIST SPECIMENS

Two glass-based ceramic blocks (IPS Empress CAD and IPS e.max CAD, Ivoclar Vivadent, Amherst, NY, USA) were cut into rectangular-shaped specimens (13 × 13 × 2 mm, N=32), according to figure 3, using a slow speed cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA) under water lubrication. Then the specimens were wet-finished using silicon carbide paper, 600-, 1200- and 2400-grit respectively and cleaned for 10 minutes in an ultrasonic bath with distilled water. IPS e.max CAD samples were sintered in a furnace (Ney CeramPress Q50, Dentsply International, USA) according to the manufacturer's instructions for final crystallization. Finally, all specimens were mounted in brass holders (figure 5) with the testing surface perpendicular to the long axis of the brass ring[49] using a customized silicone mold. Specimens were wet-finished with 1200- and 2400-grit silicon carbide paper to ensure the flat and smooth surface. Mean surface roughness values in R_a and R_q (µm) were measured using a non-contact profilometer (Proscan 2000, Scantron, Taunton, England) in four different areas assumed to be inside the wear track for each sample (figure 6). Each scanning area was limited to 1 × 1 mm using the S5/03 sensor at 10 μm step size in both *x* and *y* directions prior to wear testing.

WEAR TESTING

A two-body pin-on-disc wear testing machine (Dental Biomaterials Laboratory, Indiana University School of Dentistry, Indianapolis, IN) was used to simulate occlusal contact wear (figure 7). Zirconia sliders were mounted in the lower stations to run in a circular motion against fixed ceramic discs in the upper stations for 25,000 cycles at 1.2 Hz with distilled water running to remove wear debris under a 3 Kg load.[48] After test completion, the specimens were removed and cleaned in an ultrasonic water bath for 10 minutes.

The wear testing details are presented in Figure 8. Briefly, two groups of Y-TZP specimens were tested against the different glass-based ceramic materials. Eight samples (N=8) were evaluated per test condition.

WEAR MEASUREMENTS

Vertical substance loss was used to determine quantitative wear data of the zirconia sliders. Baseline and after-test height data were recorded using a 2D vertical digital measurement device (figure 9) in four different areas according to figure 10. For glass-ceramic tabs, surface wear was calculated by measuring vertical and volume loss using non-contact optical profilometry (Proscan 2000, Scantron, Taunton, England).[49]

The S38/3 sensor was used for scanning at 50 μm step size in both *x* and *y* directions. For height loss measurement, eight different areas along the wear track were measured by comparing with the central area of the ceramic tab outside the wear track (Figure 11 and 12). Three different spots were measured on each ceramic tab to achieve the volume loss profiles that comprised two planes in *x* and *y* axes (figure 13 and 14). Moreover, scanning electron microscopy (JEOL JSM-5310LV, Jeol Ltd, Tokyo, Japan) at different magnifications was performed to acquire additional qualitative data on wear characteristics of both glass-ceramics antagonists.

STATISTICAL METHODS

Comparisons between groups for differences in surface roughness were performed using one-way ANOVA. The effects of zirconia slider surface treatment and ceramic antagonist on antagonist height loss, antagonist volume, and slider height loss were performed using two-way ANOVA. Analyses were performed after a natural logarithm transformation of the data to satisfy the assumptions required for the ANOVAs.

SAMPLE SIZE JUSTIFICATION

In two previous studies,[34, 36] the standard deviation for wear depth was 4 μm. With eight (8) samples per polishing technique / specimen (glass-based ceramics) combination, the study had an 80% power to detect a wear depth difference of 6.1 μm between any two groups, assuming two-sided tests each conducted at a 5% significance level.

RESULTS

The representative images of the 3D surface topography obtained using Proscan2000 software of all tested groups are depicted in figure 15. Means and respective standard deviations (± SD) for surface roughness are presented in figure 17 and table I. The as-machined zirconia sliders showed the highest mean surface roughness of 0.83 ± 0.11 μm for R_a and 1.09 ± 0.15 for R_q. By contrast, e.max presented the lowest mean surface roughness of 0.17 ± 0.02 μm for R_a and 0.21 ± 0.02 μm for R_q. When comparing the two different surface treatments of zirconia sliders, one-way ANOVA revealed that the as-machined group yielded significantly higher mean surface roughness than the glazed group ($p=0.0001$ for R_a, $p=0.0018$ for R_q). Furthermore, Empress (0.20 ± 0.01 μm for R_a and 0.25 ± 0.01 μm for R_q) had significantly higher surface roughness than e.max (0.17 ± 0.02 μm for R_a and 0.21 ± 0.02 μm for Rq) at $p=0.0141$ for R_a and $p=0.0039$ for R_q. However, the differences between mean surface roughness values of the two ceramics are in a much closer range than the differences between the two zirconia groups. Qualitative SEM images (figures 18-21) suggested that the surface topography of the glazed zirconia slider was smoother than the as-machined group.

The statistical values of mean vertical substance loss and mean volume loss for all tested groups after 25,000 cycles are shown in figure 22 and table II. For the comparison between the mean vertical loss of two zirconia groups, Empress promoted significantly more slider height loss than e.max for as-machined zirconia sliders ($p<0.0001$) but there was no significant difference in slider height loss between Empress and e.max antagonists for glazed zirconia sliders ($p=0.95$). Moreover, as-machined zirconia sliders demonstrated significantly less mean slider height loss than glazed zirconia sliders ($p<0.0001$).

For wear analysis of ceramic antagonists, table II presents both mean height loss and mean volume loss of Empress ($p<0.0001$). The results were significantly less than e.max ($p<0.0001$). As-machined zirconia sliders caused significantly less antagonist height loss ($p=0.0092$) and antagonist volume loss ($p=0.0109$) than glazed zirconia sliders for Empress antagonists. By contrast, no significant differences were found in antagonist height loss ($p=0.97$) or antagonist volume loss ($p=0.79$) for e.max between as-machined and glazed zirconia groups. The summary charts are shown in figures 23 and 24. In addition, zirconia slider surface glazing did not have a significant effect on either antagonist height loss ($p=0.10$) or antagonist volume loss ($p=0.06$).

SEM images of the worn surface area of the tested ceramic antagonists at different magnifications are presented in figures 25-28. At lower magnification (75×, figure 25) Empress ceramic worn surface when opposing as-machined zirconia sliders appears to be relatively smooth; while distinct irregularities can be observed as a pattern all across the worn area of Empress antagonist against glazed zirconia sliders. More cracks and flaws indicating chipping of the materials can also be detected on the surface of Empress antagonists opposing glazed zirconia at higher magnifications images when compared with the same antagonist group against as-machined zirconia sliders (Figure 26). For e.max antagonists; however, the wear characteristics are shown to be somewhat homogenous as well as less surface irregularities (75×) with no particular differences in the wear pattern of the two e.max groups opposing as-machined as well as glazed-zirconia groups (figure 27). Similar to the group against as-machined zirconia, e.max antagonists against glazed zirconia sliders showed continuous wear grooves along the wear track following the slider movements. Equally important, material fragments were also detected at higher magnifications on the surface of e.max antagonist worn surface tested against the glazed zirconia group (Figure 28).

TABLES AND FIGURES

TABLE I

Roughness summary

Method	Type	Group	N	Mean	SD	SE	Min	Max
Ra (µm)	Ceramic Antagonist	Empress	6	0.20^{A}	0.01	0.01	0.18	0.21
		e.max	6	0.17^{B}	0.02	0.01	0.15	0.20
	Zirconia Slider	As-machined	6	0.83^{C}	0.11	0.04	0.63	0.92
		Glazed	6	0.53^{D}	0.06	0.02	0.47	0.62
Rq (µm)	Ceramic Antagonist	Empress	6	0.25^{I}	0.01	0.01	0.24	0.26
		e.max	6	0.21^{II}	0.02	0.01	0.18	0.25
	Zirconia Slider	As-machined	6	1.09^{III}	0.15	0.06	0.83	1.24
		Glazed	6	0.78^{IV}	0.10	0.04	0.68	0.93

Groups with the same superscript letter/number were not significantly different.

TABLE II

Summary of vertical substance loss (in μm) and volume loss (in mm^3) of the tested materials after 25,000 cycles of two-body wear testing

	Zirconia Slider	Ceramic Antagonist	N	Mean	SD	SE	Min	Max
Antagonist Height Loss (μm)	As-machined	Empress	8	68.4^A	9.4	3.3	46.9	78.8
	As-machined	e.max	8	146.1^B	12.9	4.6	130.0	160.7
	Glazed	Empress	8	84.9^C	18.0	6.4	63.0	112.8
	Glazed	e.max	8	146.6^A	14.8	5.2	131.2	169.4
Antagonist Volume Loss (mm^3)	As-machined	Empress	8	7.6^A	1.3	0.5	5.7	9.5
	As-machined	e.max	8	15.5^B	1.2	0.4	14.3	18.2
	Glazed	Empress	8	9.9^C	2.9	1.0	6.3	15.7
	Glazed	e.max	8	16.0^B	2.2	0.8	13.6	20.6
Slider Height Loss (μm)	As-machined	Empress	8	30.0^A	5.8	2.0	21.0	36.0
	As-machined	e.max	8	17.4^B	5.8	2.0	11.0	30.0
	Glazed	Empress	8	42.6^C	8.3	2.9	31.0	52.0
	Glazed	e.max	8	42.9^C	8.0	2.8	30.0	54.0

Groups with the same superscript letter within each height/volume loss component were not significantly different.

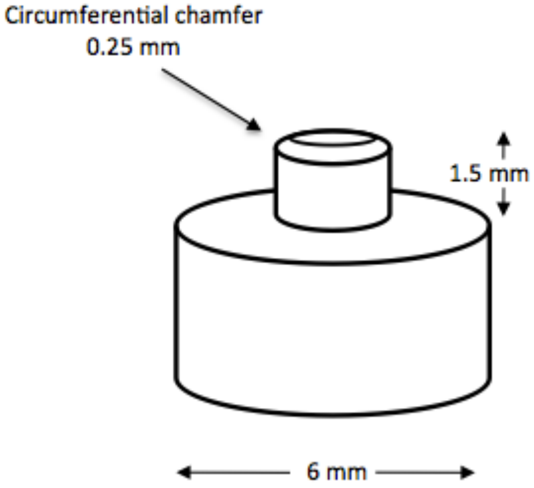


FIGURE 1. Schematic of the full-contour Y-TZP ceramic slider machined using a CAD/CAM milling unit following the predetermined geometrical dimensions (side view).

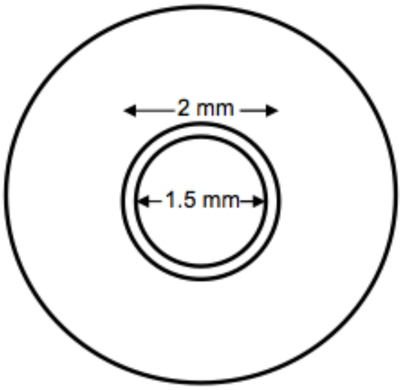


FIGURE 2. Schematic of the full-contour Y-TZP ceramic slider machined using a CAD/CAM milling unit following the predetermined geometrical dimensions (Top view).

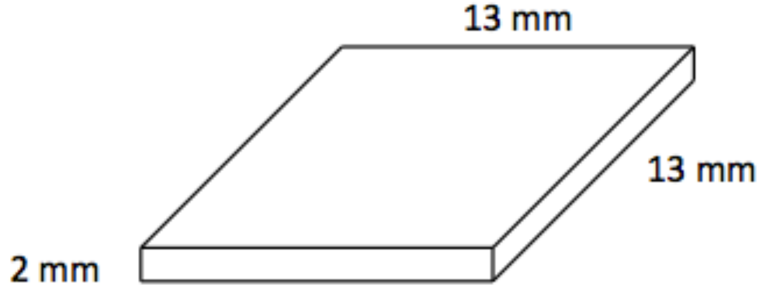


FIGURE 3. Schematic of the glass-based ceramics tabs to be used as antagonists against the Y-TZP slider in the two-body wear testing.

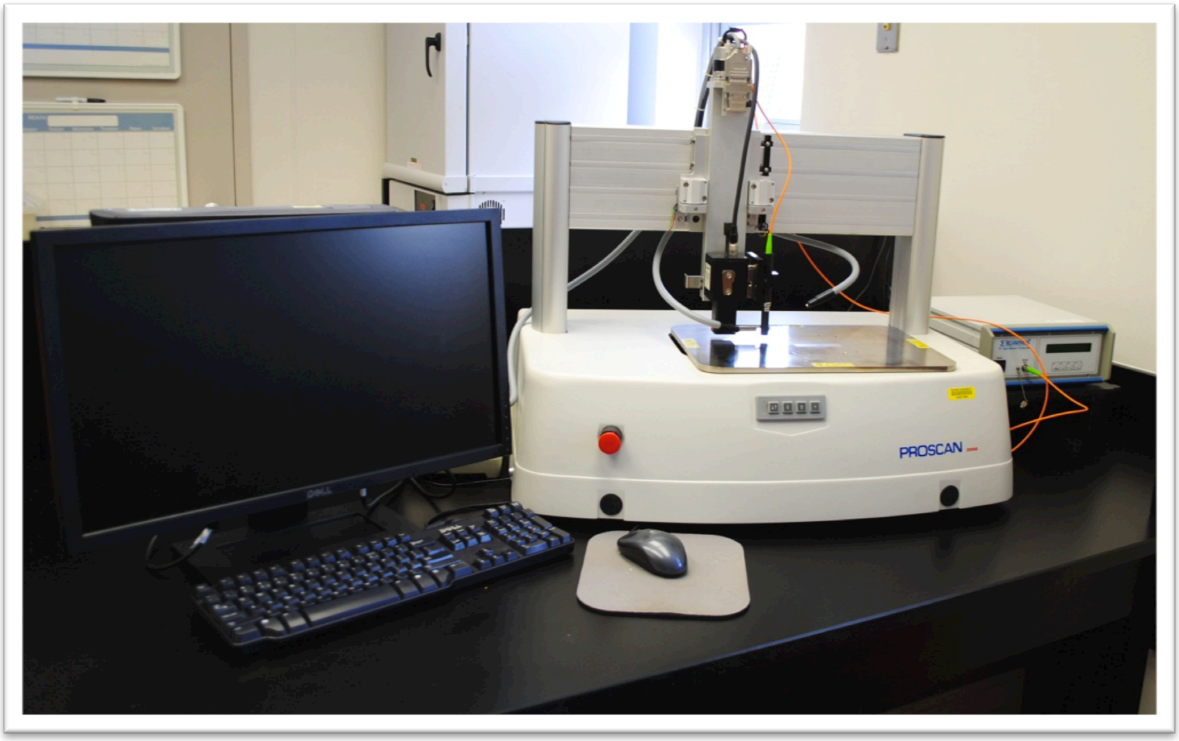


FIGURE 4. Non-contact optical profilometer (Proscan 2000, Scantron, Taunton, England).

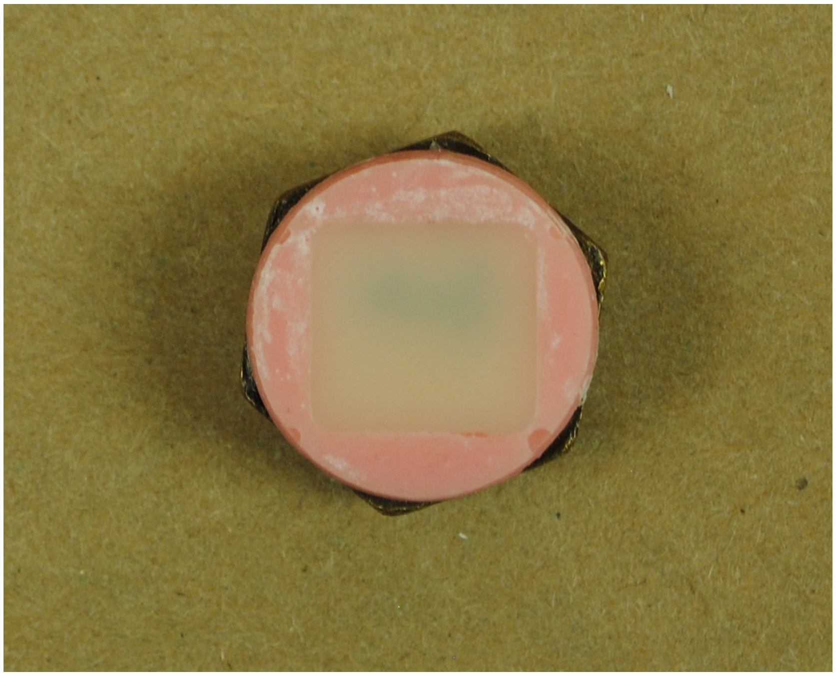


FIGURE 5. Representative ceramic (IPS Empress CAD) tab embedded in the brass holder with auto-polymerizing acrylic resin.

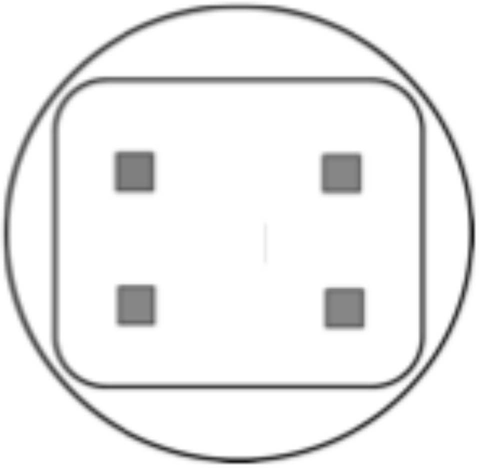


FIGURE 6. Schematic representation of the method used for roughness determination on ceramic samples.

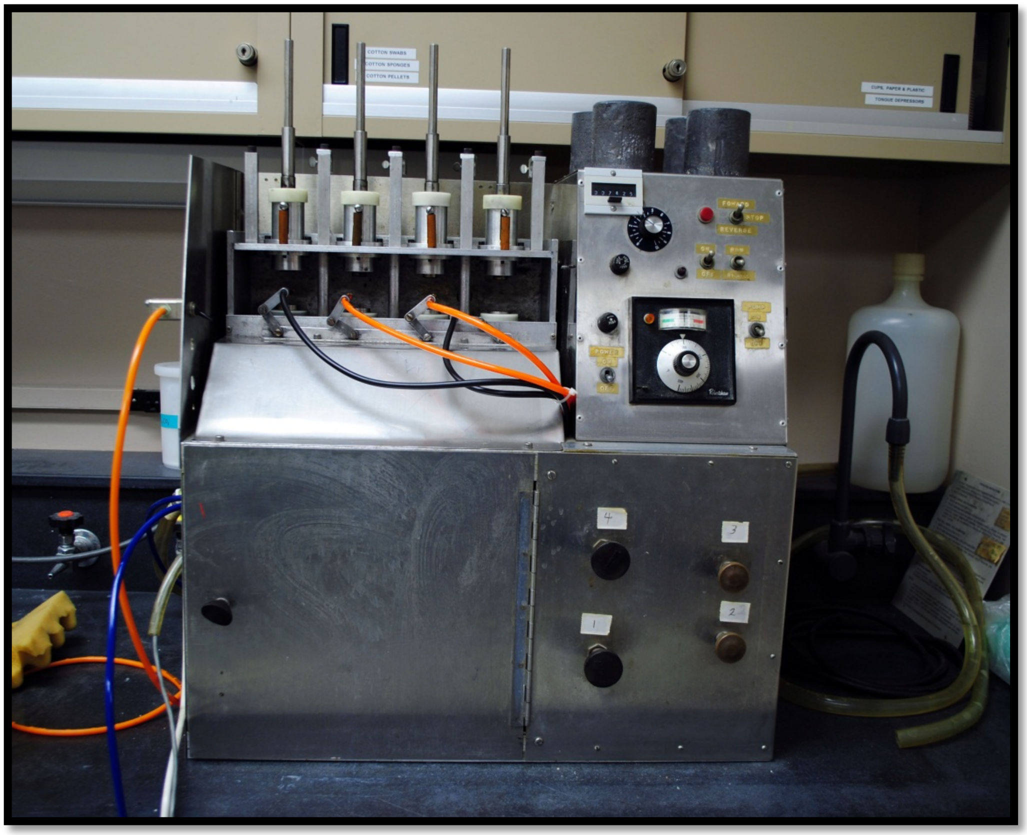


FIGURE 7. Two-body pin-on-disc wear testing machine was run for 25,000 cycles at 1.2 Hz with distilled water lubrication under a 3-Kg load during the wear test.

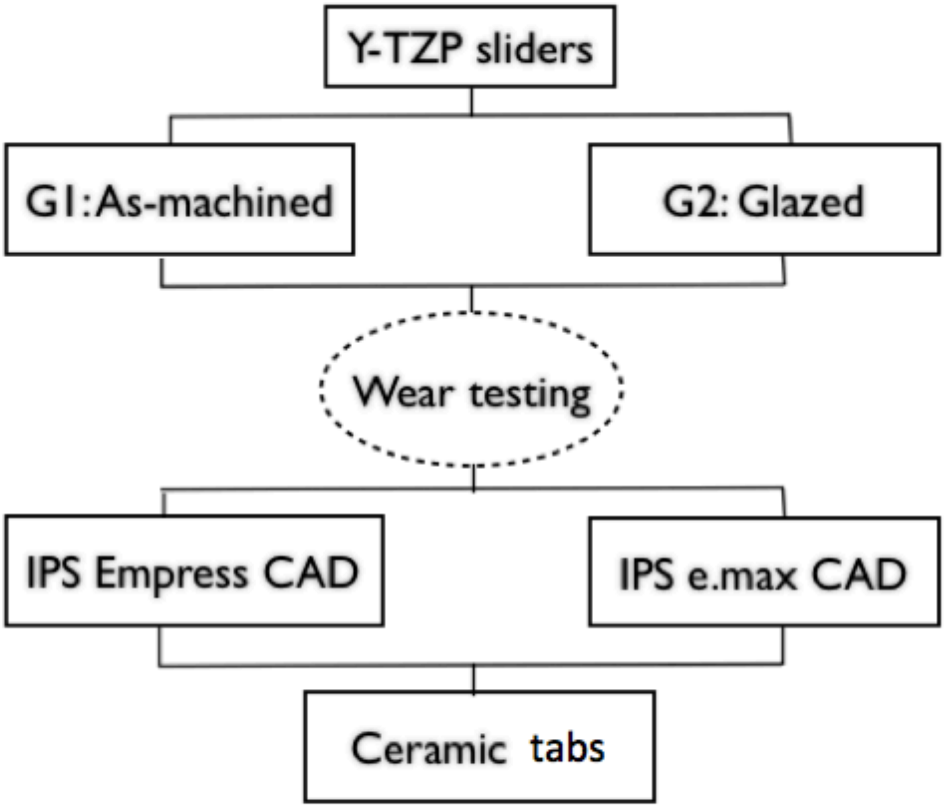


FIGURE 8. Schematic representation of the overall study design. The Y-TZP sliders were randomly allocated into two groups (n=16) as G1: as-machined and G2: glazed. Each slider group with different surface treatments was also divided in half to be tested against two machinable glass-based ceramics (n=8).

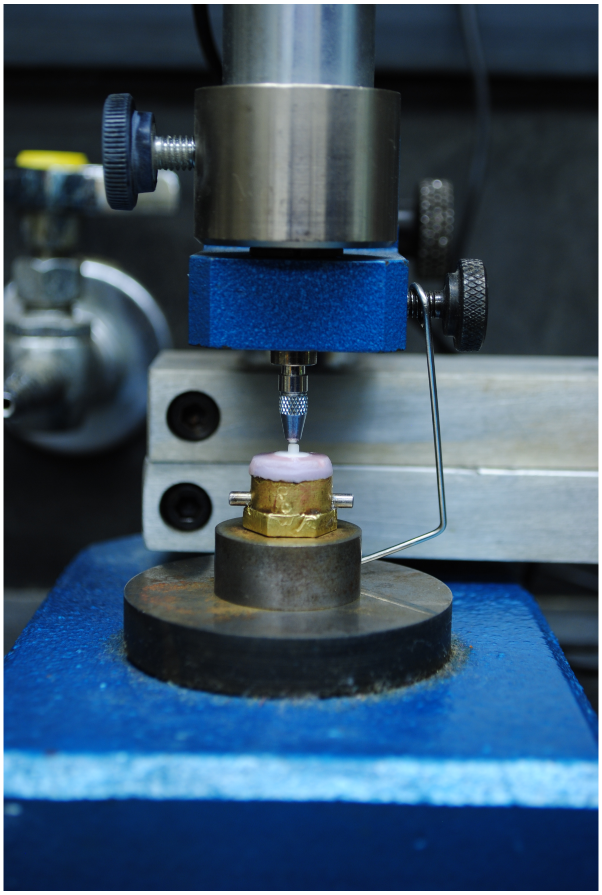


FIGURE 9. Baseline and after-test height data were recorded using a 2D vertical digital measurement device (figure 9) in four different areas.

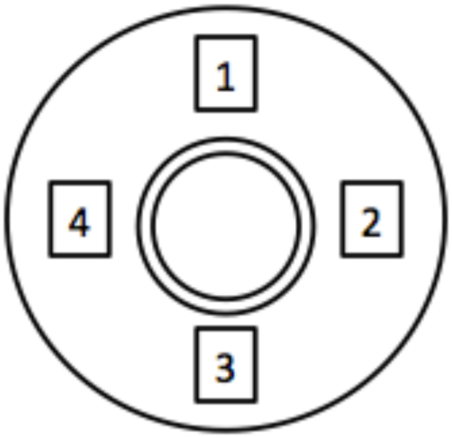


FIGURE10. Four different spots (clockwise) on the base portion of Y-TZP sliders used to measure height loss for baseline and after wear testing.

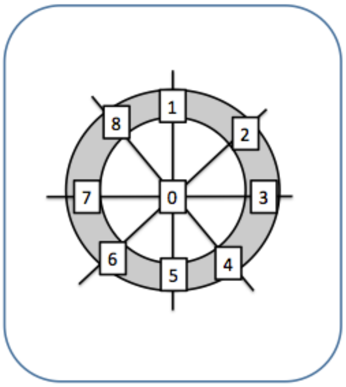
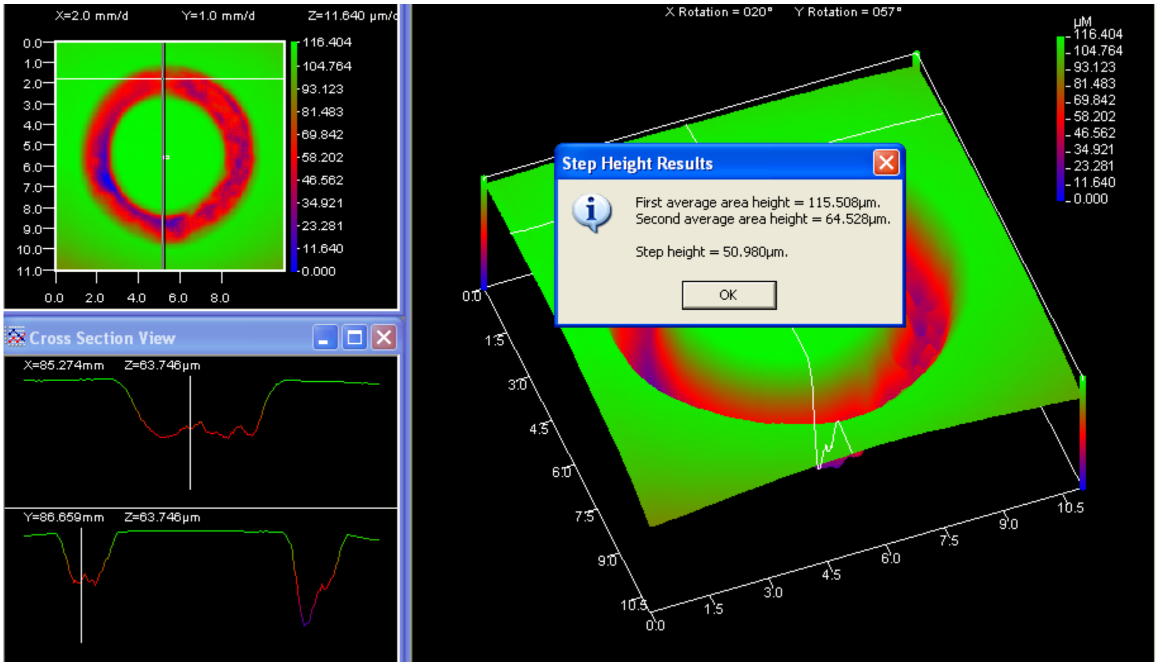


FIGURE 11. Height loss measurement for ceramic tabs. Eight different areas (clockwise) along the wear track were measured by comparing with the central area (0) of the ceramic tab outside the wear track.



First average area height = 115.508µm.
Second average area height = 64.528µm.

Step height = 50.980µm.

FIGURE 12. Height loss measurement on glass ceramic antagonist surfaces using Proscan software (Proscan 2000). Two spots were selected to calculate for height differences under the 2 point step height function. Auto level and warpage functions were used to standardize the images for better calculation.

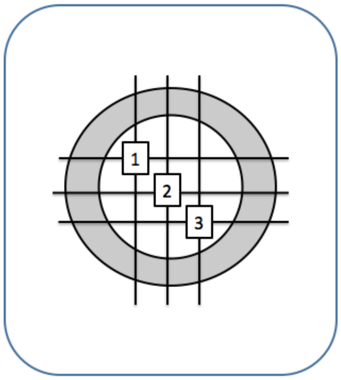
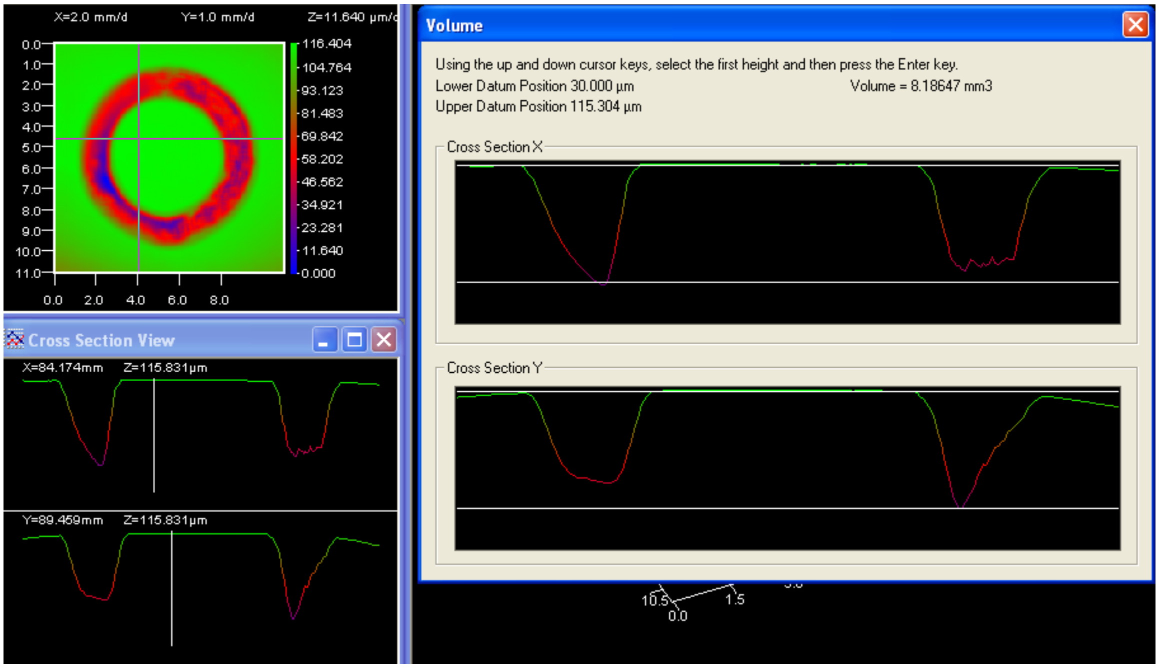


FIGURE 13. Three different spots were measured on each ceramic tab to achieve the volume loss profiles that comprised two planes in *x* and *y* axis.



Using the up and down cursor keys, select the first height and then press the Enter key.

Lower Datum Position 30.000 µm

Upper Datum Position 115.304 µm

Volume = 8.18647 mm3

FIGURE 14. Volume loss measurement on glass ceramic antagonist surfaces using Proscan software (Proscan 2000). In each spot, the lowest and the highest planes were selected on both *x* and *y* axes for volume loss calculation under the Volume function.

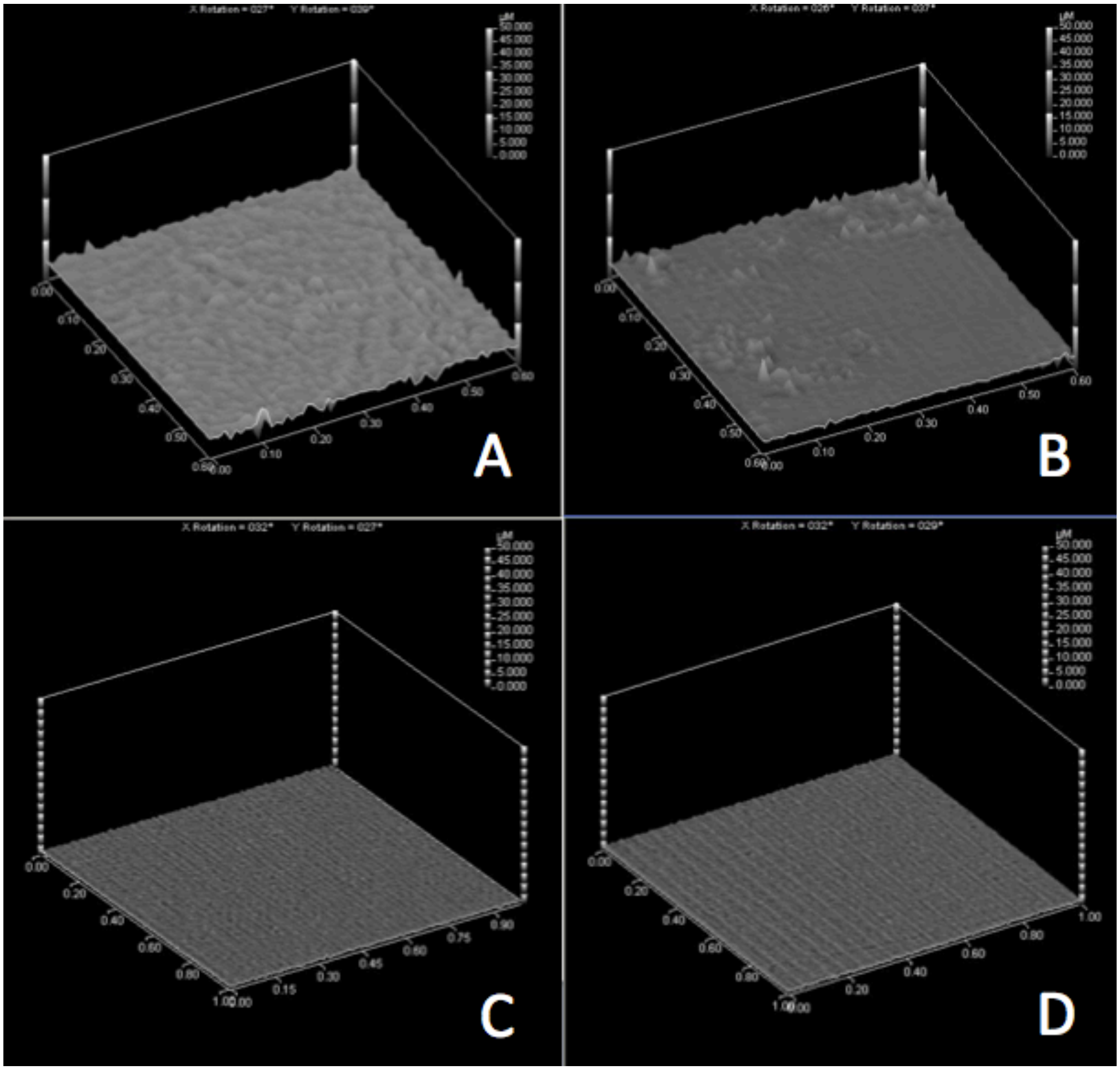


FIGURE 15. Representative image of the 3D surface topography obtained using the profilometer of the tested groups as follows: (A) As-machined Y-TZP slider; (B) glazed Y-TZP slider; (C) IPS Empress CAD and (D) IPS e.max CAD.

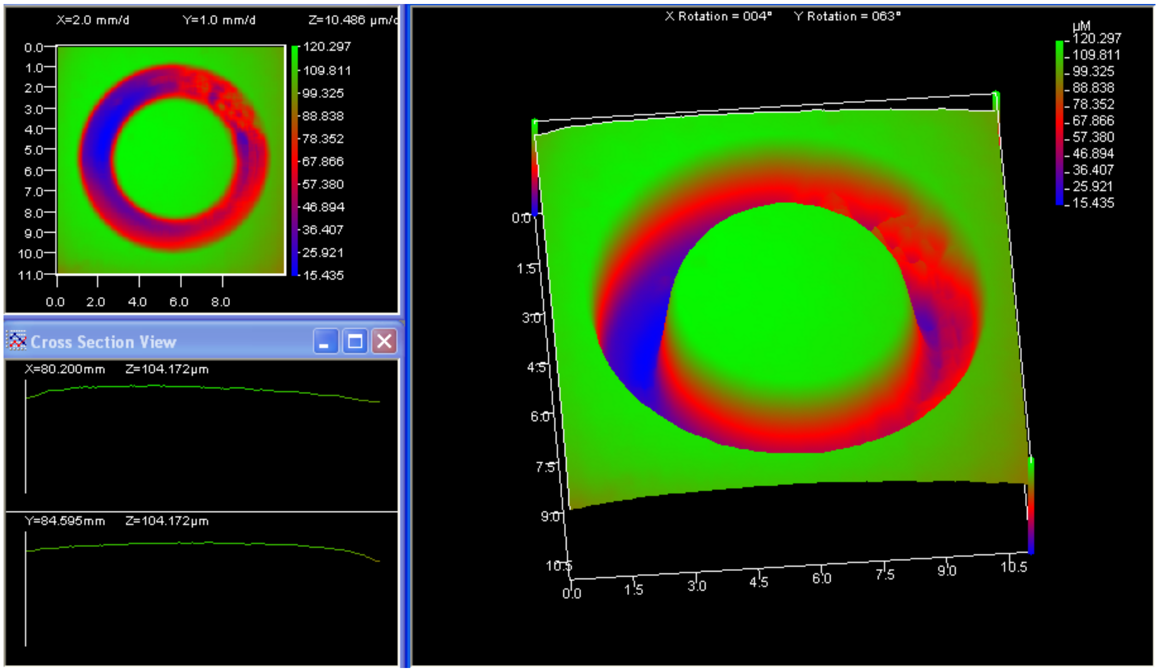


FIGURE 16. An example image of the wear track on IPS Empress CAD antagonist from non-contact optical profilometer (Proscan 2000, Scantron, Taunton, England).

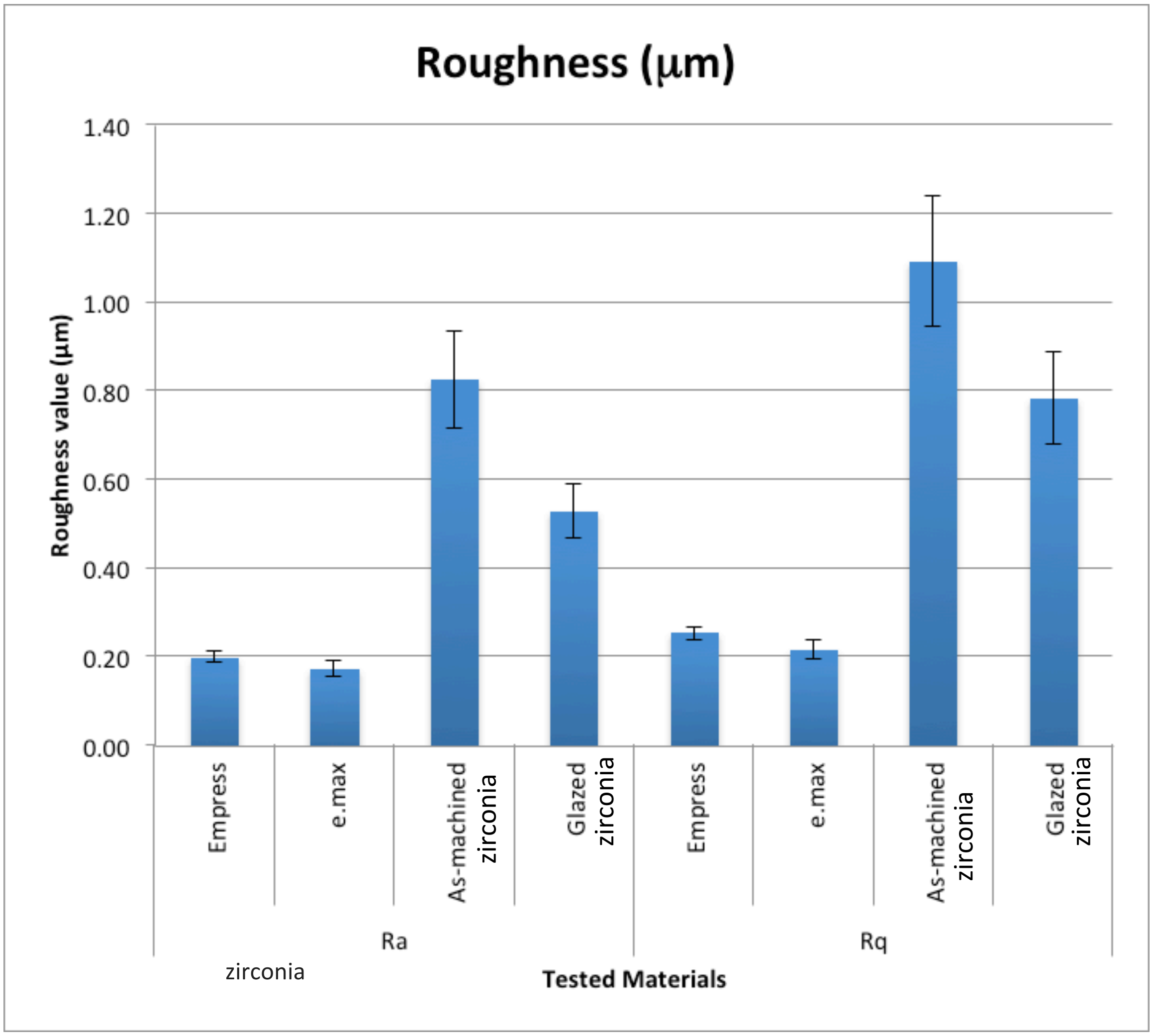


FIGURE 17. Summary of roughness measurements among the different material surfaces tested.

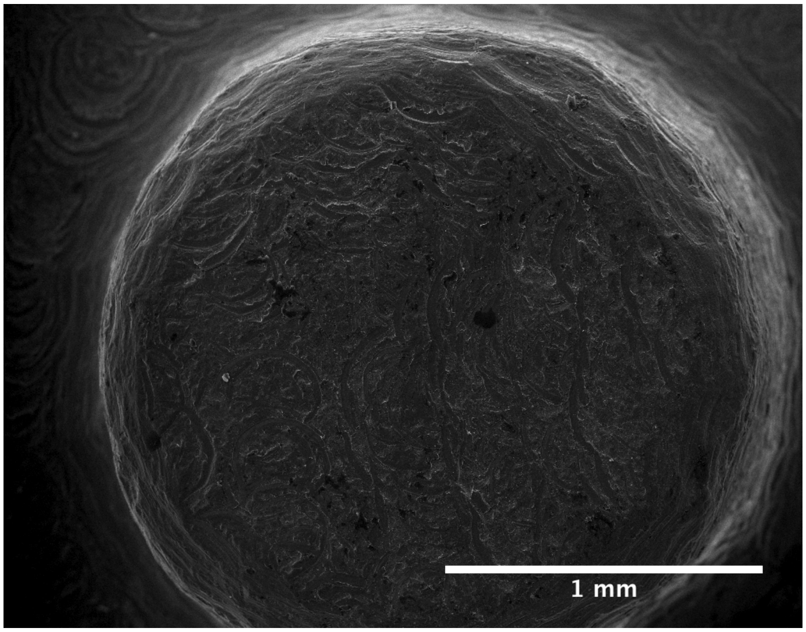


FIGURE 18. Representative SEM micrograph of as-machined zirconia slider (75 ×).

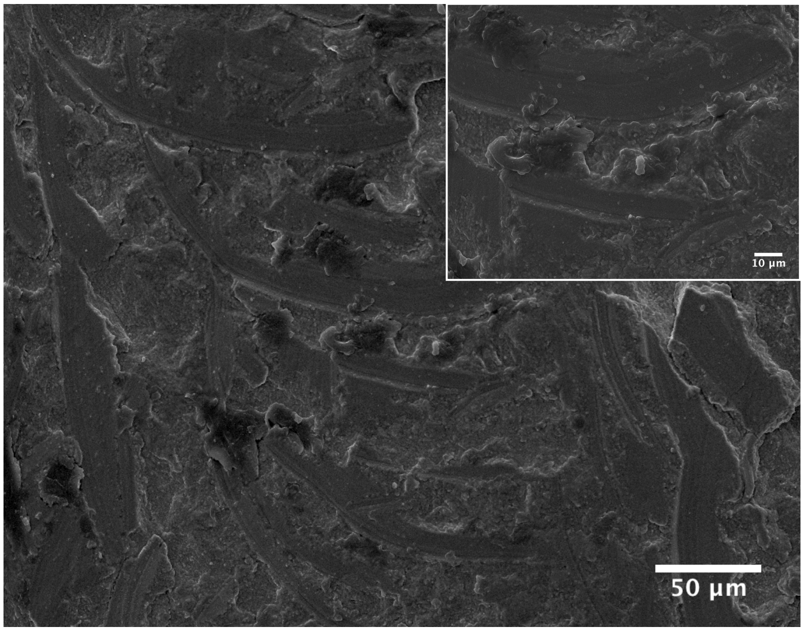


FIGURE 19. Representative SEM micrograph of as-machined zirconia group at higher magnifications (500 × and 1,500 × on the top right corner).

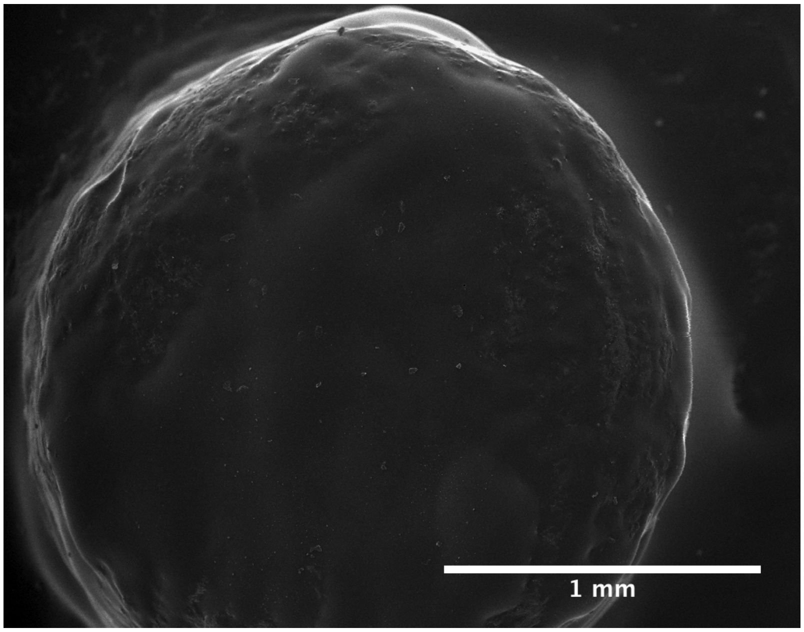


FIGURE 20. Representative SEM micrograph of glazed zirconia testing surface (75 ×).

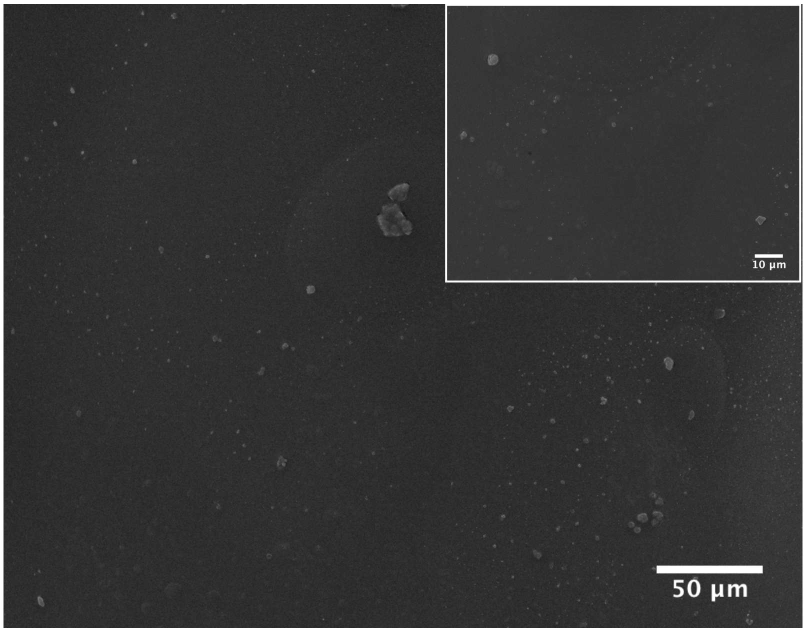


FIGURE 21. Representative SEM micrograph of glazed zirconia slider at higher magnifications (500 × and 1,500 × on the top right corner). Smooth, glass-like area is visible all across the image with a fair amount of inclusions.

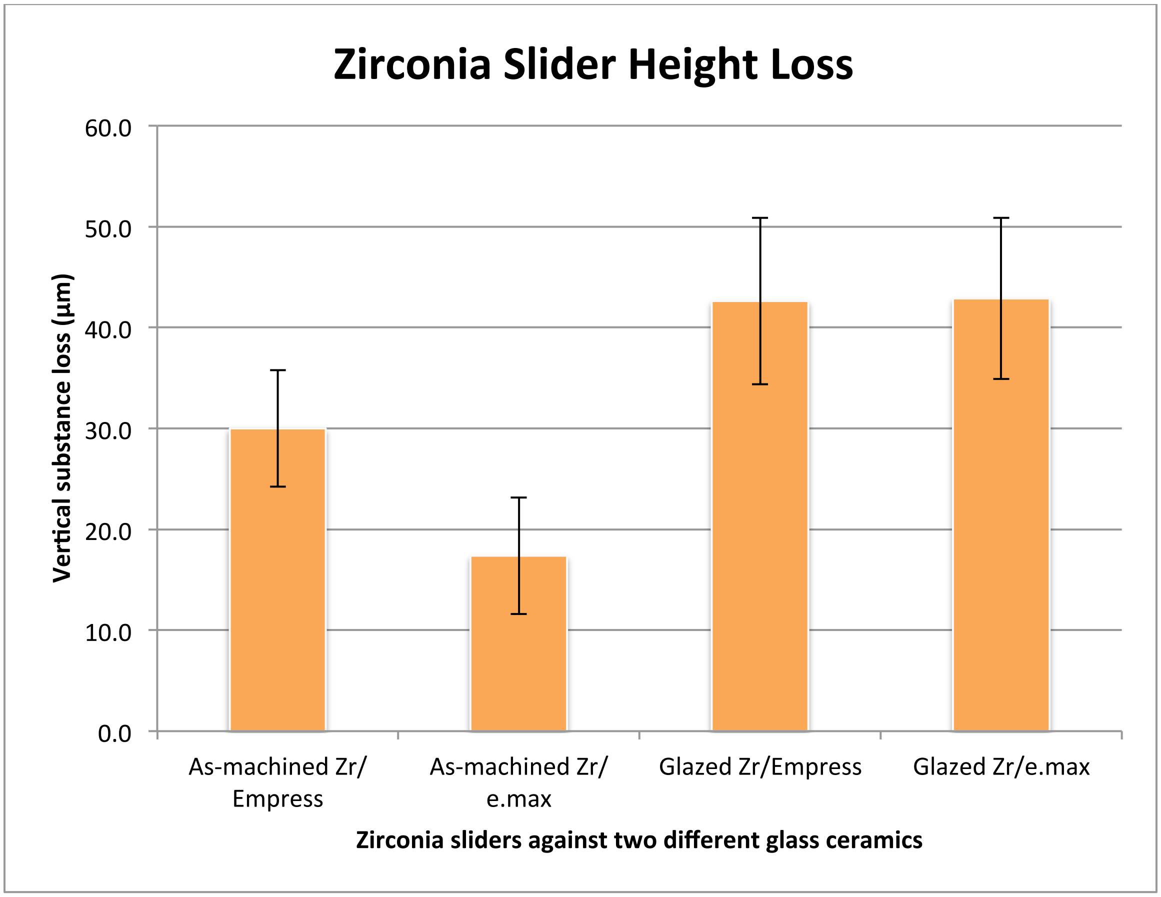


FIGURE 22. Height loss summary of Zirconia sliders (µm).

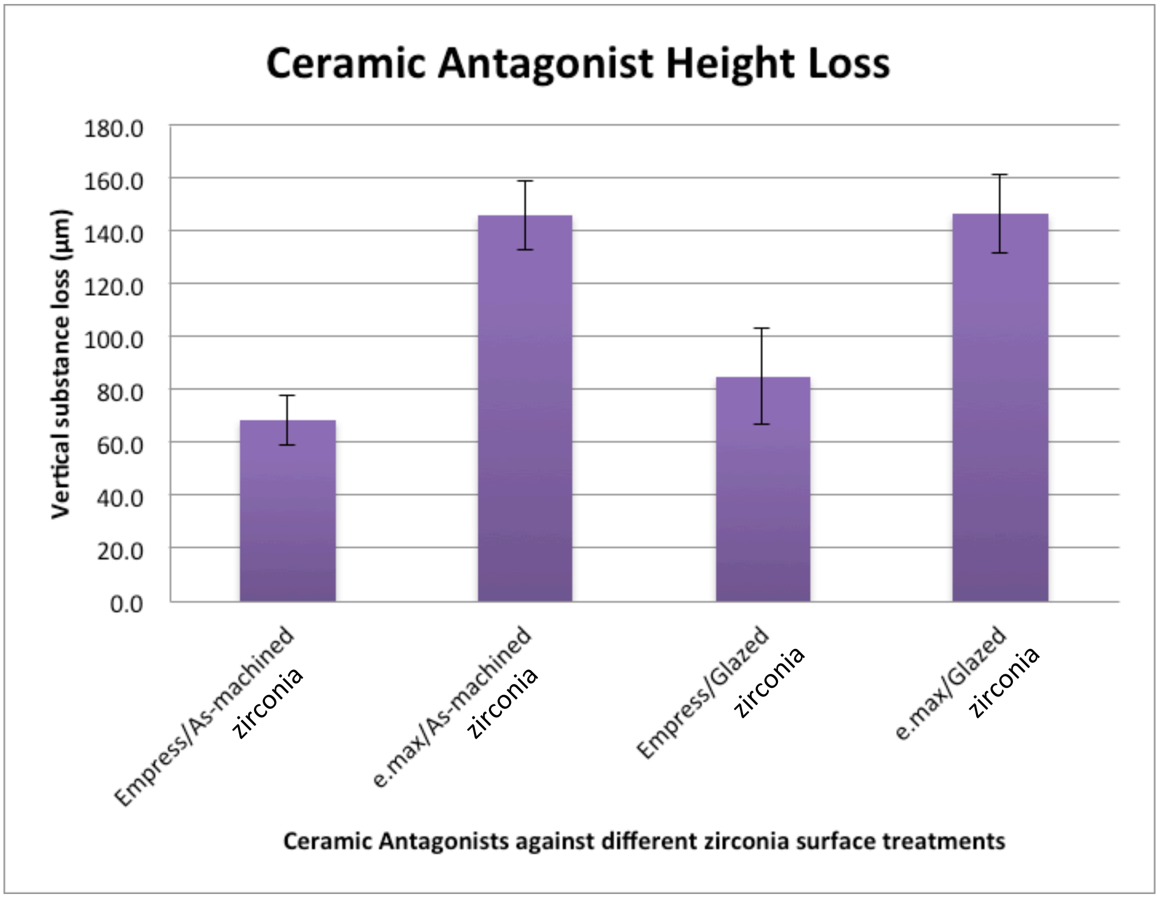


FIGURE 23. Height loss summary of ceramic antagonists (µm).

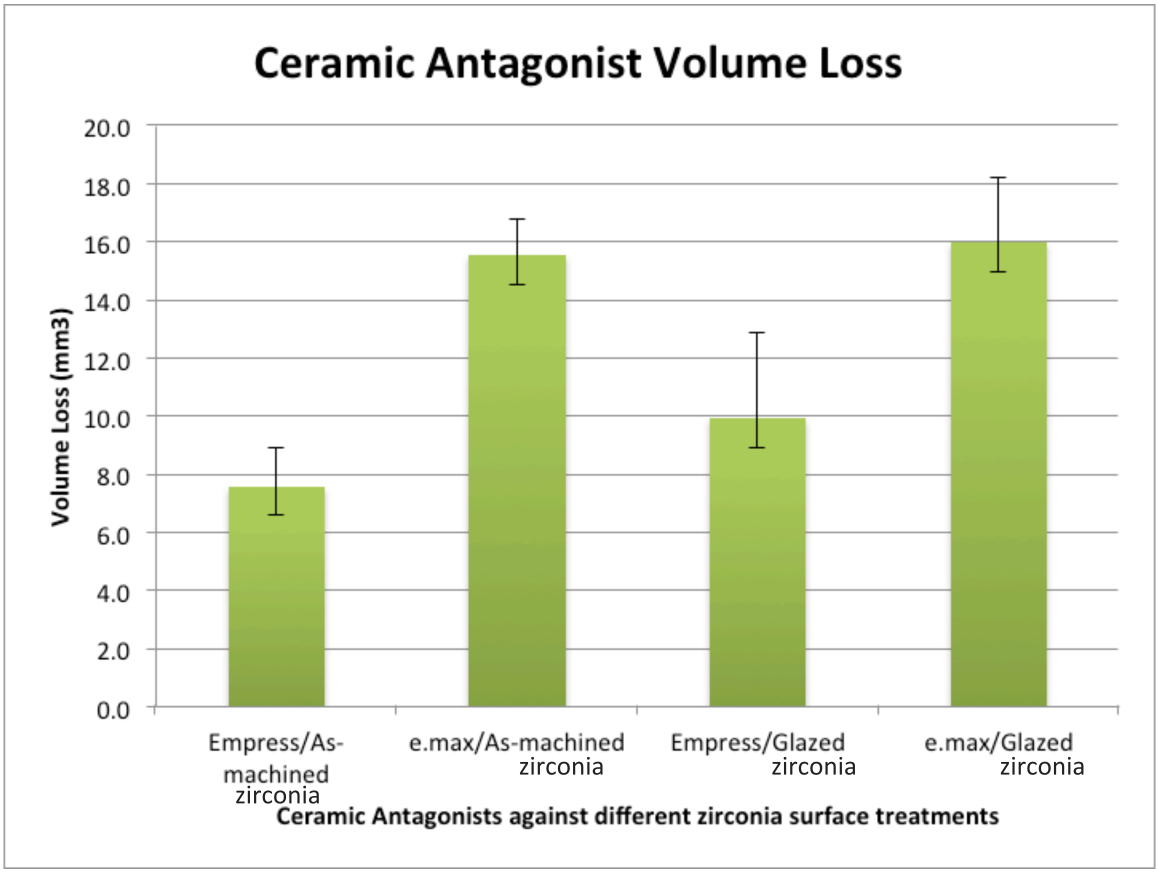


FIGURE 24. Volume loss summary of ceramic antagonists (mm^3).

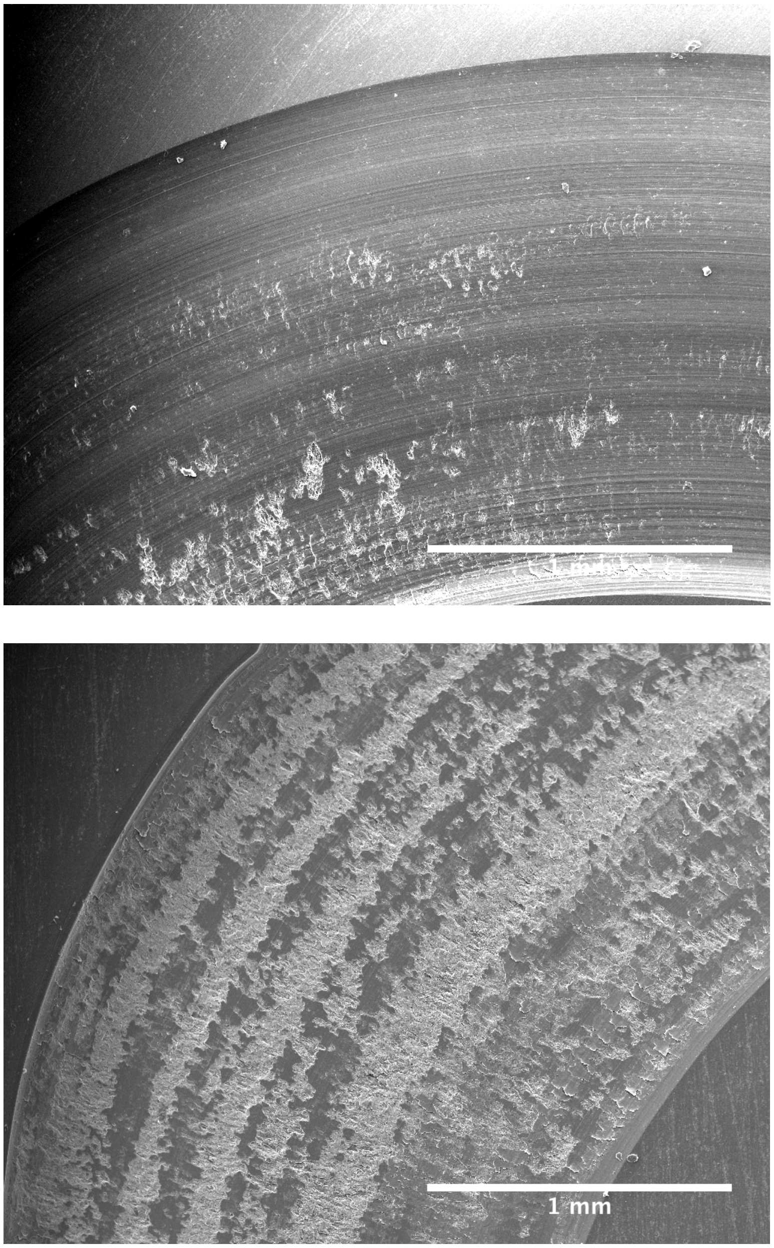


FIGURE 25. SEM images of the worn surfaces of Empress ceramic antagonists against G1(top) and G2 (bottom) at low magnification (75×). Note the rougher surface on the bottom image.

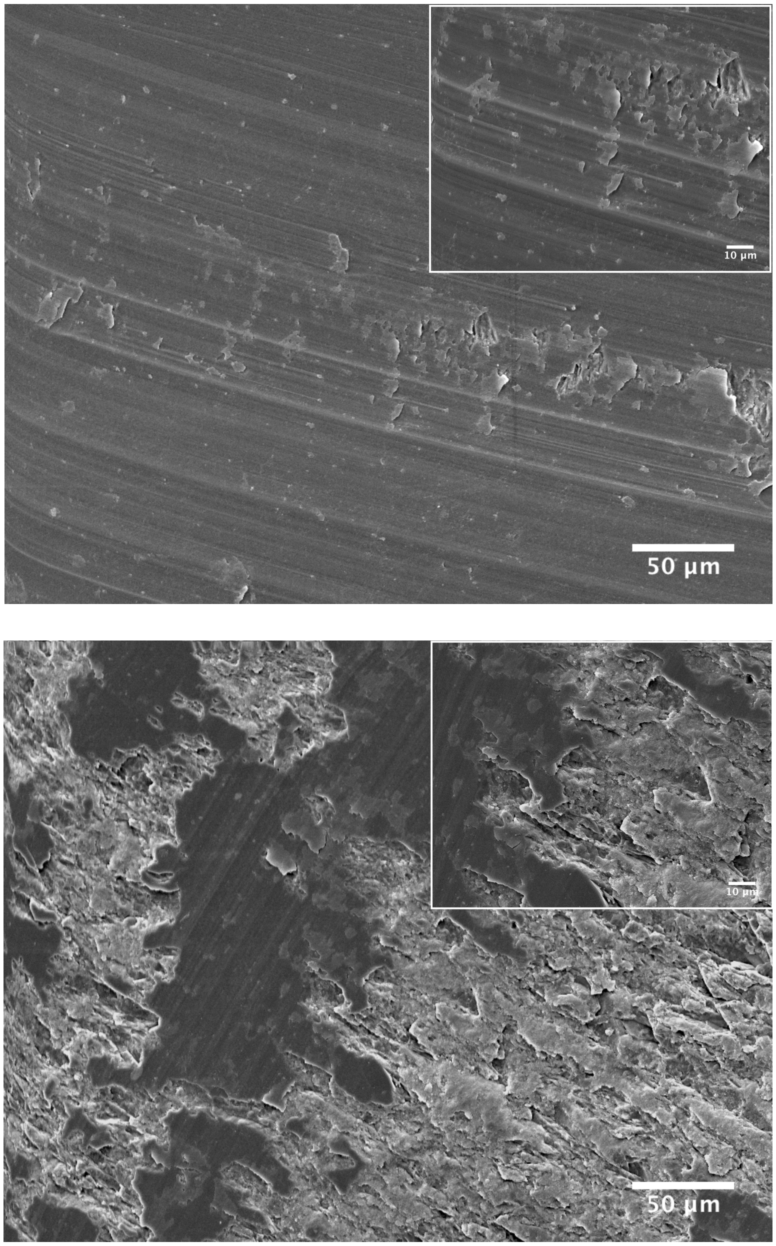


FIGURE 26. The worn surfaces of Empress CAD against as-machined zirconia group (top) and glazed zirconia group (bottom) at higher magnifications (500× and 1,500× on the top right). Plowing of the material can be seen more on the right image along with some pores.

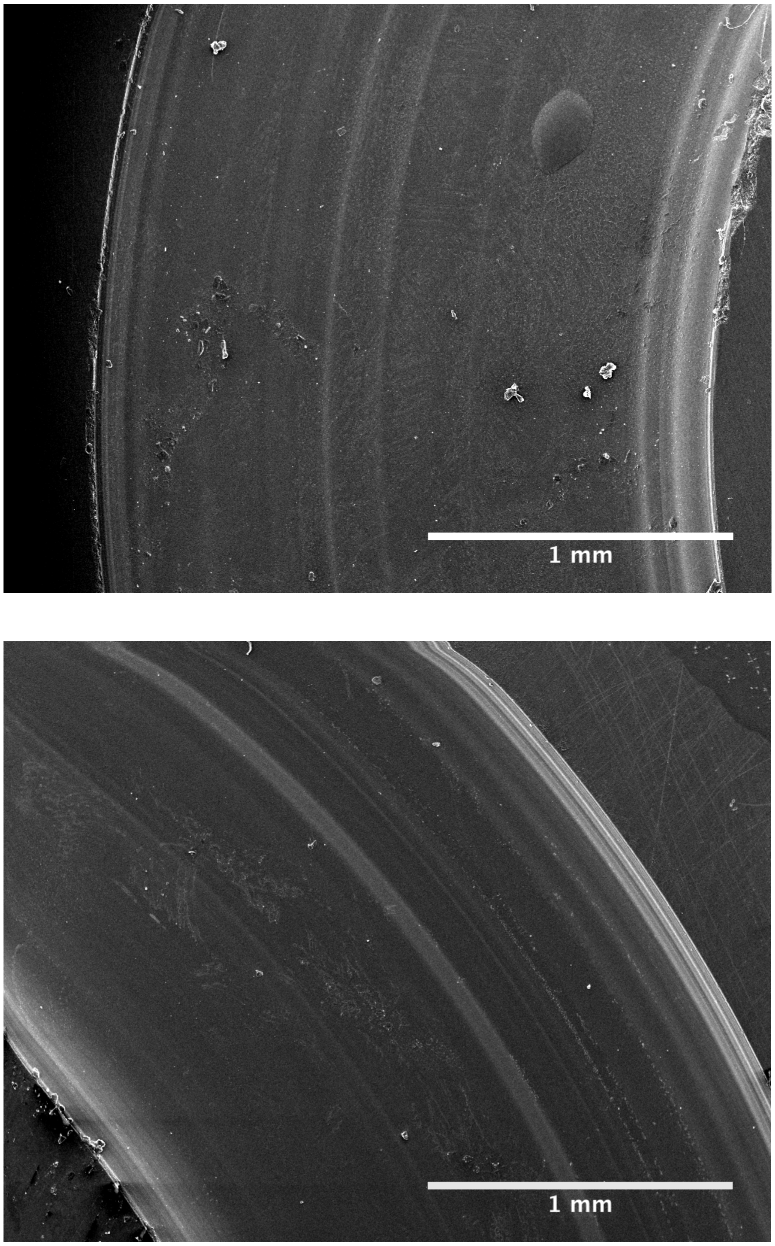


FIGURE 27. SEM images of the worn surfaces of e.max CAD ceramic antagonists against as-machined zirconia sliders (top) and glazed zirconia sliders (bottom) at low magnification (75×). Not much difference can be detected at this magnification.

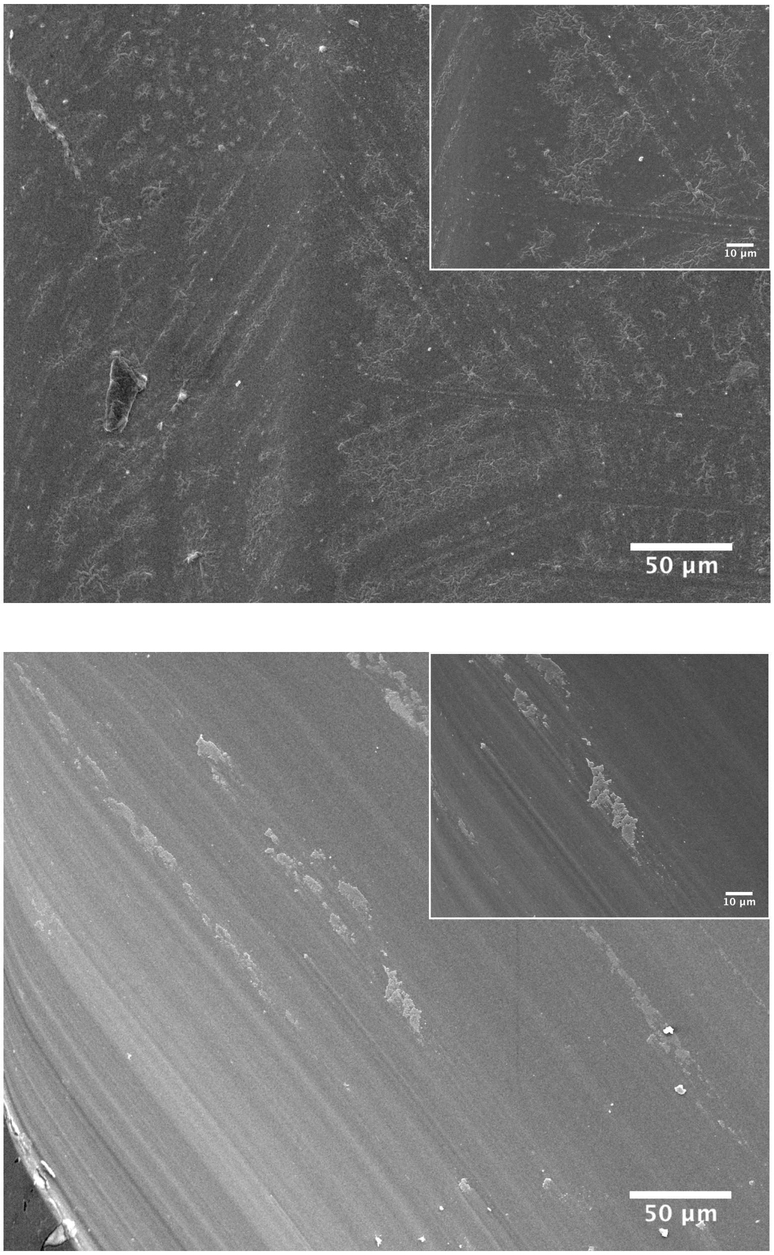


FIGURE 28. The worn surfaces of e.max CAD against as-machined zirconia group (top) and glazed zirconia group (bottom) at higher magnifications (500× and 1,500× on the top right). Irregular wear patterns can be seen on the top image while the bottom image shows uniform, longitudinal wear tracks with debris.

DISCUSSION

The null hypotheses of this study: (1) the surface roughness of Y-TZP ceramic would not be reduced due to the glaze application; (2) glass-ceramic wear would not be affected by zirconia surface roughness, and (3) the wear of glazed and as-machined zirconia against the two glass-based ceramics would not be distinguishable in spite of differences in physical, chemical and mechanical properties of the antagonist ceramics, were all rejected.

The results obtained in this study suggest that the mean vertical substance loss of the glazed zirconia group was higher than the as-machined group despite the glaze thickness of approximately 8 μm. There may be a few explanations for this finding. Glazing materials can be harder than the underlying ceramic, which could presumably result in more abrasive restoration surfaces when compared with unglazed restorations, as reported by Jacobi and Duncanson.[50] Furthermore, dislodged glaze materials may form wear debris that eventually acts as a third-body and accelerate the overall wear process.[34] Accordingly, it can be inferred that surface treatment by glazing may markedly influence the early stage of the wear process.[44] Since the wear testing in this study was carried out for only 25,000 cycles, which was considered relatively low when compared with other studies,[34-36] similar material wear of different surface treatments could have been achieved if the testing had run longer.

Though application of surface glazing on Y-TZP sliders was demonstrated to decrease the initial surface roughness values when compared with as-machined Y-TZP group, results were clear that the smoother surface by glazing did not reduce the antagonistic wear. On the contrary, it even increased antagonistic wear for Empress group.

Regarding the wear of the glass ceramics, significant differences in both vertical and volume loss were found between Empress and e.max with the Empress group wearing less than the e.max group. This finding was also supported by a review by Heintze et al[44] when they were tested against flat, ceramic antagonists. However, Albashaireh et al[34] found no differences between these two materials when polished zirconia balls were used as the antagonist. It should be mentioned that different material configurations, whether they are flat or crown specimens, as well as different study designs may affect the wear process and result in significant discrepancies in terms of wear data[44]. Previous studies have shown that differences in the substance loss of ceramic materials might be closely associated with their microstructure and the physical characteristics, specifically flexural strength and toughness. Grinding of glass and exposure of crystalline phases (e.g., leucite) during the wear test may result in roughening of the ceramic. This deterioration is influenced by the properties of a material, such as hardness, fracture toughness and/or composition.[44]

Different types of restorative material have individual wear patterns[44]. The obtained SEM images (figure 25-28) showed longitudinal wear tracks, plowing of materials, pores and fragments causing from chipping of the materials. These features indicate abrasive, adhesive and fatigue wear characteristics that were mentioned widely in the wear literature regarding ceramic wear patterns. No cracks were visible in any of the images which was similar to a previous report by Albashaireh et al[34] regarding leucitic glass ceramic (IPS Empress Esthetic). More pronounced rough surfaces and irregular concavities were prominent for lithium disilicate ceramic (IPS e.max Press). Interestingly, in our study, porosities and irregular surfaces were obvious for leucite-containing glass ceramic while more homogenous and smooth wear patterns can be seen for lithium disilicate glass ceramic (e.max). This, again, could have been due to the displacement/debonding of the glaze material which might act as an abrasive slurry causing three-body wear. The topography of the material surface may influence the abrasiveness of the glass ceramics towards the opposing slider. Based on the results from this study, as-machined Y-TZP sliders tested against e.max antagonist showed less wear than the group against Empress antagonists. There may be several explanations for this. First, the initial surface roughness of e.max measured from the present study was significantly lower than Empress. Microscopically, e.max ceramics are comprised of considerably smaller grain size of lithium disilicate crystals when compared with leucite crystals present in Empress. These features result in smoother surface characteristics for e.max ceramic. This is in agreement with Esquivel-Upshaw et al[24] who stated that, when compared with Empress 2, e.max Press caused less wear to opposing enamel due to its homogeneous crystal distribution as well as smaller grain size. Second, the lower flexural strength and fracture toughness of Empress when compared with e.max ceramics resulted in more porosities and other surface irregularities occurring by chipping of the ceramic during the wear process,[21, 44] as described earlier from the findings of this study. This mechanism may have promoted more wear to zirconia sliders by the Empress group due to their rougher surfaces.

Dental wear is a complex physiological phenomenon of opposing teeth or dental restorations sliding against each other eventually leading to tooth/ material loss and surface damage.[41] Studies have shown that restorative materials with different properties possess different wear characteristics. Surface hardness was conventionally considered to be one of the material properties that affects wear of opposing teeth or restorations. As a result, dental ceramics, especially zirconia which has extremely high hardness values, were expected to cause more wear.[35] However, strong correlation between restoration hardness and the degree of antagonist wear have not yet been established.[51] Recent studies have suggested other factors including fracture toughness, the presence of porosities, crystal size and surface characteristics as variables that define the abrasive potential of dental ceramics.[24, 44] Some authors have also suggested surface roughness as one of the major factors.[35, 36, 51] According to this study, a smoother zirconia surface did not reduce the wear of opposing glass ceramic materials.

The justification for the two-body wear testing in this study, was to simulate direct contact between the maxillary and mandibular ceramic restorations which occurs during parafunction, swallowing and dynamic occlusion movements.[36] Parafunctional activities, i.e. bruxism and clenching, have been suggested as one of the indications for full-contour zirconia restorations. For this reason, our study is clinically relevant. A load of 3 Kg was used that was determined to produce contact stresses of 10 MPa according to Jain et al.[48] Furthermore, with a smaller slider diameter of 2 mm when compared with 5 mm[45] and 8 mm[34] in other studies, a higher attrition can be expected with the reduced diameter in this study according to a report by Jaarda et al.[52] No height loss was detected in a study by Preis et al[45] for 5 mm diameter full-contour zirconia specimens after a wear testing for 120,000 cycles; while 17.4 - 42.9 μm of zirconia height loss after only 25,000 wear cycles were reported in this study.

Although *in vitro* studies have not yet been able to simulate completely nor show strong correlation to clinical conditions, they can be used as a comparative evaluation of different materials under standardized conditions.[44]

SUMMARY AND CONCLUSIONS

1. Even though surface glazing of Y-TZP ceramics decreased the roughness values, it did not seem to be relevant to their abrasive potential towards the glass ceramic antagonists.
2. For Y-TZP sliders, surface glazing affected the slider wear more than the type of antagonistic materials.
3. For glass ceramics, the material type determined the wear characteristics. Glazing on the zirconia surface did not reduce antagonist wear on either e.max or Empress.
4. IPS Empress CAD was found to be more abrasive to opposing Y-TZP sliders than IPS e.max CAD.

Several aspects regarding ceramic wear need to be addressed in future studies. First, validation of the pin-on-disc wear testing machine will need to be done since this particular machine has never been used to perform wear testing on ceramics in the literature. We attempted to use Vita Mark II feldspathic porcelain sliders with the same geometry as the Y-TZP sliders as a comparison standard in an early pilot. Unexpectedly, all Vita Mark II sliders fractured within the first 1,000 cycles. Second, incorporating the polished group as another surface treatment modality would be more beneficial and clinically relevant since most restorations are polished after intro-oral adjustments to ensure smooth contacting surfaces.

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ABSTRACT

EFFECT OF FULL-CONTOUR Y-TZP ZIRCONIA SURFACE ROUGHNESS ON WEAR OF GLASS-BASED CERAMICS

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The use of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), normally employed as a framework for all-ceramic restorations, has now started to be used without any veneering ceramics in patients with parafunctional activities.

The aims of this study were to evaluate the influence of Y-TZP surface roughness on the wear behavior (volume/height loss) against glass-based ceramics (i.e., IPS Empress CAD and IPS e.max CAD, Ivoclar-Vivadent).

Thirty-two Y-TZP full-contour zirconia (Ardent®) sliders (ϕ=2 mm, 1.5 mm in height) were milled in a CAD/CAM unit and sintered according to the manufacturer instructions. Sliders were embedded in brass holders using acrylic resin and then randomly allocated into 2 groups according to the surface treatment (n=16): G1-as-

machined and G2-glazed (Diazir®). Empress and e.max antagonists were cut into tabs (13×13×2 mm) wet-finished and also embedded in brass holders. Two-body pin-on-disc wear testing was performed at 1.2 Hz for 25,000 cycles under a 3-kg load. Non-contact profilometry was used to measure antagonist height (μm) and volume loss (mm^3). Qualitative data of the testing surfaces and wear tracks were obtained using SEM. Statistics were performed using one- and two-way ANOVAs (α=0.05).

The results indicated that G1 yielded significantly higher mean roughness values (R_a=0.83 μm, R_q=1.09 μm) than G2 (R_a=0.53 μm, R_q=0.78 μm). Regarding antagonist loss, G1 caused significantly less antagonist mean height and volume loss (68.4 μm, 7.6 mm^3) for Empress than G2 (84.9 μm, 9.9 mm^3) while no significant differences were found for e.max. Moreover, Empress significantly showed lower mean height and volume loss than e.max ($p<0.0001$). SEM data revealed morphological differences on wear characteristics between the two ceramics against Y-TZP.

Within the limitations of this study, e.max wear was not affected by Y-TZP surface roughness. However, Empress wear was greater when opposing glazed Y-TZP. Overall, based on our findings, surface glazing on full-contour Y-TZP did not minimize glass-ceramic antagonist wear when compared with as-machined group.

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