Original Article

The comparison of the surface roughness and surface morphology of sintered and chairside polished monolithic zirconia implant crown

Wasan Vatanasak, D.D.S.
Kamolporn Wattanasirmkit, D.D.S., MSc Prosthodontics, Ph.D.

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Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand

Abstract

Background: Low temperature degradation, also known as ageing, is the major challenge we cannot avoid in zirconia restorations. Changes in temperature, pressure, mechanical force and moisture are few of the factors which contribute to ageing which eventually leads to failure of zirconia restorations.

Objectives: To compare the phase transformation, surface roughness and grain morphology as indications of low temperature degradation that might occur in translucent monolithic zirconia after try in procedure which finished the restoration by chairside polishing or annealing technique.

Materials and methods: A total of 10 single implant crowns would be milled from the one Vita HT (VITA-Zahnfabrik, Germany) monolithic zirconia block. Before delivery, all the crowns would be examined under X-ray diffractometry, scanning electron microscope, and contact profilometry to serve as base line information regarding crystalline phase, surface roughness, and surface morphology. After proximal contact and occlusal surface adjustment, 5 of 10 polished specimens would be selected to undergo annealing at 1000°C for 15 minutes. Both polished and annealed groups would then be re-examined following the same methods as control. The data would be analysed by means of paired sample t-test.

Results: On the occlusal surface, the percentage of tetragonal and rhombohedral phases as well as the surface roughness showed significant difference (p-value < 0.05) between before delivery in both the polished and annealed zirconia crowns. Data obtained from the buccal surface showed significant difference between before and after delivery in percentage of te-tragonal and monoclinic phases in the both annealed and polished groups (p-value < 0.05). As for the proximal surface, significant difference between before and after delivery was seen in percentage of tetragonal phase in the annealed and polished groups, monoclinic phase in the annealed group, and the surface roughness for both groups (p-value < 0.05).

Conclusion: Within the limitations of this study, it was shown that finishing and polishing does affect the surface morphology of zirconia restoration, crystalline phase of zirconia, and surface roughness of zirconia. Annealing at 1000°C for 15 minutes did show an increase in tetragonal phase content.

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Keywords: monolithic zirconia, phase transformation, low temperature degradation, LTD, surface roughness, grains, X-ray diffractometry, XRD, zirconia

Correspondence: Kamolporn Wattanasirmkit, kamolpornw@yahoo.com

Introduction:

Zirconia is a crystalline dioxide of zirconium, known as Zirconium dioxide (ZrO_2). It is a white crystalline oxide of zirconium found in zircon, accidentally discovered by a German alchemist, Martin Heinrich Klaproth in 1789 while he was working with certain procedures that involved the heating of some gems. The first use of zirconium dioxide for medical purpose was in 1969 in orthopedics as a new material for hip head replacement instead of titanium or alumina.

Pure Zirconia crystals can be organized into three different martensitic crystalline structures which include cubic, tetragonal and monoclinic structures. At room temperature, zirconia is stable in monoclinic form. With increasing temperature, the material could transform to tetragonal by approximately 1170°C and to cubic fluorite crystalline structure at about 2370°C with melting point at 2716°C (Subbarao, 1981; Goff et al., 1999). They are characterized being diffusionless, occurring thermally implying the need for a temperature change over a range rather than at specific temperature and involving a shape deformation (Evans, 1980). Volume changes on cooling associated with these transformations are substantial enough to make the pure zirconia unsuitable for using as dental applications. Doping extra small foreign bodies like metallic oxides such as magnesium oxide (MgO), calcium oxide (CaO), or yttrium oxide (Y_2O_3) helped zirconia to obtain structural stability (Garvie et al., 1972).

Yttria-stabilized zirconia (3Y-TZP) has been used in dentistry for the fabrication of dental crowns and fixed partial dentures due to superior mechanical properties such as its flexural strength that may reach up to 900-1,200 MPa and fracture strength which ranges between 9-10 MPa·$(m)^{1/2}$.

The mechanical properties of 3Y-TZP are strongly affected by its grain size. Above critical grain size, 3Y-TZP is less stable and more susceptible to spontaneous tetragonal-monoclinic martensitic transformation. Grain sizes less than 1 µm are associated with lower transformation rate. Below a grain size of 0.2 µm, the transformation is not possible, consequently leading to a reducing in toughness. Light transmission properties with a high value shade of Zirconia substructure cause some esthetics problems. With a low translucency and white opaque color made the 3Y-TZP required veneering porcelain veneer on the outer side of the framework to achieve a more natural appearance and acceptable esthetics (Miyazaki, 2013). However, cracking or chipping of the porcelain veneer has been reported to be a major complication of these restorations (Vult von Stayen et al., 2005; Sailer et al., 2006; Sailer et al., 2007; Raigrodski et al., 2006). The possible causes of porcelain veneer cracking are: differences in coefficient of thermal expansion (CTE) between framework and porcelain, firing shrinkage of porcelain, porosities, poor wetting of veneering, flaws on the veneering, inadequate framework design to support veneer porcelain, overloading and fatigue (Miyazaki et al., 2013).

There are several solutions to overcome the veneer cracking problem due to its multifactorial nature: alternative application of techniques for veneering such as CAD/CAM veneer, modification of the firing procedures and modification of framework design (Rues et al., 2010; Rosentritt et al., 2009). Another alternative solution was to use non-veneered zirconia restorations. The light transmission properties of zirconia was developed, then full contoured monolithic zirconia restorations have become increasingly worldwide as a result of advances in CAD/CAM technology (Miyazaki et al., 2013; Stober et al., 2014). The monolithic zirconia has been used in posterior region in order to eliminate the debonding or shift of porcelain veneer problems.

However, zirconia ceramics stabilized with yttria, ceria, calcia, or magnesia are susceptible to low-temperature degradation (LTD) in a various environments over a temperature range of 65-500°C such as humid air, water vapor and other aqueous fluids (Sato et al., 1985a,b; Sato et al., 1990). The lower the temperatures, the more catastrophic effect occurs within the shorter times (Sato et al., 1990). Many researchers found that low temperature ageing occurs with the maximum rate at approximately 250°C (Sato et al., 1985a). The mechanical properties decrease over the cortical temperature range and are accompanied by an increase in the surface monoclinic content (Sato et al., 1985b).

Grinding of zirconia restorations is necessary achieve proper proximal and occlusal contacts intraorally. During this procedure, tens of microns of material would be removed by a single pass as polishing burs move across the surface being adjusted. Stresses and temperatures were high during this operation. The high stresses contribute to severe surface cracks which could lower the strength and reliability of the material. According to Kosmac et al., the critical flaw size which could initiate failure in fine grained zirconia is around 50 μm. The surface roughness of ceramic can vary according to the methods and degree of surface grinding. A rough ceramic surface decreases the physical properties of the materials, causes an excessive wear of the opposing teeth, surface discoloration and inflammation of soft oral tissue.

The degradation of properties is associated with the transformation of metastable tetragonal crystals to the stable monoclinic structures. It has even shown under certain circumstances that most total strength recovery can occur when aged specimens are annealed, thus re-transforming the monoclinic grains back to the tetragonal phase (Matsumoto, 1985).

From the problems stated, this research aims to compare the changes in crystalline phase content, surface roughness, and grain morphology after delivery with 2 finishing techniques. This first technique involved chairside polishing with Diacera (EVE Ernst Vetter GmbH, Germany) diamond impregnated polishing burs, specifically designed to polish zirconia restorations, in order to obtain the smoothest surface as possible. The other involved annealing heat treatment after chairside polishing, which according to the manufacturer, could be done to remove any potential residual stress introduced during processing of zirconia restorations and reverse transformed monoclinic phase to tetragonal phase (Guazzatom et al., 2005). Heat treatment was executed according to the manufacturer's guideline by utilizing Vita Vacummat 4000T Premium Furnace. The final temperature was held at 1000°C for 15 minutes, followed by slow cooling at the rate of 25°C/min.

Materials and methods

Patient selection

The patients who participated in this study were male and female patients that were admitted to the Faculty of Dentistry, Chulalongkorn University, Department of Oral and Maxillofacial Surgery's free implant project from academic years of 2015-2016.

The patients included in this study are the ones who needed implant placement on premolar and molar areas. The interocclusal space at the site of implant placement should be able to accommodate cement retained restoration. The antagonist tooth should be natural with the enamel surface intact. Patients should neither be presented with temporomandibular joint disorders nor parafunctional habits. All were physically and mentally healthy, and do not have past history of radiation therapy and bisphosphonate drug administration.

Methodology

All patients would undergo implant placement with Straumann bone or soft tissue level implants (Straumann group, Switzerland) according to prosthodontic treatment plan based on diagnostic wax up and dental CT images. All implants would be placed under ITI protocol by the same operator at the Graduate Oral and Maxillofacial Clinic, Faculty of Dentistry, Chulalongkorn University.

Fixture level impression of the implant would be taken by means of closed tray technique for fabricates working model following the guidelines provided by Straumann, using polyether (Impregum, 3M ESPE, USA). Straumann cement-retained titanium abutment were selected for retained monolithic zirconia crown as the final restoration. Vita HT Zirconia Blocks (Vita-zahnfabrik, Germany) monolithic zirconia was used to fabricate the final restoration with CAD/CAM process. After sintering, the crowns were stained according to VITA Classic shades (Vita-zahnfabrik, Germany) then glazed with VITA AKZENT Plus (Vita-zahnfabrik, Germany). The restorations were cement-retained implant prosthesis with a screw access hole on the occlusal surface to facilitate future retrievability.

Tables 1.1 and 1.2 show the components of VITA YZ HT Zirconia block and VITA AKZENT Plus glaze.

The restoration should be able to hold a shim stock with 8 μm thickness tight when the patient bites firmly at maximum intercuspal position (MIP). No eccentric contacts would be on the implant restoration. All proximal contacts should be tight when a dental floss passed through.

The surface that would be examined on the zirconia crowns include the occlusal, buccal, and distal surface since equal broad contact could be fabricated on first premolars and first molars included in this study. In this paper, the author would use the term "proximal

Table 1.1: Composition by wt.% of VITA YZ HT Zirconia block.

Components	wt.%
ZrO_2	90.4-94.5
Y_2O_3	4-6
HfO_2	1.5-2.5
Al_2O_3	0-0.3
Er_2O_3	0-0.5
Fe_2O_3	0-0.3

Ref: VITA YZ Technical and scientific documentation, VITA Zahnfabrik

Table 1.2: Composition by wt.% of VITA AKZENT Plus Glaze.

Components	wt.%
Ceramics	10-20
Isobutane	75-<80
Ethanol	5-10

Ref: VITA AKZENT Plus Glaze, VITA Zahnfabrik surface" to describe the results and discuss the outcomes of the experiment being carried.

Indexes to mark the area for X-ray diffractometry (XRD), contact profilometry, and scanning electron microscopy (SEM) were fabricated from Duralay pattern resin (Reliance Dental, USA). A portion of the pattern resin would be adapted to ensure that the occlusal, proximal, and buccal surfaces are aligned to the horizontal plane to facilitate proper positioning on the mounting stands of the XRD, contact profilometer, and SEM. Another portion would be fabricated to serve as the index to mark the controlled area for the surface to be adjusted and studied. On the area to be examined, a 2 x 2 mm^2 concentric circle would be drawn before being drilled with a cylindrical carbide bur with a micromotor. Figure 1 shows the pattern resin indexes that were constructed for this study.

Initial examination of the zirconia crowns was conducted prior to chairside adjustments. The zirconia crowns were subjected to X-ray diffractometry with θ-2θ diffractometer (Rigaku, Japan) using Cu-Kα radiation. The percentage of monoclinic and tetragonal phases were determined by Diffractograms with the banned from 8°-180° at a scan speed of 2°/min and a step size of 0.02°. Initial surface roughness (R_a) was examined by the use of contact profilometer (Talyscan 150, Taylor Hobson, UK). All restorations were mounted on an XY cross-table of the profilometer. Five readings would be recorded with a traveling distance of 2 mm across the surface around each point of occlusal contact. The profilometer was calibrated to have a cut off value would be 0.25 mm, stylus speed of 0.5 mm/s, and a vertical distance of 0.4 mm and the surface morphology of zirconia specimens were examined under scanning electron microscope (SEM, JSM 7800PRIME, JEOL, Japan) at 20,000X magnification at 10.00 kV.

Evaluation after polishing and annealing

The ten samples included in the study were categorized into two groups based on occlusal and proximal surface modifications prior to delivery of the restoration: the polished group and the annealed group which underwent heat treatment in Vita Vacummat 4000T Premium Furnace. Each group will contain 5 specimens consisting of restorations replacing both molars and premolars as shown in table 1.3.

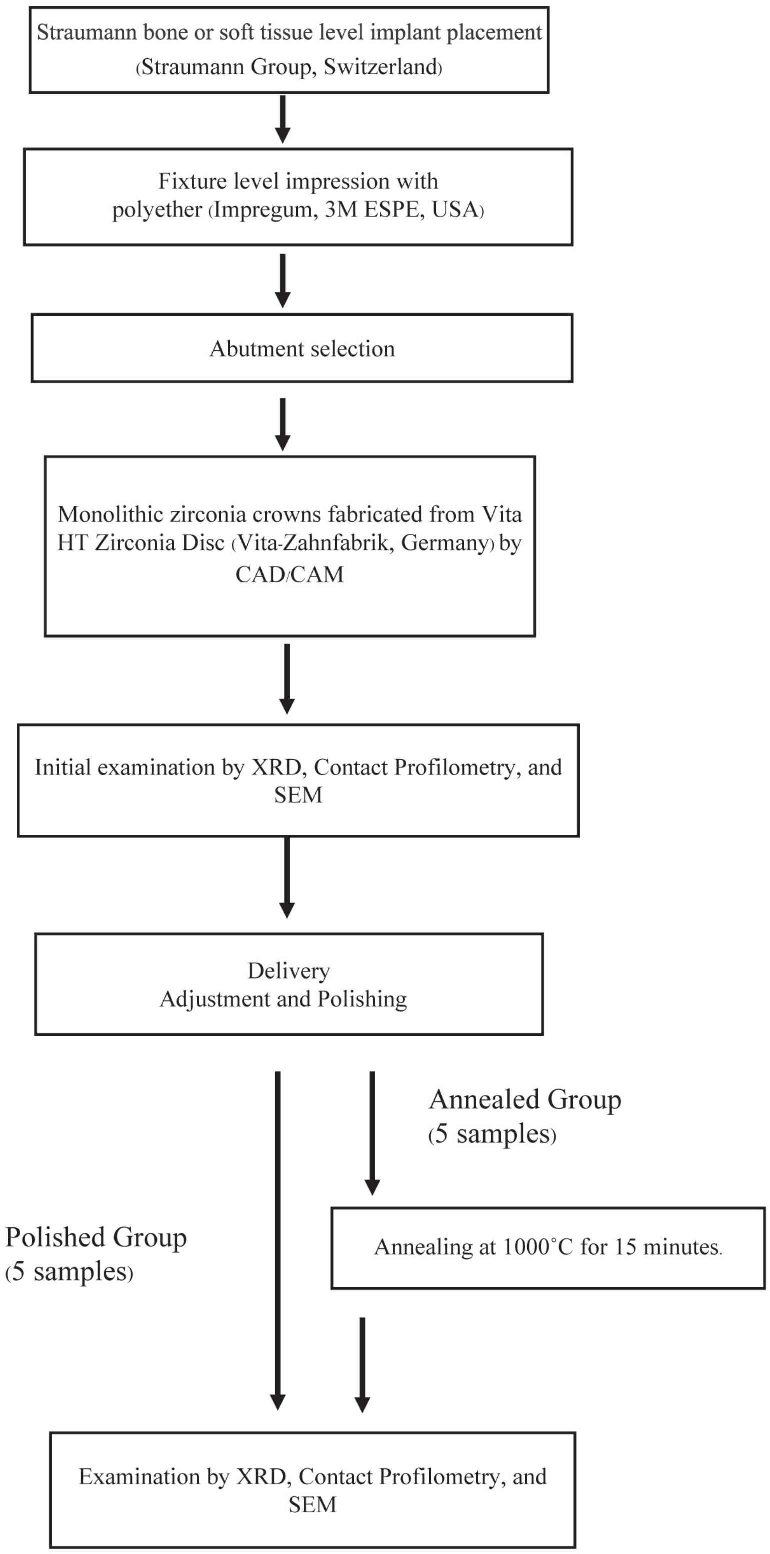
After initial examination of the restoration, all restoration were adjusted chairside during trial visit in real patients. Proper adjustments were made at the proximal contacts and occlusal surface of the restoration by a single calibrated operator. Diamond impregnated pre-polishing bur for adjusting zirconia and alumina (Diacera, EVE Ernst Vetter, GmbH, Germany) was used from gross adjustment at 10,000 rpm with a pressure of approximately 2N. The restoration should have good proximal contact when tested with dental floss and should be able to hold shim stock tight when the patient bites firmly. The occlusal and proximal contacts for the samples belonging to the polished group were polished with a medium and fine diamond impregnated silicone disc for polishing zirconia and alumina (Diacera Twist, EVE Ernst Vetter, GmbH, Germany) at the same speed and pressure as the gross

Table 1.3: Classifcation of sample groups.

Teeth	**Annealed**	**Polished**	**Total**
Premolars	2	2	4
Molars	3	3	6
Sum	5	5	10

adjustment made earlier. For the annealed group, after adjustment with Diacera polishing kit, the samples underwent heat treatment at 1000°C in Vita Vacumat Premium 4000T furnace (VITA Zahnfabrik, Germany) for 15 minutes, followed by slow cooling at the rate of 25°C/min.

The flow chart below summarized the procedure being carried for conducting the ex-periment as mentioned above:



Specimen belonging to both groups were then re-examined by means of X-ray diffractometry, contact profilometry, and scanning electron microscopy following the same protocols as during initial examination prior before cementation.

Statistical analysis

The data obtained from the two periods, before and after delivery of the final restoration, were analyzed by using statistical analysis software (SPSS 22.0, SPSS Inca, Chicago, IL, USA).

The relative amount of transformed monoclinic zirconia was first analyzed by descriptive statistics. The data obtained was then be tested for normal distribution by Shapiro-Wilk test. Data comparison between two timeframes were then compared using paired sample t-test.

Results

The crystalline phases zirconia crowns were studied under X-ray diffractometry at the occlusal, proximal and buccal surfaces, and changes in the percentage of tetragonal phase could be observed in both test groups. Unlike the buccal and proximal surfaces, rhombohedral phase was observed on the occlusal surface of all the crowns.

Table 2.1 shows mean of the percentage of the crystalline phases and surface roughness (Ra) on the occlusal surface, buccal surface and proximal surface of the crowns and the surface roughness of the two test groups.

The mean percentage of tetragonal phase at the initial point prior to polishing and annealing were 67.74 ± 15.36 and 91.97 ± 4.73 respectively on the occlusal surface, 87.84 ± 2.49 and 92.63 ± 2.67 on the

Table 2.1: Percentage of crystalline phase of the two test groups.

Surface	Modification	% Tetragonal (before:Tb)	% Tetragonal (after:Ta)	Sig. (2-tailed)	%Rhombohedral (before:Rhb)	%Rhombohedral (after:Rha)	Sig. (2-tailed)	%Monoclinic (before: Mb)	%Monoclinic (after: Ma)	Sig. (2-tailed)
Occlusal	Annealed	67.74±15.36	85.19±13.48	**0.000***	32.85±14.89	9.12±8.54	0.002	N/A	5.69±5.19	**0.070**
	Polished	91.97±4.73	89.00±5.19	**0.018***	8.03±4.73	10.17±4.58	0.057	N/A	1.44±2.33	0.241
Buccal	Annealed	87.84±2.49	97.78±4.46	**0.002***	N/A	N/A	N/A	12.15±2.49	2.22±4.46	**0.002**
	Polished	92.63±2.67	88.89±1.74	**0.014***	N/A	N/A	N/A	7.37±2.67	11.11±1.74	**0.014**
Proximal	Annealed	88.34±2.57	90.66±2.64	**0.004***	N/A	N/A	N/A	11.66±2.58	9.35±2.64	**0.004**
	Polished	87.31±1.50	84.22±2.62	**0.043**	N/A	N/A	N/A	12.76±1.34	15.77±2.62	0.051

buccal surface; and 88.34 ± 2.57 and 87.31 ± 1.50 respectively on the proximal surface. Monoclinic phase, initially, was not present in the annealed group on the occlusal surface. On the other hand, rhombohedral phase was observed with the mean of 32.85±14.89 for the annealed group and 8.03 ± 4.73 for the polished group. On the buccal and proximal surfaces, the mean percentage was 12.15±2.49 and 7.37±2.67, and 11.66 ± 2.58 and 12.76 ± 1.34 for the annealed and polished group respectively.

Table 2.2 shows the surface roughness of the two test groups. The mean occlusal surface roughness after annealing was 0.56±0.06 and 0.47±0.24 at the polished surface. For the buccal surface, the mean surface roughness was 0.31±0.08 for the annealed group and 0.25±0.10 for the polished group. On the proximal surface, the annealed group possess the mean surface roughness of 0.27 ± 0.05 and the polished group had the mean surface roughness of 0.32±0.09.

After the samples belonging to both groups had undergone polishing and annealing, changes in the crystalline phase and surface roughness was observed as shown in Table 2.1. The results on all surface tested showed general increase in tetragonal phase and a decrease in monoclinic and rhombohedral phase in the samples being annealed. The increase of surface roughness, on the other hand, were seen in both the polished and annealed groups.

Data comparison between two periods of the the experiment was done with paired sample t-test. At the occlusal surface, statistically significant difference between before try in and after try in group were founded, where p-value < 0.05, was seen in percentage of tetragonal and rhombohedral phases in both annealed and polished group, and the surface roughness of both groups.

Significant difference between before try in group and both after try in group were seen in percentage of tetragonal and monoclinic phases in the annealed and polished groups (p-value<0.05) on the buccal surface. However, the difference of surface roughness at both time intervals were not statistically significant for the two test groups.

Finally on the proximal surface, significant difference between before try in group and after try in group were seen in percentage of tetragonal phase, the annealed and polished groups, monoclinic phase in the annealed group, and the surface roughness for both groups (p-value < 0.05). Only the percentage of monoclinic phase belonging to the polished group did not exhibit significant difference.

The scanning electron microscopic image of the occlusal, buccal and proximal of zirconia crowns after undergoing polishing with Diacera Twist diamond impregnated silicone burs (EVE Ernst Vetter GmbH, Germany) and annealing in Vita Vacummat 4000T

Table 2.2: Surface roughness of the two test groups.

Surface	Modification	Ra (before: R_b)	Ra (after: R_a)	Sig. (2-tailed)
Occlusal	Annealed	0.56±0.06	0.66±0.06	0.006
	Polished	0.47±0.24	0.56±0.24	0.000
Buccal	Annealed	0.31±0.08	0.34±0.09	**0.259**
	Polished	0.25±0.10	0.25±0.10	**0.178**
Proximal	Annealed	0.27±0.05	0.31±0.05	0.000
	Polished	0.32±0.09	0.46±0.11	0.001

Premium furnace (VITA Zahnfabrik, Germany) at 1000°C for 15 minutes are shown in Figure 2. The surfaces that underwent major adjustments, namely the occlusal and proximal surfaces are presented with pores and cracks. The SEM images obtained concurred with the measurements of surface roughness by means of contact profilometer as surface roughness significantly increased. The unadjusted buccal surfaces did not exhibit visible change of the surface morphology when subjected to SEM imaging.

Figures 3-5 show the percentage of crystalline structure before and after the being polished and annealed. The tetragonal and rhombohedral peaks of monolithic zirconia crown on the occlusal surface before and after polishing and annealing is shown in Figure 3. The green line represents the peaks after annealing was done. The yellow line represents the initial tetragonal and rhombohedral peaks. The orange line represents the tetragonal and rhombohedral peaks after polishing was done. Counts of tetragonal peaks were highest in the annealed group, where as a decrease in tetragonal peaks could be observed after polishing was done as monoclinic peaks emerged.

Figure 4 shows the tetragonal and monoclinic peaks on the buccal surface before and after polishing and annealing. The green line represents the peaks after annealing was done. The yellow line represents the initial tetragonal and rhombohedral peaks. The orange line represents the tetragonal and rhombohedral peaks after polishing was done. Counts of tetragonal peaks between before and after polishing were not different since no alteration was done on the buccal surface. However, after annealing, a significant rise of tetragonal peaks could be observed.

Figure 5 shows the tetragonal and monoclinic peaks after polishing and annealing was done on the proximal surface of the monolithic zirconia crowns. The green line represents the peaks after annealing was done. The yellow line represents the initial tetragonal and rhombohedral peaks. The orange line represents the tetragonal and rhombohedral peaks after polishing was done. Similar to the XRD results from the occlusal and buccal surfaces, a shoot of tetragonal peaks could be seen after annealing was carried out. Monoclinic peaks were more frequent after polishing was done.

X-ray diffractometry showed that monoclinic peaks were not present on the occlusal surface prior to adjustments made to the milled zirconia crowns. Traces of monoclinic peak was present on the buccal and proximal surfaces. Once the crown underwent annealing at 1000°C for 15 minutes, an increase of tetragonal peak could be seen on all surfaces being inspected, especially on the unadjusted buccal surface. In contrary, the crowns that were polished showed an increase of monoclinic peaks on all surface and an increase of rhombohedral peaks on the occlusal surface.

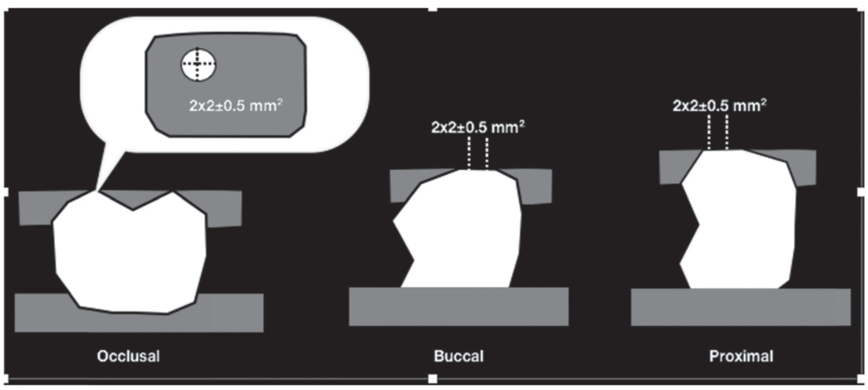


Figure 1: Pattern resin indexes made to mark the area examined under XRD, Contact Profilometry, and SEM on the occlusal, buccal and proximal surfaces.

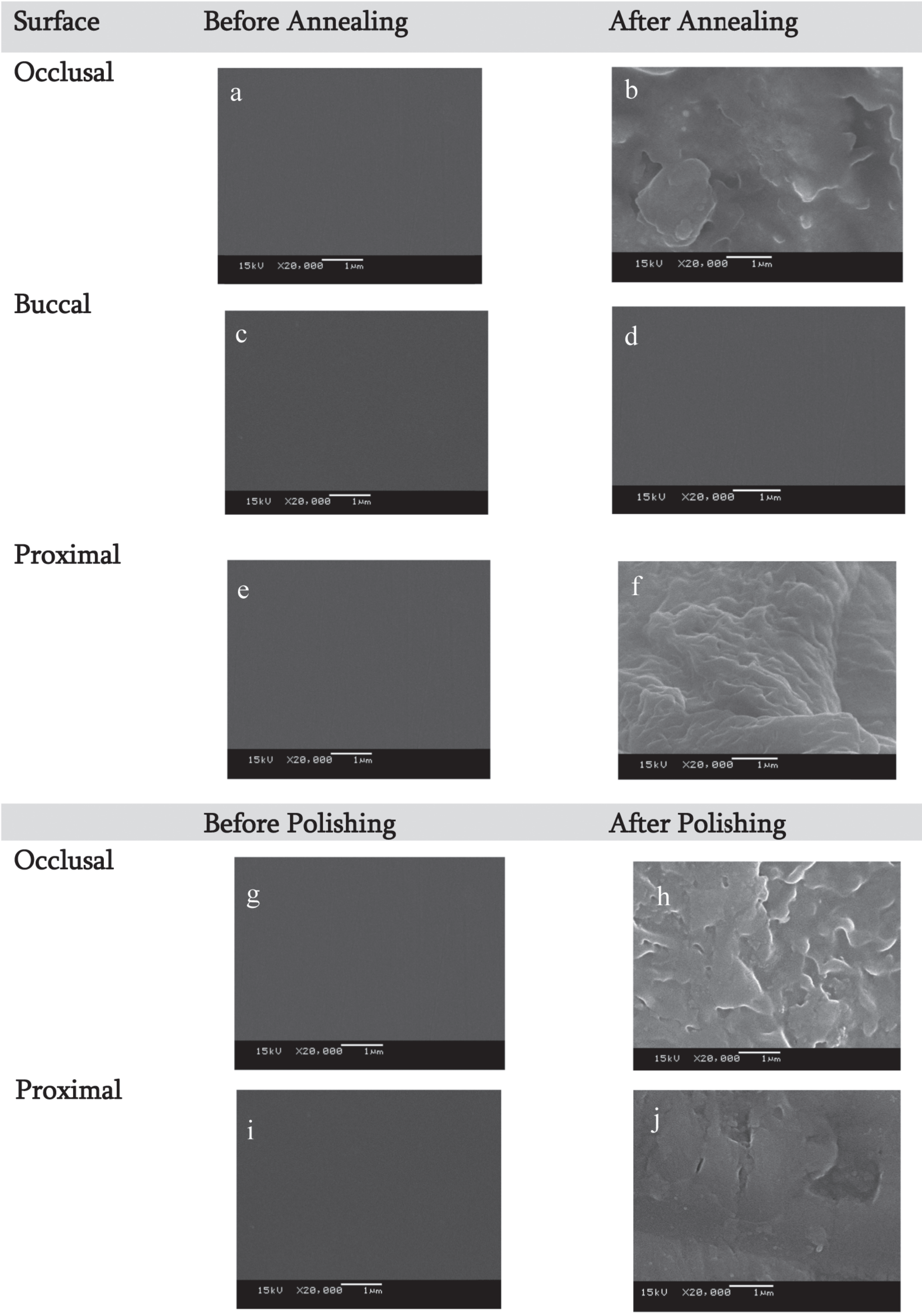


Figure 2: SEM images at 20,000X magnification of zirconia crowns before and after polishing and annealing at the occlusal, buccal, and proximal surfaces.

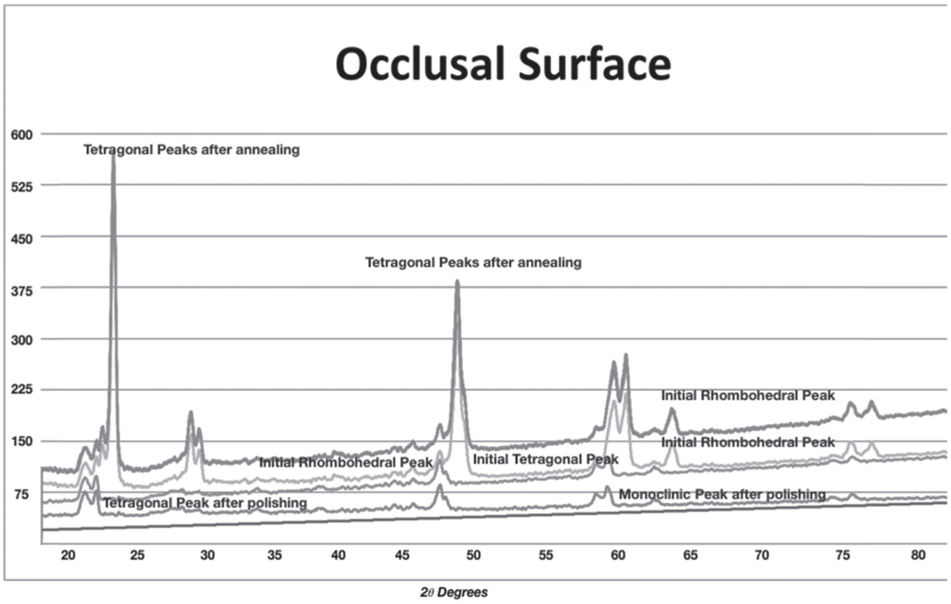


Figure 3: The tetragonal and rhombohedral peaks of monolithic zirconia crown on the occlusal surface before and after polishing and annealing.

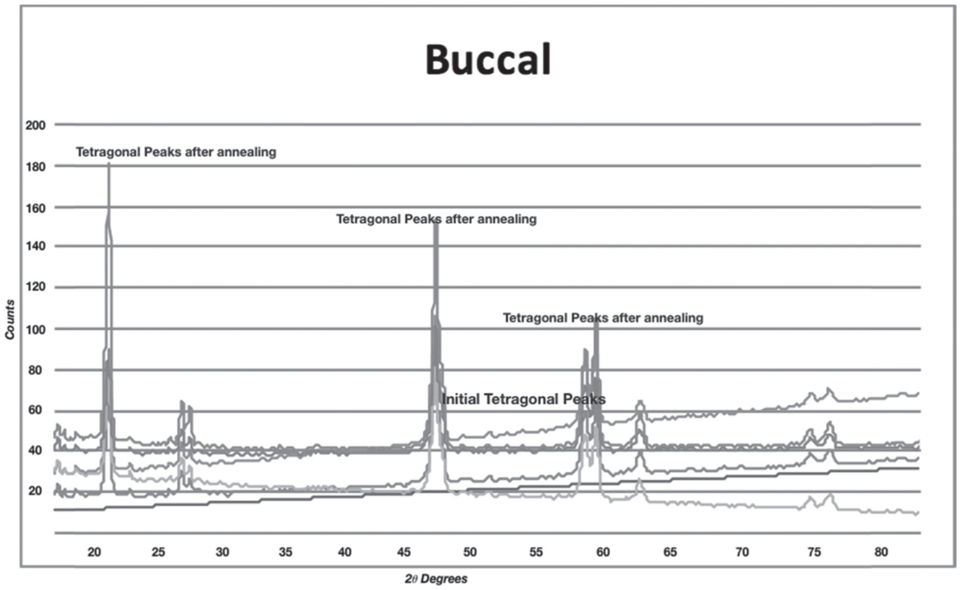


Figure 4: The tetragonal and monoclinic peaks of monolithic zirconia crown on the buccal surface before and after polishing and annealing.

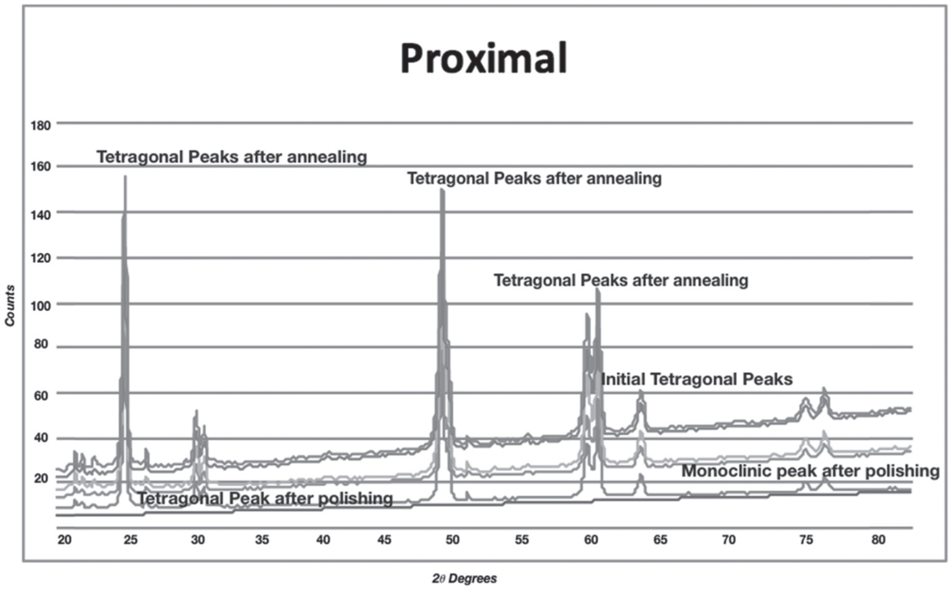


Figure 5: The tetragonal and monoclinic peaks of monolithic zirconia crown on the proximal surface before and after polishing and annealing.

Discussion

The structure of crystalline phase in zirconia was evaluated by X-ray diffractometry. It works through continuous beam of X-rays which are incident to the crystalline structures of the specimen. The diffracted radiation is intense in directions with correspond to constructive interference form waves reflected from the layers of the crystal. Computer software then detect constructive interference in forms of peaks which correspond to specific crystalline structure present in the specimen.

Changes in the percentage of tetragonal phase could be observed in both groups after the crystalline phases zirconia crowns were studied under X-ray diffractometry at the occlusal, proximal and buccal surfaces. Rhombohedral phase was only observed on the occlusal surface of all the crowns, while other surfaces exhibited only tetragonal and monoclinic phases. Significant different between before try in group and after try in group (p-value < 0.05) was observed in the percentage of tetragonal and rhombohedral phases of the resintered and polished group.

The presence of a different form of zirconia identified as rhombohedral zirconia on the abraded surfaces of partially stabilised zirconia was first reported by Hasegawa in 1983. This phase was observed at all levels of grinding and for various amounts of yttria dopant, even in the fully stabilised cubic zirconia (Hasegawa, 1993). Kim et al. showed that the low-temperature degradation of 3Y-TZP caused t-ZrO_2 to transform to r-ZrO_2 in addition to the formation of m-ZrO_2 (Kim et al., 1995). Kitano et al., and Ruiz and Ready also suggested that the r-phase on the ground zirconia surfaces was formed from t-phase through transformation under mechanical stress (Kitano et al., 1988a; Ruiz and Ready, 1996). Recently, Kondoh discussed the legitimacy of the rhombohedral phase and concluded that the shoulder observed by XRD on the (0,1,1) reflection of the tetragonal specimens after abrasion was caused by lattice distortion and not by the presence of rhombohedral phase (Kondoh, 2004).

Due to the complexity of anatomical features of the occlusal surface of posterior teeth, a more complex milling procedure was needed when compared to other smooth surfaces such as the buccal, proximal and lingual surface. Such kind of milling could result transformation of tetragonal to rhombohedral phase according to Hasegawa et al. The explanation provided by Kondoh may also be considered plausible. Abrasion of the milling tip onto the surface of the 3Y-TZP disc may as well cause lattice distortion after the procedure.

According to Denry and Holloway, the amount of strain during *t* to *r* transformation is less than of *t* to *m* transformation but with similar surface damage. Grinding of 3Y-TZP ceramic induced the formation of a rhombohedral phase and strained tetragonal zirconia phase. This led to a significant increase mean flexural strength and increased resistance to crack propagation but was associated with surface and subsurface damage, with formation of micro craters and grain pull out. Only tetragonal zirconia remained after annealing (Denry and Holloway, 2006).

On the proximal surface, significant difference was seen in percentage of tetragonal phase in the annealed and polished groups, monoclinic phase in the annealed group, and the surface roughness for both groups (p-value < 0.05). Though milling on the proximal surface may not be as complex as the occlusal surface, but several adjustments were made during try-in to seat the crown on the implant abutment.

Garvie et al. first suggested that grinding increased the strength of ceramics containing metastable tetragonal zirconia compared with fine polishing, due to *t* to *m* transformation associated with the development of surface compressive strains from unit cell volume change. However, fine polishing eliminates some of the transformed monoclinic phase, therefore reducing the surface strains (Garvie et al., 1975). Reed and Lejus confirmed the presence of lattice strain and *t* to *m* transformation after grinding of Y-TZPs. These surface changes were associated with an increase in hardness to a depth of about 4 µm (Reed and Lejus, 1977). More recent works on wear of partially stabilized zirconia have shown that the *t* to *m* transformation was indeed by wear, machining, or possibly by thermal treatment in an unstabilized powder bed. This is associated with the development of surface-compressive stresses, which prevent microcrack formation but promote subsurface damage by delimitation and grain pullout. (Hooper et al., 1989; Hu et al., 1993; He et al., 1996; Conoci et al., 1999; Kao et al., 2000)

During grinding, tens of microns of material were removed each time the bur moved back and forth across the surface and sparks were commonly observed, indicating that both stresses and temperatures were high during this procedure. Due to high stresses developed during grinding, severe surface cracks could be formed which lowered the strength and reliability of the material. According to Kosmac et al., grinding induced crack could extend into the bulk of the material about 50µm from the ground surface. Further more, the authors found that by using water spray during grinding the calculated value of critical defect size was reduced by 30%, indicating lower stresses during wet grinding. A further reduction of critical defect size can be expected by using a finer diamond bur (Kosmac et al., 1999). This is confirmed in the SEM images obtained after the adjustments were made on the occlusal and proximal surfaces as seen in Figure 2. Pitted surfaces, crack formation and surface grain pullouts could be noted on the surfaces being adjusted by diamond burs and diamond impregnated silicone burs used during adjustment and polishing. This concurred with the readings obtained from contact profilometry as increase in surface roughness was seen in all groups after delivery.

The buccal surface showed significant difference before and after annealing and polishing as seen in percentage of tetragonal and monoclinic phases between the two groups (p-value < 0.05). Though the buccal surface was never adjusted during delivery. The difference in percentage of crystalline phase obtained may be due to gripping force applied and the use of emery powder with GC pliers. Repetitive gripping with GC pliers and emery powder may result in mechanical stress due to abrasion on the buccal surface, though grinding and polishing was not done on the surface. Regardless of the force applied through the use of GC pliers during try-in, the difference of surface roughness at both time intervals were not statistically significant for the two test groups on the buccal aspect.

Unlike the polished zirconia crowns, the crowns that underwent annealing in Vita Vacummat 4000T Premium furnace (Vita-Zahnfabrik, Germany), did show an increase in tetragonal phase content. Such change is especially noted on the unadjusted surface which is the buccal surface. As seen from Table 2.1, a mean percentage of 97.78 ± 4.46 tetragonal phase could be observed at the group after annealing. The occlusal surface and proximal surface yielded the average percentage of tetragonal phase of was 85.19 ± 13.48 and 90.66 ± 2.64 respectively. This finding was similar to that of Kitano in 1988. Kitano reported that the reversal was complete after annealing at 1000°C for 1 hour. Nevertheless, a consensus linking phase transformation and surface or subsurface damage, such as roughness, microcraters, and grain pullout usually reported throughout the literature. It was shown that grinding can be detrimental to both strength and reliability of Y-TZP ceramics and confirmed that surface treatments generally trigger the t to m transformation, although there was no mention of the presence of a rhombohedral phase (Kitano et al., 1988b).

Conclusion

Within the limitations of this study, it was shown that finishing and polishing does affect the surface morphology of zirconia restoration, crystalline phase of zirconia, and surface roughness of zirconia. Changes in tetragonal phase content was significant after adjustments were made. Phase transformation from tetragonal phase to monoclinic phase, resulting in volume expansion of the crystalline structure is associated with the development of surface-compressive stresses, which prevent microcrack formation but promote subsurface damage by delimitation and grain pullout.

Annealing at 1000°C for 15 minutes with the use of Vita Vacummat 4000T Premium furnace (Vita-Zahnfabrik, Germany), did show an increase in tetragonal phase content. Higher tetragonality could lessen the development of subsurface damage from delimitation and grain pullout.

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