Topography, Microhardness, and Precision of Fit on Ready-Made Zirconia Abutment Before/After Sintering Process

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ABSTRACT

Background: Sintering porcelain on a ceramic abutment may change the microstructure and result in aging processes that influence the mechanical properties, internal strain, and the three-dimensional form of the abutment, thus causing a possible misfit between the abutment and the fixture.

Purpose: The aim was to investigate topography, microhardness, and precision of fit on yttrium-stabilized zirconia (Y-TZP) abutments before/after the sintering process.

Materials and Methods: Ten Y-TZP abutment samples were ground to a shape used in the clinical situation and divided at random into two groups: before/after sintering. After the surface roughness was measured on all abutments, the abutments were connected to fixture replicas, embedded in resin, and cut in the longitudinal axis. Both sides of the cut samples were measured with respect to microhardness and minimum distance between fixture and abutment surface. *t*-Test, one-way analysis of variance, and Bonferroni multiple comparisons were used to investigate statistical significant differences.

Results: The surface roughness (S_a and S_{dr}) after sintering was significantly higher than before sintering. The total average values of microhardness after sintering were statistically lower than before sintering with a difference of 2%. The total distance between abutment/fixture before/after sintering demonstrated no statistically significant difference. Contact between abutment/fixture was most common at the top area of the fixture.

Conclusion: A slight decrease of microhardness and contamination of porcelain particles immediately below the veneered part were found on the Y-TZP abutment after sintering. The sintering process did not affect the precision of fit.

KEY WORDS: ceramic abutment, dental implant, mechanical properties, precision of fit, surface roughness, Y-TZP

A lot of implant research has since more than a decade been focused on the bone integration toward various topographies. Treatments with oral implants have shown a high long-term survival rate on

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turned and blasted surface implants.^{1,2} Today, the research interest seems to be changing from surface topography to surface chemistry^{3,4} with the ultimate purpose to be able to load the implants early or immediately. Concerning the superstructures, an increased use of all-ceramic materials for obtaining the most esthetic results^{5,6} can be observed. At present, investigations on abutments are often related to the biological relationship between the crestal bone and abutment-fixture interface,^{7,8} and esthetic concerns have resulted in the use of alumina and zirconia abutments.^{9–11}

The alumina is characterized by good biocompatibility, corrosion resistance, mechanical properties, and low thermal conductivity, compared to silicate-based ceramics.^{12–14} However, because of the sensitivity to microstructural flaws which lead to a poor resistance to stress concentration or mechanical impact, it was

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reported that the strength of alumina abutments was only slightly higher than the estimated maximum physiologic forces on anterior teeth.¹⁵ In several clinical prospective studies on implant-supported single crowns and fixed partial dentures (FPDs), some fractures of the custom-made alumina abutments have been reported.^{16,17}

The zirconia has a flexural strength and fracture toughness about twice as high as alumina¹⁸ and has been reported to have superior biocompatibility.¹⁹ The material is frequently used as material for hip joint heads in orthopedics,^{20,21} root canal posts,²² and all-ceramic posterior FPDs²³ in dentistry. In a clinical prospective study using custom-made zirconia abutment for single-tooth restorations, no abutment fractures were reported during a 4-year follow-up period.²⁴ However, problems with screw loosening have lately been observed.^{24,25}

In general, abutment screw loosening at functional loading may be caused by a slight rotational freedom or misfit between fixture and abutment, resulting in a relative mobility between these two components. Several in vitro studies have demonstrated that the absence of the rotational freedom or misfit makes the abutment more resistant to loosening.^{26,27} Mechanically, the zirconia has a stress-induced transformation toughening mechanism by volume expansion from tetragonal to monoclinic phase transmission.²⁸ It has been suggested that the sintering process may cause changes of the microstructure²⁹ and result in aging processes by the altered thermal environment.³⁰ If these changes will influence the general mechanical properties, internal strain, and the threedimensional form of the zirconia abutment, it may cause a misfit between abutment and fixture. So far, to the knowledge of the present authors, no studies on the effects of the sintering process on the precision of the zirconia abutment-fixture contact and mechanical properties have been published.

The purpose of this in vitro study was to investigate topography, microhardness, and the precision of fit of ready-made zirconia abutments before and after the veneer porcelain sintering.

MATERIALS AND METHODS

The study design is demonstrated in Figure 1. Ten yttrium-stabilized zirconia (Y-TZP) abutment samples (Ceramic Abutment® 4.5/5.0; Astra Tech AB, Mölndal, Sweden) were used for this study. All 10 abutments were connected to fixture replicas (Fixture Replica® 5.0 ST,



Figure 1 Flowchart of study design.

Astra Tech), and were ground as in clinical practice (Figure 2) using preparation diamonds especially manufactured for zirconia grinding (Diagen® turbo grinder, Bredent, Sendent/Witzighausen, Germany) under recommended water-cooling procedure. After grinding, all samples were ultrasonic cleaned in water and thereafter steam autoclaved. The 10 samples were divided at random into two groups; five abutments were left after the grinding procedure and functioned as controls, the "grind group." Five samples were included in the "sintered group." The specimens of the sintered group were given a simplified premolar form by use of the porcelain (GC initial Zr; GC) recommended by the manufacturer



Figure 2 Detailed sample shapes after grinding and sintering.

TABLE 1 The Detailed Sample Sizes After Grinding and Sintering (After Grinding, $n = 10$; After Sintering, $n = 5$)								
	а	b	с	d	е	f	g	
Average (mm)	12.1 1	4.93	5.5	3.92	3.46	13.0 4	6.69	
SD (mm)	0.03	0.05	0.01	0.04	0.04	0.34	0.32	

(see Figure 2). The dimensions and anatomic form were standardized by use of a form guide. Detailed firing steps undertaken followed the recommended procedure from the manufacturer (low temperature: 450-480°C; high temperature: 690-830°C; temperature increase rate: 45°C/min; time of heating process: approximately 1 hour). The sample shapes and sizes after grinding and sintering are shown (see Figure 2; Table 1). All samples were manually measured by an electronic digital caliper. After the topographical measurement, all abutments in both groups were connected to fixtures (MicroThread® 4.5 ST type 13mm, Astra Tech), embedded in resin (Technovit® 7200 VLC; Heraeus Kulzer GmbH & Co., Wehrheim, Germany), and cut in the longitudinal axis of the abutment/fixture by use of a low-speed saw (Isomet® 11-1180, Buehler, Coventry, UK) with diamond wafer blade (BUEHLER®, 0.3 mm thickness). One side that had a polishing overlap width was polished by use of a polishing machine (RotoPol-21®, Struers AB, Bromma, Sweden) and resin-bonded diamond grinding disk (MD-Piano 220 and 1200, Struers).

Topography

The topography was measured by an interferometer (Micro-XAM[®], Phase-Shift, Inc., Tucson, AZ, USA). Three measuring points of each sample are shown in Figure 3. All measurements had a size of $250 \times 200 \,\mu$ m. The measuring points were defined as follows: A, the widest area of abutment; B, the external slope area between A and the abutment/fixture connection; and C, the area close to the abutment/fixture connection.

Before numerical characterization, a Gaussian filter sized $50 \times 50 \,\mu\text{m}$ was used to eliminate errors of form. For numerical evaluation, four surface roughness parameters were used to describe the topographical variation in height, space, and surface enlargement: S_a (μ m), the average height deviation from a mean plane; $S_{\rm ds}$ (1/µm²), the density of summits; $S_{\rm sk}$ (µm), the height distribution; and $S_{\rm dr}$ (%), the ratio between the measured surface in three dimensions and a complete flat reference plane (two dimensions).

The results were compared on the following conditions:

- 1. Total average values included in points A to C were compared between the two groups.
- 2. Each average of points A to C was compared between the two groups.
- 3. Multiple comparisons among points A to C were performed within each group.



Figure 3 Schema of one side measuring points in topography, microhardness, and microgap measurement. Points A to C are for topography measurement. Points a to g are for microhardness measurement. Points x to z are for microgap measurement. The other side was also measured with same to one side in all measurements.

Microhardness

The microhardness was measured with a Micro Vickers Hardness equipment (Digital Microhardness Tester FM-100e, FUTURE-TECH CORP., Tokyo, Japan). The load (500g) was applied to the surface of the abutment with a dwell time of 15 seconds. The area was observed by 50× microscope and measured with the help of a computer. Seven measuring points on the abutment were selected, a to g on each left and right side (see Figure 3), that is, a total of 14 points in each sample: a to c, the middle part of the ground area (thin area); d and e, the widest area of the abutment (thick area); and f and g, the area of the fixture top (middle thickness area).

The distances of points a, c, d, and f from surface in the grind and sintered groups were $158.2 \,\mu m \,(\text{SD} = 36.6)$ and $150.0 \,\mu m \,(\text{SD} = 39.8)$, respectively. Points b, e, and g were also defined on the middle part between the outer and inner surfaces. A total of five measurements on all of the seven points were performed on all the samples. The mean value was assumed to be a representative value of each measurement point. The results were compared on the following conditions:

- 1. Total average values included in points a to g were compared between the two groups.
- 2. Each average of points a to g was compared between the two groups.
- 3. Multiple comparisons among points a to g were performed within each group.

Precision of Fit

The precision of fitness was studied by the use of a $50\times$ metallurgical microscope (ECLIPSE ME600L, Nikon, Tokyo, Japan). The measurements were evaluated in a computer equipped with an appropriate software. Three measuring points between abutment and fixture were used and designated as x, y, and z on each left and right side (see Figure 3), that is, a total of six measuring points in each sample: x, the area at a distance of $100\,\mu\text{m}$ from the top of the fixture; y, the middle $100\,\mu\text{m}$ area of the abutment and the fixture contact area; and z, the $100\,\mu\text{m}$ area from the bottom of the abutment.

The measurement was repeated five times in each measuring point. The mean value was assumed to be a representative value of each measurement point. The results were compared on the following conditions: contact or not between abutment and fixture in the grind and sintered groups. If there was a contact in either the left side or right side, the measuring point was regarded as "contact."

The minimum distances between abutment and fixture in each measuring area were compared on the following conditions:

- 1. Total average values included in points x to z were compared between the two groups.
- 2. Each average of points x to z was compared between the two groups.
- 3. Multiple comparisons among points x to z were performed within each group.

Statistical Analysis

The population was assumed to process normal distribution in the topographical, microhardness, and precision of fitness data. Statistical significance of differences was analyzed with *t*-test (comparison between the two groups), and one-way analysis of variance and Bonferroni multiple comparisons (comparison among multipoints) at a significance level of 0.05%, respectively. A statistical add-in software for EXCEL (Esumi Corp., Tokyo, Japan) was performed for the statistical analysis.

RESULTS

External Observations

After grinding the Y-TZP abutments, a faint gray line was observed on all abutment surfaces corresponding to the top of the fixture replica made by stainless steel (Figure 4; after grinding). The gray line persisted in all samples even after they had been ultrasonically cleaned. After sintering, a faint red line was also observed at the same location on the abutment surfaces as the gray line (see Figure 4).

Topography

The total average values of the topographical parameters before and after sintering were calculated (Table 2). The total average value of the parameters S_a and S_{dr} in the sintered group demonstrated a statistically higher value (p < .05) than the corresponding grind group. The summary of the S_a and S_{dr} average values of each measuring point in the two groups are shown in Table 3. Comparing the S_a and S_{dr} average values before and after sintering, statistically significant differences (p < .05) were found only in point A. In the grind group, the S_a and S_{dr} average values of point B displayed significantly higher value than points A and C. In the sintered group,



Figure 4 External observations after (A) grinding and (B) sintering processes on yttrium-stabilized zirconia abutment. Gray line on the abutment after grinding (left side); red line on the abutment after sintering (right side).

the S_a and S_{dr} average values of point A demonstrated significantly higher value than points B and C. However, statistically significant differences (p < .05) on multiple comparisons among all the measuring points were only shown in the S_a value, that is, point A versus point B in the grind group, and point A versus point C in the sintered group, respectively.

Microhardness

The total average values of the microhardness in the grind and sintered groups were 1, 248.2 Hv (SD = 34.0) and 1, 232 Hv (SD = 25.4), respectively. A statistically significant difference (p < .01) was found between the two groups. The average value of each measuring point in

TABLE 2 Total Average Values of Four Topographic Parameters in the Grind and Sintered Groups (Statistical Difference $p < .05$)*						
	S _a (μm) Average (SD)	S _{ds} (1/μm²) Average (SD)	S _{sk} (μm) Average (SD)	S _{dr} (%) Average (SD)		
Grind	0.38 (0.25)	154, 800 (33, 393)	6.61 (10.30)	8.02 (9.35)		
Sintered	0.83 (0.62)	151, 796 (29, 001)	4.80 (8.63)	21.19 (7.59)		
p Value	0.017*	0.794	0.607	0.018*		

TABLE 3 Summary of S_a and S_{dr} Average Values of Each Measuring Point in the Grind and Sintered Groups (Statistical Difference p < .05)*

	S _a (μm)			S _{dr} (%)		
	Grind	Sintered		Grind	Sintered	
Point	Average (SD)		p Value	Average (SD)		<i>p</i> Value
А	0.22 (0.03)	1.38 (0.59)	0.011*	3.07 (2.27)	33.93 (21.60)	0.034*
В	0.62 (0.33)	0.74 (0.59)	0.681	15.38 (13.62)	20.04 (14.11)	0.610
С	0.31 (0.04)	0.37 (0.16)	0.393	5.62 (3.06)	9.60 (6.70)	0.262
		p Value			p Value	
A versus B	0.007*	0.061		0.034	0.178	
A versus C	0.490	0.006*		0.629	0.027	
B versus C	0.026	0.253		0.083	0.301	



Figure 5 Microhardness average value of each measuring point (a to g) in grind and sintered groups (*p < .05).

the two groups was evaluated (Figure 5). The average values of points a, b, d, e, and f in the grind group were higher than in the sintered group. However, a statistically significant difference (p < .01) was found only in point b. The multiple comparisons, including all the measuring points in the grind and sintered groups, showed no statistically significant difference.

Precision of Fit

In both groups, four samples out of five (4/5) in point x, one sample out of five (1/5) in point y, and one sample out of five (1/5) in point z displayed contact between the abutment and the fixture. The total average values in the grind and sintered groups were $1.17 \,\mu m \,(SD = 1.14)$ and $1.80 \,\mu m$ (SD = 1.61), respectively; however, there was no statistically significant difference. The average value of each measuring point is shown in Figure 6. Even though all the points in the grind group were lower than those in the sintered group, a statistically significant difference was not found between the two groups. The multiple comparisons among all the points in the grind group showed statistically significant difference in point x versus point z, and in point y versus point z. Also in the sintered group, there was a statistically significant difference in point x versus point z.

DISCUSSION

After grinding, there was a faint gray line in the area corresponding to the top of the fixture replica. Probably, the abutment had been slightly stained by stainless steel particles caused by wear from contact with the fixture. The change of color from gray to red may be the oxidation of the stainless steel caused by heating at sintering. If the corresponding stain occurs when a titanium fixture is used, and if this has any importance, is still not known.

From in vivo studies on implant abutments, a "threshold Ra" (Ra parameter is the same as S_a , although Ra refers to a profile, not a surface) for plaque accumulation at 0.2 µm was reported.^{31,32} The most commercially available abutments have Ra values below 0.3 µm and do not shelter a significant amount of bacteria.³³ In this study, the total S_a value averages in the grind group demonstrated slightly higher value compared to the "threshold Ra" and the other commercially available abutments Ra, while in the sintered group, twice or three times higher S_a values were found.

Before sintering, the average S_a and S_{dr} values of point B (the external slope area of abutment) displayed a significantly higher surface roughness value compared to the values of points A (the widest area of abutment) and C (the area close to the abutment/fixture connection), that is, points A and C were almost of the same surface roughness. One possible explanation may be technical difficulties during the manufacturing because of the higher tilt angle of emergence profile, resulting in a more uneven surface structure. This hypothesis is supported by the digital images of point B, which has many horizontal scratches that may have been the result of the manufacturing process (Figure 7, A and B). Therefore, the existence of the higher surface roughness part might relatively raise the total S_a values before sintering compared to other studies.^{31–33}

After sintering, a significant increase (p < .05) of the surface roughness was observed in point A only, that is,



Figure 6 Minimum distance average value of each measuring point (x to z) in the grind and sintered groups (*p < .05; **p < .01).



Figure 7 Digital images showing four different points in this study. (A) Point A before sintering (grind group), (B) point B before sintering (grind group), (C) point A after sintering (sintered group), and (D) point B after sintering (sintered group).

close to the veneered part of the abutment. Most probably, the observed variation in S_a and S_{dr} values is an effect of porcelain particle contamination during the crown buildup procedure. This hypothesis is strongly supported by the digital images of points A and B (see Figure 7, C and D), Therefore, it may be proposed that the reason for the increased S_a and S_{dr} values after sintering compared to other studies^{31–33} is not the influence of the sintering process in itself, but more probably an effect of porcelain particle contamination.

The implications of the higher surface roughness onto the Y-TZP abutment surface after sintering may be an increased possibility for plaque accumulation and eventually effects on the close-to-abutment soft tissue conditions such as loss of attachment and development of mucositis. Whether or not this will imply a clinical problem remains to be evaluated. Still, a cleaning protocol for the nonveneered part of the abutment seems rational. If not, the porcelain contaminations will sinter on the Y-TZP surface and will be impossible to eliminate unless the surface is carefully polished. Another possible implication may be effects on the fit between the abutment and the fixture. However, no statistically significant difference in marginal gap before and after the sintering process was observed.

According to Piconi and colleagues,²⁸ the Vickers Hardness of TZP is 1,200 Hv. In this study, the total averages of microhardness in the grind and sintered groups were 1,248.2 Hv (SD = 34.0) and 1,232.2 Hv (SD = 25.4), respectively. These results are on the same level as the result reported by Piconi and colleagues.²⁸ During the sintering process, the Y-TZP ceramic is exposed to temperatures up to 700 to 800°C. Mechanical strength changes of Y-TZP have been reported to depend on Y₂O₃ content, temperature, grain size, stress, and aging time.³⁴ Probably, an increased temperature and a longer sintering time will result in a spontaneous transformation of the metastable tetragonal phase into the monoclinic phase, associated with a degradation of the mechanical properties. The typical low thermal conductivity of ceramics will cause steep thermal gradients at heating and increase the thermal stresses. Therefore, the total average of microhardness after sintering was significantly lower than before sintering with a difference of 2%. However, because of the short sintering time, the thermal effects on the microstructure will be limited.



Figure 8 Schema of a possible outcome on yttrium-stabilized zirconia abutment after sintering based on the results of this study.

Kosmac and colleagues³⁵ showed that a grindinginduced crack from the ground surface into bulk of the material extended about 50μ m when a 150μ m coarse diamond was used. Because in the present study, the distance between the ground surface and point a was about 150μ m, the stress of the grinding may not reach to point a. Therefore, the influence on the microstructure by the grinding stress was not possible to detect before/after the sintering process.

In this study, the values of minimum distance between abutment/fixture at point x (the fixture top) in the grind and sintered groups were $0.62 \,\mu m \,(\text{SD} = 0.56)$ and $0.94 \,\mu m$ (SD = 1.21), respectively. These results were slightly lower than those in a study by Jansen and colleagues.³⁶ They found a marginal gap at the fixture top of the Astra Tech implant (abutment 20) of approximately 1 to $2\mu m$ as measured in SEM (20×). These differences in results between the two studies may be because of different measuring methods. The result of the present study comparing the grind and sintered groups in points x to z indicates that the contact between the abutment and the fixture tends to be focused at the top of the fixture. This may implicate that the stress from occlusal force cannot be uniformly distributed and may become one of several bone loss factors.³⁷ In the case of a ceramic abutment, this may imply an increased risk of abutment fracture and screw loosening. The total average of minimum distance between abutment/fixture after sintering was found to be 2µm or less, that is,

equivalent to findings reported by Jansen and colleagues.³⁶ Therefore, it was suggested that the level of minimum distance before/after sintering might be of minor or no influence in the clinical situation.

A possible outcome on the abutment after sintering, based on the results of this study, is shown in Figure 8. However, whether these in vitro results will provoke the in vivo problems as it stands or not need to be the subject for additional clinical studies.

CONCLUSION

The following changes were detected on the Y-TZP abutment before/after sintering of the veneering:

- 1. Slight decrease (2%) of total microhardness after sintering.
- 2. Because of contamination of porcelain particles, a significant increase in surface roughness was observed close to and immediately below the veneered part of the abutment after sintering. Therefore, a cleaning protocol for the nonveneered part after sintering is recommended.
- 3. The level of abutment/fixture minimum distance changes before/after sintering is likely to be of minor or no influence in the clinical situation.

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