FRACTURE RESISTANCE AND SURFACE TREATMENT OF Y-TZP PREPABLE CERAMIC ABUTMENTS AND BARS

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A thesis submitted to the faculty of the University of North Carolina at Chapel Hill in partial fulfillment of the requirements for the degree of Master of Science in the Department of Prosthodontics.

Chapel Hill
2006

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ABSTRACT

NIMET DIAMOND ADATIA
Fracture Resistance And Surface Treatment Of Y-TZP Prepable
Ceramic Abutments and Bars.
(Under the direction of Dr. Stephen Bayne)

Intraoral preparation of zirconia implant abutments creates deep surface defects making abutments susceptible to fracture during loading. Fracture strengths were tested for (1) HIP-processed zirconia bars (Astra-Tech) after preparation and/or surface repair treatments: no preparation (NP), dry-preparation (DP), wet-preparation (WP), or wet-preparation and 30d water storage (WP+30d), mitigating treatment of bonding agent (WP+B), sandblasting (WP+SB), or polishing (WP+P), and (2) abutment-assemblies (preparations of 0, 0.5, or 1mm margin reduction).

NP established the strength for pristine zirconia bars (1634±95MPa). DP (1144±109MPa), WP (1442±89MPa), WP+30d (1193±155MPa), and WP+B (1218±77MPa) groups had significantly ($p \leq 0.05$) reduced strengths. WP+SB (1632±134MPa) or WP+P (1664±176MPa) repairs equally well recovered original strengths ($p < 0.001$).

There were no statistically significant differences ($p > 0.05$) among different abutment-assembly groups and no logical relationship of strength to increasing amount of reduction. All fractures occurred at the interface where the abutment was connected to the analog, suggesting that fracture was unrelated to the actual abutment.

ACKNOWLEDGMENTS

I would like to thank Allah first, for getting me this far in life. Nothing in life is easy, but He has made it do-able. I would like to thank my family for understanding when I did not call home for days on end because I was writing. I would especially like to thank Agustin, who made me dinner nights when I otherwise would not have eaten, who gave up what little time we have together so that I could concentrate on writing my thesis and who always listened when I was frustrated, stressed out and needed to vent. For that and so much more, I will always be grateful.

I would like to thank my mentor, Stephen Bayne, M.S., Ph.D. His door has always been open to me, even if it was not to talk about my research. His kindness and thoughtfulness made him the best advisor I could have asked to have. Without him, this manuscript would never have been realized. It was an honor to work with him in his last year at the University of North Carolina.

I would like to thank my committee members Lyndon Cooper, D.D.S, Ph.D. and David Felton, D.D.S, M.S. who took the time to help guide me in the process of my research and in my writing. They have also both contributed immensely to my graduate program.

I would also like to thank Jeffery Thompson, PhD who provided expert counsel, expert help in understanding the ceramics engineering, research supplies, and laboratory space to perform my research. His entire laboratory team helped me at most stages of the work. I would like to thank Erica Teixeira for her patience in teaching me key procedures in the lab, guiding me in operating the Instron machine, and for troubleshooting my experiments when I had problems. Without her help, it would have taken me a lot longer to

finish. I would also like to thank Jeff Piasik for cutting the zirconia bars while he was in the midst of his thesis work. Wallace Ambrose provided instruction on the SEM and guided my efforts in that regard.

I would like to thank Astra Tech for making this project possible by HIP processing zirconia plates identical to the abutment material. Their special efforts were pivotal to this analysis.

Brasseler USA and Premier Dental Products Company supplied the disposable materials needed for this project.

ACP/3M-ESPE provided a grant to fund this research.

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LIST OF ABBREVIATIONS AND SYMBOLS

Al_2O_3	alumina or aluminum oxide
B	bonding
CAD/CAM	computer aided design/computer aided milling
cp-Ti	commercially pure titanium
c	cycle
CHS	crosshead speed
d	day
º	degrees (angular)
ºC	degrees Celsius
DP	dry-preparation
=	equals
Hz	hertz
HIP	hot isostatic pressed
hr	hour
K	Kelvin
m	meters
µm	micrometer
min	min
mm	millimeter
MPa	Megapascal (1 Mpa = 145.8 psi)
NP	no preparation

+	plus
psi	pounds per square inch
%	percentage
P	polishing
±	plus or minus
NA	nasion to inner contour of premaxilla
N	Newton
no.	number
SB	sandblasting
s	second
SEM	scanning electron microscope
sd	standard deviation
ST	single tooth
wt%	weight percentage
WP	wet-preparation
Y-TZP	yttria-stabilized tetragonal zirconia polycrystals
ZrO_2	zirconia or zirconium oxide

INTRODUCTION

Implant use in dentistry depends not only on the selection of proper materials but also on the establishment of correct orientation and dimension of the restoration. One of the more intriguing methods of approaching the orientation problem is to prepare abutments that permit compensation for implants that are aligned in less than optimal conditions.

There are only a few available material choices for prepable abutments: (a) titanium, (b) alumina, and (c) zirconia. Titanium abutments have been the standard of care for the greatest time, have high fracture resistance, and are biocompatible. Unfortunately, for patients with thin gingival biotypes or fragile tissues, the blue hue from light reflections of titanium shines through the tissues and is unesthetic (Tan and Dunne, 2004; Brodbeck, 2003). Abutments of milled or as-sintered alumina overcame the aesthetic problem (Vigolo *et al*., 2005; Andersson *et al.*, 2001) and provided favorable surfaces for fibroblast adherence (Mustafa *et al.*, 2005). However, alumina fracture resistance was poor compared to titanium or zirconia. Therefore, zirconia abutments are now being marketed to resolve both the esthetic and fracture strength issues.

Although zirconia abutments for intraoral preparation have been approved for clinical use, there have been very few laboratory studies investigating the fracture resistance of zirconia abutment assemblies. As with all ceramic materials, any surface or volume flaws may initiate potentially disastrous cracks. Some studies have looked at bur effects generated during abutment preparation (Blue *et al.*, 2003) and attempts to repair those effects with subsequent surface treatments (Kosmac *et al.*, 1999, 2000; Luthardt *et al.*, 2002; Guazzato *et al*., 2004; de Jager *et al.*, 2000). Yet, the results have been inconsistent.

During intraoral preparation of zirconia implant abutments deep surface defects are often generated which make the abutments susceptible to fracture during loading. To understand and resolve these associated problems, it's crucial to measure resulting fracture strengths of these abutments in a controlled way under a variety of conditions. This can be done by testing strengths of prepared abutments in simulated assemblies or by focusing the strength of zirconia alone to isolate the effects of surface preparation and/or surface repair treatments that mimic actual intraoral conditions. To date, no one has carefully studied the effect of surface treatments (grinding, polishing, sandblasting, or bonding film application) on HIP-processed zirconia bars to isolate and understand the fracture strength of abutment assemblies.

LITERATURE REVIEW

To understand the effect of surface flaws on the mechanical properties of prepared abutment assemblies, one must consider the background of the components involved.

A. Implant abutments

Implant replacement in the anterior may be the ideal choice to replace a single tooth, but the restoration may present challenges in the surgical and prosthetic stages (Vigolo *et al.*, 2005). Most dental implants are constructed entirely of metal (*e.g.*, cp-Ti). However, in cases demanding special considerations for esthetics, when the gingival tissues surrounding the abutment are friable, traditional metal abutments show though the gingival tissues as a dark blue-black zone. In addition, the difficulty of light transmission in this region compromises esthetics (Yildirim *et al.,* 2003, Vigolo *et al.,* 2005). Furthermore, implant abutments often require special adjustment in final orientation to accommodate for implant angulations and size discrepancies. Introduction of all-ceramic abutments solved these problems by producing more tooth-like color and allowing for individually designed emergence profiles (Yildrium *et al*., 2003).

Ceramic abutments still have some shortcomings. Ceramics are inherently brittle and are very sensitive to tensile force. Cracks which may arise can propagate, even when the implant is under very low loads, due to continual load cycling during mastication. These cracks ultimately produce failure (Yildrium *et al*., 2003).

B. Materials for abutments

Abutments theoretically could be fabricated from any material (metal, ceramic, polymer, or composite). However, materials with high fracture resistance are more likely to succeed (*e.g.*, metals and certain ceramics). Three compositions have been commercially explored up to this point – titanium (metal), alumina (ceramic), and zirconia (ceramic). These are considered in more detail as follows.

B1. Titanium

Commercially pure titanium (cp-Ti) is biocompatible and does not promote plaque adherence. It can be prepared to the correct contours. However, it does not provide optical properties of a natural tooth. Functionally, a titanium abutment provides more than enough strength and transmits force across an osseointegrated implant interface onto bone (Strub and Gerds, 2003).

Short-term mechanical failures of titanium abutments primarily involve screw joint instability that is displayed as screw loosening or screw fracture (Norton, 2000a). Norton (2000a) studied the effect of conical abutment taper on deformation rates and maximum bending moments (or failures). Failure was defined in a practical way as when deformation reached or exceeded 0.3 mm of displacement. The critical zone of deformation (or failure) for Astra uni-abutments (Astra Tech, Waltham, MA) occurred at the abutment-implant interface. Solid-screw abutments (Straumann USA, Andover, MA) fail with fracture at the head of the screw just below the base of the cone. Importantly, the loads required to produce unfavorable bending moments are higher than levels expected to occur in clinical situations.

In another study (Norton, 2000b) the abutment design was evaluated by comparing one-piece versus two-piece conical abutments (see Figure 2.1). Although one- and two-piece abutments had similar critical zones, their modes of failure were different. For one-piece abutments, failure involved the cylindrical part of the solid titanium abutments. In two-piece abutments, failure occurred at the head of the screw or at the internal hexagon at the base of the abutments.

(a) Two piece, deformation at internal hex.

(b) One piece, deformation at body of abutment.

Figure 2.1 Titanium abutment designs used to evaluate failure modes (Norton, 2000).

Strub and Gerds (2003) studied 5 different titanium abutment-implant assemblies (sterioss implants with Novosil abutment; Sterioss implant with anatomic abutment; Sterioss implant with straight HL abutment; IMZ twin with esthetic abutment; Osseotite with hexed gold from UCLA) using fatigue rather than just single-cycle load-to-failure. A 50N load was chosen as representative of the clinical range. Loads were cycled at 1.6Hz for 1,200,000 cycles. In all groups but Novosil, fracture occurred at the screw level by bending or fracturing after static loading. The IMZ test group the implant necks showed distortion. No proposed reason was given for the increased failure in the Novosil and IMZ groups. However, the physical properties of these groups were suggested to need improvement.

B2. Alumina

In 1994, the first esthetic ceramic abutment was introduced (CerAdapt, Nobel Biocare, Yorba Linda, CA) (Vigolo *et al*., 2005.) This abutment was made of densely sintered aluminum oxide (Al_3O_2) (Vigolo *et al*., 2005; Andersson *et al.*, 2001). However, it was problematic because of its radiolucency during radiological examination, fragility in handling, and low fracture resistance in service.

Alumina abutments typically have been fabricated by shaping them in their green form (pre-sintered) and sintering them to form a dense alumina abutment. Grain sizes range

from 4-7 µm, which is considered small for alumina ceramics (Blue *et al.,* 2003). Material removal by cutting produces micro-cracking along the grain boundaries and often has caused entire grains to be plucked out of the surface. The removal rate during cutting has been related to both the grain size and normal force (Blue *et al.,* 2000).

Alumina is colored closer to a natural tooth, while zirconia appears very white. Thus, alumina produces a better esthetic match than zirconia in the anterior region. Alumina is also easier to prepare than zirconia and that shortens the patient chair time necessary for final preparation (Yildirim *et al.*, 2003).

Andersson *et al.* (2001) conducted a prospective clinical study of alumina (CerAdapt) versus titanium abutments (CeraOne, Nobel Biocare, Yorba Linda, CA) over a 1-3 year period. All abutments were placed in premolar-canine-incisor areas. For CerAdapt, 12% (n=4/34) abutments fractured before loading and 7% (n=2/30) fractured during the first seven months of loading. However, for CeraOne there were no failures. CerAdapt failures were attributed to impaired abutments, excessive bending moments, and/or accidents. The authors felt that over-preparation of the abutments may have contributed to failure as well, since 17% abutments failed after using the counter-torque device, even before being loaded. It was concluded that CerAdapt abutments were more sensitive than titanium to handling techniques.

B3. Zirconia

Zirconia implant abutments are fabricated from yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) (Blue *et al.*, 2003). Zirconia is a polymorphic ceramic that occurs in three equilibrium crystalline structures: monoclinic (room temperature to 1170°C), tetragonal (1170°-2370°C), and cubic (>2370°C). After processing while zirconia is being cooled, the tetragonal transformation to monoclinic occurs at ~970°C and is associated with a 3-4% volumetric expansion. However, if small amounts of yttria are added to zirconia, then the tetragonal phase will remain all the way down to room temperature (Blue *et al.*, 2003; Guazzato *et al.*, 2005). The $ZrO_2 - Y_2O_3$ phase diagram is shown in Figure 2.2.

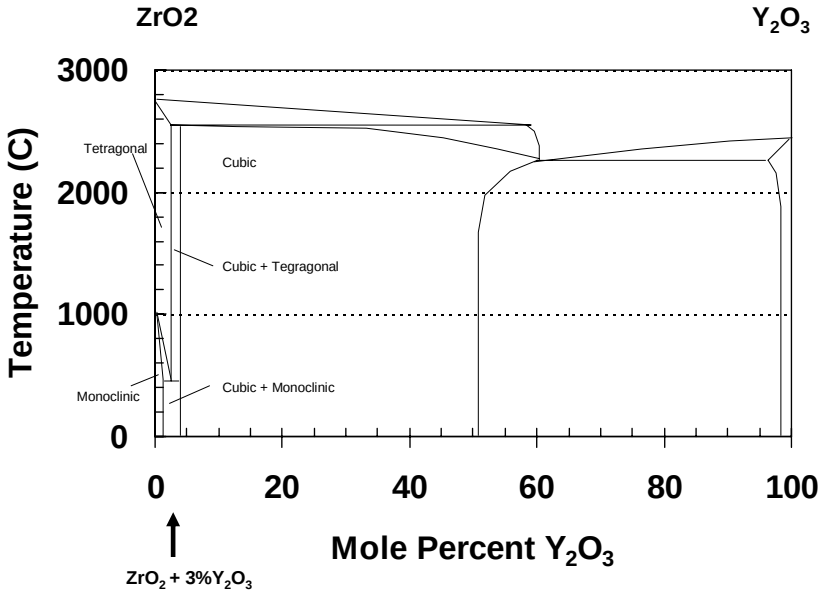


Figure 2.2 Phase diagram for $ZrO_2 – Y_2O_3$. (After Levin *et al.*, 1979)

The local volumetric change from the cubic to tetragonal transformation during cooling, results in internal compressive stresses which tend to be crack-sealing. Schematically that process can be envisioned in Figure 2.3. That is why Y-TZP has a high fracture toughness and strength when compared with conventional brittle ceramics.

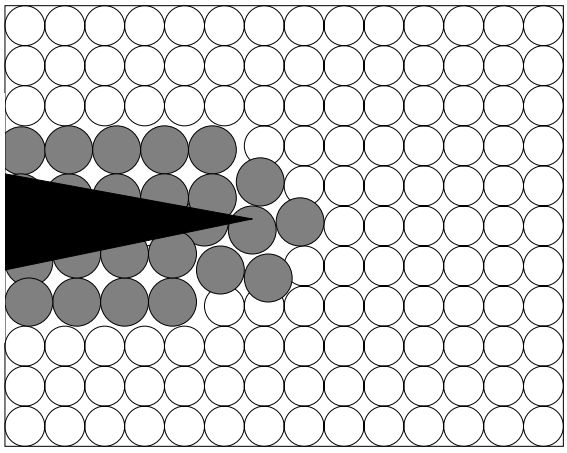


Figure 2.3 Schematic representation of effect of internal compression discouraging crack propagation. (After Jeff Thompson)

These internal compressive forces must be overcome for a crack to propagate (Guazzato *et al.*, 2005). However, preparation with burs or diamonds during preparation of Y-TZP abutments can have several negative effects. First, preparation adds mechanical energy and heat to the surface of the ceramic which encourages final transformation of material within the affected zone from the tetragonal phase to the lower-temperature monoclinic phase. Second, severe preparation can introduce deep subsurface flaws or cracks which act as stress concentrators and which could reduce strength values. Unlike alumina, partially-stabilized zirconia results in increased fracture toughness with increasing crack size (R-curve behavior). This makes the strength of partially-stabilized zirconia abutments actually less sensitive to surface flaws (Blue *et al.*, 2003) than alumina.

Yttria-stabilized zirconia has twice the strength of alumina. One recent study of the fracture loads for abutments concluded that there was a statistically significant difference between the mean oblique (30°) fracture load for zirconia (737 ± 245 N) versus alumina (280 ± 103 N) (Yildirim *et al.*, 2003). Those authors concluded that either abutment could be used successfully, but that zirconia was more likely twice as fracture resistant as the alumina abutment.

The flexure strength and fracture toughness of representative dental ceramic materials are listed in Table 2.1. Note that the flexure strength of zirconia is higher than all others.

Table 2.1 Mechanical properties of representative dental ceramic materials.

Ceramic Material	**Flexure Strength (MPa)**	**Fracture Toughness (MPa-m$^{1/2}$)**
Zirconia (3% Y_2O_3 stabilized)	900	9.00
Alumina industrial	547	3.55
Alumina slip cast	419	2.48
Dicor MGC	220	2.02
IPS Empress	182	1.77
Sintered ceramic (Omega)	85	0.99

Adapted from Brodbeck, 2003.

There are only a few hot isostatic pressed (HIP) processed zirconia abutments on the market. One of these is the ZirDesign (Astra Tech, Waltham, MA) abutment. The material properties of this abutment, according to the manufacturer, are listed in Table 2.

Table 2.2 Properties of ZirDesign abutments.

Material	**Zirconia (Y-TZP)**
Bending Strength	1000-1300 MPa
Toughness	9-10 MPa-$m^{1/2}$
Modulus of Elasticity	210 GPa
Coefficient of Linear Expansion	10.6 x 10^{-6} /°K
Color	Ivory

Adapted from Astra Tech's Ceramic Clinical and Laboratory Procedures Manual. (75344-US-0404)

Butz *et al.* (2005) compared the fracture strength of zirconia abutments (ZiReal, 3i, Palm Beach Gardens, FL) versus an alumina abutments (CerAdapt) and titanium abutment (GingiHue, 3i, Palm Beach Gardens, FL). After fatigue loading, only one CerAdapt abutment failed (load = 255 N). No failures occurred with zirconia loaded to 324 N. No failures occurred with titanium loaded to 294 N. After static loading, all abutments in the alumina group fractured, four zirconia abutments fractured and 2 screws fractured with the remaining abutment assemblies deflecting 4 mm. The titanium abutments all deflected instead of fracturing. All deflected abutments showed screw bending. This information is summarized in Table 2.3. The authors concluded that the ZiReal abutment, with its titanium collar, performed as well as the titanium abutment and could be recommended as an aesthetic alternative for implants in the anterior region.

Table 2.3 Mode and frequency of failed ceramic and titanium abutments tested in fatigue (130º from vertical (50º from vertical); 30N load, 1,200,000 cycle, 1.3Hz) (Butz *et al.*, 2005).

Assembly tested	**Number of Failures**	**Residual Strength; Location of Facture**
Titanium assemblies with metal crown and gold retaining screw	No failures after chewing cycle	324±85N; all failures were deflection of screw head to labial
Zirconia assemblies with metal crown and gold retaining screw	No failures after chewing cycle	294±53N; 4 abutment and 2 screw fractures (lingual side); remaining 10 labial deflection of screw occurred
Alumina assemblies with metal crown and gold retaining screw	One abutment failed at 9 of 1,200,000 cycles, at screw head level	239±83N; all failures were abutment fractures

C. Improved esthetics

All-ceramic restorations have become popular for restoring anterior dentition due to their exceptional translucency which allows light transmission through to the underlying tooth. They minimize gingival shadowing and yield an appearance of vitality. (Tan and Dunne, 2004). Dental implants that are restored with titanium abutments preclude translucent restorations and lead to gray color being transmitted to peri-implant tissues. Zirconia abutments do not (Tan and Dunne, 2004). Ceramic abutments can be customized to needed contours. Yet, the custom preparation may leave surface defects that create stresses which could produce future cracks (Tan and Dunne, 2004; Yildirim *et al*. 2003).

D. Surface treatments

Kosmac *et al.* (1999) examined the effects of surface textures on fracture resistance. They observed that tetragonal-to-monoclinic phase changes were associated with higher flexure strengths. Dry preparation of zirconia abutments decreased their mean flexure strength. However, sandblasting, either alone or following preparation of Y-TZP specimens, significantly improved flexure strength.

Kosmac *et al.* (2000) studied the process of aging Y-TZP ceramics to determine how the strength changed after surface treatments. They placed zirconia, as-sintered and after sandblasting, in either 4% acetic acid solution or diluted ammonia solution to simulate aging to represent conditions for clinical service. The strength of zirconia initially increased due to the progress of tetragonal-to-monoclinic phase transformation for zirconia. However, once a certain amount of monoclinic zirconia was transformed, it caused micro-cracking as a side-effect, and the overall strength decreased. Masonis *et al.* (2004) also observed that the monoclinic transformation tended to decrease strength over long periods, degradating the surface and increasing microfracture. The mechanism for this has not been established. Pre-existing monoclinic zirconia in sandblasted Y-TZP seemed to hinder the diffusion-controlled transformation during subsequent exposure to acidic and basic environments. Since the total amount of transformed zirconia was higher in this sample, the strength degradation occurred sooner than in the as-sintered group.

Luthardt *et al.* (2002) also found that preparing zirconia ceramics had a negative effect. Surface grinding of zirconia crowns significantly reduced the strength and predictability of Y-TZP zirconia compared with control samples. Analysis of flexure strength and fracture toughness revealed competing effects between strengthening from the production of surface compressive stresses versus weakening from the generation of surface flaws caused by grinding. A reduction in surface cutting depth positively influenced the residual flexure strength of zirconia. Grinding procedures for CAD/CAM fabricated Y-TZP all-ceramic restorations required special control although the method to do this was not revealed.

Guazzato *et al*., (2004) investigated the effects of grinding direction and heat treatment on zirconia strength. Grinding orientation did not significantly affect the flexure strength. They also noted that, unlike other studies, sandblasting and grinding both increased the monoclinic phase change, and both were associated with higher flexure strengths. Heat treatment tended to decrease the flexure strength of DC-Zirkon (DCS Dental, Greendale, WI)

zirconia. A similar result was also reported by Kosmac *et al.,* (2000) when zirconia samples were heated to anneal them before and after grinding. Polishing and heat treatments showed negligible monoclinic formation and were associated with lower flexure strengths.

Zhang *et al.,* (2004) found that sandblasting either alumina and zirconia caused a slight decrease in fatigue strength. Despite this trend, the strength values remained above the load values for oral function for >10 years of service life.

E. Methods of preparation for Y-TZP abutments

According to manufacturers' instructions, the ZirDesign abutment can be prepared as a tooth for a crown or bridge. Preparation is recommended with diamond wheels/burs or silicone carbide stones (Astra Tech). Brodbeck (2003) suggested using coarse diamonds to prepare ZiReal zirconia abutments. Blue *et al.* (2003) reported that smaller bur abrasive particle size was less harmful. However, preparation efficiency was enhanced by using coarse abrasives.

The amount of material removed during preparation is affected by both the composition of the ceramic substrate and the diamond abrasive particle size, but composition was more important. For zirconia abutments, there was no significant effect of bur abrasive particle size but there were some trends. Fine diamonds (50µm) lost the lowest percentage of particles during cutting. Medium diamonds (100µm) removed the greatest mass of zirconia per unit time. Coarse diamonds (150µm) lost the most particles. Those authors ultimately suggested that for zirconia abutments, a fine diamond be used with water irrigation from the start to achieve a smooth surface and potentially strengthen the abutment surface. Yet, these actual recommendations were not tested in the study. (Blue *et al.,* 2003).

Zirconia bars prepared with a coarse diamond without water irrigation lowered mean strengths and reliability (Kosmac *et al.,* 1999). Similar tests with water irrigation were not conducted. Guazzato *et al.,* (2004) prepared zirconia specimens with water coolant using a 91µm grit diamond and found a resultant increase of the strength of zirconia.

F. Fracture resistance of ceramic abutments

The fracture resistance of ceramic abutments made from pre-sintered materials (*e.g.*, Celay In-Ceram system, Mikrona, Spreitenbach) has been purported to be greater and more consistent than conventional ceramic materials for dental applications (Cho *et al.*, 2002). Pre-sintered materials have been used to produce custom-designed abutments from alumina. Fracture resistance of a ceramic abutment with an all-ceramic crown loaded vertically was significantly lower (786 N) than that of a titanium abutment (1628 N) with metal ceramic or all ceramic crown (Cho *et al.*, 2002). Under oblique loading (45°) conditions, there was no significant difference in fracture strengths among the abutments and all ceramic crowns. Failure in the ceramic abutment specimens initiated from the ceramic abutment collar near the junction between the abutment and the implant. This study used an external hex abutment joint which purportedly is weaker than an internal hex abutment joint that can contribute the mode of failure (Khraisat, *et al.*, 2002).

Tripodakis *et al.* (1995) tested sintered alumina blocks milled as ceramic abutments to determine their static fracture strength (loaded at a 30° angle) and the effects of different designs. Cemented crowns significantly improved the fracture resistance of the assemblies suggesting that a crown may reinforce the abutment during loading. The abutment screw placement was determined to significantly affect the fracture resistance of the assembly as well. Placement of a crown margin above the height of the screw head significantly lowered the fracture resistance compared to placing the margin of the crown below the head of the screw.

G. Bite strength

Peak values for occlusal force in the incisal area have been reported within the ranges of 90 to 370 N (Paphangkorakit and Osborne, 1997) and 150 to 235 N (Haraldson *et al.*, 1979). An all-ceramic implant assembly should resist these forces to be clinically successful. For this reason, Craig and Powers (2002) recommended that ceramic abutments only be used in the anterior maxilla where biting force of canines (200 N) and incisors (150 N) was lower.

H. Loading angles

According to Proffit (2000), the relationship of the upper incisor to the NA (nasion-to-A point) line ranges from 22.24º where the NA line is drawn from the nasion to the innermost point on the contour of the premaxilla. Using this as a reference, an angle of 30° from the long axis has been adopted for loading abutments in research experiments (Yildrim *et al.,* 2003). Others have adopted a 45° angle from the long axis (135 degrees to the horizontal plane) to simulate clinical conditions (Strub and Gerds, 2003). Recent prosthetic and endodontic publications report angles ranging from 45-to-150° from the long axis (Loney *et al.,* 1995; Eskitascioglu *et al.,* 2002; Heydecke *et al.,* 2002) to test maxillary central incisors. The relationships of these different descriptions of loading angles are related to each other in Figure 2.4. A summary of all the reports of loading angles is reported in Table 2.4.

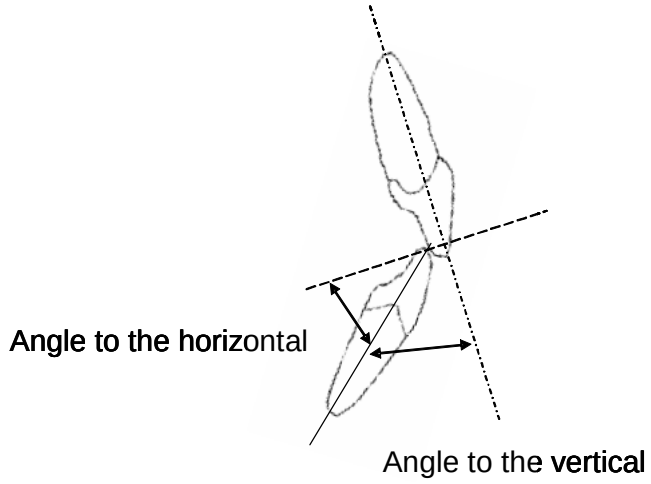


Figure 2.4 Horizontal and vertical reference lines for reporting loading angles.

Table 2.4 Extrapolated angles of loading reported in the literature.

Reference	Angle to the vertical	Angle to the horizontal	CHS (mm/min)
Proffit	(22.24º)	[66-68º]	--
Eskitascioglu *et al.*	45º	[45º]	5
Heydecke *et al.*	(40º)	[50º]	1.5
Yildirim *et al.*	30º	[60º]	0.1
Butz *et al.*	(50º)	[40º]	1.5
Strub and Gerds	(45º)	[45º]	2
Yoldas *et al.*	45º	[45º]	1
Fokking *et al.*	30º	[60º]	5
This study	[30º]	60º	0.1

Angles in brackets are 90º minus stated angle.

Angles in parenthesis are derived from author's comments.

METHODS AND MATERIALS

A. Overview

Effects of surface treatments (diamond roughening, sandblasting, polishing, and bonding film application) on Y-TZP bars (Astra Tech, Metoxit AG, Ch-8240, Thayngen, Germany), and strength of Y-TZP abutments (Astra Tech, Metoxit AG, Ch-8240, Thayngen, Germany) were compared to controls. For the first stage of testing, Y-TZP bars were prepared with diamonds to mimic clinical usage and test effects of surface treatments (see Figure 3.1) in mitigating surface damage occurring during preparation. In a second stage, actual implant abutments were prepared from the same material and evaluated for fracture strength of the assembly following different extents of surface preparation (none, 0.5 mm margins, and 1.0 mm margins) (Figure 3.2). In each stage specimens were tested using the Instron. Later, specimens were inspected using SEM analyses to characterize fractures and surface treatments.

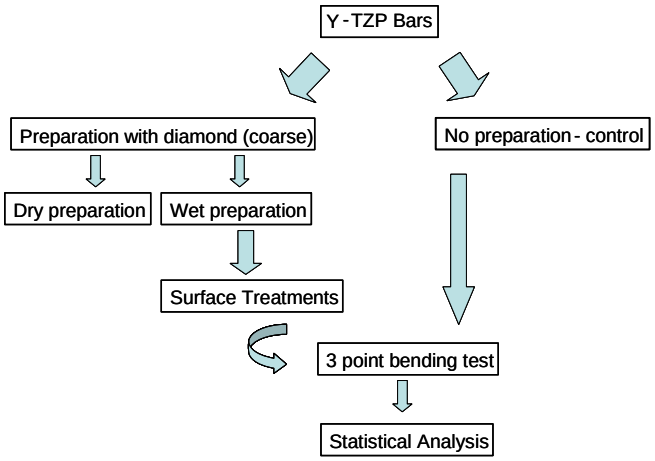


Figure 3.1 Flowchart of methods for Y-TZP preparation and testing.

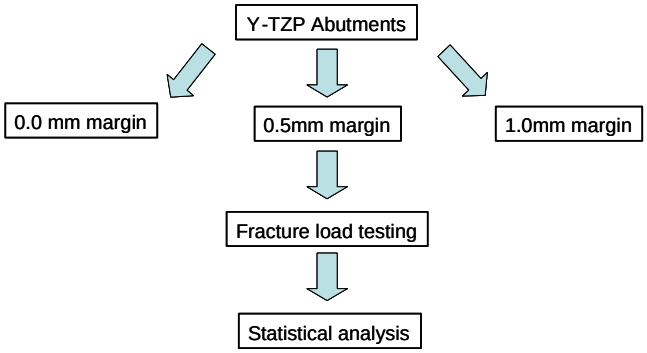


Figure 3.2 Flowchart of ZrO_2 abutment preparation and testing.

B. Composition and properties of Y-TZP

The chemical composition of yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) is reported below.

Table 3.1 Chemical composition of Y-TZP (Astra Tech,Technical brochure 75344-US-0404).

Properties	**Units**	**Theoretical**	**Measured** (Batch no. 4173)
Chemical analysis			
$ZrO_2 + HfO_2 + Y_2O_3$	Wt. %	>99.0	99.97
Y_2O_3	Wt. %	4.5-5.4	5.19
HfO_2 (abrasion resistance)	Wt. %	<5.0	1.88
Al_2O_3 (hardness)	Wt. %	<0.5	<0.005
Other oxides	Wt. %	<0.5	0.037
Radioactivity	Bq/kg	<200	<200
Physical analysis			
Density	gm/cm^3	>6.0	6.09
Average grain size	μm	<0.6	0.36

C. Prepared abutment experiments

C1. Preparation of abutments

Abutments and analogs were provided by Astra Tech Company (Waltham, MA) The ceramic abutments tested were suitable for 4.5/5.0 diameter implants and are intended for

cement-retained prostheses and for use as single-tooth restorations in the anterior, canine, or premolar regions. They are not recommended for use in the molar region. They are designed to be prepared for conventional full coverage crowns. Abutments should be attached to analogs or implants using a 25 N-cm torque.

Table 3.2 Design measurements of Y-TZP abutments

Design	mm
Height (H)	13.7
Height above Fixture (A)	10
Diameter (Ø)	5.5

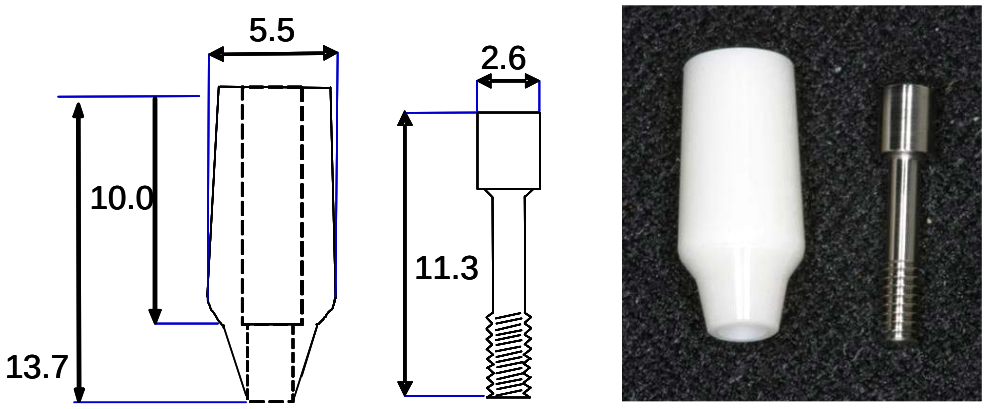


Figure 3.3 Abutment and abutment screw specifications and image

Figure 3.4 Abutment and analog assembled

D. Zirconia bar experiments

D1. Fabrication of zirconia bars

Hot-isostatic-pressed (HIP-processed) zirconia plates (5.19 wt% yttria stabilized, Metoxit AG, Ch-8240, Thayngen, Germany, Batch no. 4173) were provided by Astra Tech Company. Thirty bars (20 x 60 x 2 mm) were cut from each zirconia plate using a Hi-Tec diamond saw (PIA Series, Santa Clara, CA).

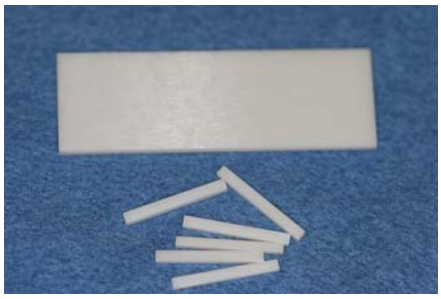


Figure 3.5 Image of Y-TZP plates and cut bars.

Table 3.3 Dimensions of Y-TZP materials.

	Width (mm)	Thickness (mm)	Length (mm)
Plates	~20	~2	~60
Bars (average)	2.065	2.045	~20

Each bar was labeled using an ultra fine permanent ink pen (Sharpie Pen, Sanford, Oakbrook, IL) to indicate HIP sides. Bars were then lighted polished using copious water irrigation (MetaServ 2000 grinder/polisher, 125 rpm, 600 grit paper, Buehler, France) to slightly round along all 90-degree edges and eliminate any potential stress concentrations from the original sectioning.

D2. Surface treatment of zirconia bars

A 2 mm distance was measured from either end of the HIP-surface side on each bar and marked with a thin line using an ultra fine marking pen. This delineated the area and the side to be prepared.

Bars were divided into seven groups of 12 for testing with:

(1) no preparation (control group) (NP),

(2) wet prepped (WP),

(3) wet prepped bars and stored in water for 1 month (WP+ 30d),

(4) wet prepped and polished to recover from surface damage (WP+P),

(5) wet prepped and sandblasted to recover from surface damage (WP+SB),

(6) wet prepped and coated with bonding agent to fill in surface damage cracks (WP+B), and

(7) dry prepped (DP).

To simulate abutment preparation, bars were roughened with a diamond bur (Two-striper, Premier, Plymouth Meeting, PA, 515.7, ISO 110, Lot 330) using a high-speed handpiece (Brasseler USA, Savannah, GA, USK America, NL-85S). A single bur was used to prepare six bars before being replaced. One group (NP = no preparation) was tested without any surface treatment. Each bar requiring bur treatment was swiped with 7 passes of the bur (maximum speed of 430,000 rpm) using moderate hand pressure under copious irrigation.

The WP+30d group was prepped and was fully immersed left in 32°C sterile water for 1 month and then tested. This group was designed to test any potential effects of water immersion on stress corrosion (propagation of existing cracks).

The WP+P group was wet prepped and then polished using ceramic polishing burs (Dialite burs, Brasseler USA, Savannah, GA). Polishing occurred in a stepped sequence using the adjusting bur, pre-polishing bur, and then high shine bur until no scratches were visible to the naked eye. The steps required about 10 sec per bur. No sparks were generated. No significant heat was thought to be generated as the bars were able to be handled directly after polishing.

The WP+SB group was wet prepped and then sandblasted (Basic Master, Renfert, Hilzingen, Germany). Using 50 psi pressure and 50um Al_20_3, the sandblasting nozzle was held at an 80° angle about 5mm from specimen and moved back and forth over specimen for about 10 sec (Figure 3.6).

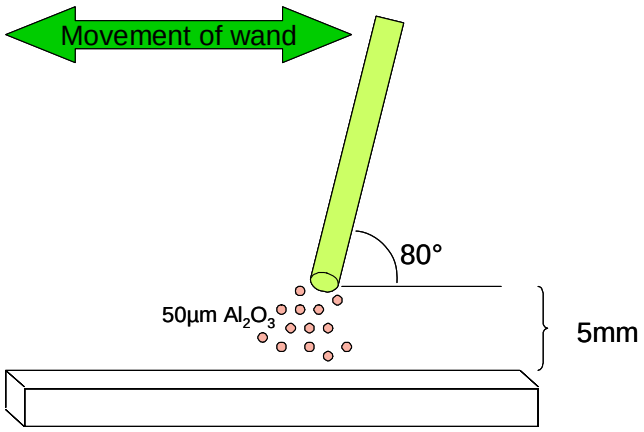


Figure 3.6 Diagram of sandblasting method.

The WP+B group was wet prepped and then surface sealed with Optibond-Solo-Plus (Kerr, Orange, CA, Lot 410822). Optibond-Solo-Plus was chosen since this product can be readily found in the dental office as a multifunctional adhesive for direct and indirect bonding applications. It was chosen due to its claim that the fillers within the material can penetrate the dentin tubules (www.kerrdental.com). After preparation, the surfaces were painted with two coats of material, using a green microbrush. Each time the brush was moistened, wiped on the side of the carrier to remove the excess, then brushed evenly over the surface in a light brushing motion. The material was gently air dried with an air/water syringe for five seconds and then light cured twice for 20 sec each cycle. A new unidose package of bonding agent was utilized for each group of six bars.

The DP group was prepared without any irrigation in the same speed and pressure as the wet prepared groups.

All groups except NP and DP were prepared under copious water irrigation. Groups WP+P, WP+SB, WP+B and DP were prepared and then tested 48 hr later to allow release of any residual stresses that may have arisen during the preparation.

D3. Testing of zirconia bars

Bars were tested for flexure strength at room temperature in air in three-point bending as shown below (Guazzato *et al.*, 2005).

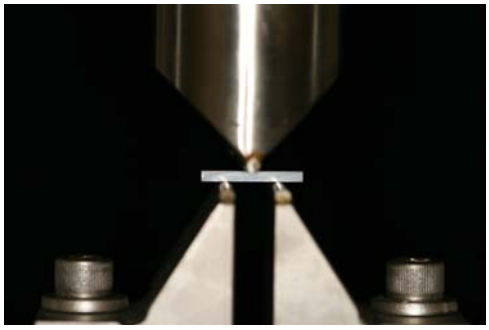


Figure 3.7 Testing apparatus for bar specimens

D4. Calculation of flexure strength

The 3 point bending stress is calculated by computer using the following equation (ISO 6872, 1985)

$\sigma_{3p} = 3WI/2bd^2$

where σ_{3p} is the flexure strength (N/mm^2 where $1N/mm^2 = 1MPa$), W is the fracture load (N), I is the test span (center-to-center) distance between support points (mm), and b and d are the width and thickness of the specimen (mm), respectively.

Each bar was individually measured and the flexure strength was calculated based on these measurements. Width and thickness of each bar can be found in Appendix A.

D5. Statistical analysis of zirconia bar groups

Means and standard deviations for each group of 3-point flexure strengths were calculated. Statistical differences among groups were determined by 1-way ANOVA ($\alpha \leq 0.05$, Bonferoni's post-hoc test).

E. Abutments

E1. Preparation of abutments

Actual ceramic abutments (4.0 standard) were prepared wet with identical burs but without including any surface modifications after preparation that paralleled the zirconia bar experiments. Effects of different margin positions were tested using assemblies that mimicked the intraoral condition of abutment use with an implant. Three groups of abutment

assemblies were tested. Margins were all placed at 1.0 mm above the height of contour and included 2.0 mm of occlusal reduction using a Premier coarse chamfer diamond (Figure 3.8).

(1) Group 1 was tested without being prepped (*i.e.*, control).

(2) Group 2 was wet prepped with a chamfer margin of 0.5 mm.

(3) Group 3 was wet prepped with a chamfer margin of 1.0 mm.

The manufacturer recommends the margin be 0.8mm in size, therefore test specimens with margins aimed higher and lower than this recommended margin size were selected.

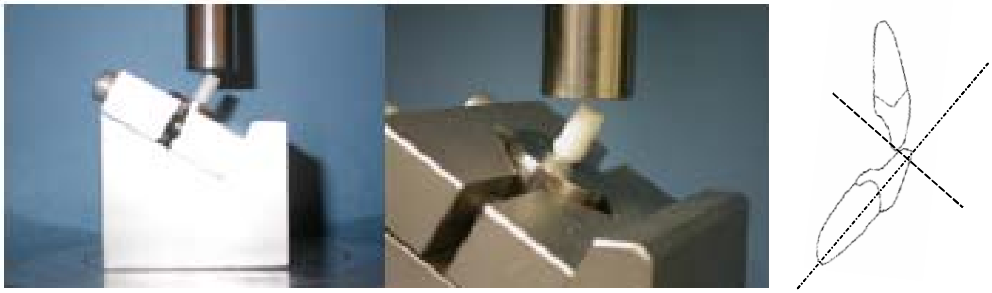
E2. Creation of assemblies

For testing, each analog was first positioned with a stainless steel cylinder with its neck parallel to the top of the cylinder and fixed in place using Field's metal alloy (www.scitoys.com). Abutments were then connected to implant analogs using a torque of 25 N-cm (Astra Tech torque wrench). This combination is referred to as the assembly or implant assembly (Figure 3.7). The cylinders with assemblies were placed onto a stainless steel fixture which was inclined at 30° to the vertical in preparation for loading (Figure 3.9) in a universal testing machine (Instron, Model 4411, Grove City, PA).

Figure 3.8 Abutment assembly fixed within implant analog with Field's metal.

E3. Testing of assemblies

An angled load (see Figure 3.9) was applied to the incisal edge (crosshead speed = 0.1 mm/min , 25°C) until failure was detected as a maximum load during testing or the abutment failed. To prevent inadvertent damaging effects by the loading stylus on the ceramic abutment and to ensure an even load, a thin layer (0.1 mm) of Mylar film was inserted between the stylus and the abutment. Fracture of the abutment was accompanied by an audible pop.



A. B.

Figure 3.9 Assembly testing regime. A. Testing apparatus for loading assemblies containing prepared abutments. B. Schematic of forces applied.

E4. Statistical analysis of assembly results

The mean fracture loads for each group of prepared abutments were compared ($\alpha \leq 0.05$, 1-way ANOVA) using personal computer software (Analyse-It, www.analyse-it.com, UK). A regression analysis was used to determine the amount of abutment reduction that could be achieved while maintaining acceptable fracture resistance.

F. SEM analysis of specimens

SEM analysis (JEOL JSM 6300 scanning machine, Peabody, MA) was performed without coating zirconia materials by using 2.0 KeV, a probe current of 13 ma, and a working distance of 39 mm. Each bar was examined on its prepared surface for surface texture and at a 45-degree angle to look at the fractured edge surface. Specimens were observed at 100x and 500x magnification. Abutments from assemblies were examined on

the prepared surface for texture, and the abutments were also observed at a 90 degree angle to examine the fractured collar of the implant abutment. Each abutment specimen was observed at 100x and 500x magnification.

RESULTS

Results for flexure strength testing of zirconia bars and zirconia abutments are presented in the tables that follow. The individual experimental results are recorded in Appendices A1-A7.

A. Flexure strength of zirconia bars

Results for 3-point flexure strength testing for all experimental groups are reported below in Table 3.1 and graphically displayed in Figure 3.1. The control group with no preparation (NP) and the two groups that represented wet preparation with sandblasting (WP+SB) or polishing (WP+P) after preparation were statistically equal. All other groups were statistically lower than the unprepared control (NP). Statistical differences among groups are indicated in the table at the far right with small letters to distinguish differences.

During grinding with burs, sparks were commonly observed, and preparation of the bars left a scratched yet shiny surface. Sandblasting left the surface of the bars with a matted finish. Polishing created a visually smooth, unscratched surface (Figure 4.2).

Table 4.1 Flexure strength of Y-TZP bars.

Specimen Group:	*Prep Type:*	*Repair Treatment:*	*Group Goal:*	*3-point-Flexure: (MPa±sd)*	
NP	None	None	Control (un-prepared)	**1634±95**	[a]
DP	Dry	None	Control (dry damage)	1144±109	[b]
WP	Wet	None	Control (wet damage)	1442±89	[c]
WP+30d	Wet	None (stored 30d)	Control (water effects)	1193±155	[b,d]
WP+B	Wet	Optibond-Solo+	Repair defects	1218±77	[b,e]
WP+SB	Wet	Sandblasted	Reduce large defects	**1632±134**	[a,f]
WP+P	Wet	Polished	Remove large defects	**1664±176**	[a,g]

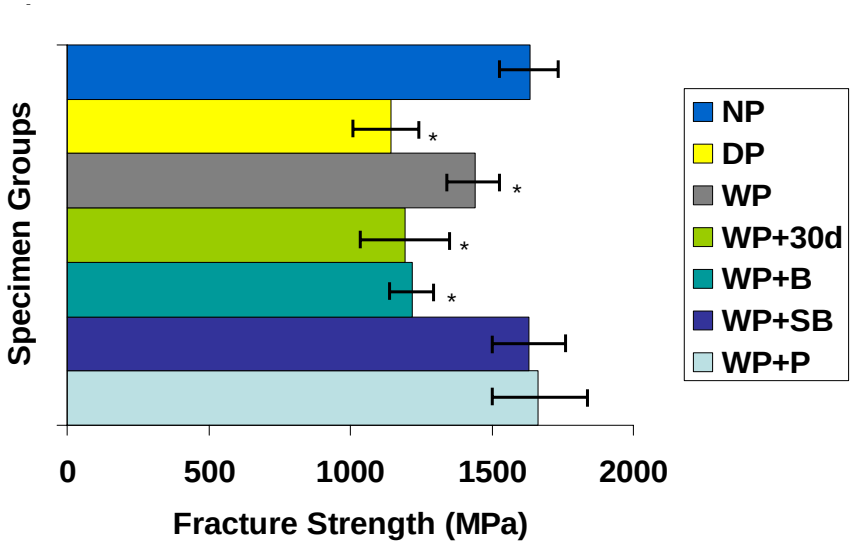


Figure 4.1 Flexure strength of Y-TZP bars reported in Table 4.1.

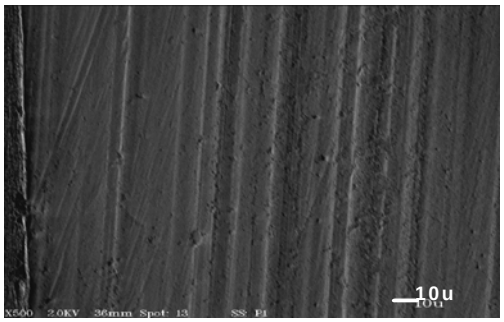
B. SEM examination of zirconia bar surfaces

To examine the potential surface flaws which may either have been introduced during preparation or which may have remained after attempts to repair the surfaces, SEM examinations of representative samples were performed. Micrographs representing each surface condition are shown in Figure 4.2.

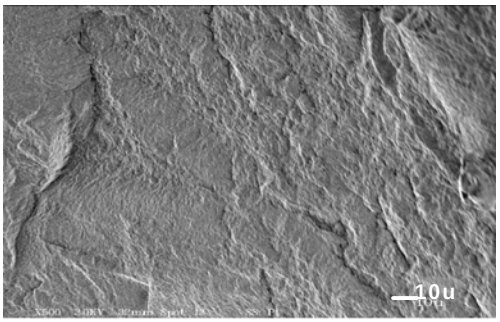
The unprepared surface (NP) was not as smooth as one might have expected. It appeared to contain some vertical striations, perhaps from the molds from which it was HIP-processed (Figure 4.2A). The striation pattern changes to a horizontal pattern when prepared by diamonds. The dry polished (DP) samples actually appeared smoother than the NP samples (Figure 4.2C). This might have been due to the presence of a fine smear layer embedded onto the surface that had not been effectively removed by any water during the procedure. Wet prepared surfaces (WP) were rough, as expected, but were not much rougher than the unprepared surface. However, these surfaces did seem to show some small pits where material may have been torn from the surface (Figure 4.2E). There was not much difference between the samples stored for 30 days in water (WP+30d) and the wet prepared ones (WP). Surfaces on specimens that had been coated with bonding agent (WP+B) were smoother but had some texture due to the unevenness of the bonding agent film. There

appeared to be crystalline particles on the surface, which could not be detected when looking at the fractured surface, although the surface of the bars was sticky to the touch (Figure 4.2I). Wet prepared specimens that were subsequently sandblasted (WP+SB) were the roughest looking of all groups and seemed to be crinkled. The crinkled appearance may simply have been due to the build up of a damage zone or smear layer. The area appeared to be uniformly damaged with rough surfaces with cracks randomly oriented (Figure 4.2K). Finally, the smoothest looking surface of all was associated with specimens which had been wet prepared and then polished (WP+P). However, several randomly-oriented scratches were still visible on them as well (Figure 4.2M).

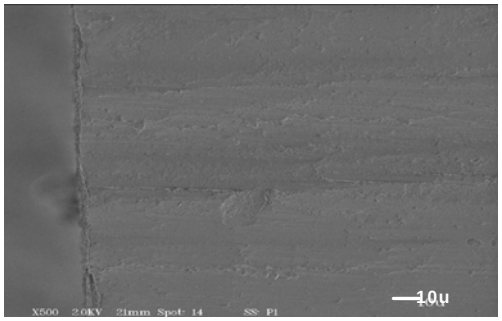
All the end surfaces of fractured bars looked approximately the same. There was evidence of fracture patterns that were all similar.



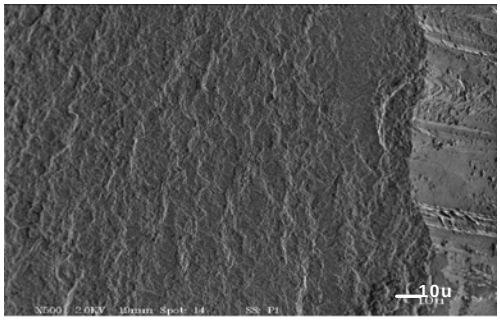
A. NP – HIP surface



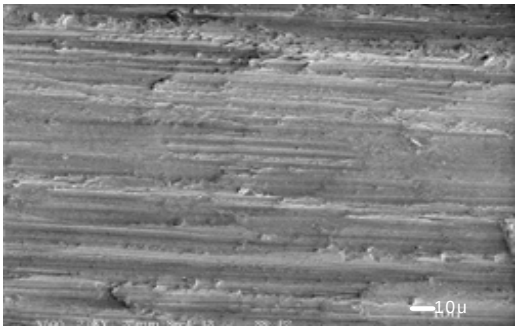
B. NP – fractured surface



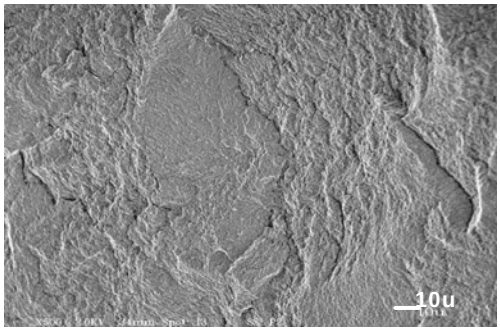
C. DP – prepared surface



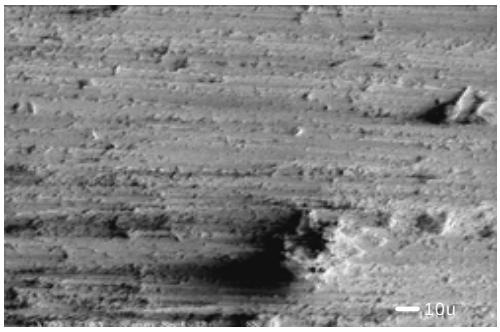
D. DP – fractured surface



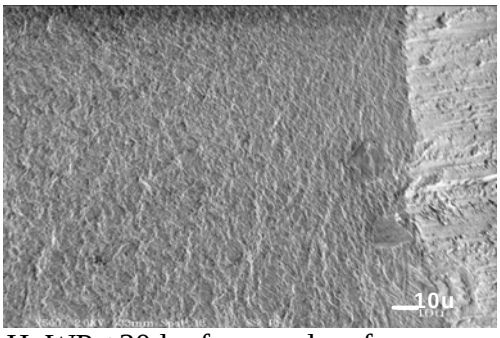
E. WP – prepared surface



F. WP – fractured surface

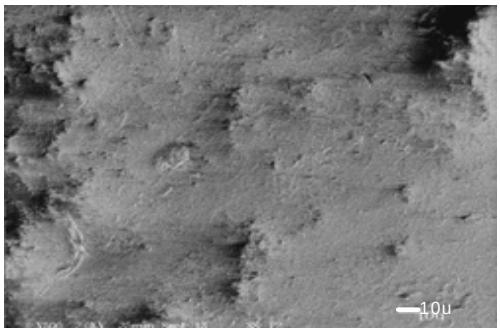


G. WP + 30d – prepared surface

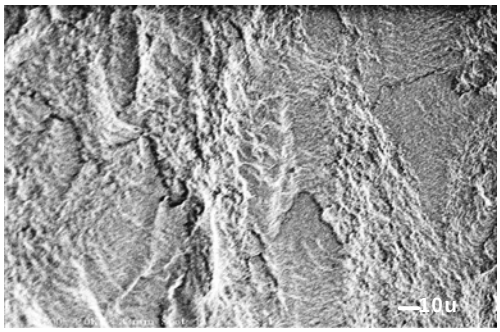


H. WP +30d – fractured surface

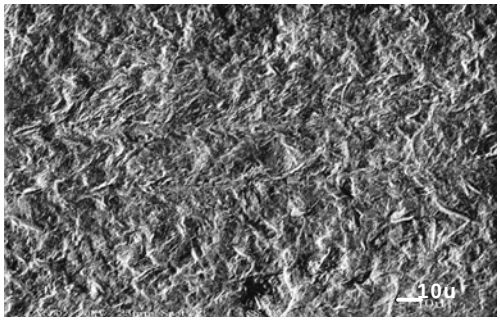
Figure 4.2 Representative SEM micrographs of the treated surfaces of test bars compared to the fractured surfaces of the test bars (control groups).



I. WP + B – prepared surface

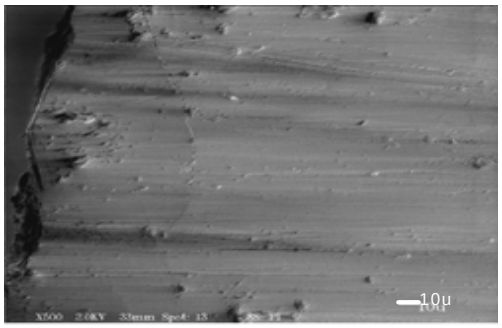


J. WP + B – fractured surface

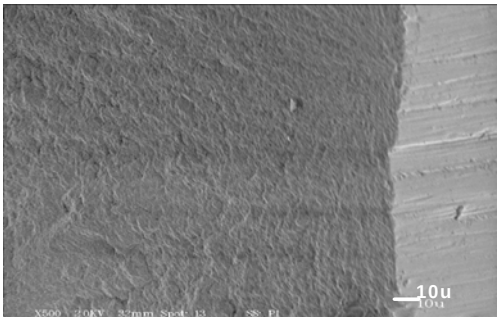


K. WP + SB – prepared surface

L. WP + SB – fractured surface



M. WP + P – prepared surface



N. WP + P – fractured surface

Figure 4.2 (continued) Representative SEM micrographs of the treated surfaces of test bars compared to the fractured surfaces of the test bars, (repaired groups).

C. Fracture loads for prepared zirconia abutments

During preparation of the zirconia abutments, sparks were commonly observed in the same way as with the preparation of the zirconia bars. Assemblies were all torqued to 25 N-cm, but by the end of testing, all screws had loosened.

The actual amount of axial reduction for individual abutments was measured and has been reported for each group in Table 4.2 below. While the original aim for reduction for the 0.5 mm group was achieved reasonably well, the intended reduction for the 1.0 mm group fell short of the target.

Table 4.2 Mean reduction of abutments in each specimen group

Specimen Aim	Mean Reduction (mm)		
	cervical	middle	incisal
0.5 margin	0.5±0.1	0.38±0.1	0.62±0.2
1.0 margin	0.8±0.1	0.72±0.2	0.94±0.2

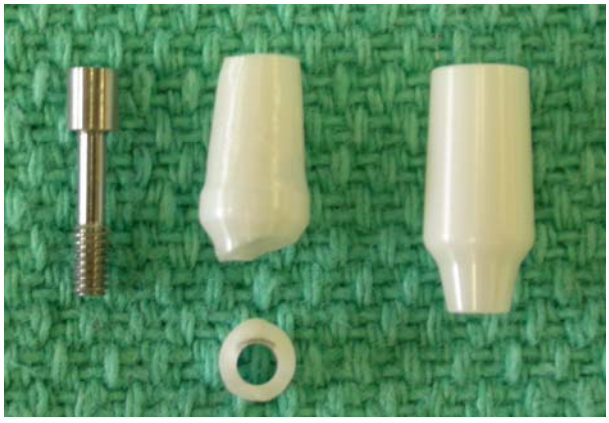


Figure 4.3 Indication where typical abutment fracture occurred.

Using the values from the mean reduction, it was possible to calculate the mean volumes of reduction as well and they have been reported in Table 4.3 below. Actual calculations for this transformation can be found in Figure D.1 in Appendix D. The failure loads for the assemblies for each group have been reported in the table below as well.

Table 4.3 Calculated mean volumes and strengths for each sample group.

Group	n	Mean Total Volume (mm^3)	Abutment Strength (N)	Screw Strength (N)	Assembly Strength (N)
Control	10	150±0	282±59	246±111	429±140
0.5mm	10	125±6	205±62	371±123	576±120
1.0mm	10	108±7	172±48	375±110	547±139

For each group a regression analysis was performed to determine the relationship among the failure loads and the volume of reduction. The regression coefficients (R^2) are shown below in Table 4.4 and were quite low, indicating that there did not seem to be any particular relationship at all. Part of the explanation may be related to the events associated with the failure that were revealed on the failure curves.

Table 4.4 Calculated regression coefficients and slopes for adjusted peak strengths of various groups.

Group, y (MPa) versus x (mm^3)	y=mx+b	R^2
Assembly Strength vs. Volume	y= -1.54x+736	0.0619
Abutment Strength vs. Volume	y= -3.67x+731	0.1320
Screw Strength vs. Volume	y= -3.76x+802	0.2850

A schematic representation of a typical direct loading curve for a prepared abutment assembly is shown below in Figure 4.4. Curves had been dissected into two different regions that seemed to represent the dominating events (abutment changes, screw changes) by guessing the contributions of the components to the overall behavior of the assembly.

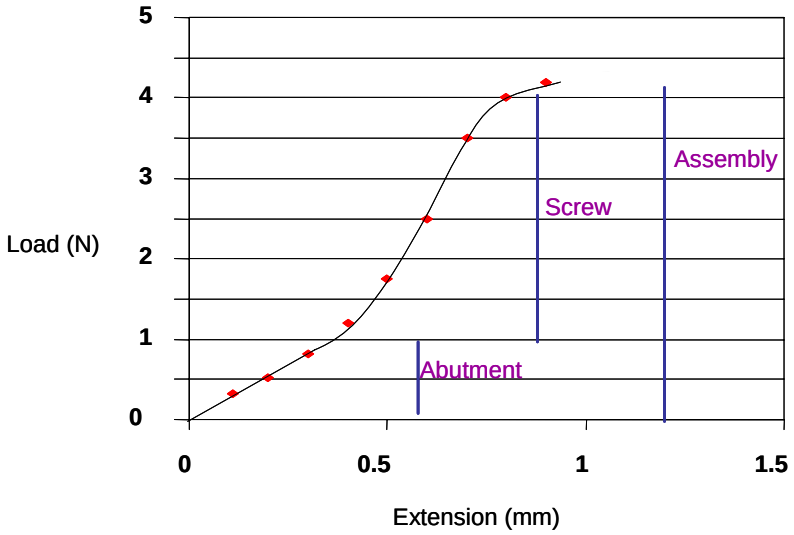


Figure 4.4 Schematic representative of typical load-deformation curve. (Vertical lines indicate the different assembly parts seemingly associated with the major deformations.)

Loads at failure may have been associated more with the screw in the assembly than the abutment. However, there was no straight-forward way to distinguish these two events.

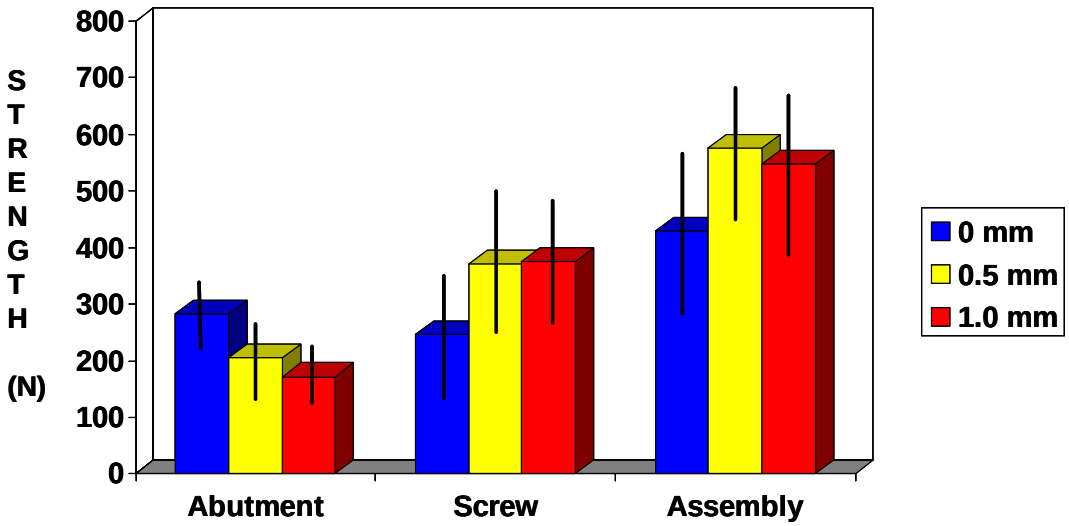
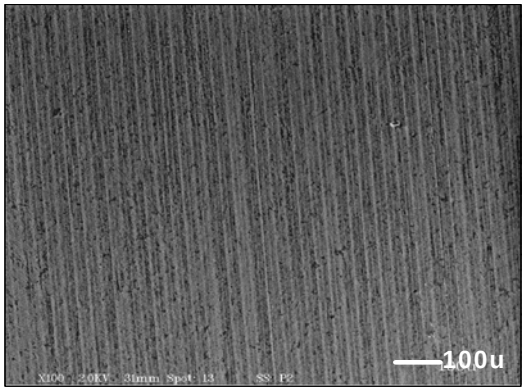


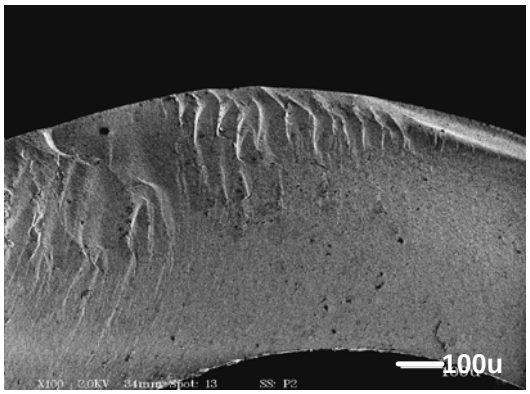
Figure 4.5 Peak load for each portion of the load-deformation curve depicted above in Figure 4.4 for each assembly group.

D. SEM of zirconia abutments

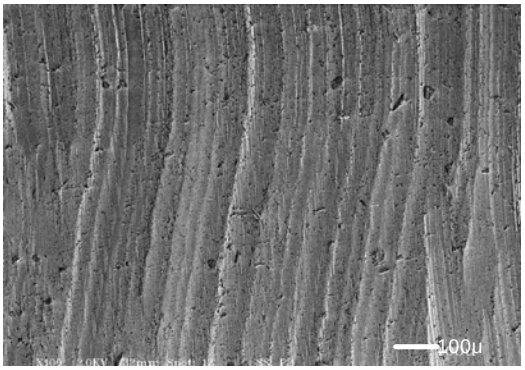
SEM images of the zirconia abutments are shown in Figure 4.6 below. The unprepared zirconia abutment surface showed evidence of what appeared to be the pattern from the inside of the HIP-processing mold that seemed to be a series of parallel ridges and valleys that were about equally spaced. The appearance was very similar to the one on the HIP-processed bars that had not been prepared (Figure 4.2A). The wet-prepared zirconia abutments were remarkably similar in appearance to the wet-prepared bars. The surfaces showed some roughness with occasional evidence of small pits as though material had been ripped from the surface during diamond bur cutting.



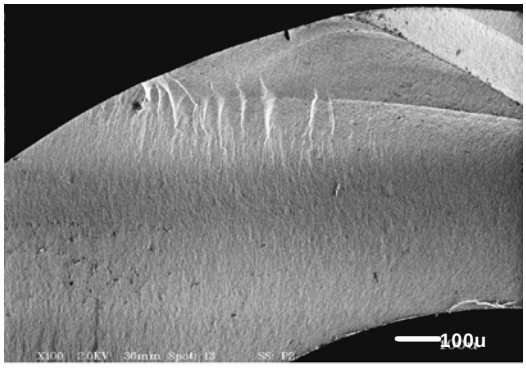
A. Control – HIP surface



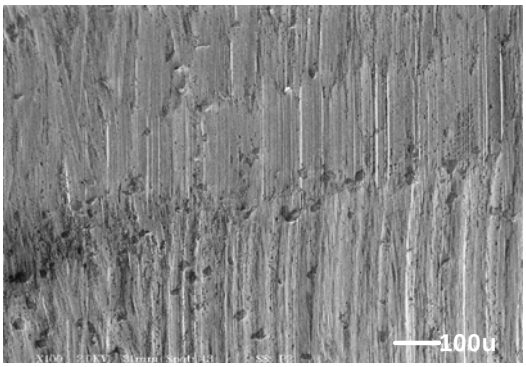
B. Control – fractured surface



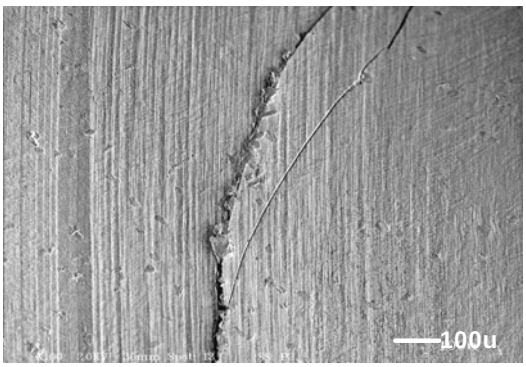
C. 0.5mm margin – prepared surface



D. 0.5mm margin – fractured surface



E. 1.0mm margin – prepared surface



F. 1.0mm margin – fractured surface

Figure 4.6 Representative SEM micrographs of the prepared surface of the abutments compared to the fractured surfaces of the abutments at different axial reductions.

DISCUSSION

The following discussion is divided into a careful reconsideration of the experimental design, an interpretation of the results, comparison of the results to the published literature, interpretation of the real clinical value of the results, and a list of recommendations for future research.

A. Critique of the experimental design

This study was designed to observe the effect of clinically-relevant surface treatments on the flexure strength of HIP-processed Y-TZP zirconia. The first experiment was designed to identify the effects of various surface treatments applied to HIP-processed bars. By testing the ceramic alone, all other variables associated with the assembly were removed from the experiment. Production of the bars was not simple, but the set-up of this experiment was. There was not much scatter in the results (see Table 4.1), indicating good intra-specimen reliability. In the second experiment, the actual effects on assembly failure of the amount of axial and marginal reduction on HIP-processed Y-TZP abutments (ZirDesign) from Astra Tech were tested.

A1. Testing of prepared abutment assemblies

The abutment assembly was oriented to test only the fracture of the ZirDesign abutment. The results, however, showed that the screw inside the abutment may have played a considerable role in the final strength values. It may be of some value to repeat the experiments utilizing a stainless steel screw, instead of the titanium screw provided, which would resist bending forces better and perhaps allow for a better visualization of the fracture

strength of the abutment alone. That being said, it is important to realize that the screw of the abutment will play a role *in vivo,* and it is important to know at what level it will fail.

Implant analogs were used in this study rather than implants. Any potential differences in the abutment torque into an analog versus an implant were not considered, but probably should be investigated. The analogs were made of stainless steel and the implants are made of titanium. Therefore, one could expect some difference in the interaction of the screw with analog versus implant. However, the bending of the screw seemed to be the first location that change was observed.

To delineate the events associated with the load-deformation curve for the abutment assemblies, it might be worthwhile to stop the testing prior to failure and examine the position and appearance of various parts of the assembly. It was assumed from analyzing the stress-strain curves that the abutment peak load averaged 282 N for the control group. To determine when the abutment actually began to fail and when the screw bending took place, assemblies could be loaded in stages. For example, an assembly could be loaded to 50 N, embedded in acrylic, cut lengthwise, and examined to determine what had occurred. This could be done at loads of 50, 100, 150, 200, 250, 300 and 350 N.

During preparation of the specimens, an effort was made to ensure that the amount of material removed was uniform and that all specimens were treated equally. However, different lot numbers of abutments were provided by the manufacturer for these tests. The small number of specimens being tested may have explained why the standard deviation was so large.

A2. Testing of the assemblies as a function of different variables

As reported in the literature, various angles have been used to test maxillary central incisors to replicate clinical setting *in vitro.* Only one prior study has observed the effects of varying the angle at which the teeth are loaded (Loney *et al.,* 1995). They reported that angles of 110 and 130° from the horizontal were significantly different than 150° from the

horizontal. Mean fracture loads increased as the angle increased and the loading angle approached being parallel to the long axis of the tooth.

The present study tested the assemblies at 60° from the horizontal (or 120°), which corresponds to the values given by Proffit (2000) as 22-24° from the NA line. Assuming the NA line is perpendicular to the horizontal, this would be equivalent to 66-68° from the horizontal.

Assemblies were loaded at 0.1 mm/min crosshead speed, and that was a lower loading rate than other studies which used rates of 1-2 mm/min (Norton, 2000; Strub and Gerds, 2003). Typically, increasing the loading rate by of an order of magnitude such as this would increase the results by 20-50% in value. Assemblies could have been tested at a higher loading rate to determine any effects. The rate of 0.1 mm/min, however, should be low enough to simulate intraoral loading. For most dental materials testing, a low loading rate is presumed. A typical range of choices is 0.1 to 1.0 mm/min depending on the specimen dimensions. This is a typical loading rate chosen in most ADA or ISO testing regimes and is assumed to reflect the typical intraoral situation.

A3. Testing of bars

Bars were tested 48 hours after surface preparation. Specimens could have been stored much longer after surface preparation to be sure all residual stresses had been eliminated before 3-point testing. Most bars were kept dry prior to testing. Perhaps bars should be stored wet in future tests. However, first it is important to understand the differences between dry and wet conditions on the strength.

Bars were tested at a crosshead speed of 0.1 mm/min. Other studies have used 0.1 mm/min (de Jager *et al.*, 2000), 0.5mm/min (Guazzato *et al.*, Albakry *et al.*) and 1 mm/min (Luthardt *et al.*, 2002). The International Standards ISO 6872 recommends a speed of 0.5 mm/sec for their standard geometry. For any particular specimen size, the loading rate needs to be adjusted appropriately to produce the same actual specimen strain rate. Thinner specimens would require a lower loading rate to produce the same strain rate. The loading

rate of 0.1 mm/min used for the current experiments also seemed to be low enough to appropriately mimic intraoral loading rates and produce proper strain rates.

Heat-treatment of the bars was considered as a potential effect in the planning of the study but rejected as impractical for most clinical situations. Clearly, heat treatment would be able to alter the microstructure and presumably recover tetragonal zirconia.

A4. Flexure tests for bars

There are several testing modes for the mechanical properties for dental ceramics including tensile tests, compressive tests, flexure tests, hardness tests, fracture toughness tests, and diametral tensile tests (Jin *et al.*, 2004). Although it is commonly known that brittle materials such as dental ceramics should be weaker in tension than in compression (Jin *et al.*, 2004, Zeng *et al.*, 1996), flexure tests (and not tensile tests) are frequently used to more conveniently test ceramics. (Jin *et al.*, 2004, Zeng *et al.*, 1996, Vallo, 2002)

Several options are available for flexure test analyses such as the 3-point bending test, 4-point bending test, and variations on the design of the biaxial flexure test. The American Society for Testing and Materials (ASTM) and the International Standards Organization (ISO) both now recommend the biaxial flexure test for determining the strength of ceramic substrates. However, ISO standards for dental applications currently recommend 3-point bending for ceramic specimen tests (Jin *et al.*, 2004).

There has been much debate as to the merits of 3-point versus 4-point bending tests. It has been shown that 3-point tests give a significantly higher measured flexure strength than the 4-point test on the same material (Jin *et al.*, 2004, Vallo, 2002). This seems to be due to the fact that the 3-point test has a smaller amount of surface or volume subjected to a maximum tensile stress versus a larger loading span of the 4-point test (*i.e.*, 1.6 mm diameter loading area versus 8 mm loading area)(Jin *et al.*, 2004; Zeng *et al.*, 1996). The 3-point bending test has been used to predict biaxial flexure strength. These two methods may lead to the same statistical failure stress provided the area of the specimen under the maximum load is considered (Zeng *et al.*, 1996). Since it has been shown that the 3-point bending test

can provide similar failure stress as the biaxial flexure test, and it has been recommended by the ISO. Thus, the 3-point bending test was selected for the present study.

B. Interpretation of the results

B1. Stages of assembly failure

A schematic curve representing the abutment assembly failure was shown in Figure 4.3. The first part of the curve seemed to be associated with the abutment becoming loaded. This part of the curve was usually a linear. At some point, the angle of the curve began to change into a sigmoidal curve which was interpreted as the plastic deformation of the screw, ending with the screw peak load. As mentioned earlier, it was difficult to dissect each part of the curve without performing segmented sequential loads and evaluating the abutment under SEM to see when crack formation occurs. Some authors have reported that the use of gold screws and a controlled torque should reduce the rate of failure as compared to titanium screws (Butz *et al.*, 2005; Strub and Gerds, 2003). However, no evidence for this was uncovered in the literature.

The titanium retaining screw provided by the manufacturer was used throughout the current study. This was the original hexed screw with a square head. The new screw that Astra Tech now provides with the abutment system is a rounded Ti-alloy screw head. The dimensions of the screw are the same, but the head itself has been rounded to reduce stress points and allow for more material bulk.

Yildirim *et al.* (2003) and Mitsias (2003) both found that when zirconia abutment assemblies failed, they failed at the cervical portion of the abutment, near the gold screw and platform of the implant. Yildirim *et al.* tested the Branemark flat-top hex implant-abutment system. Mitsias tested the AstraTech internal conical seal system. Fixtures with an external-hex have shown an increase in strain at the cervical area under horizontal load, while internal-hex fixtures produce more strain at the fixture tip area. It has been suggested that fixtures with internal-hex show more widely spread force distributions down to the fixture tip as compared with external hex ones (Maeda *et al.*, 2006). Khraisat *et al*. (2002) found that

the effect of joint design on the fatigue strength and failure mode of the ITI internal hex system was significantly better than that of the Branemark external hex single-tooth implant system. Yildirim *et al.* (2003) presumed this area to be an area of the highest torque and stress concentrations due to the levering effects. It would be nice to attempt a finite element analysis (FEA) to see if stresses were actually distributed in this manner.

B2. Failures of bars

The impact of dry preparation (no water irrigation) caused the greatest decrease in flexure strength of the Y-TZP bars. Although the level of monoclinic change was not measured in this study, it was assumed that any heat generated from dry grinding caused microstructural changes that reversed the effects of compressive strengthening. Water irrigation seemed to provide a sufficient cooling for the present experiments so that the bars did not overheat. This was inferred by comparing the dry prepared bars with the wet prepared bars under the same conditions. The fracture strength was statistically higher for the wet prepared group. Blue *et al.* (2003) has argued that water irrigation has the effect of increased preparation efficiency by cleaning the debris from away from the abrasive particles on diamonds.

Sandblasting the bar surface created damage that was detected in the SEM pictures. Despite the fact that sandblasting induced surface flaws, the resultant flexure strength increased. This appears to be due to the fact that a tetragonal to monoclinic phase transformation occurred on the sandblasted surface. Although this was not measured directly in this study by analyzing the phases, this effect has been reported in the literature (Guazzato *et al.*, 2005). The transformation to a monoclinic phase creates a layer of compressive stresses which counteracts the sandblasting damage. Kosmac et al. (1995) found that the actual surface flaw sizes which were introduced by sandblasting did not exceed the thickness of the compressive stress layer and, therefore, the strength increased instead.

Polishing the surfaces of bars after diamond preparation also increased the flexure strength of the bars. This is in contrast to what has been stated in the literature (Guazzato *et*

al., 2005) who found that fine polishing decreased the monoclinic phase content of the bar's surface and resulted in lowered flexure strength. Bars in the current experiments were polished until no scratches could be detected with the naked eye. Yet, analysis in the SEM quickly revealed that scratches were still evident along the tested surface. It was presumed that these scratches were small enough that they did not represent defects of sufficient size to contribute to crack formation and failure.

Optibond-Solo-Plus was used to create a polymer film on the surface of zirconia bars and fill in the defects left by preparation. This was an attempt to see if it was possible to buffer the effects of preparation and cause a recovery in flexure strength. In fact, this did not work at all, and this treatment group had the third lowest flexure strength. The polymer film appeared to embed some of the debris on the prepared surface. If the bonding film treatment had no effect at all, then strength should have been comparable to the wet prepared group. However, this group was actually worse. The reason for this has remained elusive.

The wet prepared group which was then left in water for one month was intended to simulate short-term intraoral conditions. The reason for dramatic reduction in strength seems to be related to the action of water encouraging crack propagation. Marx *et al.* (2004) found that zirconia was extremely sensitive to humidity. They reported that zirconia which had been exposed to 100% humidity fractured at a level 25% lower than if it were exposed to only 60% humidity (*i.e.*, dental laboratory conditions). The present results are in agreement with those of Marx *et al.* since the currently observed strengths were 17 % below those for the un-stored specimens.

C. Comparison of the results to the literature

C1. Comparison of results of assemblies to the literature

A summary table of all the published results for static and fatigue testing are reported in Table 5.1 for reference. Differences in testing conditions and implant designs led to a wide range of strengths simulations of implant assemblies.

Table 5.1. Comparison of different studies observing fracture strength involving implant abutments.

Reference	**Assembly tested**	**Test dimensions**	**Testing from vertical**	**Fracture or residual strength**
Butz *et al.*	Al_2O_3 abutment; Prepared; metal crown; gold retaining screw	Fatigued 1,200,000 c; 30 N load at 1.3Hz	50°	239±83 N
Butz *et al.*	Titanium abutment; Prepared; metal crown; gold retaining screw	Fatigued 1,200,000 c; 30 N load at 1.3Hz	50º	324±85 N
Butz *et al.*	Zirconia abutment; Prepared; metal crown; gold retaining screw	Fatigued 1,200,000 c; 30 N load at 1.3Hz	50º	294±53 N
Yildirim *et al.*	Al_2O_3 abutment; Prepared; all-ceramic crown; gold retaining screw	Static loading; 5 N preload; CHS = 0.1 mm/min	30º	280±103 N
Yildirim *et al.*	Zirconia abutment; Prepared; All-ceramic crown; gold retaining screw	Static loading; 5 N preload; CHS = 0.1 mm/min	30º	788±273 N
Strub and Gerds	Titanium abutment (Steri-Oss/Novostil); metal crown; gold retaining screw	Controlled static loading; CHS = 2 mm/min; Fatigued 1,200,000 c; 50 N load at 1.6Hz,	45º	*Control:* 537 N *Fatigued:* 694 N
Strub and Gerds	Titanium abutment (Steri-Oss anatomic abutment); metal crown; titanium retaining screw	Controlled static loading; CHS = 2 mm/min; Fatigued 1,200,000 c; 50 N load at 1.6Hz	45º	*Control:* 817 N *Fatigued:* 750 N
Strub and Gerds	Titanium abutment (Steri-Oss Straight HL); metal crown; titanium retaining screw	Controlled static loading; CHS = 2 mm/min; Fatigued 1,200,000; 50 N load at 1.6Hz	45º	*Control:* 893 N *Fatigued:* 867 N
Strub and Gerds	Titanium abutment (IMZ/Esthetic abutment); metal crown; titanium retaining screw	Controlled static loading; CHS = 2 mm/min; Fatigued 1,200,000 c; 50 N load at 1.6Hz	45º	*Control:* 473 N *Fatigued:* 484 N
Strub and Gerds	Titanium abutment (Osseotite/UCLA); metal crown; gold retaining screw	Controlled static loading; CHS = 2 mm/min; Fatigued 1,200,000 c; 50 N load at 1.6Hz	45º	*Control:* 743 N *Fatigued:* 750 N
Mitsias	Titanium abutment; metal crown; titanium retaining screw	Static load; CHS not mentioned	30º	1475±625 N
Mitsias	Zirconia abutment; Prepared; metal crown; titanium retaining screw	Static load; CHS not mentioned	30º	690±430 N
Current Study	Zirconia abutment; Prepared; titanium retaining screw	Static load; CHS 0.1 mm/min	30º	*Control:* 429±140 N *0.5mm:* 576±120 N *1.0mm:* 547±139 N

Fracture loads reported by Butz *et al.* (2005) for fatigued zirconia abutments were 294 ± 53 N. This value was reported for abutments loaded 50º from the vertical. They found that in 63% of their specimens, abutment screw deformation occurred, despite the use of a gold screw. They did, however, find no screw loosening after fatiguing the zirconia specimens. In the present study, all titanium screws were loose after static loads were applied. This could be due to the plastic deformation of the titanium screw occurring over a longer period of time (CHS = 0.1 mm/min compared to 1.5 mm/min (Butz *et al.*, 2005)), as well as other factors, such as the lack of an excellent fit into the analog.

Yildirim *et al.* (2003) prepared zirconia abutments with a 1.0 mm chamfer, 1.5 mm axial reduction, and 4 mm clearance. They tested the abutment assemblies (abutment, gold retaining screw, luting cement, and all-ceramic crowns) under static load until failure. They found that for 40% of the specimens the all-ceramic crown failed before the abutment failed, in 30% of the assemblies the abutment fractured before the all-ceramic crown fractured, and in the remaining 30% of assemblies the gold screw failed before either the abutment or the all-ceramic crown failed. The fracture strength of zirconia abutments was 788 N and ranged from 619 to 1366 N. The authors claim that since the all-ceramic crown was the weakest portion of the assembly, the abutment did not affect the fracture toughness of the assembly. In the present study, the choice was made to eliminate this variable and test only the abutment assembly consisting of the abutment and retaining screw.

In unpublished data, Mitsias (2003) reported in his pilot study, ZirDesign abutments had static flexure strength of 690 ± 429 N. Abutment assemblies were tested (abutment, titanium retaining screw, luting cement, and crown). He found the weakest part of the ceramic abutment was at the hex portion of the abutment body. Additionally, the abutment body fractured into many pieces. Abutments tested in the present study were non-ST components, and did not contain a hex. Therefore, the observed failures were within the abutment body, as well as in the area where the abutment connected to the implant analog (*i.e.*, the conical seal).

Flexure strength of the ZirDesign abutments under static loading of assemblies was lower than reported by Mitsias (2003) or Yildirim *et al.* (2003). The flexure strength of the control group (no preparation) was statistically different ($p<0.05$) from both of the tested groups but no trend was immediately apparent. To try to understand this situation, the results were corrected on the basis of the total remaining volume of the abutment versus the flexure strength, but no correlation was observed. This seemed to indicate that although there appeared to be a difference in the flexure strength of control versus prepared abutments, this might not be a real difference. With greater sample group size, there may turn out not to be a real difference (Type I error).

C2. Comparison of results of bars to literature

No study has yet shown that applying surface treatments to prepared surfaces will increase the flexure strength of zirconia. Guazzato *et al.*, found that sandblasting HIP-processed bars gave flexure strengths of 1540 MPa. Unfortunately, their results did not include any control group. Kosmac *et al*. (1999) found that sandblasting specimens produced higher flexure strengths (1239MPa) than the original as-sintered control group. They also found that sandblasting specimens produced lower local temperatures encouraging less tetragonal-to-monoclinic transformation. Results reported from the present study showed similar high values for sandblasting (1632 MPa) that were within the range of what has been reported by other researchers. Zhang *et al.* (2004) found that sandblasting caused very large reductions (up to 30%) in the fracture strength for zirconia when fatigue cycled to simulate oral conditions. It was stipulated that the sandblasting induced true microcracks which are not detectable by SEM. Cyclic loading exacerbated the crack formation by mechanical degradation, perhaps by continual "reduction of friction of microcrack walls in repeated shear sliding" (Zhang *et al.*, 2004).

Results of the current study showed that step-wise polishing of zirconia bars increased the flexure strength (1664 MPa) to levels equal to unprepared bars. Guazzato *et al.* (2005) found that polishing unprepared HIP bars actually reduced the flexure strength

(1095MPa). They attributed this change to the negligible amounts of monoclinic phase found after polishing. It may be, however, that grinding treatments which had been shown to increase the monoclinic phase change, may have plateaued with polishing to an amount that created favorable compressive stresses to counteract the grinding flaws. There was no information available about whether polishing produced any heat or not that could have contributed to a monoclinic transformation. Guazzato *et al.* (2005) also found that wet grinding did not cause a significant fall in the flexure strength, although results from Kosmac *et al*. (1999) and Luthardt *et al*. (2002) both supported results from the current experiment that both wet and dry grinding do decrease flexure strength. Guazzoto *et al.*, (2005) tested their specimens at a speed of 3300 rpm with a 91µm grit diamond under water coolant, where as Kosmac *et al.,* tested his specimens with a coarse diamond under dry conditions with 150000 rpm. The differences in preparation conditions probably altered the specimen temperatures and microstructural phases, resulting in differing tetragonal and monoclinic phase contents. Kosmac *et al*. (1999) attributed the fall in flexure strength to deep surface flaws whose length exceeded the depth of the grinding induced compressive layer. Kosmac *et al.* (1999) found that dry and wet grinding caused 53% and 63% reduction in flexure strength, respectively. They found no difference between wet and dry grinding, unlike the results of the present study, which found that dry and wet grinding caused a 70% and 88% reduction in flexure strength, respectively.

No one yet has examined the effect of a bonding or glazing film on the mitigation of potential surface damage caused by grinding. Guazzato *et al.* did look at heat treatments to high temperatures characteristic of ceramic glazing procedures and found that monoclinic-to-tetragonal phase transformation did occur, almost eliminating the monoclinic phase and resulting in lower flexure strengths. They assumed that heat treatments released the compressive stresses gained by the monoclinic phase transformation that occurred during preparation or surface treatments, thereby revealing the full effects of the defects produced by either sandblasting or diamond preparation.

Kosmac *et al.* (2000) found that when Y-TZP was exposed to an aqueous environment above 100ºC over long periods of time, that Y-TZP spontaneously started to transform to the monoclinic structure. This transformation was diffusion controlled and was accompanied by extensive microcracking that led to strength degradation. This strength reduction was similar to what was found in the current experiments when placing wet prepared specimens into water at 25 ºC for 30 days. Pre-existing monoclinic zirconia on the surface of the sandblasted zirconia hindered the propagation of the diffusion-controlled transformation during subsequent exposure to aqueous environments. However, low temperature strength degradation of sandblasted material was likely to occur sooner because the total amount of transformed zirconia was higher than for the surface of material that had less monoclinic phase after the same exposure time (Kosmac *et al.* 2000).

D. Clinical meaning of the results

The separation of the present experiment into two stages revealed information about the overall assembly behavior and the separate behavior of the zirconia itself. These are now considered separately.

D1. Clinical meaning of static results of assemblies and bars

It may seem clinically desirable to have a large amount of monoclinic phase due to its compressive effect and potential increase in flexure strength. However, large amounts of monoclinic phase on the surface also may lead to microcracking and predispose the material to a more rapid moisture-assisted transformation with time and/or energy from mechanical loading than for a surface with a low monoclinic content (Guazzato *et al.*, 2005). Procedures which provide an initially weaker, but more stable material may be more desirable. Polishing of zirconia has been shown to leave very little monoclinic phase on the surface. However, it still increased the flexure strength of zirconia. Polishing may be the most promising clinical treatment that can be accomplished by either the lab or by the dentist at chairside.

The current study only considered static loading. Zhang *et al.* (2004) showed that although static loading showed only small changes in flexure strength with sandblasting,

cyclic loading showed a substantial (30%) decrease in flexure strength. Abutments intraorally failed from continual cyclic loading via subsequent crack propagation.

Some *in vitro* studies have been conducted using cemented crowns. To date, however, effects of the cement and restoration on stress-shielding and/or blunting of any surface defects on the abutment have not been examined. There have been no published clinical trials utilizing the ZirDesign or ZirReal abutments. However, a clinical trial utilizing the CerAdapt alumina abutment was performed by Andersson *et al.* (2001) who found that 12% of the abutments failed before loading and 7% failed after seven months. In contrast, there was 100% success with CeraOne titanium abutments.

D2. Restorations linked to abutment success.

It is very important to understand the potential clinical success of zirconia abutments. These abutments are used for cemented restorations and a catastrophic fracture of these abutments would require not only replacement of the abutment, but also replacement of the overlying crown. Zhang *et al.* (2004), using cyclic studies, predicted that zirconia abutments could last over 10 years. Unfortunately, their study did not consider cyclic loading in an aqueous environment or in an acidic/alkaline environment. Therefore, it would not be wise to extrapolate those results to the mouth.

E. Suggestions for future research

In order to explain the effects on zirconia abutments, it is important to fully understand the nature of the zirconia itself. There should be a method of characterizing the bulk phases present before and after testing. This would permit much better structure-property understandings to occur.

It is now apparent that several other variables are important and need further consideration. These are discussed as follows.

E1. Need to test effects of water on bars

Humidity effects on zirconia need to be evaluated. It is important to conduct an experiment testing the effects of storage time within an aqueous environment on crack

propagation and measured flexure strength. Bars could have their surfaces prepared wet and then be stored in water for 30 d, 6 m, and/or 1 y before being tested for residual strength.

The protective effects of cement and a crown could be simulated using a thin layer of cement and porcelain over top the prepared surface. These specimens could then be stored for similar time periods in water or artificial saliva to determine if pH and/or proteins in the saliva might have an effect on the resultant strength.

E2. Fatigue stressing of bars

Since static loading does not simulate intraoral conditions, cyclic testing should be conducted on bars after similar surface treatments to the present study. Polymer films can be excluded in future experiments, since that treatment did not cause any improvement in flexure strength. The most practical approach of examining fatigue effects has been to control the maximum load, cycle the bars for different times (100,000 or 500,000 or 1,000,000 cycles), and then test for residual strength. This should provide more meaningful estimates of actual clinical performance.

E3. Surface treatments on dry preparation of bars

The effect of surface treatments on dry preparation of bars should be evaluated as well. It is quite possible that surface treatments on zirconia may not be properly conducted by different operators with adequate water cooling. This could contribute a further decrease in the fracture strength.

E4. Testing of other zirconia abutment designs

All of the current tests were performed on a single abutment design. As intimated in the interpretation of the testing of assemblies, there seemed to be major contributions of other components in the assemblies such as those of the screw. It would be instructive to compare several assemblies for this reason.

E5. Monitoring of the phase reactions on surfaces of treated bars

It would be very helpful to characterize the state of phase transformation (tetragonal to monoclinic) on the surfaces of zirconia specimens. For flat bars, this might be done easily

as low angle x-ray diffraction to explain the relative strength of the surface layer after dry preparation, wet preparation, or sandblasting treatments.

CONCLUSIONS

Within the limitations of these experiments, the following could be concluded:

1) Any type of grinding on zirconia will cause a reduction in its fracture strength.

2) Dry preparation significantly reduces the fracture strength of zirconia bars.

3) Wet preparation defects can be repaired to pristine strengths using sandblasting or polishing.

4) Water appears to cause an increase in crack propagation, giving lower fracture strengths, similar to dry preparation.

5) Polymer film on prepared zirconia surface was not effective in repairing defects and recovering fracture strength.

6) Margin preparation of abutments up to 1.0 mm did not seem to adversely affect the fracture strength of abutment assemblies.

7) The weakest point of the abutment assemblies seemed to be the abutment-analog interface.

APPENDICES

The following appendices contain tables describing the individual experimental specimen data that was summarized in the Results section of this thesis. All of the statistical analysis associated with these experiments is reported in tables as well.

A. Flexure strength of bars

Twelve HIP-processed Y-TZP bars were tested after bur roughening and subsequent surface treatments. All but two groups were roughened under water irrigation and tested for fracture strength with a three point bending test, two days after they were bur roughened. The results of individual specimen tests are reported in the following tables.

Table A1. Flexure strength testing of HIP processed Y-TZP bars unprepared.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.03	2.05	965	1697
2	2.08	2.05	799	1372
3	2.01	2.05	976	1733
4	2.06	2.04	980	1715
5	2.08	2.05	918	1575
6	2.07	2.06	957	1634
7	2.05	2.04	939	1650
8	2.13	2.04	960	1625
9	2.07	2.04	955	1663
10	2.07	2.04	915	1594
11	2.08	2.04	970	1680
12	2.05	2.04	948	1667
Mean	**2.07**	**2.05**	**940**	**1634**
St Dev	***0.03***	***0.01***	***49***	***95***

All bars were tested on the same day within the same time frame (3/18/2005)

Table A2. Flexure strength testing of HIP-processed Y-TZP bars prepared without water irrigation.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.02	2.02	584	1062
2	2.03	2.00	670	1238
3	2.03	2.01	669	1223
4	2.64	2.01	760	1069
5	2.03	2.01	681	1245
6	2.03	2.02	557	1009
7	2.04	2.02	637	1148
8	2.33	2.01	808	1288
9	2.04	2.01	653	1189
10	2.04	2.01	570	1037
11	2.05	2.01	535	969
12	2.03	2.03	695	1246
Mean	**2.11**	**2.01**	**652**	**1144**
St Dev	***0.19***	***0.01***	***82***	***109***

All bars were tested on the same day within the same time frame (6/03/2005)

Table A3. Flexure strength testing of HIP-processed Y-TZP bars prepared under water irrigation.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	1.97	1.93	797	1629
2	1.94	1.94	681	1399
3	1.97	1.97	765	1501
4	1.98	1.97	738	1441
5	1.97	1.97	745	1463
6	1.97	1.96	712	1411
7	2.04	2.01	814	1482
8	2.04	2.02	813	1466
9	2.03	2.00	743	1373
10	1.98	2.02	697	1294
11	2.02	2.02	734	1336
12	2.03	2.01	829	1515
Mean	**2.00**	**1.99**	**756**	**1443**
St Dev	***0.03***	***0.03***	***48***	***89***

All bars were tested on the same day within the same time frame (6/03/2005)

Table A4. Flexure strength testing of HIP-processed Y-TZP bars prepared under water irrigation, and then left for 30 days in sterile, room temperature water.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.20	2.04	714	1170
2	2.06	2.03	689	1217
5	2.10	2.03	855	1482
7	2.07	2.03	690	1213
9	2.07	2.03	549	966
10	2.20	2.04	869	1424
11	2.27	2.01	789	1291
12	2.04	2.06	664	1151
13	2.32	2.05	744	1145
14	2.05	2.06	558	961
15	2.06	2.05	684	1186
16	2.05	2.06	642	1107
Mean	**2.12**	**2.04**	**704**	**1193**
St Dev	***0.10***	***0.02***	***100***	***155***

All bars were tested on the same day within the same time frame (4/18/2005)

Table A5. Flexure strength testing of HIP-processed Y-TZP bars prepared under water irrigation, followed by surface treatment with Optibond -Solo-Plus bonding agent.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.03	2.06	700	1219
2	2.04	2.04	735	1298
3	2.03	2.04	733	1302
4	2.02	2.04	670	1196
5	2.01	2.05	779	1383
6	2.02	2.04	667	1190
7	2.03	2.03	624	1118
8	2.02	2.03	653	1176
9	2.03	2.02	682	1235
10	2.05	2.05	676	1178
11	2.02	2.05	634	1121
12	1.74	2.03	575	1202
Mean	**2.00**	**2.04**	**677**	**1218**
St Dev	***0.08***	***0.01***	***55***	***77***

All bars were tested on the same day within the same time frame (6/03/2005)

Table A6. Flexure strength testing of HIP-processed Y-TZP bars prepared under water irrigation, followed by sandblasting (50 μm Al_2O_3) surface treatment.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.03	2.01	1079	1973
2	2.03	2.03	958	1718
3	2.03	2.03	757	1357
4	2.01	2.03	764	1383
5	2.03	2.01	966	1767
6	2.02	2.02	895	1628
7	2.03	2.03	929	1666
8	2.03	2.03	968	1735
9	2.03	2.03	867	1555
10	2.02	2.02	1005	1830
11	2.02	2.01	957	1758
12	1.72	2.02	747	1596
Mean	**2.00**	**2.02**	**908**	**1664**
St Dev	***0.09***	***0.01***	***105***	***176***

All bars were tested on the same day within the same time frame (6/03/2005)

Table A7. Flexure strength testing of HIP-processed Y-TZP bars prepared under water irrigation, followed by polishing (low speed Dialite burs) surface treatment.

Specimen	Width (mm)	Thickness (mm)	Peak Load (N)	3pt Flexure Strength (MPa)
1	2.04	2.04	989	1747
2	2.03	2.01	980	1792
3	2.03	2.00	881	1627
4	2.03	2.01	935	1709
5	2.04	2.02	948	1708
6	2.04	2.03	927	1653
7	2.04	2.04	798	1410
8	2.05	2.01	799	1446
9	2.01	2.01	960	1773
10	2.29	2.03	901	1433
11	2.04	1.98	857	1607
12	2.02	2.01	917	1685
Mean	**2.06**	**2.02**	**907**	**1632**
St Dev	***0.07***	***0.02***	***64***	***134***

All bars were tested on the same day within the same time frame (6/03/2005)

B. Fracture strength of Y-TZP abutments

Three groups of Y-TZP abutments were prepared under water irrigation with 0 mm, 0.5 mm or 1.0 mm margins. All abutments were tested on a 60-degree angle using the Instron machine.

Table B1. Measured failure load of HIP processed Y-TZP abutments at a 60 degree angle, unprepared.

Specimen Number	N (Load to Fracture)	Date Tested
1	645	6/7/2005
2	218	6/7/2005
3	394	6/7/2005
4	230	6/7/2005
5	327	6/7/2005
1	584	8/25/2004
2	518	8/25/2004
3	392	8/25/2004
4	528	8/25/2004
5	509	8/25/2004
Mean	435	
St Dev	146	

Table B2. Measured failure load of HIP-processed Y-TZP abutments at a 60-degree angle, with a 0.5 mm margin and prepared with 2 mm of occlusal reduction.

Specimen Number	N (Load to Fracture)	Dates Tested
1	407	6/6/2005
2	477	6/6/2005
3	574	6/6/2005
4	391	6/6/2005
5	617	6/6/2005
1	756	8/25/2004
2	796	8/25/2004
3	663	8/25/2004
4	579	8/25/2004
5	603	8/25/2004
6	624	8/25/2004
7	795	8/25/2004
11	719	8/25/2004
12	658	8/25/2004
13	535	8/25/2004
Mean	613	
ST Dev	126	

Table B3. Measured failure load of HIP processed Y-TZP abutments at a 60 degree angle, prepared with a 1.0mm margin and 2mm occlusal reduction.

Specimen Number	**N (Load to Fracture)**	**Dates Tested**
1	512	8/27/2004
2	590	8/27/2004
3	468	8/27/2004
4	439	8/27/2004
5	599	6/6/2005
6	589	6/7/2005
7	393	6/7/2005
8	748	6/7/2005
9	770	6/7/2005
10	358	6/7/2005
Mean	547	
St Dev	139	

C. Abutment reduction

Each abutment is reported that was involved with a group associated with 2 mm occlusal reduction and either 0.5 mm margin or 1.0 mm margin. All abutments were manually prepared after marking the margin placement at 1.0 mm above the height of contour.

Table C1. Abutment reductions in control and 0.5 mm specimen groups.

Spec	Before preparation (mm)			After preparation (mm)			Reduction (mm)			Date Measured
	cerv	mid	inc	cerv	mid	inc	cerv	mid	inc	
1	5.5	5.2	5.0	5.0	4.9	4.5	0.5	0.3	0.5	6/5/2005
2	5.5	5.2	5.0	5.0	5.0	4.6	0.5	0.2	0.4	
3	5.5	5.2	5.0	4.9	4.7	4.5	0.6	0.5	0.5	
4	5.5	5.2	5.0	5.0	5.0	4.6	0.5	0.2	0.4	
5	5.5	5.2	5.0	5.0	4.8	4.3	0.5	0.4	0.7	
1	5.5	5.2	5.0	4.9	4.6	4.1	0.6	0.6	0.9	8/25/2004
2	5.5	5.2	5.0	5	4.7	4.3	0.5	0.5	0.7	
3	5.5	5.2	5.0	5.1	4.8	4.3	0.4	0.4	0.7	
4	5.5	5.2	5.0	5.05	4.85	4.1	0.5	0.4	0.9	
5	5.5	5.2	5.0	5.1	4.9	4.5	0.4	0.3	0.5	
Mean	**5.5**	**5.2**	**5.0**	**5.0**	**4.8**	**4.4**	**0.5**	**0.4**	**0.6**	
sd	**0.0**	**0.0**	**0.0**	**0.1**	**0.1**	**0.2**	**0.1**	**0.1**	**0.2**	

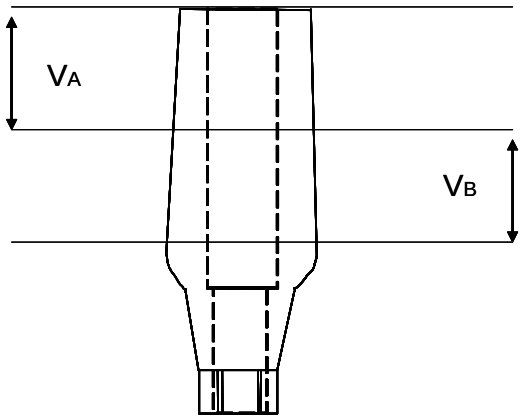
Table C2. Abutment reductions in control and 1.0 mm specimen groups.

Spec	Before Preparation (mm)			After preparation (mm)			Reduction (mm)			Date Measured
	cerv	mid	inc	cerv	mid	inc	cerv	mid	inc	
1	5.5	5.2	5.0	4.7	4.5	4.2	0.8	0.7	0.8	6/5/2005
2	5.5	5.2	5.0	4.7	4.5	4.0	0.8	0.7	1.0	
3	5.5	5.2	5.0	4.6	4.5	4.2	0.9	0.7	0.8	
4	5.5	5.2	5.0	4.8	4.6	4.2	0.7	0.6	0.8	
5	5.5	5.2	5.0	4.8	4.5	4.4	0.7	0.7	0.6	
6	5.5	5.2	5.0	4.7	4.7	4.2	0.8	0.5	0.8	
1	5.5	5.2	5.0	4.8	4.5	3.9	0.7	0.7	1.1	8/27/2004
2	5.5	5.2	5.0	4.7	4.5	3.9	0.8	0.7	1.1	
3	5.5	5.2	5.0	4.8	4.4	3.9	0.7	0.8	1.1	
4	5.5	5.2	5.0	4.4	4.1	3.7	1.1	1.1	1.3	
Mean	**5.5**	**5.2**	**5.0**	**4.7**	**4.5**	**4.1**	**0.8**	**0.7**	**0.9**	
sd	**0.0**	**0.0**	**0.0**	**0.1**	**0.2**	**0.2**	**0.1**	**0.2**	**0.2**	

D. Analysis of abutment assemblies

Each curve generated by the load to fracture test was analyzed by looking at inflection points that seemed to indicate failures associated with either the screw or the abutment. Strengths extrapolated from the dissected curves were then plotted against the volume of the abutment.

Figure D1. Volume calculations of abutment after preparation



Cone = ((pi)(h)/12)(db^2 +dbdt + dt^2), where

pi = 3.14
h= height of cone
db = diameter of bottom
dt = diameter of top

Prepable Abutment Volume = Cone volume A + Cone Volume B

Example. Control group calculations

Total h =	7.0
d incisal =	5.00
d middle =	5.20
d cervical =	5.50

Cone1 = ((3.14)(3.5)/2)(5.5^2 + (5.5)(5.2) + 5.2^2))/12 = 71.50 mm^3

Cone 2 = ((3.14)(3.5)/12)(5.2^2 + (5.2)(5.0) + 5.0^2)) = 78.70 mm^3

Prepable abutment volume = 39.35 mm^3 + 35.75 mm^3 = 150.20 mm^3

Table D1. Volume calculation for 0.5 mm specimens.

Date:	Specimen	V(A)	V(B)	Vol	Volume Change	Failure Load	Peak Load	Abutment Load	Screw Load
6/6/05	1	67.36	60.76	128	22.09	407	407	230	177
6/6/05	2	68.72	63.37	132	18.12	477	477	265	212
6/6/05	3	63.34	58.18	122	28.69	574	574	250	324
6/6/05	4	68.72	63.37	132	18.12	391	391	90	301
6/6/05	5	66.01	56.97	123	27.23	617	617	165	452
8/25/04	1	62.04	52.07	114	36.09	756	756	222	534
8/25/04	2	64.68	55.70	120	29.83	796	695	275	420
8/25/04	3	67.38	56.97	124	25.87	663	663	135	528
8/25/04	4	67.36	55.18	123	27.67	579	579	252	327
8/25/04	5	68.73	60.76	129	20.72	603	603	165	438
8/25/04	6	xxx	xxx	xxx	xxx	xxx	624	195	429
8/25/04	7	xxx	xxx	xxx	xxx	xxx	795	238	557
8/25/04	11	xxx	xxx	xxx	xxx	xxx	719	190	529
8/25/04	12	xxx	xxx	xxx	xxx	xxx	658	240	418
8/25/04	13	xxx	xxx	xxx	xxx	xxx	535	255	280

*Volumes associated with some specimens could not be calculated because the dimensions have not been measured.

Table D2. Volume calculation for 1.0 mm specimens.

Date:	Specimen	V(A)	V(B)	Vol	Volume Change	Failure Load	Peak Load	Abut. Load	Screw Load
6/7/05	5	58.18	52.04	110	40.00	599	599	220	379
6/7/05	6	58.18	49.71	108	42.32	589	589	195	394
6/7/05	7	56.91	52.04	109	41.26	393	393	175	218
6/7/05	8	60.73	53.26	114	36.22	748	748	235	513
6/7/05	9	59.46	54.44	114	36.31	770	770	235	535
6/7/05	10	60.72	54.49	115	34.99	358	358	115	243
8/27/04	1	59.46	48.57	108	42.18	512	512	155	357
8/27/04	2	58.18	48.57	107	43.46	590	590	110	480
8/27/04	3	58.20	47.40	106	44.61	468	468	120	348
8/27/04	4	49.67	41.85	92	58.69	439	439	160	279

Table D3. Volume calculations for control specimens (unprepared).

Date:	Spec	V(A)	V(B)	Volume	Volume Change	Failure Load	Peak Load	Abut. Load	Screw Load
8/25/04	1	71.5	78.7	150.2	0	584	525	240	285
8/25/04	2	71.5	78.7	150.2	0	518	518	185	333
8/25/04	3	71.5	78.7	150.2	0	392	392	175	217
8/25/04	4	71.5	78.7	150.2	0	528	528	165	363
8/25/04	5	71.5	78.7	150.2	0	509	509	270	239
6/6/05	6	71.5	78.7	150.2	0	645	645	190	455
6/6/05	7	71.5	78.7	150.2	0	218	218	85	133
6/6/05	8	71.5	78.7	150.2	0	394	394	225	169
6/6/05	9	71.5	78.7	150.2	0	230	235	90	145
6/6/05	10	71.5	78.7	150.2	0	327	327	200	127

E. Summary of statistical analysis details

The results of personal computer statistical analyses are summarized below.

E1. One-way ANOVA for Y-TZP bar tests

Zirconia Bar Experiments	n	Mean	SD	SE
NP	12	1633.7	94.8	27.36
DP	12	1143.5	109.0	31.46
WP	12	1442.4	88.9	25.67
WP+30d	12	1192.7	155.3	44.83
WP+B	12	1218.2	77.0	22.24
WP+SB	12	1663.8	176.0	50.82
WP+P	12	1632.4	134.2	38.73

Source of variation	SSq	DF	MSq	F	p
Zirconia Bar Experiment	3834882.2	6	639147.0	41.50	<0.0001
Within cells	1185940.2	77	15401.8		
Total	5020822.4	83			

Contrast	Difference	95% CI		p<0.05
NP v DP	490.2	331.0	to 649.4	(significant)
NP v WP	191.3	32.1	to 350.4	(significant)
NP v WP+30d	441.0	281.8	to 600.2	(significant)
NP v WP+B	415.5	256.3	to 574.7	(significant)
NP v WP+SB	-30.1	-189.3	to 129.1	
NP v WP+P	1.2	-158.0	to 160.4	
DP v WP	-299.0	-458.2	to -139.8	(significant)
DP v WP+30d	-49.2	-208.4	to 110.0	
DP v WP+B	-74.7	-233.9	to 84.5	
DP v WP+SB	-520.3	-679.5	to -361.1	(significant)
DP v WP+P	-489.0	-648.2	to -329.8	(significant)
WP v WP+30d	249.7	90.5	to 408.9	(significant)
WP v WP+B	224.3	65.1	to 383.5	(significant)
WP v WP+SB	-221.4	-380.6	to -62.2	(significant)
WP v WP+P	-190.0	-349.2	to -30.8	(significant)
WP+30d v WP+B	-25.5	-184.7	to 133.7	
WP+30d v WP+SB	-471.1	-630.3	to -311.9	(significant)
WP+30d v WP+P	-439.7	-598.9	to -280.5	(significant)
WP+B v WP+SB	-445.6	-604.8	to -286.4	(significant)
WP+B v WP+P	-414.3	-573.5	to -255.1	(significant)
WP+SB v WP+P	31.4	-127.8	to 190.6	

E2. One-way ANOVA for breaking loads for Y-TZP abutment/analog assemblies

Prepped Abudments	n	Mean	SD	SE
0-mm	10	434.500	145.915	46.1424
0.5-mm	16	596.688	137.932	34.4831
1-mm	10	546.600	139.448	44.0974

Source of variation	SSq	DF	MSq	F	p
Prepped Abudments	162966.885	2	81483.442	4.12	0.0252
Within cells	652012.338	33	19757.950		
Total	814979.222	35			

Contrast	Difference	Bonferroni, 95% CI		p<0.05
0-mm v 0.5-mm	-162.188	-305.103	to -19.272	(significant)
0-mm v 1-mm	-112.100	-270.651	to 46.451	
0.5-mm v 1-mm	50.088	-92.828	to 193.003	

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