ACP | AMERICAN COLLEGE OF PROSTHODONTISTS
Your smile. Our specialty.

The Effect of Multiple Firings on Microtensile Bond Strength of Core-Veneer Zirconia-Based All-Ceramic Restorations

Somayeh Zeighami, DDS, MSc, Hoseinali Mahgoli, DDS, MSc, Farzaneh Farid, DDS, MSc, & Abbas Azari, DDS, MSc

Assistant Professor, Dental Research Center and Department of Prosthodontics, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran

Keywords
All-ceramic; zirconia; veneering porcelain; multiple firing cycles; microtensile bond strength; delamination; layered restoration; mode of failure; coefficient of thermal expansion; Lucite content.

Correspondence
Somayeh Zeighami, Dental Research Center and Department of Prosthodontics, School of Dentistry, International Campus, Tehran University of Medical Sciences, Tehran, Iran. E-mail: somayeh.zeighami@gmail.com

This study was supported by grant No 649 from the Dental Research Center of Tehran University of Medical Sciences.

The authors deny any conflicts of interest.

Accepted February 12, 2012

doi: 10.1111/j.1532-849X.2012.00889.x

Abstract

Purpose: Differences in core and veneer coefficients of thermal expansion, firing shrinkage, and speed of increasing and decreasing the temperature may generate stress in veneered all-ceramic restorations. Given the necessity of performing multiple firing cycles to achieve improved contour, color, and esthetics, the purpose of this study was to determine the effect of multiple firing cycles on the microtensile bond strength (MTBS) of zirconia core to the porcelain veneer in zirconia-based all-ceramic restorations.
Materials and Methods: Thirty blocks ($12 \times 12 \times 4$ mm^3) of semi-sintered zirconia were machined and sintered according to manufacturer's instruction. Specimens were placed in three groups based on the number of firing cycles (4, 6, 8) for the veneering process. After veneering, the specimens were sectioned into microbars with 8 mm length and 1 mm cross-section. Twenty sound microbars in each group were stressed to failure in a microtensile tester machine at 1 mm/min. Fractured specimens were surveyed under a scanning electron microscope and classified as cohesive in core, cohesive in veneer, and mixed. MTBS data were analyzed using one-way ANOVA and Tukey test ($p < 0.05$).
Results: The mean MTBS (MPa) after 4, 6, and 8 firing cycles were 30.33 ± 2.13, 27.43 ± 1.79, and 25.06 ± 1.76, respectively. There was a statistically significant difference between the bond strengths of each of the three groups ($p < 0.001$).
Conclusion: Increase in firing cycles decreased MTBS. Most of the failures (90–95%) in all three groups were cohesive in the veneering porcelain and did not change as the number of firing cycles increased.

In recent years, demands for esthetic dental materials have increased. Along with the growth of public knowledge regarding newly developed materials and advanced manufacturers' products, the restorative dentist faces a challenge considering the use of the latest inventions in esthetic dental materials. Although ceramics are mostly regarded as the best materials in esthetic dentistry, these materials all suffer from inherent brittleness.[1]

In the last decades, several attempts have been made to modify the ceramic microstructures to make them more durable. Recently, zirconium-based ceramics have been introduced. They use the principles of computer-aided design/computer-aided manufacturing (CAD/CAM) for fabrication of the crowns and fixed partial dentures (FPDs).[1]

Zirconia is resistant in chemical environments, and has a high fracture strength. Thus, zirconia is one of the best materials for fixed restorations. To gain a more esthetic appearance, zirconia frameworks are layered with veneering porcelain that gives the definitive restorations appropriate optical characteristics.[2]

In bilayer restorations, veneering porcelain may be cracked or delaminated during function, so the core/veneer bond strength must have a minimum value. Because the stress release pattern in bilayer restorations has more complexity than that in mono-layer restorations, there are more elements to study in the zirconia-based restorations.[2]

A review article found that bulk fracture of zirconium-based FPDs rarely happened.[3] Instead, problems such as crazing and/or chipping of the layered ceramics may cause problems for clinicians following the zirconia core veneering process.[4-7] Both cohesive fracture of the layered veneer itself and delamination of veneering porcelain from the core material have been reported.[8]

Table 1 Material properties according to manufacturer's data

Materials	Manufacturer	Batch	Composition	Coefficient of thermal expansion (ppm/°C)
Cercon Base	Degudent GmbH, Hanau-Wolfgang, Germany	18000134	Zirconium oxide (92%vol), yttrium oxide (5%vol), hafnium oxide (2%vol), alumina and silica (<1%vol)	10.5
Cercon Ceram Kiss Paste liner	Degudent GmbH, Hanau-Wolfgang, Germany	53096	Selenium, feldspathic porcelain	10.3
Cercon Ceram Kiss Shoulder Porcelain	Degudent GmbH, Hanau-Wolfgang, Germany	53965	Feldspathic porcelain	9.5
Cercon Ceram Kiss Dentin	Degudent GmbH, Hanau-Wolfgang, Germany	53319	Feldspathic porcelain	9.2
Cercon Ceram Kiss Add-On Porcelain	Degudent GmbH, Hanau-Wolfgang, Germany	53865	Feldspathic porcelain	8.3
Ducera Liquid SD	Degudent GmbH, Hanau-Wolfgang, Germany	55103	Water based glycerin-containing liquid	—

In layered all-ceramic restorations, the bond strength of zirconia core to veneering porcelain is not sufficiently strong.[2] Differences in core and veneer coefficients of thermal expansion, firing shrinkage, and speed of increasing and decreasing the temperature may generate stress in core-veneer all-ceramic restorations.[2]

Because of the differences in thermal behavior of the materials, heating and cooling cycles could produce stress in layered restorations. Generated stress may have two statuses, passing and/or remaining. If the passing stress has sufficient value, the porcelain will crack instantaneously, while remaining stresses will reduce the restoration lifetime. Application of more loads to these restorations may cause them to be fractured. Thus, the clinical survival of a metal-ceramic or an all-ceramic restoration is dependent on the thermal characteristics of the used substances displayed via thermal expansion/contraction coefficient.[9]

The thermal mismatch of core and layering ceramic in all-ceramic systems follows the same concept used in metal-ceramic systems. In a perfect all-ceramic restoration, the differences between thermal contraction coefficients of layering ceramic and core material should not be notable.[9]

The thermal manner of core and layering ceramic in metal-ceramic restorations is simpler than that in all-ceramic restorations. Porcelains in metal-ceramic systems, which are composed of Lucite crystals embedded in a glassy matrix, display a variation in thermal dimension after firing cycles. This is because a modification in Lucite crystal amounts to a decoupling of Lucite from the glassy matrix throughout the cooling procedure and then recoupling to the glassy matrix through firing. In other words, dental ceramics display a nonlinear thermal dimensional manner, and as a result of a modification in phases after heat treatment, their structure modifies. Although this variation in thermal dimension does not lead to a problem in metal-ceramic restorations, it may have an effect on the thermal mismatch of core and veneer substances in all-ceramic restorations.[9]

To achieve improved contour, color, and esthetics, multiple firing procedures are necessary. The effect of multiple firing cycles on microtensile bond strength (MTBS) of core-veneer zirconia-based all-ceramic restorations is not clear, and no study has been done in this field. Therefore, the purpose of this study was to determine the effect of multiple firing cycles on the MTBS of zirconia core to the porcelain veneer.

Materials and methods

Preparation of zirconia core

To prepare the test blocks, a cubical aluminum block ($12 \times 12 \times 4$ mm^3) was made by machining process. This block was used as a pattern to prepare 30 blocks from the semi-sintered zirconia (Cercon Heat, Degudent GmbH, Hanau-Wolfgang, Germany; Table 1) by milling. All specimens were then fired, sandblasted (120 μm Al_2O_3 particles at 350 kPa pressure), ultrasonically cleaned, and randomly categorized in three groups based on the number of firing cycles (4, 6, and 8) for veneering process (10 specimens in each group; Table 2).

Veneering procedure

To make the shapes similar after baking and to limit the veneering ceramic, a specially designed aluminum cubic mold ($12 \times 12 \times 8$ mm^3) was used. According to firing procedures in each group (Table 2), veneering procedure was started and followed step-by-step based on the manufacturer's instructions (Table 3). The veneering steps (liner, margin, dentin, glaze, and

Table 2 Porcelain veneer condensation procedures used in each testing group

Groups (firing cycle)	Firing cycle procedure
4	Liner1/Margin1//Dentin1/Glaze
6	Liner1/Margin1/Dentin1/Dentin2/Glaze/Correction
8	Liner1/Margin1/Dentin1/Dentin2/Glaze1/Glaze2/Correction1/Correction2

Table 3 Firing cycles for veneering porcelain according to manufacturer's data

	Standby temperature (°C)	Final temperature (°C)	Drying time (min)	Heating time (min)	Holding time (min)	Vacuum Time (min)
Paste liner1	575	970	9.00	6.00	1.00	6.00
Paste liner2	575	960	9.00	6.00	1.00	6.00
Margin1	450	850	9.00	6.00	1.00	6.00
Margin2	450	850	9.00	6.00	2.00	6.00
Dentine1	450	830	9.00	6.00	1.30-2.30	6.00
Dentine2	450	820	9.00	6.00	1.00-2.00	6.00
Glaze	450	800	9.00	6.00	1.00	0.00
Correction	450	800	9.00	6.00	1.00	6.00
Final shoulder	450	800	9.00	6.00	1.00	6.00

correction) (Table 2), were accomplished through condensation methods, and each layer was fired according to manufacturer's recommendations for a 4 mm thickness (Table 3).

Preparation of microbars for microtensile test

All specimens were mounted with a specially prepared metal mold and then cut into microbars with a diamond-coated disk under copious water irrigation (Mecatome T201A, Technimetal, Persi, Grenoble, France). Several microbars with 8 mm length and 1 mm cross-section ($8 \times 1 \times 1$ mm^3) were obtained, and the accurate dimensions of all microbars were further assessed by digital caliper. All microbars were carefully checked by stereomicroscope at 40× magnification (SZX9, Olympus, Tokyo, Japan). During the cutting procedure to prepare the microbars many specimens were completely fractured or cracked. Microbars with any detectable cracks were discarded from the study (Fig 1).

Microtensile bond strength test

Twenty sound microbars were randomly selected from each testing group. They were ultrasonically cleaned for 5 minutes, washed with warm water, and then dried. The prepared specimens were all stressed to failure within a microtensile tester machine (Bisco, Schaumburg, IL) at a 1 mm/min crosshead speed.

Scanning electron microscopy (SEM)

Fractured specimens were ultrasonically cleaned, carefully stacked to a sheet of metal by carbon double-sided tape

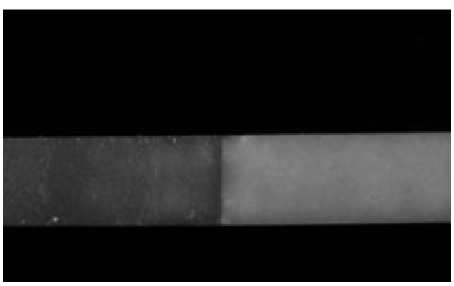

Figure 1 Stereomicroscope image (40× magnification) of a sound microbar ready for microtensile test.

Table 4 Core/Veneer MTBS and mode of failure in each group

Firing cycle	N	MTBS (MPa) (SD)	Cohesive in core	Cohesive in veneer	Mixed
4 times	20	30.33 (2.13)	-	19	1
6 times	20	27.43 (1.79)	-	19	1
8 times	20	25.06 (1.76)	1	18	1

(Nisshin EM Co.Ltd, Tokyo, Japan), coated with gold by a coater instrument (Auto Sputter Coater E5200, Bio-Rad, Hercules, CA), and then examined under SEM. Fractured surfaces were evaluated by SEM (CamScan MV2300, Oxford, UK) under 100× and 250× magnification. Based on the elemental analysis, all failures were classified into three modes:

(1) Cohesive in zirconia core: Only the remnants of zirconia core were seen in the fractured surface.
(2) Cohesive in porcelain veneer: Only the remnants of porcelain veneer were seen in the fractured surface.
(3) Mixed: Both material remnants (Zirconia and porcelain veneer) were detected in the fractured surface.

Statistical analysis

To determine the effects of firing cycles on MTBS of specimens, one-way ANOVA was used ($p < 0.05$). Tukey's post hoc-test was also used to determine the paired differences of groups. SPSS 16.0 v3.0 (SPSS, Inc., Chicago, IL, USA) was used.

Results

The mean MTBS (MPa) after 4, 6, and 8 firing cycles are listed in Table 4. One-way ANOVA showed a statistically significant difference between the three groups ($p < 0.001$). Tukey's post-hoc test on mean MTBS of paired groups also showed that each paired group was significantly different ($p < 0.001$).

Modes of failure of specimens in three groups are shown in Table 4. The majority of specimens (90–95%) fractured cohesively in porcelain veneer, and only one in each group had mixed failure. Cohesive failures in veneering porcelain originated and propagated in the veneer ceramic (Fig 2), but in mixed failures, fracture originated at the zirconia/ceramic interface that left exposed zirconia surface and then followed in the veneer ceramic (Fig 3).

Discussion

The first attempt to use direct ceramic machining in dentistry was made in 2001.[10] Since then, to simplify the production processes, many manufacturers have promoted techniques that use Y-TZP. Today, CAD/CAM zirconia frameworks usually are made of semi-sintered Y-TZP blocks, which convert to workable form by expending a final sintering stage.[3]

Very few clinical studies report the longevity of dental restorations with zirconia core, but a 90% success rate has

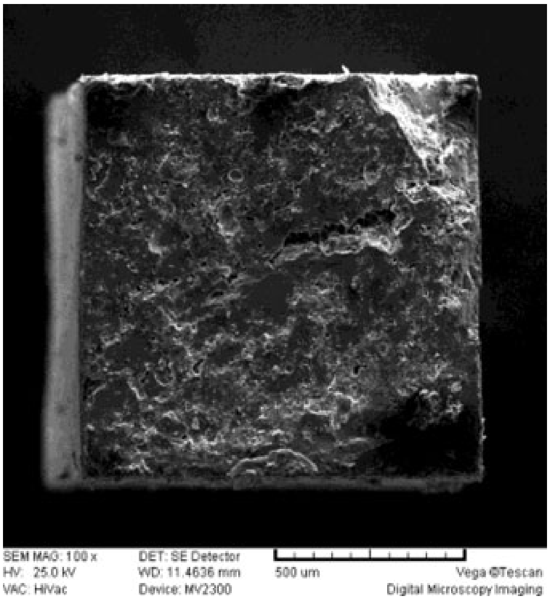

Figure 2 SEM image (100× magnification) of a cohesive failure in veneering porcelain. Fracture originated and propagated in the veneer ceramic.

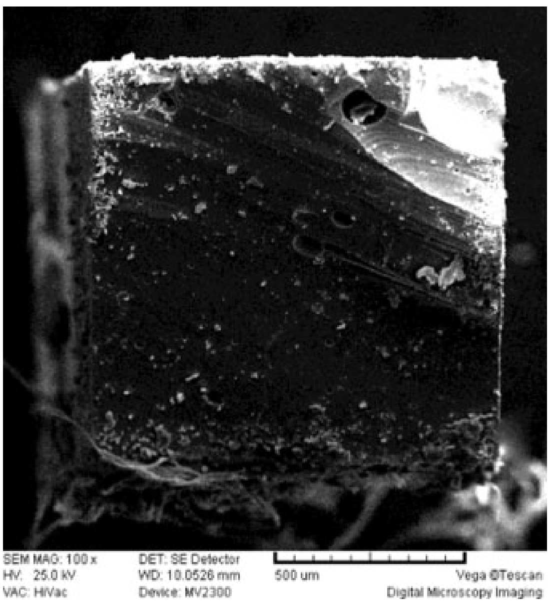

Figure 3 SEM image (100× magnification) of a mixed failure. Fracture originated at zirconia/veneer interface, leaving exposed zirconia grains.

been reported over 5 years of service.[11,12] The main type of failure has been reported as porcelain veneer fracture with little or no bulk fracture of core material.[3]

It has been shown that thermal expansion/contraction coefficients mismatch between layered ceramics, and zirconia core may accumulate residual stresses in the interfacial regions of the layered ceramic restorations.[13] The repeated firing seems to be a major problem in changing thermal specifications of the layered restorations, and so the main purpose of this article was to investigate whether multiple firing cycles of layered zirconia have the potential to adversely affect the MTBS of zirconia core to porcelain veneer.

It has been shown that if the MTBS test were used in measuring the core/veneer bond strength, the results would be more reliable and accurate, because application of perpendicular forces to a limited cross-section of microbars will reduce possible structural defects.[2]

Veneering ceramic has lower strength than zirconia core (i.e., MTBS of Cercon core in Aboushelib et al[14] was 339.5 MPa, while ceramic veneer's MTBS was 28.7 MPa). Thus, in core/veneer zirconia-based all-ceramic restorations, fracture occurs in the porcelain veneer or the core/veneer interface, and cohesive failure in the core is very rare. This fact is compatible with this study, in which 90–95% of failures were cohesive in the veneer.

Most failures in all three groups were cohesive in the veneering porcelain and did not tend to change as the number of firing cycles increased. With the increase in firing cycles, the core/veneer interfacial bond strength might be reduced, but this reduction was lower than that of the veneering ceramic. This was why most failures in this study were cohesive in the veneering ceramic, and delamination failure was rare. Furthermore, the delaminating fracture pattern observed in the interfacial zone of some of the microbars seems to be a result of chemically damaging mechanisms or weak contact between zirconia core and veneer in the interfacial zone.

The coefficient of thermal expansion of veneering ceramic in the layered all-ceramic systems has been changed by manufacturers and is compatible with zirconia core. Thus, in the cooling process, the veneering ceramic undergoes compressive strength; however, differences in coefficients of thermal expansion of core and veneer can produce stress in the core/veneer interface when multiple firing cycles occur. This is a reason for MTBS reduction.

Firing shrinkage of ceramic is 27–45%. It can produce stress in the core/veneer interface. Therefore, exact control of condensation and firing technique is necessary to diminish porcelain shrinkage[15] and stress production in the core/veneer interface.

All-ceramic restorations may need multiple heat treatments, which are required for the condensation process of veneering porcelain and color or contour modification. The results of this study clearly showed that an increase in firing cycles from 4 to 8 cycles decreased the MTBS, and all three groups have statistically significant differences in MTBS. The effect of multiple firings on the reduction of veneering ceramic fracture strength in metal-ceramic systems has been previously shown.[10]

After firing, porcelain consists of two phases**:** crystalline phase and glassy matrix. The crystalline phase is Lucite, which controls porcelain's coefficient of thermal expansion. Lucite also has a major role in porcelain strength.[15] During the porcelain cooling process, Lucite crystalline transforms from the cubic to the tetragonal phase. Lucite crystals contract more than the glassy matrix because larger thermal expansion coefficients and compressive stresses will be produced around Lucite crystals.[16]

Finally, although differences in core and veneer coefficients of thermal expansion, firing shrinkage, and speed of increasing and decreasing the temperature may lead to MTBS reduction of the core-veneer zirconia-based all-ceramic restorations, the maximum failures were in the veneering porcelain. The major reasons for this phenomenon seem to be:

1. Change in Lucite content of veneering porcelain after multiple firing cycles.
2. Microcrack formation between Lucite phase and glassy matrix because of the differences in coefficients of thermal expansion of Lucite and glassy matrix.

The clinical implication of this study is that in the zirconia core-veneering process, firing cycles should be limited as much as possible; however, more investigations are needed. This study was in vitro, and intraoral factors such as exposure to a moist environment, thermocycling, and cyclic loading were not evaluated. Additionally, only one zirconia system (Cercon) was evaluated.

Conclusion

Under the limitations of this study, the following conclusions were drawn:

1. An increase in firing cycles from 4 to 8 cycles decreased the MTBS.
2. All three groups had statistically significant differences in MTBS.
3. In each group most of the failures (90–95%) were cohesive in the veneering ceramic, and the mode of failure did not tend to change as the number of firing cycles increased.

Acknowledgments

The authors are grateful to Dr. Farid Zaeri for his statistical consultation, the Reference Laboratory of the Dental Research Center of Tehran University of Medical Sciences, and also Ms. Zohreh Dehghan and Mr. Esmaeel Hasanzadeh for their technical assistance in the laboratory work. We acknowledge Ms. Seyedeh Nastaran Pezeshki for her help in the grammar editing of this paper.

References

1. Nikzad S, Azari A, Dehgan S: Ceramic (Feldspathic & IPS Empress II) versus laboratory composite (Gradia) veneers: a comparison between their shear bond strength to enamel: an in vitro study. J Oral Rehabil 2010;37:569-574
2. Aboushelib MN, Kleverlaan CJ, Feilzer AJ: Microtensile bond strength of different component of core veneered all-ceramic restorations. Part 3: double veneer technique. J Prosthodont 2008;17:9-13
3. Denry I, Kelly JR: State of the art of zirconia for dental applications. Dent Mater 2008;24:299-307
4. Vult von Steyern P, Carlson P, Nilner K: All-ceramic fixed partial dentures designed according to the DC-Zirkon technique. A 2-year clinical study. J Oral Rehabil 2005;32:180-187
5. Kohorst P, Brinkmann H, Dittmer MP, et al: Influence of the veneering process on the marginal fit of zirconia fixed dental prostheses. J Oral Rehabil 2010;37:283-291
6. Larsson C, Vult von Steyern P, Sunzel B, et al: All-ceramic two- to five-unit implant-supported reconstructions. A randomized, prospective clinical trial. Swed Dent J 2006;30:45-53
7. Donovan TE: Factors essential for successful all-ceramic restorations. J Am Dent Assoc 2008;139(Suppl):14S-18S
8. Aboushelib MN, de Kler M, van der Zel JM, et al: Microtensile bond strength and impact energy of fracture of CAD-veneered zirconia restorations. J Prosthodont 2009;18:211-216
9. Isgro G, Kleverlaan CJ, Wang H, et al: The influence of multiple firing on thermal contraction of ceramic materials used for the fabrication of layered all-ceramic dental restorations. Dent Mater 2005;21:557-564
10. Barghi N, McKeehan-Whitmer M, Aranda R: Comparison of fracture strength of porcelain-veneered-to-high noble and base metal alloys. J Prosthet Dent 1987;57:23-26
11. Griggs JA: Recent advances in materials for all-ceramic restorations. Dent Clin North Am 2007;51:713-727
12. Kelly JR: Dental ceramics: current thinking and trends. Dent Clin North Am 2004;48:513-530
13. Isgro G, Wang H, Kleverlaan CJ, et al: The effects of thermal mismatch and fabrication procedures on the deflection of layered all-ceramic discs. Dent Mater 2005;21:649-655
14. Aboushelib MN, Jager ND, Kleverlaan CJ, et al: Microtensile Bond strength of Different component of core veneered all ceramic restorations. Dent Mater 2005;21:984-991
15. Power JM, Sakaguchi RL: Craig's Restorative Dental Materials (ed 12). St. Louis, Mosby, 2006
16. Mackert JR, Butts MB, Fairhurst CW, et al: The effect of the Lucite transformation on dental porcelain expansion. Dent Mater 1986;2:32-36