

THE INFLUENCE OF DIFFERENT SURFACE TREATMENTS ON MICRO-SHEAR BOND STRENGTH OF TWO RESIN LUTING AGENTS TO MONOLITHIC ZIRCONIA

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ABSTRACT

Purpose. Establishing a reliable bond to zirconia-based materials has proven to be difficult which is the major limitation against fabricating adhesive zirconia restorations. The purpose of this study was to evaluate the micro-shear bond strength of 2 dual-cured adhesive cements to monolithic zirconium oxide ceramic after different surface conditioning treatments.

Material and methods Eighteen 14 × 14 × 1 mm monolithic zirconia ceramic plates were sliced from their respective block by using a low speed diamond saw \. The plates were divided into three groups, and three different surface treatments were performed: (1) no treatment (NT); (2) airborne-particle abrasion with 110- μ m alumina particles (SB); (3) silica coating with Rocatec soft system (aluminum oxide of 30 μ m grain size modified with silica) (CT). Each group was further subdivided into two subgroups according to the resin cement; Panavia v5 with clearfil primer plus (Kurary, Japan) and RelyX Unicem (3M/ESPE, USA). Then, ten composite resin cylinders (0.8-mm diameter × 0.5-mm height) were light-polymerized onto the ceramic plates in each subgroup. Each specimen was subjected to a shear load at a crosshead speed of 0.5 mm/min until fracture occurred. The fracture sites were examined with scanning electron microscopy (SEM) to determine the location of failure during debonding and to examine the surface treatment effects. Data were statistically analyzed using two-way ANOVA and multiple comparisons were made using Fisher's test at $p < 0.05$.

Results. Micro-shear bond strength was significantly affected by the surface treatment and by the type of resin cement. Panavia v5 showed higher significant results in comparison to RelyX Unicem. Surface treatment with CT was highly significant with both cements, followed by SB and then by NT. SEM examination revealed predominantly cohesive failures within the resin cements for CT group, mixed failures within SB group and predominantly adhesive failure at the interfacial area within NT group.

Conclusions: The micro-shear bond strength of resin cement to partially stabilized zirconia ceramics varied significantly depending on the type of resin luting agent and surface treatment method. The tribochemical silica coating of zirconia surfaces in combination with MDP-containing primer- resin cement (Panavia v5) showed a superior performance

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INTRODUCTION

Interest for using high-strength zirconium oxide ceramics for oral rehabilitation is growing in recent years¹. The unique mechanical properties, chemical stability, and biocompatibility make zirconia an attractive core material for fabrication of all-ceramic restorations.² Combined with CAD/CAM technology, the fabrication of complex restorations incorporating zirconia cores has become a relatively simple procedure³. Patients are usually concerned about the reduction of sound abutment teeth and request more conservative solutions. Adhesively bonded all-ceramic zirconia restorations are, thus, a treatment option.⁴ Recently, high translucency zirconia has been developed for clinical use. In addition (CAD/ CAM) technology has facilitated the design of frameworks and complete contour restorations as well as the processing of monolithic zirconia crowns and fixed dental prostheses (FDPs).⁵⁻⁷ Complete-contour monolithic zirconia restorations may provide adequate esthetics in the molar area. To enhance the translucency of zirconia, residual pores and impurities which create volumes of differing refractive indexes and lead to optical scattering on the surface and reduction of translucency must be reduced.⁸⁻¹⁵ Alumina, which is added to zirconia improve the mechanical properties and prevent low temperature degradation (LTD), is the most common impurity.¹⁶⁻²²

The retention and the stability of these restorations primarily depend on the adhesive bond strength, which must be strong enough to resist the expected functional loads. Although conventional cementation of zirconium oxide restorations with traditional luting agents (such as zinc-phosphate or resin-modified glass ionomer cements) may provide adequate clinical fixation, adhesive cementation is preferable for ensuring better retention and marginal adaptation^{23,24,25}.

The achievement of reliable adhesion to ceramics conventionally requires surface pre-treatments. However, neither hydrofluoric acid

etching nor silanization result in a satisfactory resin bond to zirconia because of the high crystalline content and the limited vitreous phase (below 1%) of this high-strength core ceramic^{24,26,27,28,29}. As a consequence, alternative conditioning methods have been proposed. Many studies reported that airborne abrasion may increase the surface area, resulting in acceptable micrometer scale roughness facilitating resin/ceramic micromechanical interlocking formation^{30,31}.

Advances in adhesive dentistry have resulted in the recent introduction of modern surface conditioning methods. One such system is silica coating by the Rocatec soft system (3M/ESPE). In this technique, the surfaces are air abraded with aluminium trioxide particles modified with silica^{32,33,34}. The blasting pressure results in the embedding of these silica coated alumina particles on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents.^{35,36}

Cement selection is a prerequisite for ensuring effective bond strength to zirconia. Phosphate monomer-based luting agents have been proposed for cementation^{29,37} such as 10-MDP containing luting systems Panavia v5 with clearfil primer plus (Panavia, Kurary).

Self-adhesive cements that rely on a single step application, have also been proposed for luting zirconium-based restorations^{30,38}. The resin matrix of these systems consists of multifunctional acid methacrylates that should react with the substrate and contribute to the adhesion mechanism³⁹.

Although investigators have used a variety of bond strength methods, shear testing has become a very popular method. Shear stresses are believed to be major stresses involved in in-vivo bonding failures of restorative materials^{40,41,42}. In this study, bond strengths were assessed by means of a micro-shear bond test that measured bonding to small areas of the substrate⁴³.

The aim of this study was determining the bond strength of both adhesive and self-adhesive dual-cured adhesive cements to zirconium oxide ceramic after different surface conditioning treatments (airborne-particle abrasion and tribochemical silica coating).

MATERIALS AND METHODS

Construction of ceramic samples

Eighteen 14 x 14 x 1 mm machinable monolithic zirconia ceramic plates (Bruxzir Glidewell, California, USA) were sliced from their respective blocks by using a low speed diamond saw (Buehler-Isomet LakeBulff, IL, USA) After wet slicing, the zirconia plates were cleaned ultrasonically in distilled water and then sintered in a ceramic sintering furnace (InFire HTC, Sirona), for 7 hours at 1550°C. Fully sintered plates were inspected and measured to verify the dimensions. If necessary, they were adjusted with diamond stones at high speed and water coolant, and the surfaces were smoothed with 1200 grit silicon carbide abrasives. Afterwards, all samples were placed in a ceramic furnace at 1000°C for a process of stress relief.

Conditioning of ceramic samples

The plates were then assigned to three groups according to the type of surface treatment:

Group NT: No surface treatment applied.

Group SB: Airborne particle abrasion with 110-µm aluminium oxide particles at 35 psi from a distance of approximately 10 mm for 15 seconds and cleaned with compressed oil-free air for 30 seconds, with no silane application.

Group CT: silica coating using Rocatec soft with particle size of 30 µm were used (3M/ESPE, USA). Particles were ejected at a pressure of 2.8 bars for a period of 15 seconds and at a distance of 10 mm, Silane coupling agent (RelyX Ceramic Primer, 3M/ESPE) was then applied and left to dry for one minute.

Preparation of resin samples and bonding procedures:

The materials used in the bonding procedures are described in table 1. For each group, six zirconia plates were further subdivided into two subgroups according to the type of resin cement used. Half of the samples were bonded to RelyX Unicem dual cure resin cement (3M/ESPE, USA), and the other half to Panavia v5 with clearfil primer plus (Kuraray, Japan) dual cure resin cement.

The methodology developed by Shimada et al²⁵ was used to prepare specimens for the microshear test. Surfaces of ceramic samples were treated with bonding resin. Prior to light-curing of bonding resin, cylindrical plastic translucent molds with an internal diameter and a height of approximately 0.75 and 0.5 mm, respectively, were positioned over the treated surface of each ceramic plate. Bonding site was then cured for 10 seconds. Following which, application of clearfil primer plus for panavia group then freshly mixed resin cement (Panavia v5 or RelyX Unicem according to grouping) was applied into the molds to fill their internal volume using a C-R syringe (Centrix Dental, Shelton, CT, USA). Light curing was performed for 40 seconds for each sample. In this manner, very small cylinders of resin, approximately 0.75 mm in diameter and 0.5 mm in height were bonded to the ceramic surface at 3 to 4 locations.

The specimens were stored at room temperature (23°C) for 1 h prior to removal of the plastic tubing. Ten resin specimens were created for each group combination. The specimens were then stored in water for 24 h. Before bond strength testing, all samples were checked for defects under an optical microscope at 25x magnification. Samples showing air bubble inclusions, interfacial gaps, and other defects were discarded.

Micro-shear bond strength evaluation

Each zirconia sample was attached onto a testing device mounted in a universal testing machine

TABLE (1) List of the main materials used in the study showing their composition.

Material	Type	Composition	Manufacturer
Panavia v5	Dual polym-erizing resin luting agent	1. Bisphenol A Diglycidyl-methacrylatw 2. Triethylene glycol dimethacrylate 3. Silanated barium Glass 4. Silica 5. Aluminum oxide 6. Aromatic dimethacrylate 7. Aliphatic dimethacrylate 8. Camphorquinone 9. Accelerators 10. Pigaments	Kurary Medical, Inc Okayama, Japan
Clearafil ceramic primer plus	Universal Primer	1. Ethanol 2. Silane 3. MDP	Kuraray, Osaka, Japan
RelyX Unicem	Dual polym-erizing resin luting agent	Base/catalyst Methacrylated phosphoric ester, dimethacrylate, inorganic fillers, fumed silica, chemical and photoinitiators	3M ESPE, USA

(Lloyd, UK) for the micro-shear bond strength test. A thin orthodontic wire (0.2 mm in diameter) was looped around the resin cylinder, making contact with half of its circumference and gently held flushed against the resin-zirconia interface. The resin-zirconia interface, the wire loop, and the center of the load cell were aligned as straight as possible to ensure the desired orientation of the shear force. Each cylinder was then subjected to a shear force

at a crosshead speed of 0.5 mm/min until failure occurred. Interfacial shear strength was calculated by dividing the maximum load recorded on failure by the circular bonding area in square millimeters and expressed in MPa.

Data were statistically analyzed using two-way ANOVA and multiple comparisons were made using Fisher's test at $p < 0.05$. SPSS statistical software for windows was used for data analysis.

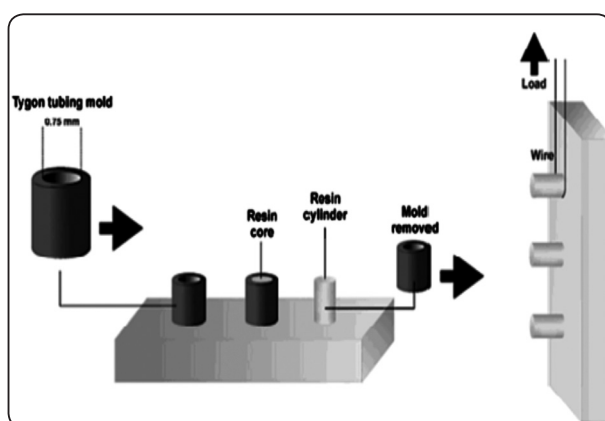


Fig. (1): Schematic illustration of the process of bonding and testing

Morphological study using scanning electron microscope (SEM)

Following shear testing, all fractured interfaces were examined under SEM examination, to determine the mode of failure and observe the topographic changes. They were recorded as:

- Mode 1: adhesive (between resin and zirconia),
- Mode 2: cohesive in adhesive layer,
- Mode 3: cohesive in resin or cohesive in zirconia,
- Mode 4: mixed failures (comprising two types).

RESULTS

The mean shear bond strength and standard deviation values are shown in table 2. Two way ANOVA indicated that the micro-shear bond strength was significantly affected by the surface treatment and by the type of resin cement evaluated at $p < 0.05$. Tukey’s test showed that Panavia v5 cement bonded to zirconia treated with the rocatec soft system showed the highest significant mean micro-shear bond strength value, and the lowest value was with RelyX Unicem with no surface treatment. Surface treatment with CT was highly significant with both cements, followed by SB and then by NT. With Panavia v5 there was a high significant difference in micro-shear bond strength values between CT and both SB and NT groups, while there was a very small significant difference between SB and NT groups. With RelyX Unicem, there was a high significant difference between the 3 surface treatment groups. Tukey’s test also showed that Panavia v5 showed a high statistical significant difference in comparison with Rely X with all types of surface treatments.

TABLE (2) Mean micro-shear bond strength of different surface treatment groups for tested resin cements, showing standard deviations and statistical significance

Resin Cement	NT Group			SB Group			CT Group		
	Mean	Sd	Sig	Mean	Sd	Sig	Mean	Sd	Sig
Panavia F 2.0	14.39	2.37	C	15.92	2.88	B	21.59	1.66	A
RelyX Unicem	8.81	1.14	E	11.67	1.17	D	14.58	1.28	C

Sig: Statistical significance. Values with different letters indicate significant difference.

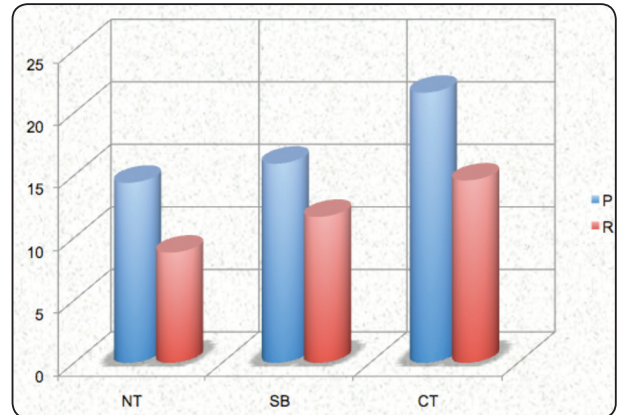


Fig. (2) Column chart showing the mean micro-shear bond strengths of the different treatment protocols with the two types of resin cements.

SEM examination with $\times 3.000$ magnifications of the fractured interfaces showed variations among groups. Mode of failure analysis revealed predominantly cohesive failures (mode 2 and 3) within the resin cements within CT group, mixed failures (mode 4) within SB group and predominantly adhesive failure at the interfacial area (mode 1) within NT group.

Representative SEM images of zirconia samples are reported in figures 3,4,5,6. Specimens conditioned with Al_2O_3 showed a change in surface texture with the formation of micro-retentive grooves. Tribochemical silica coating produced only a slight modification of zirconia surface. Cement residuals are detectable on the zirconia surface in the CT and SB groups, while complete detachment from the ceramic surface occurred with the NT group.

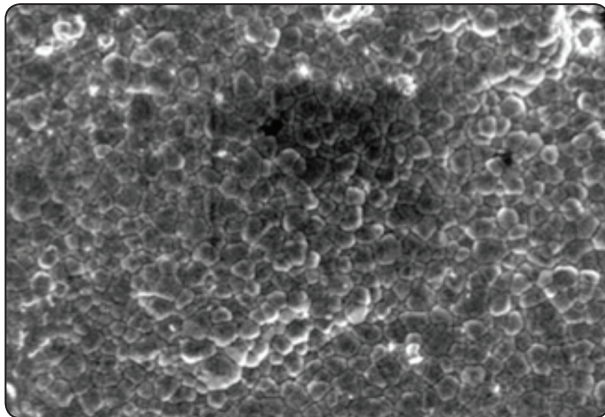


Fig. (3) SEM of NT as-sintered zirconia surface

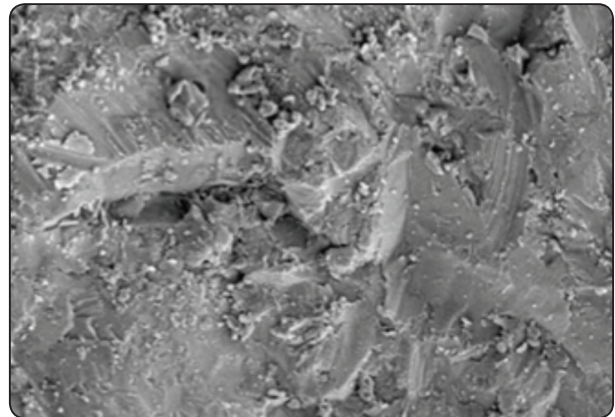


Fig. (4) SEM of SB sandblasted zirconia surface

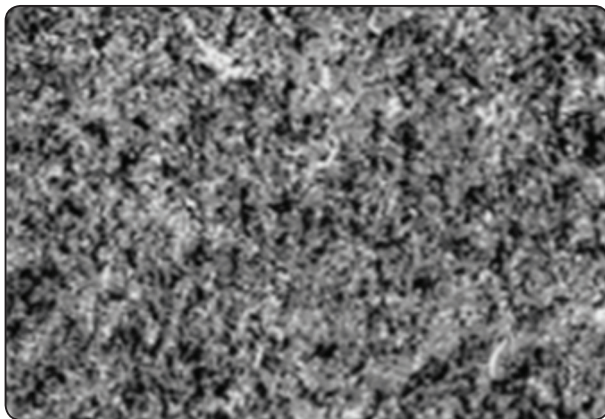


Fig. (5) SEM of CT silica-coated zirconia surface before cementation

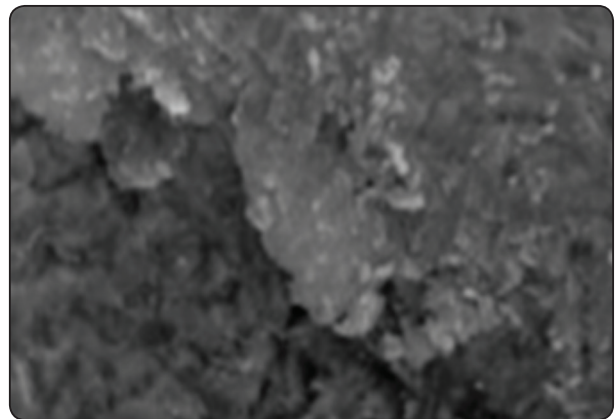


Fig. (6) SEM of CT zirconia showing resin cement covering the surface

DISCUSSION

Zirconium-oxide ceramics have been proven to resist fracture loads and show optimum strength *in vitro*,⁴⁴ All ceramic restorations have become popular due to esthetic appearance and metal free structure⁴⁵. Zirconia (ZrO_2)-based ceramics demonstrates superior mechanical properties such as high fracture strength and fracture toughness, enabling its use with posterior fixed partial dentures (FPDs)⁴⁶. Due to optical opacity of these materials, zirconia is used as substructure material that is veneered with feldspathic ceramics. In clinical application, limited number of studies reported seldom zirconia substructure fractures

but chipping of the veneer is described to be the most frequent occurrence that reduces the success rate of zirconia FPDs⁴⁷⁻⁵⁰. In order to overcome this problem, translucent tooth-colored zirconia (monolithic zirconia) which enables the fabrication of restorations without using veneering ceramic has been developed. Advantages of monolithic zirconia restorations include limited amounts of defects due to fabrication of the restoration from presintered homogeneous blocks with Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) technique and reduced production time/cost. Also, permitting a minimal material thickness of 0.5 mm due to high mechanical strength contributes to the preservation of tooth substance and enables

the use of ceramic restoration in case of limited interocclusal space⁵¹.

But their use also requires a reliable bond with the luting agent. Concerns still remain regarding the identification of the best luting methodology. The purpose of this investigation was that of evaluating the influence of conditioning treatments and cement type on bonding to zirconia.

Sliced zirconia surface is smooth and evenly flat with a regular crystal grain pattern.

Untreated zirconium oxide ceramic is a relatively inert substrate with low surface energy and wettability^{52,53}, and this explains the low micro-shear strength values associated with NT group. SEM analysis revealed a significant increase in surface roughness after sandblasting. Airborne-particle abrasion with Al₂O₃ is the preferred surface treatment method for high-strength ceramic materials.^{29, 54-60} Surface roughening methods increase surface energy and, therefore, wettability.⁵⁴ However, despite the increase in bond strength, application of airborne particle abrasion on such ceramics is controversial, due to the possible introduction of flaws and micro-cracks. The micro-porosities that surface-treatment methods create may function as crack initiators and, therefore, weaken ceramic materials. However, it has been shown that resin luting agents, proven to provide durable resin bonds, have the ability to “heal” minor surface flaws created by airborne-particle abrasion and, therefore, significantly strengthen ceramic materials.^{25,44} This is more relevant with glass ceramics. With Y-ZTP ceramics, there is a particular concern about the effect of sandblasting on crystalline transformation of the tetragonal zirconia to the monoclinic form, producing transformation toughening at such an early stage. This might require a phase reversal procedure through heat treatment of zirconia to return to the tetragonal phase before clinical fixation. Nevertheless, as expected, in the present study, the application of airborne-particle abrasion resulted in

a significant increase in micro-shear bond strength.

The Rocatec soft system is composed of blasted silica-modified Al₂O₃ particles, which promote surface roughness and a silica coat for resin bonding via silane agents.⁶¹ This tribochemical reaction produces a high temperature contact area that can hold the blasted particles and/or the silica layer on the ceramic surface.⁶² Microscopic analysis of the blasted surface reveals a thin and micro retentive layer,^{35,63} which should increase the bond strength to resin chemically and mechanically.⁵⁴ The silane agent used for silanization also contributes to the bond strength by promoting a chemical bond to resinous materials via cross-linkages with methacrylate groups, and also increases the substrate surface energy and improves the surface wettability to resin.^{36,54,64} This adhesive mechanism explains the high bond strength values observed for the CT-treated group, thus making for an interesting option for surface treatment of high crystalline ceramics. In this tribochemical procedure, changes in particle size and application time may be further evaluated, although more aggressive treatments may expedite surface micro-cracks formation, thus compromising the quality of the substrate⁶⁰.

Luting cement selection seems to be a relevant factor when bonding to zirconium oxide ceramics. The adhesiveness of phosphate monomer-containing cements increased bond strength, revealing the capability of acidic functional monomers of reacting with the substrate.

The application of MDP-containing silane/cement system attained the best overall results. The adhesive potential of 10-MDP to densely sintered zirconia may depend on the presence of a passive coating of zirconium oxide on the ceramic surface. Chemical reactions involving the hydroxyl groups of the layer and the phosphate ester monomers of the MDP may occur at the interfacial level^{65,66}. Moreover, the functional monomer has been rated as relatively hydrolysis stable, due to the presence

of a long carbonyl chain. A relatively strong poly-molecular layer may be responsible of the ceramic–resin cement bond⁶⁷. However, the longevity of these interfaces should be further evaluated^{3,68}.

RelyX Unicem showed the capability of bonding the substrate, regardless of the ceramic surface treatment and without additional coupling agent application. Lower bond strengths were attained if compared to the 10-MDP based cement. Bonding mechanism of RelyX Unicem is reminiscent of the self-adhesiveness of glass ionomer cements and a possible improvement in bond strength may occur after cement maturation overtime⁶⁹.

Comparing the effect of sandblasting of zirconia surface on the improvement of bond strength for both cements, it is obvious that it was more significant with RelyX Unicem. This is probably due to the fact that the molecular size of the MDP-containing primer could be larger than the micro-roughness produced by air abrasion of zirconia, in comparison to the low molecular size of Bis-GMA present in RelyX Unicem. Panavia v5

Some studies have shown that blasting with Al_2O_3 particles combined with a monomer-phosphate-based resin cement allows a significant bond strength to yttrium oxide-partially stabilized zirconia ceramic. This study showed that the groups treated with Rocatec soft system combined with the monomer-phosphate-based resin cement presented higher bond strength compared to samples blasted with Al_2O_3 particles.

The results from the present study are consistent with those of Bottino et al,²⁷ who found that silica coating followed by silanization increased the bond strength of zirconium-oxide ceramic to an MDP-containing composite resin relative to the use of airborne-particle abrasion with zirconium-oxide ceramic. In contrast, another study found that a durable resin bond to zirconium-oxide ceramic was obtained after airborne-particle abrasion of the ceramic and the use of a composite resin containing

an adhesive phosphate monomer, while the zirconium-oxide ceramic treated with silica coating and bonded with Bis-GMA resin luting composite failed.²⁹

Therefore it is safe to suggest that using a tribochemical system combined with monomer-phosphate-based resin cements is the best alternative for the cementation of zirconia ceramics.

Measurement of bond strength, regardless of the technique chosen, is a controversial topic in dental adhesion.⁷⁰ Conventional shear and tensile bond tests have generally been used to evaluate resin to ceramic bonding; however, the most commonly used shear bond test often produces fracture away from the adhesion zone.⁷¹⁻⁷⁵ Such failures of the substrate prevent measurement of interfacial bond strength and limit further improvements in bonding systems.

Several studies have identified nonuniform stress distributions along bonded interfaces.^{71,76} The nonuniform interfacial stress distribution generated for conventional tensile and shear bond strength tests initiates fractures from flaws at the interface or in the substrate in areas of high stress concentration. Recently researchers have preferred to use the microtensile method and fracture mechanics to understand the properties of the adhesive interface.⁷⁷ Unfortunately, the microtensile bond test, although an effective method in terms of testing a small area, is difficult to conduct and time-consuming for specimen preparation, especially in the case of ceramic samples.

In this study micro-shear bond tests was performed to measure the bond strength between resin cement and ceramic surfaces. Compared to the 'micro-tensile bond test,' trimming of the sample after the bonding procedure is not necessary for the micro-shear bond test, and the bonding surface was intact. In addition, preparing the specimens for this test is so facile that multiple samples, even using brittle materials, can easily be made. In the micro-

shear test method, stress distribution is uniform because an ultra-small area of bonding interface was tested.

Much criticism converges on the large variation of shear bond test results and clinical relevance. It has been shown that the stress distribution in the tested interface is uneven^{76,78}. Although it is difficult to duplicate in-vivo conditions, as described by SoÈ derholm⁴⁰, an understanding of the conditions which impact the test protocol is imperative in using in-vitro testing. A wide variety of configurations are used including wire loops, points and knife edges to apply the shear force^{40,42,43,79,80,81}. The use of a wire loop rather than a knife edge for shear bond tests is reported to reduce the stress concentration magnitude adjacent to the interface⁷⁸. While these are considerably less than those occurring in the shear test arrangement, it is by no means suggested that tensile bond strength is ideal⁸². In case of the tensile bond test, although stress inhomogeneities due to geometry are avoided, the interfacial stress should not be uniformly tensile due to the changes in elastic moduli⁸². Various investigators have suggested shear bond testing as a viable screening mechanism for predicting clinical performance. Clearly differing methods of load application lead to differing stress distributions. Thus, one must expect uneven stress distributions and acknowledge that the bond strengths reported are nominal values and need cautious interpretation. The use of bond strength data based on static load-to-failure tests should be restricted to comparisons of relative effects of material properties, material microstructure, and treatment conditions that may enhance the resistance to fracture⁷⁸.

Only the early bonding ability of resin cement to zirconia ceramic was investigated in the present study. The early bond strength of resin cement is quite important because unfavorable clinical situations such as debonding and fractures of ceramic restorations usually occur during or soon

after the setting process. If resin cement could produce sufficient bonding to the tooth as well as ceramic restorations soon after setting, the ceramic restoration would in effect be part of the tooth, and, as a result, the occurrence of debonding and fractures would be decreased. Of course, the effects of aging on bonding materials should also be taken into consideration and investigated.

CONCLUSIONS

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

1. Micro-shear bond strength for zirconium oxide ceramic differed significantly depending on luting agents and surface treatments.
2. The phosphate monomer-containing luting system (Panavia v5 with clearfil primer plus) showed superior results and seems to be the most suitable to bond zirconia ceramic surfaces if compared to self-adhesive resinous cement (RelyX Unicem). However, the durability of these ceramic-to-composite chemical bonds should be further evaluated.
3. On the effects of different surface conditioning methods, chairside tribochemical silica coating followed by silanization improved the bonding of zirconia ceramic significantly compared to aluminum oxide abrasion.

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