



Effect of various surface treatments on the interfacial adhesion between zirconia cores and porcelain veneers



Hyung-In Yoon^{a,*}, In-Sung Yeo^b, Jung-Suk Han^b

^a Department of Dentistry, School of Medicine, Ewha Womans University, Seoul, Republic of Korea

^b Department of Prosthodontics and Dental Research Institute, Seoul National University School of Dentistry, Seoul, Republic of Korea

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ABSTRACT

Predictable success of zirconia-based all-ceramic restoration is dependent on the stable bonding between ceramic veneer and zirconia core. The study investigated the effect of surface treatments on interfacial binding between zirconia and ceramic veneer under thermal stress. The surfaces of 60 yttria-stabilized zirconia beams were polished. Half underwent sandblasting. The polished-only and polished-abraded specimens then received a liner, a glass-based bonder, or no intermediate layer ($n=10$ /subgroup). All specimens were veneered, subjected to thermal cycling, and subjected to the four-point bending test to calculate the energy release rate. Five Raman spectra were obtained with a line scan across the interface. The data were analyzed with two-way ANOVA with a significance level of 0.05. Scanning electron microscopy revealed the interfacial crack pattern. The interfaces with the intermediate ceramics were significantly more resistant to four-point bending than others ($p=0.011$). Raman imaging suggested the interfacial diffusion between the zirconia cores and porcelain veneers. The crack failure mode was adhesive for all specimens. Under simulated physiological conditions, strong adhesion at the interface of porcelain-veneered zirconia complexes could be achieved with the application of intermediate ceramic layers.

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1. Introduction

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is widely utilized in clinical practice, including in the fields of dentistry and orthopedics [1]. Zirconia serves as a core ceramic in fixed dental restoration as it has excellent biocompatibility, wear resistance, flexural strength, and fracture toughness. However, veneers of feldspathic porcelain or fluorapatite glass ceramic must still be applied to single crowns and multi-unit frameworks of zirconia-based all-ceramic prostheses; this yields esthetic restorations with tooth-like optical properties and suppresses the progressive spontaneous transformation of the metastable tetragonal phase into the monoclinic phase, which is more likely to occur in wet environments such as that inside the mouth [1–4].

The survival and complication rates of the all-ceramic and metal–ceramic tooth-supported restorations were reported in the recent systematic reviews [5,6]. An estimated 5-year survival was 95.7% for metal–ceramic single crowns, while 91.2% for zirconia-based crowns [5]. For the multiple-unit fixed dental prostheses

(FDPs), the 5-year survival rates of the metal–ceramic and zirconia FDPs were estimated at 94.4% and 90.4%, respectively [6]. The annual failure rates of the metal–ceramic and zirconia FDPs were 1.15% and 2.02%, respectively [6]. The relative complication rate of ceramic chipping and fracture was significantly higher for zirconia-based FDPs than metal–ceramic FDPs [5,6].

Veneer chipping and delamination are caused by several factors: differences between the zirconia framework and veneering porcelain in terms of the coefficients of thermal expansion (CTE), poor wetting of the zirconia core by the veneering material, inadequate framework support, low thermal conductivity of zirconia and different porcelain cooling schedule [4,7]. Since the intact surface of the Y-TZP substrate is relatively inert with low surface energy and wettability [8], the zirconia surface must be treated with a procedure that cleans it, increases its surface roughness, and improves its wettability, thereby enhancing interface adhesion [9]. The two most commonly used zirconia core surface treatments are airborne-particle abrasion and liner application. Several studies have assessed the interfacial bond strengths that result from these treatments [10–14], but which treatment is best remains unclear. Moreover, studies examining why the bilayer systems fail have yielded conflicting results [13,15–17]. These disparities may reflect not only the differences between the surface treatment techniques, but also the methods used to evaluate

* Correspondence to: Department of Dentistry, School of Medicine, Ewha Womans University 1071, Anyangcheon-ro, Yangcheon-gu, Seoul, South Korea.
Tel.: +82 2 2650 5042; fax: +82 2 2650 5764.

E-mail address: prosyhi@ewha.ac.kr (H.-I. Yoon).

Table 1
Chemical composition and physical properties of the materials, as stated by the manufacturers.

Material	Manufacturer	Chemical composition	Elastic modulus (GPa)	Poisson's ratio (ν)	CTE ^a ($\times 10^{-6}/K$)
ZirBlank-PS	Acucera, Pocheon, South Korea	ZrO ₂ , Y ₂ O ₃ , HfO ₂	200	0.32	10.5
IPS e.max ceram	Ivoclar Vivadent, Schaan, Liechtenstein	SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, CaO, P ₂ O ₅ , F, other oxides	70	0.27	9.5
IPS e.max ceram ZirLiner (liner)	Ivoclar Vivadent, Schaan, Liechtenstein	SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, ZnO, CaO, P ₂ O ₅ , F, other oxides			9.8
Hotbond zirconnect (bonder)	DCM, Rostock, Germany	Unknown ^b			9.7

^a Coefficient of thermal expansion.

^b The manufacturer did not provide the authors with specific information regarding this material.

Table 2
Firing schedules of the veneering ceramics used in this study, as stated by the manufacturers.

Material	Pre-heating temperature (°C)	Pre-heating time (min)	Heating rate (°C/min)	Firing temperature (°C)	Holding time (min)
Hotbond zirconnect	450	2	60	1000	1
IPS e.max ceram ZirLiner	403	4	40	960	1
IPS e.max ceram	403	4	40	750	1

the strength of the interfacial bonding between the Y-TZP core and the veneering porcelain [18]. Traditionally, the shear and micro-tensile bond tests have been used to assess the interfacial binding strength, but both fail to estimate the non-uniform stress distribution at the interface [19]. Moreover, non-interfacial failure that is started by surface tensile stresses away from the interface can compromise the stress values that are yielded by such conventional measures [19]. A better test of the bond strength, at least at metal–ceramic interfaces, may be the interfacial four-point bending test, which is based on the concept of fracture mechanics [20]. This method was first described by Charalambides et al. and was later adapted for testing dental materials. It eliminates non-uniform stress distribution during loading and ensures stable crack growth at the interface [20,21]. Hence, this has been proven to be reliable for adhesion analyses of bimaterial dental restorative systems [20].

Clinical performance of all-ceramic dental restorations may be influenced by the moisture-induced internal stress within the veneered all-ceramic materials [22]. This stress can arise because the interfaces of these restorations are located in a wet environment whose temperature changes irregularly [22]. Thermal cycling in hot and cold fluids can simulate the hydrothermal aging condition at the interface of bilayer complex [2,22].

The purpose of the present study was to use the four-point bending test to evaluate the effect of various surface treatments on the interfacial fracture toughness between zirconia and the veneer after thermal aging in an aqueous environment. The surface treatment methods were airborne-particle abrasion and application of different intermediate ceramic layers (a liner, a glass-based bonder, or no bonding layer). The bonded interfaces of bilayers were analyzed with micro-Raman spectroscopy. The null hypothesis to be tested was that the bonding quality of the zirconia/veneer interface would not be influenced by the surface treatment conditions.

2. Materials and methods

2.1. Preparation of bilayer specimens and thermal aging

Table 1 lists the chemical compositions and mechanical properties of the materials that were used in this study. Sixty fully-sintered 3 mol% yttria-stabilized tetragonal zirconia beams (ZirBlank-PS,

Acucera, Pocheon, South Korea) were prepared (40 mm \times 5 mm \times 1.5 mm) and a testing face on each zirconia specimen was polished with a Beta grinder-polisher (Bueher, Germany) to produce a standardized surface. The polishing protocol involved first polishing the zirconia with abrasive papers whose grain size decreased from 120 to 2400 grit. Thereafter, fine polishing was performed with a succession of discs with diamond suspensions that decreased in size from 6 μ m to 1 μ m. The specimens were then cleaned with ethanol in an ultrasonic bath for 5 min. Of the 60 zirconia beams, 30 were then subjected to airborne-particle abrasion with alumina particles that were 125 μ m in diameter (Cobra; Renfert, Hilzingen, Germany) at 0.2 MPa of pressure and a distance of 10 mm (Basic master; Renfert). The polished-only and polished-abraded groups of specimens were then randomly divided into three subgroups of ten specimens according to whether they did not receive an intermediate bonding layer or received a liner (IPS e.max Ceram ZirLiner, Ivoclar-Vivadent, Schaan, Liechtenstein) or a glass-based bonder (Hotbond Zirconnect, DCM, Rostock, Germany). Thus, there were six groups of specimens ($n=10$) in total. The liner was layered evenly on the polished surface of the zirconia specimen and fired to a thickness of approximately 0.1 mm (Table 2). The glass-based bonder was sprayed onto the test surface of the specimen and the specimen was fired to obtain a glassy surface coating that was approximately 20 μ m thick (Table 2). All specimens were then veneered with a fluorapatite glass ceramic (IPS e.max Ceram, Ivoclar-Vivadent) according to the firing schedule proposed by the manufacturing company (Table 2). After the porcelain was fused to the zirconia, the specimens were ground with a water-cooled diamond saw (ISOMET 2000, Buehler, UK) on the porcelain side to a total plate thickness of 3 mm, which corresponded to a core–veneer thickness ratio of 1:1. Each veneered Y-TZP block was then trimmed perpendicularly to the interface so that each specimen had a 4 mm \times 3 mm cross-sectional plane. There were no premature failures during this process. As an aging treatment, all specimens were subjected to thermal cycling for 25,000 cycles. Each cycle consisted of serial transfers of the specimens from a 5 °C water bath to a 55 °C water bath (ISO 11405). The specimens were immersed in each bath for 20 s and the transfer time between baths was 2 s. After aging, the porcelain–zirconia interface at the middle of the veneered surface of the test bilayer plates (40 mm \times 4 mm \times 3 mm) was notched by using a water-cooled diamond saw blade (600 μ m) (ISOMET 2000, Buehler, UK). The dimensions of the notch were 0.4 mm in width and 1.4 mm in depth. One side of the notch (a 3 mm-wide face) was mirror-

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