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Original Paper

Effect of Zirconia Surface Treatments on the Shear Strength of Zirconia/Veneering Ceramic Composites

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Aim of the investigation was to assess the effect of different surface treatments on the bond strength of veneering ceramics to zirconia.

In a shear test, the influences of polishing, sandblasting, and silica-coating of the zirconia surface on bonding were assessed with five different veneering ceramics. In addition the effect of liner application was examined. With one veneering ceramic, the impact of regeneration firing of zirconia was also evaluated. Statistical analysis was performed with one-way ANOVA and *post hoc* Scheffé's test.

Failure in every case occurred in the veneering ceramic adjacent to the interface with a thin layer of ceramic remaining on the zirconia surface, indicating that bond strength was higher than the cohesive strength of the veneering ceramic. Shear strength ranged from 23.5±3.4MPa to 33.0±6.8MPa without explicit correlation to the respective surface treatment. Regeneration firing significantly decreased the shear strength of both polished and sandblasted surfaces.

Findings of this study revealed that bonding between veneering ceramics and zirconia might be based on chemical bonds. On this note, sandblasting was not a necessary surface pretreatment to enhance bond strength and that regeneration firing was not recommended.

Key words: Zirconia, Veneering ceramic, Shear strength

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INTRODUCTION

Yttria-stabilized tetragonal zirconia (Y-TZP) opens new vistas for all-ceramic restorations. High flexural strength and fracture toughness afford its application as framework material for fixed partial dentures even in loaded reconstructions in the molar region1,2). According to theory, tensile stress in crack tips leads to a phase transition of the lattice structure from the metastable tetragonal to monoclinic. The phase transition is correlated with a volume increase of 4−5%. This volume increase inhibits crack propagation by creating a compressive stress3).

Clinical failures of veneered Y-TZP frameworks — due to chipping of the veneering ceramic — are reported to be 13.0% after an observation period of three years4) and 15.2% after five years5). Sufficient bond strength between the veneering ceramic and the substructure is therefore a concern for the long-term clinical success of zirconia restorations. Bond strength is determined by a host of factors: strength of the chemical bonds, mechanical interlocking, type and concentration of defects at the interface, wetting properties, and the degree of compressive stress in the veneering layer due to a difference in the coefficients of thermal expansion between zirconia and the veneering ceramic6-8). Few studies have dealt with the influence of different surface treatments on bond quality, and the mechanism of bonding is not completely understood9-11).

On enhancing bond strength, sandblasting is a popular means used to achieve this purpose by increasing surface roughness and providing undercuts9,11). However, sandblasting also initiates phase transition, thus affecting mechanical strength and most probably, the bonding capacity of the material12,13). This is because the coefficient of thermal expansion of monoclinic zirconia ($7.5 \cdot 10^{-6}$/K)14) is significantly lower than that of tetragonal zirconia ($10.8 \cdot 10^{-6}$/K)15). On this score, the effect of sandblasting on the mechanical strength of Y-TZP and the bond quality to veneering ceramics is an intensely discussed subject12,16,17).

For veneering zirconia, silicate ceramics are used. Silica coating of zirconia, therefore, may be considered to enhance bond strength. Silica coating has been applied on zirconia to enhance its bond strength to resins18). As such, silica-coated alumina particles with a grain size of 110 μm are blasted onto the substrate surface. Due to the high kinetic energy of the particles at the impact, silica is fused to the substrate surface. Depending on the bond strength of the silica layer to zirconia, the bond strength of the veneering ceramic might also be enhanced. However, no information is available to support the hypothesis.

With one manufacturer (Vita Zahnfabrik, Bad Säckingen, Germany), a regeneration firing of the zirconia framework for 15 minutes at 1000℃ prior to veneering is recommended. This was done to the end of re-establishing the tetragonal lattice after sandblasting or grinding, and thereby to obtain better bond strength[19]. It is supposed that a superficial layer of the monoclinic phase, created by sandblasting or grinding, leads to tensile stress in the veneering layer due to the quite low coefficient of thermal expansion of the monoclinic phase. However, no scientific evidence is provided to prove this theory. In contrast, a phase transition from monoclinic to tetragonal phase, following thermal treatment, reduces the compression layer at the surface, thus reducing the flexural strength[12,13,16,20].

To measure the bond strength of all-ceramic systems, shear tests[9,21] or microtensile tests[10,11] are generally used to evaluate the influence of the substrate surface on bond quality. Laboratory studies have shown that failure of the veneer primarily occurs near the zirconia-veneer interface with residual veneering ceramic remaining on the zirconia[21]. This could be interpreted as a good chemical bond between both layers. Polishing or sandblasting the zirconia surface revealed no differences in microtensile bond strength[10], indicating that enhanced surface roughness generated by sandblasting is probably not necessary to obtaining a good bond quality. However, in that study, only one veneering ceramic was investigated, thus barring a general conclusion to be drawn.

A further aspect on bond strength to be considered is the application of liners. Liners can be applied as an intermediate layer between the zirconia substrate and the veneering ceramic to mask the framework and to increase the wetting property on the zirconia surface. This additional intermediate layer in some material combinations dramatically affected bond strength[11].

To date, limited knowledge is available on the bonding of veneering ceramics to zirconia, especially on the effects of sandblasting and liner application. The hypothesis of this study was that (1) sandblasting, (2) silica coating, (3) liner application, or (4) regeneration firing will increase the bond strength of zirconia to veneering ceramics.

MATERIALS AND METHODS

Materials used

Table 1 lists the five veneering ceramics investigated in this study. To measure the bond strength between the core material and the veneering ceramic, a shear test according to Schmitz-Schulmeyer[9] was applied. Cubes of Y-TZP (YZ Cubes, Vita Zahnfabrik, Bad Säckingen, Germany) were prepared in the white state and densely sintered (ZYrcomat T, Vita Zahnfabrik). Final edge length was 10 mm, and one face of each cube was polished with diamond paste down to 3 μm. The cubes were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath with ethanol.

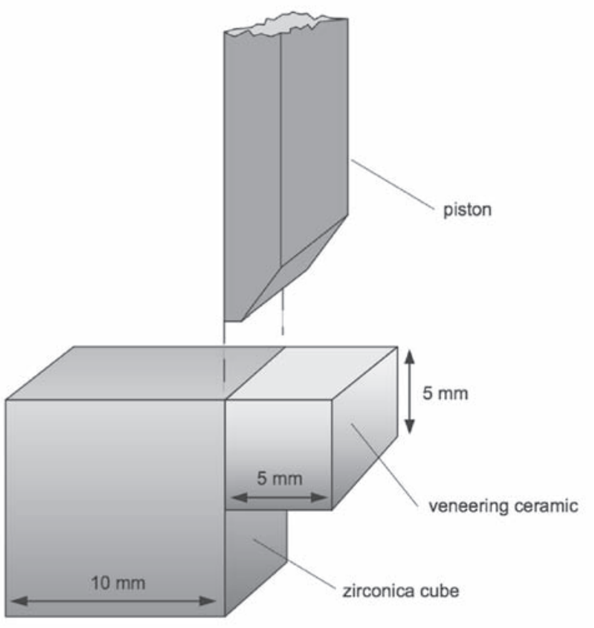


Fig. 1 Schematic illustration of specimen size and test design.

Table 1 Veneering ceramics used in this investigation

Veneering ceramic	Lot No. Liner	Lot No. Liquid	Lot No. Dentin	Lot No. Liquid	Manufacturer	Code
Cerabien ZR	OE720	OEY01	201611	OEY01	Noritake, Nagoya, Japan	CZ
IPS e.max	H30927	H33669	H24320	H33669	Ivoclar-Vivadent, Schaan, Liechtenstein	EM
Triceram	006A	015A	019	042005	Dentaurum, Ispringen, Germany	TC
Vintage ZR	PN9019	090301	120502	090106	Shofu, Kyoto, Japan	VZ
VM9	15420	7728	30580	10780	Vita, Bad Säckingen, Germany	VM

Surface pretreatments

Three different surfaces were investigated: polished, sandblasted, and silica-coated. Sandblasting was performed on the polished face with 110-μm alumina for 10 seconds at a pressure of 0.2 MPa and at a distance of 10 mm between the nozzle and the surface (CEMAT NT4, Wassermann, Hamburg, Germany). Silica coating (Rocatector Delta, 3M ESPE, Seefeld, Germany) was performed on the polished face with Rocatec Pre (3M ESPE) for 10 seconds and subsequently Rocatec Plus (3M ESPE) for 12 seconds, and both at 0.28 MPa.

Ceramic veneering, liner application and regeneration firing

For each combination, 10 specimens were veneered. On the prepared face of each cube, a 5-mm layer of veneering ceramic was fired, covering an area of 5 mm × 10 mm at one edge of the face (Fig. 1). A separable steel mold was used to layer the ceramic. The mold was isolated (Ceramic Separating Stick, Ivoclar Vivadent, Schaan, Liechtenstein) to avoid the adhesion of ceramic powder to the mold during layering. Ceramic powder was mixed with an appropriate amount of the respective liquid as per the common practice in a dental laboratory, and filled into the mold. Excess liquid was sucked off with a tissue. Solely dentin, but no enamel, was layered. Firing was done in a ceramic oven (Austromat D4, Dekema, Freilassing, Germany) according to the manufacturers' recommendations (Table 2). In a second firing under the same conditions, dentin was added to compensate the shrinkage of the sintering process. Prior to the second firing, the slurry was jiggled in the mold for two seconds at 50 Hz (Porex Elektro Vibrator, Renfert, Hilzingen, Germany).

To investigate the effect of liner application, a second series of specimens was produced with the respective liner prior to veneering.

With VM9, the effect of an additional heat treatment after sandblasting (*i.e.*, regeneration firing) was assessed. To this end, a series of polished specimens and a series of sandblasted specimens were heated for 15 minutes at 1000℃ in the ceramic oven prior to veneering as recommended by the manufacturer.

Shear test

Finished specimens were fixed in a special sample holder and placed in a universal testing machine (Z010, Zwick, Ulm, Germany). The ceramic block was loaded up to failure with a chisel-shaped piston at the interface parallel to the zirconia surface with a crosshead speed of 1 mm/min (Fig. 1). Shear strength was calculated as the mean of 10 specimens from the load at fracture and the surface area of the zirconia-veneer interface.

Surface morphology and surface roughness evaluations

Zirconia surface morphologies after different surface treatments and the fractured areas after shear test were documented by means of SEM (CS4, CamScan, Waterbeach, UK). Surface roughness of the polished, sandblasted, and silica-coated specimens was measured (MarSurf GD25, Mahr, Göttingen, Germany) on five specimens for each type of surface treatment.

Statistical analysis

Results of the shear test were statistically analyzed with one-way ANOVA, followed by a *post hoc* Scheffé's test. Surface roughness measurement results were statistically analyzed with a t-test (SPSS 15.0, SPSS Inc., Chicago, IL, USA; $p<0.05$).

Table 2 Firing schedules of the veneering ceramics

Veneering Ceramic	Pre Drying		Heating Rate (℃/min)	Firing Temperature (℃)	Holding Time (min)
	Temperature (℃)	Time (min)			
Liner (vacuum during heating)					
Cerabien ZR	700	2	65	1090	1
IPS e.max	400	4	60	960	1
Triceram	500	4	65	800	1
Vintage ZR	500	8	45	940	1
VM9	500	6	55	930	1
Dentin (vacuum during heating)					
Cerabien ZR	600	5	45	930	1
IPS e.max	400	4	50	750	1
Triceram	500	6	55	760	2
Vintage ZR	650	6	45	920	1
VM9	500	6	55	910	1

RESULTS

Shear strength

Shear strength on the polished surface was in a wide range of 23.5±3.4MPa for IPS e.max to 31.0±7.1MPa for Triceram. However, no statistical significant differences were found (Fig. 2). The effects of different surface treatments and liner application were quite consistent between the different brands, with only few significant differences found within the groups (Fig. 2). Sandblasting in no case enhanced shear strength significantly. On the contrary, sandblasting and silica coating reduced the shear strength of Triceram significantly. Vintage ZR also showed a significant decrease in shear strength after sandblasting in conjunction with liner application. Regeneration firing significantly decreased the shear strength of VM9 on both polished and sandblasted surfaces (Fig. 3).

Failure analysis

In all the specimens, the fracture started at the core-veneer interface and proceeded into the veneering ceramic, observable in the SEM image after debonding (Fig. 4). Veneering material remaining on the zirconia surface was clearly visible.

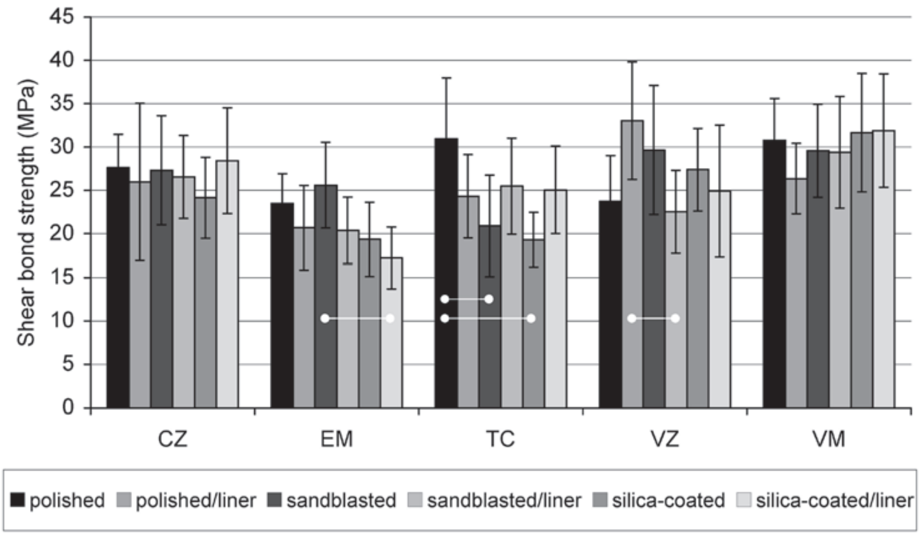


Fig. 2 Shear strengths of veneering ceramics after different surface treatments. Statistically significant differences within the groups are marked with •——•.

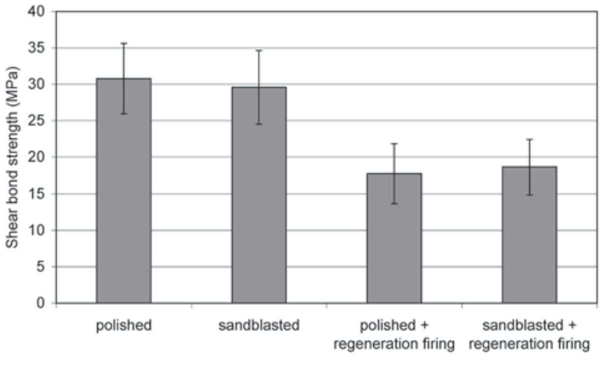


Fig. 3 Shear strengths of VM9 after regeneration firing.

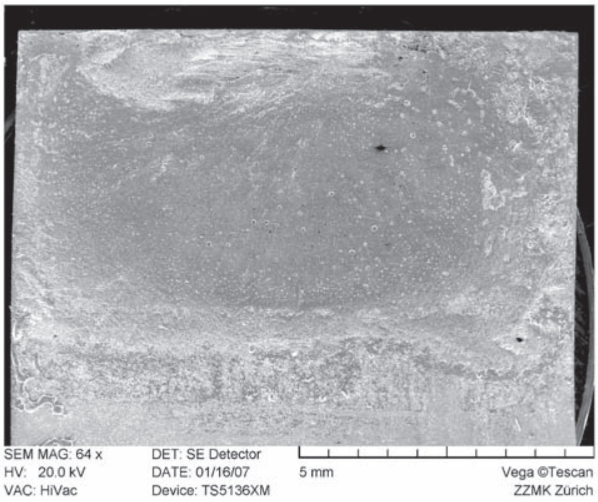
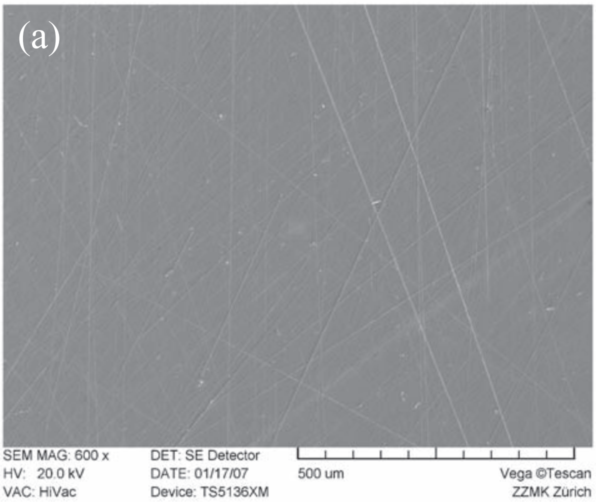
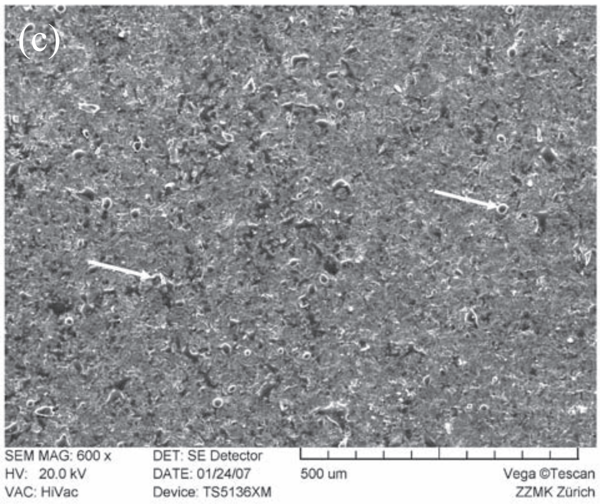


Fig. 4 Scanning electron microscope image of VM9 specimen after the shear test, zirconia side. Veneering ceramic remained on the zirconia surface.

Table 3 Surface roughness (Ra) values of the different zirconia surfaces

	polished	sandblasted	silica-coated
without regeneration firing	0.07 (±0.00)	0.63 (±0.01)	0.67 (±0.01)
with regeneration firing	0.06 (±0.00)	0.60 (±0.01)	





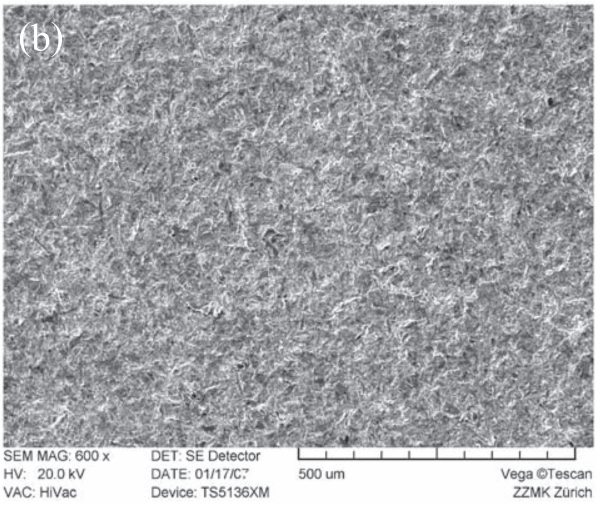


Fig. 5 Scanning electron microscope images of zirconia surfaces: (a) Surface after polishing. Grooves due to the polishing process were visible; (b) Surface after sandblasting with 110-μm alumina; (c) Surface after silica coating. Small nodules were attached to the surface (arrows).

Surface morphology

SEM revealed the different surface morphologies of polished, sandblasted, and silica-coated surfaces (Fig. 5). On the polished surface (Fig. 5a), grooves due to the polishing process were visible on an otherwise smooth surface. Sandblasting led to a distinctly rough surface (Fig. 5b). After silica coating, nodules were observed on the surface (Fig. 5c). Corresponding to these findings, surface roughness significantly increased after sandblasting with alumina or silica coating (Table 3). No statistical significant differences in surface roughness were found between the sandblasted and silica-coated specimens. After regeneration firing, surface roughness was slightly — but not significantly — reduced.

DISCUSSION

After the shear test in the present study, veneering ceramic remained on the zirconia surface for every specimen group. Thus, it could be concluded that the bond strength between zirconia and the veneering ceramic was higher than the cohesive strength of the veneering ceramic. In other words, the weakest link was not the interface but the veneering ceramic itself. This also showed that the applied test design analyzed not the bond strength, but the shear strength of the veneering ceramic adjacent to the interface. These findings were consistent with the observations of Al-Dohan *et al.*[21] who also used a shear test. This failure result could be ascribed to the stress that was generated and which peaked near the interface, due to a difference in the coefficients of thermal expansion between both layers. This led to a stress concentration parallel to the interface, preventing crack propagation along the interface.

Based on the present results, the hypothesis that surface treatments of zirconia will increase its bond strength to veneering ceramics could neither be accepted nor rejected.

On the effect of surface treatments, neither sandblasting nor polishing improved the shear strength. This conclusion was made on the premise that it was the cohesive strengths of the ceramics, but not the bond strength of the interface, which were tested in this study. In one specimen group (Triceram), the polished surface yielded the highest shear strength among all the six conditions tested, and which was significantly higher when compared to the sandblasted and silica-coated surfaces. Different wetting conditions or different surface energies might have accounted for these results.

Strong bonding of the veneering ceramics to polished zirconia surfaces suggested that chemical bonds were established between both materials during firing. Consequently, surface roughness as created by sandblasting was not necessary to enhance bond strength. Bonding of a veneering ceramic to a polished zirconia surface has been investigated previously[10]. With veneering ceramic not included in the present investigation, no differences in shear strength were found between the polished and sandblasted surfaces, as shown by three out of five ceramics in the present investigation. In the aforementioned study[10], polishing was performed with 1200-grit SiC paper, corresponding to a mean grain size of $3.0 \pm 0.5\,\mu$m[22], and was thus comparable to the surface finish obtained in the present investigation. However, the bond strength values in the aforementioned study[10] could not be compared with those of the present study because of the following reasons: (1) microtensile bond strength test was used *versus* the shear test in the present study; and (2) data for the polished and the sandblasted surfaces were not given separately.

In comparison to another study[9] that employed the same test design to investigate the bond strength of an experimental veneering ceramic to yttria-stabilized zirconia, the strength values obtained in the present investigation were lower than the reported value of 36.2 MPa. At this juncture, it warrants a cautious note that comparison of test results must be done discerningly. In the cited study[9], the specimen size was different from that of the present study: edge length of 5.8 mm *versus* a dimension of 10 mm the in the present investigation. According to Weibull theory[23], strength values of larger specimens are lower than those obtained with smaller ones because larger specimens exhibit a higher probability for surface defects and therefore a higher risk of early failure. Other parameters must also be considered with regard to the different shear strength values. The greater mass of the Y-TZP cubes implied a greater heat capacity, which might slow down the sintering process and the cooling of the veneering ceramic. In addition, the oven and the firing schedule were most probably different. However, no details concerning sample preparation were given in the cited study[9]. It is also noteworthy that in the cited study[9], an experimental veneering ceramic was used, which was probably different in chemical composition and thermal expansion.

In the present study, liner application did not affect shear strength. In contrast to our findings, liner-applied microtensile specimens were doubled in bond strength if a proper contact were established between the veneering ceramic and zirconia surface[10]. However, in the same study[10] with an experimental veneering ceramic, a complete denudation of the zirconia surface after microtensile testing was observed. Microspaces between zirconia and the liner, which were interpreted as a result of poor wetting, accounted for such a failure.

Regeneration firing for 15 minutes at 1000℃ strongly and adversely affected shear strength. The manufacturer's claim that phase transition after sandblasting or grinding must be reversed to retrieve the coefficient of thermal expansion of the tetragonal zirconia surface could not be proved or verified in this study. Phase transition from monoclinic to tetragonal occurs at temperatures above 900℃[16]. However, microcracks do not close during heating at 1000℃. The reduced shear strength observed in this study might be interpreted as follows. During regeneration firing, the compression layer at the surface relaxed, thus reducing the internal compression. Further, microcracks did not close at this temperature. Consequently, the overall strength of zirconia decreased[12,13,16,20], thus affecting the shear strength of the veneering ceramic adjacent to the interface. Differences in failure mode, however, were not observed in the present study.

Since the bond strength of the interface was higher than the cohesive strength of the ceramic, it was concluded that the veneering ceramic was the weakest link. To realize the benefit of the high strength of zirconia frameworks, the strength of veneering ceramics has to be improved.

CONCLUSION

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

1. Increased surface roughness of zirconia did not enhance shear strength.
2. Application of a liner did not enhance shear strength.
3. Regeneration firing, as recommended by one manufacturer, decreased shear strength.
4. To realize the benefit of the high strength of

zirconia frameworks, the strength of veneering ceramics has to be improved.

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REFERENCES

1) Sturzenegger B, Fehér A, Lüthy H, Schärer P, Gauckler LJ. Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM). Int J Comp Dent 2001; 4: 89-106
2) Lüthy H, Filser F, Loeffel O, Schuhmacher M, Gauckler LJ, Hämmerle CHF. Strength and reliability of four unit all-ceramic posterior bridges. Dent Mater 2005; 21: 930-937.
3) Garvie RC, Hannink RH, Pascoe RT. Ceramic steel? Nature 1975; 258: 703-704.
4) Sailer I, Fehér A, Filser F, Lüthy H, Gauckler LJ, Schärer P, Hämmerle CHF. Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow-up. Quintessence Int 2006; 37: 41-49.
5) Sailer I, Fehér A, Filser F, Gauckler LJ, Lüthy H, Hämmerle CHF. Five-year clinical results of zirconia frameworks for posterior fixed partial dentures. Int J Prosthodont 2007; 20: 383-388.
6) al-Shehri SA, Mohammed H, Wilson CA. Influence of lamination on the flexure strength of dental castable ceramic. J Prosthet Dent 1996; 76: 23-28.
7) Isgro G, Pallav P, van der Zel JM, Feilzer AJ. The influence of the veneering porcelain and different surface treatments on the biaxial flexure strength of a heat-pressed ceramic. J Prosthet Dent 2003; 90: 465-473.
8) De Jager N, Pallav P, Feilzer AJ. The influence of design parameters on the FEA-determined stress distribution in CAD-CAM produced all-ceramic crowns. Dent Mater 2005; 21: 242-251.
9) Luthardt RG, Sandkuhl O, Reitz B. Zirconia-TZP and Alumina — Advanced technologies for the manufacturing of single crowns. Eur J Prosthodont Rest Dent 1999; 7: 113-119.
10) Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Dent Mater 2005; 21: 984-991.
11) Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: Zirconia veneering ceramics. Dent Mater 2006; 22: 857-863.
12) Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater 1999; 15: 426-433.
13) Guazzato M, Quach L, Albakry M, Swain M V. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. J Dent 2005; 33: 9-18.
14) Patil RN, Subbarao EC. Axial thermal Expansion of ZrO_2 and HfO_2 in the range room temperature to 1400℃. J Appl Cryst 1969; 2: 281-288.
15) Fischer J, Stawarczyk B. Compatibility of machined Ce-TZP/Al_2O_3 nanocomposite and a veneering ceramic. Dent Mater 2007; 23: 1500-1505.
16) Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. J Biomed Mater Res (Appl Biomater) 2000; 53: 304-313.
17) Zhang Y, Lawn BR, Malament KA, Thompson VP. Damage accumulation and fatigue life of particle-abraded ceramics. Int J Prosthodont 2006; 19: 442-448.
18) Lüthy H, Loeffel O, Hämmerle CHF. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 2006; 22: 195-200.
19) Vita Zahnfabrik. Veneering material VitaVM9. Working instructions. Vita Zahnfabrik, Bad Säckingen, 2007.
20) Sundh A, Molin M, Sjogren G. Fracture resistance of yttrium oxide partially-stabilized zirconia all-ceramic bridges after veneering and mechanical fatigue testing. Dent Mater 2005; 21: 476-482.
21) Al-Dohan HM, Yaman P, Dennison JB, Razzoog ME, Lang BR. Shear strength of core-veneer interface in bi-layered ceramics. J Prosthet Dent 2004; 91: 349-355.
22) FEPA-Standard 42-2: 2006. Grains of fused aluminum oxide, silicon carbide and other abrasive materials for bonded abrasives and for general industrial applications Microgrits F230 to F2000. Federation of European Producers of Abrasives, Paris, 2006.
23) Weibull W. A statistical distribution function of wide applicability. J Appl Mech 1951; 18: 293-297.

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