Effect of Thermal Misfit between Different Veneering Ceramics and Zirconia Frameworks on *in vitro* Fracture Load of Single Crowns

Jens FISCHER¹, Bogna STAWARCZYK¹, Milos TOMIC², Jörg R. STRUB² and Christoph H.F. HÄMMERLE¹ ¹Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Center for Dental and Oral Medicine, University of Zurich, Plattenstrasse 11, CH-8032 Zurich, Switzerland ²Department of Prosthodontics, School of Dentistry, University Hospital Freiburg, Hugstetterstrasse 55, D-79106 Freiburg, Germany Corresponding author, Jens FISCHER; E-mail: jens.fischer@zzmk.uzh.ch

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Thermal misfit is deemed as one reason for the chipping of veneered zirconia restorations. Aim of the present study was to assess the effect of thermal misfit on the fracture load of veneered zirconia frameworks and to evaluate the applicability of a universal veneering ceramic for both zirconia and titanium frameworks. Fracture loads of zirconia and titanium frameworks veneered with different ceramics were measured. Differences in the thermal expansion coefficients of core and veneer ($\Delta \alpha$), as well as differences between glass transition temperatures of the veneering ceramics and room temperature (ΔT) — which is considered as the effective temperature range for stress formation, were determined. In the zirconia group, fracture load ranged from 818.0 ± 127.2 to 935.2 ± 186.3 N without significant differences (Student's t-test, p<0.05). Moreover, zirconia and titanium crowns veneered with the universal veneering ceramic revealed high fracture load. Results also showed a correlation to the product $\Delta \alpha \cdot \Delta T$, such that if $185 \cdot 10^{-6} < \Delta \alpha \cdot \Delta T < 1120 \cdot 10^{-6}$, a veneering ceramic adapted for titanium might be likewise applicable for zirconia.

Keywords: Zirconia, Thermal misfit, Fracture load

INTRODUCTION

Yttria-stabilized zirconia (Y-TZP) frameworks provide high mechanical strength^{1,2)} and high biocompatibility^{3,4)}. By virtue of these favorable characteristics, they are increasingly used in restorative dentistry. For esthetical reasons, these frameworks are layered with a veneering ceramic. A crucial point for clinical, long-term success is the compatibility of the core and its veneering material. For metal-ceramic restorations, it is known that the quality of bond between both layers is, among other parameters, affected by the difference in the coefficients of thermal expansion (α) of core and veneering material⁵⁾.

A thermal mismatch between framework and veneer leads to compressive or tensile stress in the veneer, depending on whether the thermal expansion of the veneering ceramic is lower or higher than that of the framework material. Ceramics are susceptible to tensile stress while compressive stress is less critical. A slight compressive stress in the veneer is favored since the veneering ceramic is reinforced and the fracture strength increased. Hence, thermal expansion of the veneer must be lower than that of core material to generate such a compressive stress during cooling⁵⁾. For metal-ceramic restorations, the exact range of compatible values for thermal expansion is not determined^{5,6)}. As a rule of thumb, it is generally accepted that the coefficient of thermal expansion of the veneer should be about 10% below that of the metal substrate⁷⁾.

In clinical studies with zirconia frameworks,

chipping of the veneering ceramic revealed to be a major problem. Complication rates are reported to be 15% after two years⁸⁾ or 13% after three years⁹⁾, which is significantly higher compared to metal-ceramic restorations¹⁰⁾. Thermal misfit between framework and veneer is suggested to be one reason for this high failure rate¹¹⁾.

Some investigations have dealt with the thermal compatibility of zirconia and veneering material. For instance, spontaneous debonding occurred with microtensile bond strength samples when an experimental ceramic with a thermal expansion coefficient of 12.5 μ m/m·K was used on 5Y-TZP ($\alpha = 10.5 \mu$ m/m·K), while a commercial ceramic with a thermal expansion coefficient of 9.5 μ m/m·K showed good results¹¹). On the other hand, a strong thermal mismatch in a bilayered sample with the veneering layer having a thermal expansion coefficient of 5 μ m/m·K led to tensile stress in the underlying zirconia. This stress was revealed by a Vickers indentation in the zirconia layer close to the interface, leading to strong crack propagation into the zirconia¹².

Stress in the veneering ceramic may develop only below the glass transition temperature (Tg). Above Tg, a thermal misfit is compensated by plastic flow. Therefore, in addition to the mismatch of thermal expansion coefficients, the magnitude of the interval between Tg of the veneering ceramic and room temperature has to be considered as a second criterion for stress formation. To the knowledge of the authors, no recommendations concerning the adequate coefficient of thermal expansion of veneering materials for zirconia frameworks are available.

Titanium shows an α which is about 10% lower compared to zirconia. For that reason, two different types of veneering ceramics are offered for these two core materials. With a view to consolidating the wide range of veneering materials available for titanium and zirconia frameworks, a universal veneering ceramic suitable for both frameworks was developed (Triceram, Dentaurum, Pforzheim, Germany). To avoid tensile stress in the veneer, the coefficient of thermal expansion of such a ceramic must be lower than that of titanium. Consequently, the difference against the coefficient of thermal expansion of zirconia is quite high. However, to date, no evidence for the functional efficiency of this universal veneering ceramic is published.

The aim of the present investigation, therefore, was to assess the influence of varying a's and Tg's of commercially available veneering ceramics on the *in vitro* fracture load of layered zirconia frameworks and to verify if a veneering ceramic matching to titanium is likewise applicable for zirconia.

MATERIALS AND METHODS

Frameworks

To produce standardized frameworks, a metal tooth analog with the shape of a prepared upper canine with a chamfer, cast from a CoCr alloy¹³⁾ was duplicated in plaster.

1. Zirconia frameworks

To fabricate zirconia frameworks, the plaster die was scanned (Lava Scan, 3M ESPE, Seefeld, Germany). A coping was designed with an anatomical support at the incisal edge, as per the common practice in dental laboratories (Fig. 1). A total of 70 copings were produced (Lava Frame, 3M ESPE).

2. Titanium frameworks

Twenty titanium copings were manufactured to evaluate the fracture strength of titanium frameworks



Fig. 1 Schematic illustration of the veneered crowns, left: lateral view, right: frontal view (dotted line: location of the frontal section).

veneered with Triceram and Vita Ti (Table 1). To produce titanium copings identical in shape to the zirconia frameworks, one zirconia coping was duplicated in silicone (Sil-Tech Plus, Ivoclar Vivadent, Schaan, Liechtenstein, Germany) and wax patterns (Aqua Green, Kerr Lab, West Collins Orange, USA) were cast. The wax patterns were invested in sets of five (Trinell, Dentaurum, Ispringen, Germany), and casting was performed with titanium grade 1 in a standard procedure (Autocast Universal 230, Dentaurum). 3. High gold alloy frameworks for control

As a control, 10 frameworks of a high gold alloy (Degudent U, DeguDent, Hanau, Germany) were produced by casting technique using the same wax patterns as for the titanium copings. Wax patterns in this case were invested in a phosphate bonded investment (Ceramicor, Cendres & Métaux SA, Biel, Switzerland). Casting was performed with an oxygen-propane flame and a centrifuge (Multicast, Degu-Dent). The molds were bench-cooled before divesting. Casting procedures were consistent with the alloy manufacturer's instructions.

Veneering of the frameworks

The copings were divided into sets of 10 and veneered with different ceramics according to Table 1. The sprues of the metal frameworks were cut, and all metal frameworks were sandblasted on the surface with 50- μ m alumina particles at a pressure of 0.2 MPa and at a distance of 10 mm between nozzle and workpiece. The zirconia copings were veneered without further surface treatment. On all zirconia copings, a liner was applied according to the manu-

Table 1 Veneering ceramics used in the investigation

Core material	Veneering ceramic	Manufacturer
	┌─ IPS e.max	Ivoclar Vivadent,
		Schaan, Liechtenstein
Y-TZP	Cerabien ZR	Noritake, Nagoya,
		Japan
	Lava Ceram	3M ESPE, Seefeld,
		Germany
	Zirox	Wieland, Pforzheim,
		Germany
	Rondo Zirconia	Nobel Biocare,
		Gotenborg, Sweden
	Vita VM9	Vita, Bad Säckingen,
		Germany
	– Triceram	Dentaurum, Ispringen,
Ti		Germany
	Vita Ti	Vita, Bad Säckingen,
		Germany
De mala de II	Vita VM13	Vita, Bad Säckingen,
Degudent U		Germany

		Liner/opaque ii	ring (vacuun	during neating)		
Core Material	Veneering Ceramic	Pre Drying		Heating Rate	Firing	Holding Time
Core Material		Temperature (°C)	Time (min)	(°C/min)	(°C)	(min)
ZrO_2	e.max	400	4	60	960	1
	CerabienZR	700	2	65	1090	1
	Lava	no liner available				
	Zirox	575	2	45	930	1
	RondoZirconia	575	2	45	945	1
	VM9	500	6	55	930	1
	Triceram	500	4	65	800	1
titanium	Triceram	500	4	65	795	1
	Vita Ti	400	2	98	790	1
Au alloy	VM13	500	6	55	890	1
		Dentin (v	acuum durin	g heating)		
ZrO_2	e.max	400	4	50	750	1
	CerabienZR	600	5	45	930	1
	Lava	450	6	45	800	1
	Zirox	575	3	45	900	2
	RondoZirconia	575	5	45	925	1
	VM9	500	6	55	910	1
	Triceram	500	6	55	760	2
Titanium	Triceram	500	6	55	755	1
	Vita Ti	400	6	53	770	1
Au alloy	VM13	500	6	55	880	1
		Gla	zing (no vacu	ium)		
$ m ZrO_2$	e.max	400	6	60	725	1
	CerabienZR	600	5	50	930	4
	Lava	480	2	45	820	1
	Zirox	575	1	45	880	1
	RondoZirconia	575	1	55	945	1
	VM9	500	0	80	900	1
	Triceram	500	2	55	760	1
Titanium	Triceram	500	2	55	755	1
	Vita Ti	400	0	93	770	1
Au alloy	VM13	500	0	80	880	2

Table 2 Firing schedules of the veneering ceramics according to the manufacturers

facturers' recommendations except for Lava Ceram, where no liner was available. The firing schedules strictly followed the instructions of the manufacturers (Table 2), using an appropriate ceramic oven (D4, Dekema, Freilassing, Germany) and a procedure commonly used in dental laboratories. To achieve a standardized shape and size of the veneers, a silicone key was used.

Fracture load test

The crowns were one by one fit to the metal tooth analog as per the common practice in dental laboratories, and subsequently cemented with a glass ionomer cement (Ketac Cem, 3M ESPE). During cementation, the crowns were secured with finger pressure for one minute. Setting of the glass ionomer cement was allowed for 10 minutes at ambient conditions. Subsequently, the analog was fixed in a metal mounting support and specimens were loaded in a universal testing machine (Z010, Zwick, Ulm, Germany). Extra axial load was induced with a flat cylindrical piston with a diameter of 9 mm on the palatal side of the incisal edge at an angle of 45 degrees to the long axis of the tooth. Force was applied at a crosshead speed of 1 mm/min and recorded up to failure. To avoid force peaks, a piece of 0.5-mm tin foil was placed between the incisal edge and the loading piston.

Linear thermal expansion coefficient measurement

Six samples for each material were prepared for linear thermal expansion coefficient measurement. The metal samples were cylindrical with a diameter of 5 mm and a length of 25 mm. All ceramic samples were prismatic with a dimension of $25 \times 5 \times 5$ mm. Samples of the veneering ceramics were fabricated using an appropriate mold according to ISO 9693: 1999. One dentin firing was performed following the manufacturers' instructions. The zirconia samples were cut and densely sintered (Lava Therm). Titanium and high gold alloy samples were fabricated from wax patterns by using the same casting procedures as described above. High gold alloy samples were annealed for 10 minutes at 950°C in the ceramic oven to simulate ceramic firing.

Measurement was effected at a heating rate of 5 K/min up to 650°C (DIL 402C, Netzsch, Selb, Germany). Linear thermal expansion coefficient between 25°C and 500°C was determined as the mean of six measurements. Further, glass transition temperatures of the veneering ceramics designed for zirconia frameworks were determined from these measurements by extrapolation, as per the common practice in thermal analysis.

Statistical analysis

Fracture strength values of the crowns were statistically analyzed with a Student's t-test (SPSS Inc., Chicago, IL, USA; p<0.05). In addition, Weibull statistical analysis was performed.

RESULTS

Fracture loads of zirconia crowns ranged from 818.0 ± 127.2 N (IPS e.max) to 935.2 ± 186.3 N (VM9) (Table 3). Zirconia frameworks veneered with Triceram showed the second highest load capacity in the all-ceramic group (930.5 \pm 145.8 N). With the zirconia frameworks, fracture occurred either in the veneering ceramic or that a complete failure of veneer and framework was observed — with no preference for the one system or another. Titanium frameworks veneered with Triceram fractured at 898.6 ± 105.7 N, which was higher than the value obtained for the specimens veneered with Vita Ti (762.9 \pm 105.6 N). As for the fracture load of high gold control group (VM13/Degudent U), it reached 990.0±161.3 N. Statistically significant differences were found only between VM13/Degudent U on one side and IPS e.max/zirconia (p=0.005), Cerabien ZR/zirconia (p= 0.041), and Vita Ti/titanium (p=0.004) on the other side. Weibull statistics showed only slight differences between the systems.

For the veneering ceramics intended for use on zirconia frameworks, coefficients of thermal expansion ranged from 8.7 μ m/m·K (Triceram) to 10.4 μ m/m·K (IPS e.max) (Table 4). The thermal expansion coefficient of Triceram was very close to that of Vita Ti (8.5 μ m/m·K). Glass transition temperatures ranged from 486.6°C (IPS e.max) to 603.9°C (VM9).

Table 3 Fracture loads and respective Weibull parameters of veneered crowns (SD: standard deviation;* : significantly different to Degudent U (p<0.05)).

Core material	Ceramic	Load at frac- ture (N) mean (±SD)	Weibull Modulus m	Weibull strength $\sigma_{63.21\%}$ (N)
Y-TZP	IPS e.max	818.0 (±127.2)*	7.4	872.2
	Cerabien ZR	836.0 (±117.3)*	8.3	885.9
	Lava Ceram	852.3 (±131.9)	7.5	907.7
	Zirox	855.2 (±145.8)	7.0	914.1
	Rondo Zirconia	849.9 (±119.2)	8.3	900.1
	VM9	935.2 (±186.3)	5.9	1009.4
	Triceram	930.5 (±145.8)	7.5	990.4
Ti	Triceram	898.6 (±105.7)	10.0	943.4
	Vita Ti	762.9 (±105.6)*	8.5	806.8
Degudent U	VM13	990.0 (±161.3)	7.1	1057.9

Table 4 Coefficients of thermal expansion between 25° C and 500° C (α) and glass transition temperatures (Tg) of the materials used (SD: standard deviation)

Material	$lpha$ (μ m/m•K) mean (±SD)	Tg (K) mean (±SD)
IPS e.max	10.4 (±0.0)	759.6 (±4.1)
Cerabien ZR	$9.9(\pm 0.3)$	832.1 (±8.1)
Lava Ceram	$9.9(\pm 0.1)$	$821.2 (\pm 2.3)$
Zirox	$9.8 (\pm 0.1)$	849.0 (±2.2)
Rondo Zirconia	$9.7~(\pm 0.1)$	$845.3 (\pm 2.5)$
VM9	$9.3 (\pm 0.1)$	876.9 (±3.3)
Triceram	8.7 (±0.3)	830.7 (±1.9)
Vita Titan	$8.5 (\pm 0.2)$	
VM13	$13.2 (\pm 0.3)$	
zirconia	$10.8 (\pm 0.1)$	
titanium	9.8 (±0.1)	
Degudent U	14.0 (±0.1)	



Fig. 2 Glass transition temperature (Tg) plotted against coefficient of thermal expansion (α).



Fig. 3 Fracture load of veneered zirconia crowns plotted against the product of the difference between the thermal expansion coefficient of veneer and core $(\Delta \alpha)$ and the difference between glass transition temperature of the veneering ceramic and room temperature (Δ T).

With the exception of Triceram, α and Tg were strongly correlated (Fig. 2). A linear least squares regression showed a coefficient of determination of $R^2=0.95$, when the value of Triceram was excluded.

As the difference in thermal expansion coefficient between core and veneer ($\Delta \alpha$), coupled with the difference in temperature range between glass transition temperature and room temperature (Δ T), may have an impact on stress formation in the samples, the product expression $\Delta \alpha \cdot \Delta T$ may act as a measure for the residual stress¹⁴. As such, a plot of obtained fracture strength values against $\Delta \alpha \cdot \Delta T$ should reveal the impact of thermal properties on fracture strength. Indeed, as shown in Fig. 3, the respective plots showed a slight linear correlation with R²=0.85 (Δ T in K).

DISCUSSION

Results of the present investigation displayed a variation in α for the veneering ceramics designed for zirconia frameworks, ranging from 8.7 μ m/m·K to

10.4 μ m/m·K. However, effect of this variation on the overall strength of the crowns was found to be minimal: no significant differences were observed between fracture load values within the zirconia group. To provide a plausible explanation for these results, it must first be put into perspective that stress formation arose due to a thermal misfit that occurred during the cooling down phase after firing. At temperatures higher than Tg, the ceramic is in a plastic state and adapts to deformations of the rigid core material by plastic flow. The effective temperature range for stress formation is therefore between Tg and room temperature, and a low Tg thus reduces the stress magnitude. A basic approach to rank the residual stress in the different systems is to presume Poisson's ratio and Young's modulus to be identical for the different veneering ceramics and to thereby calculate $\Delta \alpha \cdot \Delta T^{14}$. In this investigation, a plot of the fracture load values against $\Delta \alpha \cdot \Delta T$ revealed a positive linear correlation between both parameters, thus proving the hypothesis that a thermal mismatch can increase the fracture strength of layered crowns, although in this case only slightly. From the results obtained in this study, it could be concluded that as long as $\Delta \alpha \cdot \Delta T$ was kept between $185 \cdot 10^{-6}$ and $1120 \cdot 10^{-6}$, no excessive stress would be generated in the veneer.

The notion of using a single veneering ceramic for both titanium and zirconia frameworks revealed to be justifiable. Although Triceram had an extremely low α , the fracture load values were quite high, indicating that residual stress was not exceeding a critical level. In addition, the Weibull modulus - which is a measure of the reliability of the respective system — was highest for Triceram/titanium. For Triceram/zirconia, the Weibull modulus was in the mid-field range. If Triceram would follow the same conditions as the other veneering ceramics for zirconia, Tg would be in the range of 980 K ($\approx 705^{\circ}$ C). But Tg was successfully decreased to 831 K (≈558°C) in order to reduce the residual stress when used on zirconia. These findings thus supported the approach to use one single veneering ceramic for both zirconia and titanium, provided that $\Delta \alpha \cdot \Delta T$ stay below 1120 $\cdot 10^{-6}$. Such a universal ceramic is a valuable contribution in the effort to simplify the range of products.

Material testing with standardized, non-anatomical specimens leads to basic information but leaves the effect of the complex geometry of dental restorations unconsidered¹⁵. Conversely, a laboratory test setup with full crowns can be very close to clinical situations¹⁶. Therefore, to assess the overall strength of complete restorations, specimens that closely simulate real clinical situations should be used. However, currently, no standardized test setup is available. Nonetheless, a shear test using a veneered crown to measure the strength of metal-ceramic restorations¹⁷)

present investigation. At this juncture, it must also be clarified that there are some weak points in the test design. For example, strength is measured by static loading; the test is not performed in a humid environment; and the material properties of the metal die are different from dentin. Despite these weak points, the test is close to clinical conditions. As for the design of the samples, it mimics a real clinical situation. The restoration is cemented, and a force transmission — which simulates the loading of an upper canine by its antagonist — is chosen. With such a test design, the materials investigated can at least be classified in a relative order, especially when an approved metalceramic system is used as a control. In addition, it seemed that the shear test applied in this investigation was a sensitive test because the differences in thermal properties resulted in slight differences in fracture load values.

It could be stated that the fracture strength results in all cases exceeded the maximum bite forces, which were reported to be up to about 400 N in the molar region²¹⁾. However, zirconia and titanium crowns did not reach the strength of the gold alloy control group, whereby the differences were significant for these combinations: e.max/zirconia, Cerabien ZR/zirconia, and Vita Ti/titanium. These results suggested that certain cautiousness is recommended in the clinical application of zirconia restorations.

At this juncture, it should also be clarified that the indication for use of zirconia restorations should be kept restricted until sufficient clinical long-term results are available.

Further parameters, which might influence the overall strength of layered all-ceramic frameworks, must be considered. First, the strength of veneering ceramic itself should have an influence on the test results. Reliable strength data of the veneering ceramics used in this investigation are presently not available. In addition, the bond strength between the veneering ceramic and core material might also influence the overall strength of the samples¹⁵). In this light, the effects of both the strength of the veneering ceramic and its bond strength to zirconia are the subject of a current research. The results obtained will be presented in the near future.

CONCLUSIONS

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

1. Glass transition temperature Tg and thermal expansion coefficient, α , of veneering ceramics for zirconia can be adjusted in a wide range as long as the product $\Delta \alpha \cdot \Delta T$ is kept between

185•10⁻⁶ and 1250•10⁻⁶ with:

- $\Delta \ \alpha = {\rm difference}$ between the thermal expansion coefficients of veneering ceramic and core material, and
- $\Delta T =$ difference between Tg of veneering ceramic and room temperature (in K).
- 2. From a technical viewpoint, it is possible to use one single veneering ceramic for both zirconia and titanium frameworks.

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