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Abstract

OBJECTIVES: The flexural strengths of veneering ceramics for zirconia were compared. METHODS: With 10 different veneering ceramics for zirconia (test group) and three different veneering ceramics for the metal-ceramic technique (control group) three-point flexural strength and biaxial flexural strength according to ISO 6872: 1995 as well as four-point flexural strength according to EN 843-1: 2005 were measured (n=10). Statistical analysis was performed with one-way ANOVA and post hoc Scheffé test (SPSS, p<0.05). RESULTS: For the test group, three-point flexural strength ranged between 77.8+/-8.7 and 106.6+/-12.5MPa without any statistically significant differences, biaxial flexural strength between 69.1+/-4.8 and 101.4+/-10.5MPa with three homogeneous groups and four-point flexural strength between 59.5+/-6.2 and 89.2+/-9.5MPa with five homogeneous groups. The control group showed three-point flexural strength values ranging from 93.3+/-13.5 to 149.4+/-20.5MPa, biaxial flexural strength values from 93.4+/-10.0 to 141.2+/-11.6MPa, and four-point flexural strength values from 82.7+/-8.5 to 116.9+/-9.8MPa. In every case, the results of the four-point flexure test were significantly lower than those obtained in the three-point flexure test. The three-point flexural strengths of the test group are similar to those of two ceramics of the control group. The flexural strength of one ceramic of the control group significantly exceeded the strengths of all other ceramics investigated. CONCLUSION: Three-point flexural strength values of veneering ceramics for zirconia are similar to those of veneering ceramics for the metal-ceramic technique. The four-point flexure test among all three tests showed highest discrimination between the different ceramic materials.

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Flexural strength of veneering ceramics for zirconia

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ABSTRACT

Objectives: The flexural strengths of veneering ceramics for zirconia were compared.

Methods: With 10 different veneering ceramics for zirconia (test group) and 3 different veneering ceramics for the metal-ceramic technique (control group) 3-point flexural strength and biaxial flexural strength according to ISO 6872:1995 as well as 4-point flexural strength according to EN 843-1:2005 were measured (n=10). Statistical analysis was performed with one-way ANOVA and post-hoc Scheffé test (SPSS, $p < 0.05$).

Results: For the test group 3-point flexural strength ranged between 77.8±8.7MPa and 106.6±12.5MPa without any statistically significant differences, biaxial flexural strength between 69.1±4.8MPa and 101.4±10.5MPa with 3 homogeneous groups, and 4-point flexural strength between 59.5±6.2MPa and 89.2±9.5MPa with 5 homogeneous groups. The control group showed 3-point flexural strength values ranging from 93.3±13.5MPa to 149.4±20.5MPa, biaxial flexural strength values from 93.4±10.0MPa to 141.2±11.6MPa, and 4-point flexural strength values from 82.7±8.5MPa to 116.9±9.8MPa. In every case the results of the 4-point flexure test were significantly lower than those obtained in the 3-point flexure test. The 3-point flexural strengths of the test group are similar to those of 2 ceramics of the control group. The flexural strength of 1 ceramic of the control group significantly exceeded the strengths of all other ceramics investigated.

Conclusion: 3-point flexural strength values of veneering ceramics for zirconia are similar to those of veneering ceramics for the metal-ceramic technique. The 4-point flexure test among all 3 tests showed highest discrimination between the different ceramic materials.

INTRODUCTION

Yttria-stabilized zirconia (Y-TZP) provides a sufficient mechanical strength to be used in frameworks for all-ceramic fixed partial dentures[1,2]. For esthetical reasons these frameworks have to be veneered with an appropriate veneering ceramic. In clinical application the veneering ceramic revealed to be the weakest link in such reconstructions[3-5]. Chipping of the veneer is described to be the most frequent reason for failure with a failure rate of 15.2% after a service time of 35.1±13.8 months[5].

Among other reasons failure of a veneer may be caused by insufficient bond strength[6-8], excessive tensile stress due to a thermal mismatch between veneer and framework[9], or excessive load due to premature contacts[10]. The bond strength was intensely investigated[11-14]. It revealed to be in the range of that measured with metal-ceramic systems. The tensile stress in the veneering ceramic is established during cooling after firing, when an unequal thermal contraction of both layers happens. The coefficients of thermal expansion should be adjusted in a way that during cooling a slight compression of the veneering ceramic occurs to enhance its strength[15]. In metal-ceramic systems, excessive stress to some extent may be compensated by thermal creep of the alloy, i. e. plastic flow, especially if a high gold alloy is used[16,17]. In all-ceramic systems the ceramic framework is rigid and does not yield to the stress induced by a thermal mismatch to that extent. Therefore, the risk of destructive stress formed in the veneer layer might be higher in all-ceramic systems and thus would require a high mechanical strength for veneering materials for all-ceramic systems. Hence the strength of the veneering ceramic is a crucial parameter for the clinical long-term success. For metal-ceramic restorations failure rates after 5 years, caused by chipping of the veneer are reported to be 0.4% for single crowns[18] and 2.9% for fixed partial dentures[19]. Hence, veneering ceramics for zirconia should at least show a flexural strength, which is similar to that of veneering ceramics for alloys.

Flexural strength can be measured in a 3-point flexure test, a 4-point flexure test, or a biaxial flexure test. In all cases static load is applied until failure. In the 3-point flexure test a nonuniform central stress field is created, while in the 4-point flexure test the stress field is uniform between the two loading pistons. In the biaxial flexure test, where a disk is loaded in the center, the probability of edge failures is reduced[20]. The results of the 3-point flexure test and the 4-point flexure test are correlated[21]. Lower values were found for the 4-point flexure test compared to both other tests, but the relation between 3-point flexure test and biaxial flexure test was not uniform for all ceramics investigated.

To the knowledge of the investigators no systematic investigation of the flexural strength of veneering ceramics for zirconia is available.

Aim of the present study therefore was to measure the flexural strength of a variety of commercially available veneering ceramics for zirconia to provide a comprehensive analysis of the mechanical strength of these products.

MATERIALS AND METHODS

Three-point flexural strength, four-point flexural strength and biaxial flexural strength of 10 different veneering ceramics for zirconia according to Table 1 were measured. As control 3 ceramics for the metal-ceramic technique were additionally included (Imagine Reflex, IPS d.sign, and VM13).

Specimens were prepared according to ISO 6872: 1995 (three-point and biaxial flexural strength) or DIN EN 843-1: 2005 (four-point flexural strength). Separable steel molds were used to layer the ceramic. Ceramic powder and an appropriate amount of the respective liquid were mixed to form a sticky slurry, which was filled into the mold. Excess liquid was sucked off with a tissue. Only dentin was layered. Firing of the specimens was performed in a ceramic oven (Austromat D4, Dekema, Freilassing, Germany) according to the recommendations of the manufacturers (Table 2). The specimens were placed on a tray, which was covered with a layer of silica powder. After firing, the specimens were ground to the final dimensions using SiC discs P220, P500 and P1200 according to ISO 6344-1:1998. As required by the standards the two faces of the specimens did not differ more than 0.05mm in parallelism. Ten specimens were prepared for each series. The dimensions of the samples were measured to the next 0.01mm. The specimens were placed in the appropriate sample holder and loaded in a universal testing machine (Z010, Zwick, Ulm, Germany) with a cross-head speed of 1mm/min until failure. The flexural strength was calculated as mean of the 10 results.

Statistical analysis between different test methods and between the ceramics were analyzed with one-way ANOVA, followed by a post-hoc Scheffé test (SPSS Inc., Chicago, IL, USA; $p < 0.05$).

3-point flexural strength

Specimens with a final size of 4±0.25mm in width, 1.2±0.2mm in thickness and a length of at least 20mm were produced.

The sample holder had a span between the two bearers of 15mm. Supports and loading piston were steel knife edges, rounded to a radius of 0.8mm. Load was applied at the midpoint of the specimens. The flexural strength was calculated according to the equation

$$\sigma = 3Fl/(2bh^2)$$

σ = maximum center tensile stress (MPa)
F = load at fracture (N)
l = distance of the two supports (mm)
b = width of the specimen (mm)
h = height of the specimen (mm)

4-point flexural strength

Specimens with a final size of 2.5±0.25mm in width, 2.0±0.2mm in thickness and a length of at least 25mm were used.

The sample holder had a span between the two bearers of 20mm. The distance between the two loading pistons was 10mm. Supports and both loading pistons were steel knife edges, rounded to a radius of 1.25 mm. The flexural strength was calculated according to the equation

$$\sigma = 3Fd/(2bh^2)$$

σ = maximum center tensile stress (MPa)
F = load at fracture (N)
d = difference in the distance of the two supports and the distance of the two loading pistons (mm)
b = width of the specimen (mm)
h = height of the specimen (mm)

Biaxial flexural strength

Disk-shaped specimens, 12±0.2mm in diameter and 1.2±0.2mm in height were prepared. The specimens were tested in a biaxial flexure jig with a piston on three balls design as described in the standard. The balls had a diameter of 3.2mm and were arranged in an angle of 120° to each other on a circle of 10mm in diameter. Loading at 1mm/min was applied in the center of the specimen with a 1.5mm diameter steel rod. Calculation of the biaxial flexural strength was performed with the following equation:

$$\sigma = - 0.2387 \cdot F \cdot (X - Y) / d^2$$

σ = maximum center tensile stress (MPa)
F = load at fracture (N)
$X = (1 + \nu) \ln(r_2 / r_3)^2 + [(1 - \nu) / 2] (r_2 / r_3)^2$
$Y = (1 + \nu) [\ln(r_1 / r_3)^2] + (1 - \nu) (r_2 / r_3)^2$

In which
ν = Poisson’s ratio;
r_1 = radius of support circle (mm)
r_2 = radius of loaded area (mm)
r_3 = radius of specimen (mm)
d = specimens thickness at fracture origin (mm)

Poisson’s ratio was taken as 0.25 for all ceramics according to the recommendation in the standard.

RESULTS

Means and respective standard deviations for 3-point flexural strength, 4-point flexural strength and biaxial flexural strength are shown in Table 3 and Fig. 1. For every ceramic the values of the three-point flexural strength were significantly higher than those of the four-point flexural strength. Statistical significant differences were found between 3-point flexural strength and biaxial flexural strength for the following ceramics: Cerabien ZR, Initial ZR and Vintage ZR, while significant differences between biaxial flexural strength and 4-point flexural strength occurred with Cerabien ZR, Rondo Zirconia, Lava Ceram, Triceram and Zirox and VM13. In table 3 the homogeneous groups with no statistically significant differences between the different ceramics are marked. In the 3-point flexure test the strength values of the veneering ceramics for zirconia showed no statistically significant difference (group a). In the biaxial flexure test 3 different homogeneous groups (c, d, e) of veneering ceramics for zirconia can be distinguished and in the 4-point flexure test there were found 5 different groups (g, h, j, k, l) by statistical analysis. In the 3-point flexure test the values of the veneering ceramics for zirconia were similar to those of Reflex and IPS d.sign. In the biaxial flexure test the flexure strengths of Cerabien ZR and Vintage ZR and in the 4-point flexure test the flexure strengths of Cerabien ZR, Vintage ZR, IPS e.max, Zirox, Lava Ceram and Initial ZR were significantly lower than those of the veneering ceramics for the metal-ceramic technique. The flexural strength of VM13 in every case significantly exceeded those of the other ceramics investigated.

Linear regression analysis revealed the following coefficients of determination:

3-point/4-point: $R^2 = 0.89$; $\sigma_{3\text{-pt}} = 1.24\ \sigma_{4\text{-pt}}$

3-point/biaxial: $R^2 = 0.90$; $\sigma_{3\text{-pt}} = 1.07\ \sigma_{\text{biax}}$

biaxial/4-point: $R^2 = 0.92$; $\sigma_{\text{biax}} = 1.16\ \sigma_{4\text{-pt}}$

DISCUSSION

The results of this study revealed that the 3-point flexural strength values of veneering ceramics for zirconia are in the same range as those of veneering ceramics for metal-ceramic systems. The regression analysis showed that the results of all three test methods are correlated. However, the 3-point flexure test yielded the highest values. Compared to the 4-point flexure test this difference was significant for all materials, compared to the biaxial flexure test only for 3 out of 13 ceramics. The biaxial flexure test in turn showed significantly higher values compared to the 4-point test results for 6 out of 13 ceramics. But in general it can be concluded that all three test designs provided the same relative order of the results. The 4-point flexure test provided highest discrimination between the different ceramic materials, resulting in statistically significant differences between some veneering ceramics for zirconia and the control.

Similar biaxial flexural strength results as obtained in the present investigation are reported for leucite reinforced ceramics[22-24]. IPS d.sign showed a biaxial flexural strength of 98.19±5.71MPa[24], which is comparable to the value measured in the present investigation (95.5±7.8MPa). A further investigation employed biaxial flexure test and 4-point flexure test[25]. Comparably low values for a body and an opaque ceramic for the metal-ceramic technique were found, but the relation between the results of both test methods was the same as in the present study. In another investigation it is reported that IPS d.sign had a flexural strength in the 3-point, 4-point and biaxial flexure strength test of 124.3±12.4MPa, 77.9±7.9MPa, and 114.3±13.3MPa, respectively[21]. These values are quite high compared to the present investigation. Nevertheless the authors also found a correlation between the three test methods, which was in the same order as in the present study. In a further study it is reported that the 3-point flexure strength of alumina was higher than that obtained in a biaxial flexure strength while this value was higher than the results obtained in a 4-point flexure test, which again is in accordance with the present findings[26].

The difference in the results of the three different test designs may be explained as follows. Flexural strength obtained with the 4-point flexure test is generally lower because the probability to have a surface crack between the two loading pistons is higher than in the more limited area beneath the loading piston of a 3-point flexure test. In the biaxial flexure test the force is applied in the center of the specimen. Defects at the edges, which most probably lead to an early failure, are less effective. Nevertheless the probability of a crack in the vicinity of the loading piston is higher than in the three-point flexure test because the loaded area is larger[20]. Consistent with Ban and Anusavice[25] it can be concluded that for screening tests, for instance during the development of ceramics, the biaxial flexure test is most appropriate because preparation of the samples is easy, compared to the 3- and 4-point flexure tests. But, according to the present results, when a scientific approach is intended, the 4-point flexure test should be prefered.

The fact that the strength of veneering ceramics for zirconia is in the same order as that of veneering materials for metal-ceramics could be interpreted in the sense that the strength of the veneering ceramics are not the limiting factor for the clinical long-term success of zirconia restorations. Nevertheless, compared to metal-ceramics excessive chipping is observed in clinical studies with zirconia restorations[3-5]. To explain this effect, two aspects have to be considered. One aspect is the stress, built during cooling after firing of the veneering ceramic. In metal-ceramic systems, this stress may be at least partially relaxed by an elastic or plastic deformation of the substructure[16]. Especially high-gold alloys show a low sag-resistance[17]. A zirconia substructure in contrast is rigid, which leads to higher stress formation. Hence, compared to metal-ceramics a higher flexural strength of the veneering ceramic is favorable to provide a high reliability of the veneer. The present investigation has shown that, depending on the test method and the brand, the flexural strength of veneering ceramics for zirconia is rather similar or even lower than that of veneering ceramics for the metal-ceramic technique. Therefore, the effort to improve the veneering ceramics for zirconia should be directed to the optimal adjustment of the thermal expansion and the increase of mechanical strength, which is in accordance with the appraisal of other authors. A second point is the fact that in the oral cavity water exposure may cause hydrolysis of the Si-O-Si bonds, thus affecting the mechanical properties of the ceramic. Flexural strength values are obtained at ambient laboratory conditions. The increased failure rate of veneering ceramics for zirconia under humid conditions in the oral cavity may be attributed to a different chemical composition compared to ceramics for the metal-ceramic technique, resulting in a higher susceptibility for hydrolytic attack. Further investigations are scheduled to test this hypothesis.

CONCLUSION

Within the limitations of this *in-vitro* study, the following conclusions can be drawn:

(1) 4-point flexural strength values of all materials tested were significantly lower than those obtained with the 3-point flexure test. The biaxial flexural strength in general was between the 4-point flexural strength and the 3-point flexural strength.

(2) Strength values for zirconia veneering ceramics are similar to those of veneering ceramics for the metal-ceramic technique.

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Legend to Figure

Fig. 1: Flexural strength values and standard deviations of veneering ceramics.

Tables

Table 1. Veneering ceramics used in the investigation. Veneering ceramics for the metal-ceramic technique are highlighted.

Veneering Ceramic	**Manufacturer**
Cerabien ZR	Noritake, Nagoya, Japan
Creation ZI	Metalordental, Oensingen, Switzerland
IPS e.max	Ivoclar-Vivadent, Schaan, Liechtenstein
Initial ZR	GC, Tokyo, Japan
Lava Ceram	3M Espe, Seefeld, Germany
Rondo Zirconia	Nobel Biocare, Gothenborg, Sweden
Triceram	Dentaurum, Ispringen, Germany
Vintage ZR	Shofu, Kyoto, Japan
Vita VM9	Vita, Bad Säckingen, Germany
Zirox	Wieland, Pforzheim, Germany
Reflex	Wieland, Pforzheim, Germany
IPS d.sign	Ivoclar-Vivadent, Schaan, Liechtenstein
Vita VM13	Vita, Bad Säckingen, Germany

Table 2. Firing schedules of the veneering ceramics. Vacuum was used until the final temperature was reached. Veneering ceramics for the metal-ceramic technique are highlighted.

Veneering Ceramic	**Pre Drying**		**Heating Rate (°C/min)**	**Firing Temperature (°C)**	**Holding Time (min)**
	Temperature (°C)	**Time (min)**			
CerabienZR	600	5	45	930	1
Creation ZI	450	6	45	810	1
IPS e.max	400	4	50	750	1
Initial ZR	400	6	45	780	1
LavaCeram	450	6	45	800	1
Rondo Zirconia	575	5	45	925	1
Triceram	500	6	55	760	2
Vintage ZR	650	6	45	920	1
VM9	500	6	55	910	1
Zirox	575	3	45	900	2
Reflex	575	7	75	900	2
IPS d.sign	403	6	60	869	1
VM13	500	6	55	880	1

Table 3. Flexural strength values of the veneering ceramics (mean±SD), arranged in ascending order of the values for the 4-point flexural strength. Identical letters following the values indicate homogeneous groups. Veneering ceramics for the metal-ceramic technique are highlighted.

Veneering Ceramic	**3-Point Flexural Strength (MPa)**		**Biaxial Flexural Strength (MPa)**		**4-Point Flexural Strength (MPa)**	
CerabienZR	77.8 ± 8.7	a	69.1 ± 4.8	c	59.5 ± 6.2	g
Vintage ZR	84.9 ± 11.2	a	71.3 ± 8.4	c	64.8 ± 6.3	gh
IPS e.max	85.7 ± 20.5	a	73.2 ± 10.4	cd	69.2 ± 5.1	ghj
Zirox	102.9 ± 14.7	a	95.1 ± 7.6	e	71.7 ± 4.4	ghjk
LavaCeram	90.0 ± 9.0	a	86.1 ± 7.0	cde	74.0 ± 5.9	ghjkl
Initial ZR	102.8 ± 10.2	a	87.0 ± 8.5	cde	80.2 ± 6.3	hjkl
Creation ZI	98.7 ± 17.2	a	92.3 ± 9.6	de	82.1 ± 9.1	jkl
Rondo Zirconia	99.8 ± 14.7	a	97.4 ± 14.2	e	82.6 ± 10.1	jkl
Reflex	100.5 ± 10.2	a	93.4 ± 10.0	de	82.7 ± 8.5	jkl
IPS d.sign	93.3 ± 13.5	a	95.5 ± 7.8	e	83.1 ± 5.4	jkl
Triceram	105.5 ± 12.4	a	101.4 ± 10.5	e	86.8 ± 13.4	kl
VM9	106.6 ± 12.5	a	98.9 ± 13.0	e	89.2 ± 9.5	l
VM13	149.4 ± 20.5	b	141.2 ± 11.6	f	116.9 ± 9.8	m

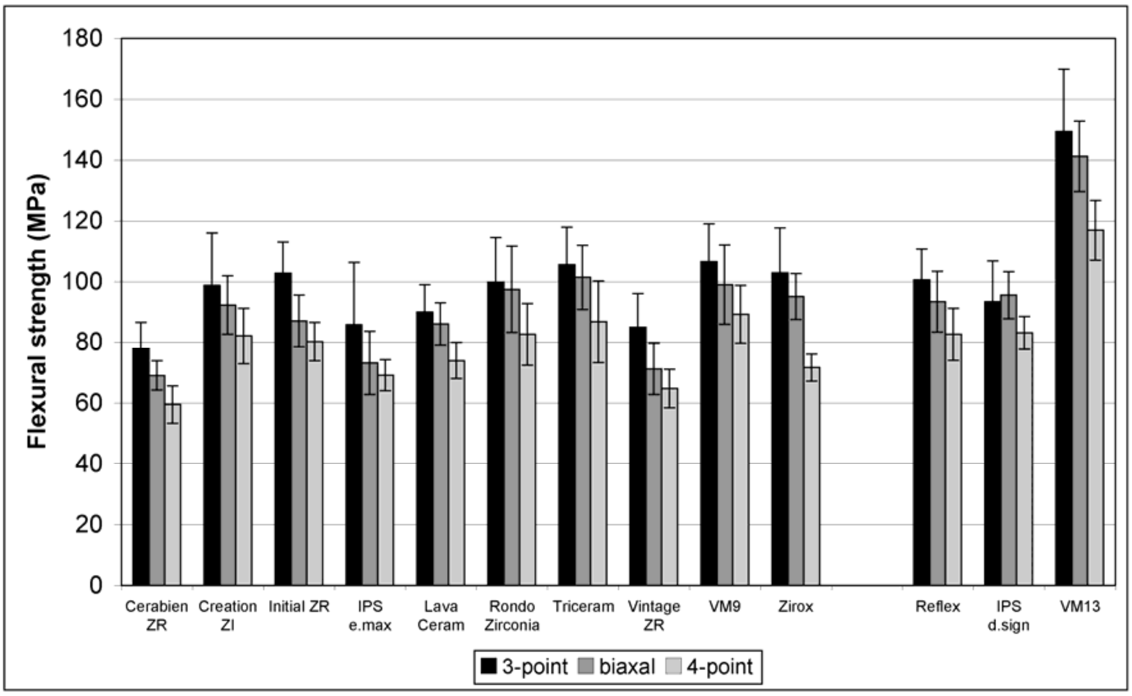

Fig. 1: Flexural strength values and standard deviations of veneering ceramics.