Strength of a feldspar ceramic according to the thickness and polymerization mode of the resin cement coating

Susana Maria SALAZAR MAROCHO¹, Renata Marques de MELO¹, Luis Guilherme Scavone MACEDO¹, Luiz Felipe VALANDRO² and Marco Antonio BOTTINO¹

¹Prosthesis and Dental Materials Department, Sao Paulo State University-UNESP, Av. Engenheiro Francisco Jose Longo 777, 12245-820, Sao Jose dos Campos, Sao Paulo, Brazil

²Dental School; Department of Restorative Dentistry, Rua Marechal Floriano, 1184, 97015-372, Santa Maria, SP, Brazil

Corresponding author, Renata Marques de MELO; E-mail: marquesdemelo@gmail.com

The aim of this study was to evaluate, *in vitro*, the biaxial flexural strength (ISO 6872) of a feldspathic ceramic (VM7, Vita Zahnfabrik) coated with a resin cement with different thicknesses and polymerization mode. Control groups consisted in VM7 with and without acid etching with 10% hydrofluoric acid for 1 min. Experimental groups comprised VM7 etched, silanated and coated with Variolink II (Ivoclar Vivadent) cement, that was polymerized through the porcelain in light-cure and dual-cure modes, with thickness $\leq 150 \mu\text{m}$ or $>150 \mu\text{m}$. The specimens ($n=15$) were subjected to the biaxial flexural strength essay and analyzed fractographically. Parametric (Dunnett, Anova 2-way, Tukey) and non-parametric tests (Weibull) were used to evaluate results. Ceramic disks coated with resin cements of either activation modes and thicknesses exhibited higher flexural strength while the Weibull moduli did not present significant differences for a confidence interval of 95%.

Keywords: Dental porcelain, Resin cement, Flexural strength, Weibull modulus

INTRODUCTION

A good adhesive bond of tooth, ceramic and resin cement is essential for the longevity of veneer restorations¹. As the fracture of dental ceramics has been associated to occlusal functional loads, static and fatigue studies on the role of the support material in resisting fracture, mainly the penetration of the cement², the cement layer thickness and elastic modulus^{2,4,5} have arisen.

The ceramic surface modification, to improve interaction with cements was first proposed by Horn⁶, who suggested the use of hydrofluoric acid (HF) on ceramic veneers. Later, Bertolotti⁷ *et al.* showed that the acid etching in glass ceramics could create a rough surface, similar to that on the tooth enamel. In low concentration, the hydrofluoric acid dissolves mainly the crystalline phase, whereas in higher concentrations it increases the etched surface area by removing the silica glassy phase. Thus, extending the existing flaws and exposing others on the ceramic surface, the acid etching with HF diminishes the ceramic strength but increases the strength of cemented specimens².

The idea of a hybrid ceramic-composite layer as the strengthening mechanism exerted by resin coatings on veneering porcelains was recently introduced by Addison *et al.*⁴. According to the authors the defects created by the roughened surfaces are penetrated by the resin, creating a region sensitive to the elastic modulus of the resin. This has been the most accepted rationale to explain the strengthening mechanism exerted by resin cements, rather than the previously reinforcement hypothesis that the cracks are healed by the cement⁸ or that stress contraction leads to crack closure⁹. In general, higher elastic moduli are associated with light-polymerization¹⁰.

Besides occlusal stresses, thermal variation and saliva¹¹, moisture from dentin may also affect the ceramic-cement interface. The water absorption causes the expansion of the polymer matrix, decreasing the strength and the elastic modulus of the cement. When water reaches the cemented surface, ceramic crowns may be distorted, resulting in stresses that favor the subcritical crack growth and fracture⁵.

The thickness of the cement also has the potential to change the restoration performance under static loading or fatigue. Very thin layers of cement caused an apparent increase of elastic modulus^{12,13}.

Ceramic strength data is generally not normally distributed about the mean because it depends mainly on the flaws distribution inside the material. More general distributions, such as the Weibull 2-parameter distribution give a better description of the strength variability. The two parameter estimates are the Weibull modulus (m) and the Weibull characteristic strength (σ_0) that respectively represent the material reliability (based on the strength data distribution) and the stress responsible for 63.21% of the sample failures^{14,15}.

It is not yet known how ceramic-cement specimens of varying cement thickness and polymerization mode behave in water. To answer this question we assessed the biaxial flexural strength (σ) and Weibull associated parameters (m and σ_0) of coated and non-coated porcelain specimens. Fractographic analysis of the specimens fractured under static loading was also performed.

MATERIALS AND METHODS

Specimen preparation

Ninety disk-shaped specimens were made out of a micro-

particulate feldspathic ceramic (Vita VM7, VITA Zahnfabrik, Bad Säckingen, Germany) and subjected to the biaxial flexural test, in wet environment, based on the guidelines of ISO 6872¹⁶⁾ (Fig. 1). The materials used in the study are listed in Table 1.

The ceramic powder and the modeling liquid (Vita Modeling fluid, Vita) were mixed and inserted under vibration in a metallic matrix 12% larger than the final

Fig. 1 Biaxial flexural strength test set-up and specimen.

specimen to compensate the ceramic contraction. The powder was condensed and the excess of liquid was removed with absorbent paper. The disks were sintered to a firing cycle (Table 2) according to the manufacturers' instructions in a vacuum furnace (Vita Vacumat 40, Vita).

After being air cooled, the disks were flattened with SiC #120, 400, 600, 800 and 1200, under coolant irrigation, at 300 rpm. Thirty disks were assigned to the control groups. Half were conditioned with Hydrofluoric acid (10% Hydrofluoric acid, Angelus, Londrina, Parana, Brazil) for 1 min (V7_{HF}, negative control) and rinsed with tap water, and the other half received no treatment (V7, positive control). The remaining groups were treated as V7_{HF}, silanated with Monobond S, allowed to dry for 20 s and coated with Variolink II (VL) in light cure (L) or dual cure (D) mode. The thickness of the cement layer was controlled through a digital micrometer (Series 369, Mitutoyo America Corporation, Aurora, Illinois, USA) having two parallel plates.

Firstly, the specimen and a mylar sheet were compressed between the plates and the micrometer was zeroed at this position. The plates were then separated, the cement was put on the specimen surface, covered with the strip and the set was compressed again until the micrometer marked 50 μ m or 100 μ m, resulting in cement layers of ≤ 150 μ m (70–150 μ m), and > 150 μ m (170–250 μ m) respectively. These thicknesses were chosen because an *in vivo* study showed that the cement thickness of ceramic crowns is around 150 μ m, depending on the crown region¹⁷⁾. The experimental groups formed were V7_{HF}/VL ≤ 150 L, V7_{HF}/VL ≤ 150 D, V7_{HF}/VL > 150 L, and V7_{HF}/VL > 150 D. The specimens' final dimension was approximately 12 mm diameter and 1.2 mm thickness.

Table 1 Commercial names, manufacturer, composition and batch numbers of the materials used in the study

Material	Manufacturer	Average Composition	Batch #
Vita VM7 (1M2 shade)	VITA Zahnfabrik, Bad Säckingen, Germany	Micro-particle feldspathic ceramic	11616
Variolink II (Yellow/Universal shade, High viscosity)	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, Triethyleneglycol dimethacrylate, Urethane dimethacrylate, Benzoylperoxide, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments	Base 46354 Catalyst 3628
Monobond S	Ivoclar Vivadent, Schaan, Liechtenstein	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	M63563

Table 2 Firing cycle of Vita VM7 according to the manufacturer's recommendation

Material	Pre-drying temp.	Pre-drying Time	Heating time	Heating temp. per min	Initial temp. under vacuum/ hold time	Vacuum time	Final temp. under vacuum
Vita VM7 Dentine	500°C	6 min	7.27 min	55°C	910°C/1 min	7.27 min	600°C

For the groups that received the coating of dual-cured resin cement, equal amounts of cement base and catalyst were dispensed and mixed. For all experimental groups, the resin cement was polymerized through the porcelain for 80 s (750 mW/cm², Demetron Optilux 501, SDS Kerr, Danbury, CT, USA). Further polymerization of 20 s was attained after the specimen was removed from the micrometer to ensure the polymerization of the cement at the center of the disk. The specimens were kept in distilled water at 37°C for 24 h before testing.

Mechanical testing

The specimens were placed onto three stainless-steel balls with diameter of 3.2 mm, equally spaced along a support circle (ISO 6872¹⁶). The device for testing was placed in the universal testing machine (Emic DL-1000, Emic, São José dos Pinhais, PR, Brazil), aligned with respect to the piston to ensure that the loading was symmetrical and concentric (Fig. 1). During the flexural test, the specimens were immersed in distilled water at a constant temperature of 37°C. The load (50 kgf) was applied at a constant speed (1 mm/min) until fracture

occurred. The coated surfaces of the experimental groups were put on the tensile side.

Using equations 1–3 according to the guidelines of ISO 6872¹⁶, the biaxial flexural strength (MPa) was calculated.

$$\sigma = 0.2387P(X-Y)/b^2 \quad (1)$$

Whereas σ is the maximum tensile stress in Pascal, P is the total load causing fracture in Newton, and b is the specimen thickness at fracture origin in millimeters. X and Y were determined as follows:

$$X = (1+\nu)\ln(r_2/r_3)^2 + [(1-\nu)/2](r_2/r_3)^2 \quad (2)$$

$$Y = (1+\nu)[1 + \ln(r_1/r_3)^2] + (1-\nu)(r_1/r_3)^2 \quad (3)$$

In which, ν is Poisson's ratio (use Poisson's ratio=0.25²¹); r_1 the radius of support circle, in mm, r_2 the radius of loaded area, in mm; r_3 is the radius of specimen, in mm; d is the specimen thickness at fracture origin, in mm.

Parametric statistics was used to compare the

Table 3 Groups, number of specimens (n), flexural strength means (σ), and statistical parameters (m and σ_0) obtained from the Weibull Distribution of the initial mechanical strength. Numbers in parentheses are standard deviations (SD)

Groups	n	σ	m	σ_0
V7	15	49.7 (5.83)	9.2 (1.7)	52.4 (1.5)
V7 _{HF}	15	47.5 (7.12)	8.0 (1.6)	50.4 (1.7)
V7 _{HF} /VL _{≤150D}	15	68.5 (11.2)	6.7 (1.3)	73.4 (2.9)
V7 _{HF} /VL _{>150D}	15	62.0 (12.7)	5.8 (1.2)	67.0 (3.1)
V7 _{HF} /VL _{≤150L}	15	55.6 (10.5)	6.8 (1.4)	59.6 (2.3)
V7 _{HF} /VL _{>150L}	15	64.6 (11.6)	6.2 (1.2)	69.5 (3.0)

Fig. 2 Topography of V7 (a) and V_{HF} (b). The polished surface presents some pores (a). The etched surface in (b) shows rough parts with grooves (white arrows) and insufficiently etched parts with smoother aspect (black arrows).

biaxial flexural strength mean values. The control group where compared to the experimental ones with Dunnett test (IC=95%). Two-way analysis of variance (ANOVA) and Tukey tests ($p < 0.05$) were conducted for all groups ($n=15$).

The strength data obtained (initial mechanical strength data) (σ) were also analyzed and described using Weibull statistics, using the following equations:

$$\ln \ln \frac{1}{1-F} = m \ln \sigma - m \ln \sigma_0 \quad (4)$$

$$F_x = \frac{x-0.5}{n} \quad (5)$$

where F_x is the failure probability of the “x” specimen after ranking the specimens in ascending order according to their strength (σ), n is the total number of specimens tested, σ_0 is the characteristic mechanical strength or scale parameter that represents the strength at which 63.21% of the specimens fail, and m is the Weibull modulus or shape parameter of the distribution of strength data as a function of failure probability. The Weibull parameters, σ_0 and m indicate the magnitude and scattering, respectively, of the measured strength data. The σ_0 and m were calculated from the y-scale intercept and the slope, respectively, of linear fittings to the strength data when plotted in a $\ln \ln(1/(1-F))$ versus $\ln(\sigma)$ graph.

Surface and fracture analysis

The fracture surfaces of the specimens were examined under the combination of optical stereomicroscope (STEMI2000-C; Carl Zeiss, Thornwood, New York, USA) and scanning electron microscope (SEM, JEOL Ltd, Tokyo, Japan) in order to identify the fracture origin and the mode of fracture. For the SEM observations, all specimens were gold-sputtered under vacuum. The fractographic analysis of several specimens was performed in order to see how the fracture of control and experimental groups differed. Stereoscope examination under various lighting configurations was performed in order to find features that give an indication of the fracture origin. The specimens were then gold sputter-coated for SEM viewing. Additional examination of the specimen surface composition was made through EDS analysis (SEM, JEOL Ltd, Tokyo, Japan).

RESULTS

The mean values (MPa) and standard deviations are displayed in Table 3. There were no significant differences between the control group means V7 (49.7 MPa) and V7_{HF} (47.5 MPa), whereas the differences between them and the experimental groups were statistically significant (Dunnett test, IC=95%). The Anova 2-way for the experimental groups revealed that the main factors (polymerization mode ($p=0.08$) and thickness ($p=0.68$) did not result in any significant differences, but the

interaction factor ($p=0.0097$) did, showing that V7_{HF}VL_{≤150D} (68.5 MPa) had higher mean strength values than V7_{HF}VL_{≤150L} (55.6 MPa).

The Weibull statistical parameters (m and σ_0) are also presented in Table 3. The Weibull modulus ranged from 6.4–13.2 (V7), 5.4–11.8 (V7_{HF}), 4.5–10.2 (V7_{HF}/VL_{≤150L}), 4.5–9.8 (V7_{HF}/VL_{≤150D}), 4.2–9.1 (V7_{HF}/VL_{>150L}), and 3.8–8.7 (V7_{HF}/VL_{>150D}). The cement coating did not compromise the overall structure of the specimens as no significant differences were observed among the groups, observed by the overlapping of the 95% confidence intervals of the Weibull modulus (m).

The compositional analysis with EDS showed that the major component elements included Si (25.11%), Ca (4.79%), Al (5.5%), Ba (2.3%), K (22.1%) and O (40.2%). The surface topography of the specimens pertaining to the control groups are showed in Fig. 2.

Stereoscope and SEM images of the fracture surfaces are showed in Fig. 3. For coated and non-coated specimens radial cracks emanating from the cementation surface (tension side) were frequent.

DISCUSSION

Recently, a study that measured the nano-hardness of the resin luting cement Variolink II polymerized in dual-cure and light-cure modes showed that the elastic modulus of the dual cure (7.4 GPa) material is not so different from the light-cure one (8.8 GPa)⁹. Based on that, the greatest strengthening would be expected for the light-cure higher modulus groups, but this was not confirmed by the observations in the current study. In fact the mean values of biaxial flexural strength of all cemented groups in the present study revealed a strengthening tendency, whereas the structure reliability was reduced as evidenced by the decrease in the Weibull modulus (m). This reduction was not significant though, as the 95% Confidence Intervals of the Weibull moduli overlapped.

The weakening effect of the acid etching on V7 was also not significant, not differing from the polished group regarding the flexural strength means and pattern of variability. The percentage of silica in the ceramic composition is high (25.11%), which makes it very sensitive to the acid reaction¹⁴. But, the nature and distribution of flaws on the porcelain surface are dependent on the HF acid etching time and concentration. While we used 10% HF for 60 s, following the manufacturer's instructions, most studies^{2-4,14} reported using HF 9.5–20% for 90 s or above.

Where the defect frequency is low, the least strengthening is observed¹⁸. Therefore, less of an etching effect on the ceramic surface may also have had an impact on the Weibull parameters of the fracture stress data of the cement coated groups. The influence of cement thickness and polymerization mode was probably biased by the lack of an efficient cement wetting, decreasing the structure reliability represented by m .

The differences between V7_{HF}VL_{50D} and V7_{HF}VL_{50L} can be understood in terms of level of cure. The degrees

Fig. 3 a-b, c-d, e-f

of conversion of light cure cements is higher than dual-cure ones. That leads to higher volume contraction that probably caused the interfacial gaps seen on the ceramic bottom surface of the light-cured specimens. The presence of discontinuities can harm the bonding quality

by exposing the silane hydrolytically unstable layer. The V7_{HF}VL_{100L} specimens also presented gaps, but the higher thickness might have impaired fast water sorption especially at the center area of the specimens. In spite of that, the Weibull data suggested that the flaws

Fig. 3 g-h, i-j

Fig. 3 Fracture surfaces of specimens after the biaxial flexural strength test: a-b (V_{7HF}); c-d ($V_{7HF}VL_{\leq 150D}$); e-f ($V_{7HF}VL_{\leq 150L}$); g-h ($V_{7HF}VL_{>150D}$) and i-j ($V_{7HF}VL_{>150L}$). a, c, e, g and i are stereoscope images. Side lighting was used to better identify the failure. Hackles are therefore highlighted by a change in shading at the fracture surface. b, d, f, h and j are SEM secondary electron images. Note several wake hackles are visible starting from pores within the ceramic specimen. Black arrows indicate the direction of crack propagation (dcp) goes from bottom to top. Small squares in Figure 3d and 3j show a detailed view of the site of fracture initiation. 3c and 3i show stereoscope analysis of gold-coated fracture surfaces. Tension surfaces are at the bottom.

distributions were not affected by the cement thickness and mode of cure.

Round defects on the ceramic surface coated or not with cement were more likely the points of fracture initiation, located at the tension surface of the specimens⁵. In all SEM images presented in this study, fracture patterns and fracture surface markings such as hackles¹⁹ from pores trapped during the ceramic condensation were recognizable, and provide a wealth of clear interpretable information. Mainly, maps of crack propagation direction are constructed, through observations of the wake hackle markings which are considered helpful indicators of the direction of crack propagation²⁰. In the present study, those markings emanated from pores and, based on the former

information, were selected as reference for determination of the location of fracture origin in the specimens, as displayed in Fig. 3. Therefore, the fracture process could be explained with respect to the crack's origin and propagation.

For some coated surfaces the fracture features were richer, meaning that greater stresses were necessary to fracture them. The cracks propagated in a radial manner from the ceramic bottom surface to the top and, for the coated specimens, they extended to the cement layer.

We have made comparisons between uncoated and cement coated dental ceramic bi-axial failure stress data using the mono-layered solution established by ISO 6872¹⁶. Addison and Fleming²¹ pondered that bi-layered solutions that take into account elastic constants should

be used instead, especially in specimens having cement thickness higher than 40 μm . The authors also stated that the symmetry of the distribution for coated specimens is the same when mono-layered or bi-layered solutions are used. Thus, although the ISO solution works well for either coated or uncoated specimens, one limitation of the present study was applying just one solution to compare both types. Further studies with the recommended solutions in concert with fractographic analysis are needed to validate our findings.

CONCLUSIONS

Within the limitations of the present study, the following conclusions are possible:

1. Etching the ceramic surface for 1 min did not weaken the ceramic.
2. Water storage for 24 h may degrade the strength of ceramics coated with light-cured cement layers thinner than 150 μm .
3. Coating the ceramics in light or dual cure modes, with thicknesses higher or lower than 150 μm , increased the fracture strength of the ceramic, but did not increase the structure reliability.
4. The fracture of coated and non-coated specimens initiated on round defects on the center of the bottom surface of the ceramics.

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