

ORIGINAL ARTICLE

The influence of the resin-based cement layer on ceramic-dentin bond strength

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Abstract

The purpose of the study was to measure the cement thickness obtained when ceramic rods were luted to dentin and to analyze the relation between cement thickness and the previously published tensile bond strength of similar test specimens. In addition, the ISO standard 4049:2019 method was used to determine the film thickness of the used cements. Zirconia (n = 100) and lithium disilicate (n = 50) rods were cemented to bovine dentin using one of five different resin-based cements. The ceramic-dentin test specimens were cut into two slices and the cement thickness was measured using a scanning electron microscope and compared to the bond strength values of similar specimens already published. The mean cement thickness recorded for ceramic rods cemented to dentin was in the range 20–40 μm, which was larger than the cement film thickness found by the ISO method. The cement film thickness determined according to ISO standard methods did not concur with the results obtained when cementing ceramic rods to dentin. For cementing ceramic restorations, a cement thickness in the range 25–35 μm seems to be favorable for the bond strength.

KEY WORDS

finite element analysis, glass ceramics, resin cements, tensile strength, zirconium oxide

INTRODUCTION

Resin-based cements are commonly used for cementing ceramic restorations [1,2]. Their major advantage is greater adhesion between cement and ceramic and between cement and dental tissue compared to water-based cements [3–5]. Resin-based cements are easy to handle, have a fast and regulated setting, and the potential for both mechanical and chemical adhesion [6,7]. Resin cements with different setting modes are available. Exclusively light-activated setting cements are used for cementing thin ceramic veneers, but for cementing restorations with a greater dimension, dual-setting cements

are preferable to increase the degree of conversion [8]. Commonly used dual-setting resin cements consist of a resin matrix, activator-initiator systems, silane coupling agent, pigments, and a variable filler content [9–11]. Dual-setting reflects the number and type of initiator: one that is activated by light and the other by a chemical substance [8,12]. RelyX Unicem is a self-adhesive dual-setting resin cement. This cement has a different chemistry compared to other resin cements and is based on glass-ionomer technology reinforced with a light-activated polymerizing resin system [13].

The dimension of the gap allowed for cement between the restoration and preparation is an important factor determining

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the success and survival of ceramic restorations. May et al. [14] recommended a pre-cement gap around 50–100 µm for resin cements and ceramic crowns. Bonding benefits were lost when cement thickness approached 450–500 µm due to polymerization shrinkage stresses [14,15]. A standard protocol for laboratory cement testing requires that the cement thickness for dual-setting resin cements should not exceed 50 µm [16].

In a previous study [17], the fracture morphologies of test specimens composed of ceramic, resin cement, and bovine dentin were studied in a light microscope to identify crack propagation in tensile testing, a recommended method to test adhesive materials [18]. The results showed a relation between the tensile bond strength and fracture morphology. The test specimens with the lowest tensile bond strength had a higher prevalence of cohesive fractures (that is, crack propagation through the cement alone). This was in accordance with results from bond strength testing by Seitz et al. [19], where the highest frequency of cohesive fractures was observed for the lowest bond strengths.

The aim of the study was to measure cement thickness of dentin-ceramic test specimens and analyze the relation between the thickness and previously published tensile bond strength of similar test specimens [17]. A null hypothesis of no relation between cement thickness and tensile bond strength was tested. In addition, the cement film thickness of the used products was measured according to ISO standard 4049:2019 to investigate if a standardized method could foresee the results obtained when cementing ceramic rods to dentin.

MATERIAL AND METHODS

Preparation of specimen

Specimens were prepared as in the previous study [17]. Cylindrical zirconia (n = 100, Dental Direct Bio ZW; Dental Direkt) and lithium disilicate (n = 50, IPS e.max CAD; Ivoclar Vivadent) rods (diameter = 5 mm) were produced to copy previously used test specimens [17].

Bovine incisors [20,21] were extracted, cut, and embedded in epoxy resin. The buccal surface was ground flat using P500 silicon carbide on Planopol (Struers) rotating grinding machine to create a minimum of 5 x 5 mm exposed dentin surface.

Surface treatment of zirconia and lithium disilicate rods

The surface treatment procedures were performed as in the previous study [17]. The zirconia rods were randomly

assigned to one of two surface treatment groups (n = 50 each group): (i) Zir-A: air borne particle abrasion by 50 µm aluminum oxide (Al₂O₃, Korox; Bego), or (ii) Zir-E: hot etching by potassium hydrogen difluoride (KHF₂) [22]. The lithium disilicate rods (LDS, n = 50) were etched with 4.5% hydrofluoric acid (HF, IPS Ceramic Etching Gel; Ivoclar Vivadent) for 20 s.

Cementation

Rods of each ceramic material were cemented to dentin using one of the five dual-setting resin cements being tested (n = 10 rods for each cement) (Table 1). Cementation was performed according to each manufacturer's instructions for use. Specimens were loaded with 8.7 N in a cementation jig during setting.

Cutting of specimens

After 24 h storage in distilled water, the test specimens were embedded in epoxy resin and mounted in a Micracut Precision (Kemet International) cutting machine. Two vertical slices of 2 mm were cut from each specimen with the ceramic rod centered (Figure 1). The slices were kept moist in closed containers. One slice of each specimen was selected for scanning electron microscope study and coated using a combination of platinum (80%) and palladium (20%).

Measurement of cement thickness

A scanning electron microscope (HITACHI SU1510 Variable Pressure SEM; Hitachi High-Tech) was used for studying the cement layer between the ceramic rod and bovine dentin. Each cement layer was imaged in back-scattered

TABLE 1 Cements used in the present study

| Cement | Manufacturer | Filler content | Reference |
|-----------------------|-------------------------|----------------|-------------------------|
| Variolink Esthetic DC | Ivoclar Vivadent | 60%–68% | Ivoclar Vivadent [25] |
| Multilink Automix | Ivoclar Vivadent | 61% | Ivoclar Vivadent [25] |
| Duo-Link | BISCO Dental | 62% | Lee et al. [9] |
| Panavia F2.0 | Kuraray Noritake Dental | 76% | Hirabayashi et al. [10] |
| RelyX Unicem | 3 M | 70% | 3 M [26] |

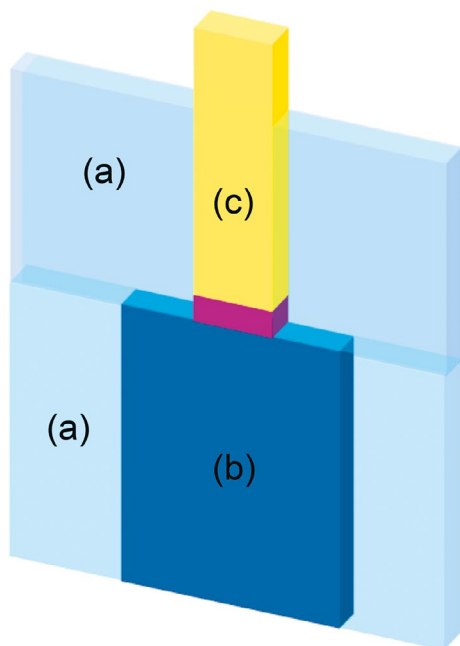


FIGURE 1 Illustration of the specimen after being cut into slices: epoxy resin (A), embedded dentin (B), ceramic rod (C) with resin cement (red)

or secondary electron mode and the thickness measured at five evenly distributed points at 300 x magnification.

ISO cement film thickness

The ISO cement film thickness was measured according to ISO 4049:2019 [16]. Cement was placed between two glass-plates and loaded with 150 N for 2 min before light-activated polymerization. ISO cement film thickness was measured using a micro-meter (Mitutoyo). The procedure was repeated five times for each cement and the median values were calculated.

Geometry of the cement layer

Measurements of the thickness of the cement in the sectioned test specimens were undertaken at five evenly spaced positions across the full width of each section. For some of the test-specimens, it appeared that the ceramic rod surface was oriented with a slight inclination angle to the dentin surface. The thickness at the central point of the cement layer (called cement thickness) and the inclination angle were both estimated for each specimen by linear regression ($\text{Thickness} = \text{constant} + \beta \cdot \text{distance of measurement}$) based on the five measuring points. In addition, the thinnest part of the cement layer was derived from the regression parameters and located at the periphery of the rods due to the inclination angle. This was called peripheral cement thickness.

Finite element analysis

Finite element analysis (FEA) was performed to examine the uncertainty in the measured strength introduced by variation in the cement thickness, and to see whether correlation between the average variation for a test specimen and the outcome of the experiments could be explained. A 1292-element and 21168-element models of a rod-cement-dentine-epoxy mounted tensile-test specimens were created in Lisa (version 8.0.0, Lisa-Finite Element Technologies) with refinement of element size down to 1.5 μm for the outer edge of the cement layer in the later model. In a half-section model (Figure 2), the components were divided into sixteen segments of 11° and four segments of 1° about the cylinder axis. To validate the test conditions, the circumference of the epoxy moulding was constrained to zero displacement along the axial direction. The origin along this axis was set at the cement-rod interface.

The three fully bonded components of a specimen (ceramic rod, dentin, and epoxy mould) were assigned the elastic tensile moduli and Poisson ratios given in Table 2. A tensile force summing to the mean force found in tensile tests for each cement was distributed uniformly over the top face of the ceramic rod. Maximum tensile stress was then evaluated for combinations of elastic modulus and Poisson ratio of the cement. The principal stresses in the nodes at and near the edges of the cement section were evaluated for cement layers with uniform thicknesses of 24, 30, 36, and 48 μm and for specimens tilted at the average angle found for that cement. The principal stresses are the three components of stress in a system of coordinates for which shear stresses are zero. They include the largest tensile and compressive stresses acting on the material at the position.

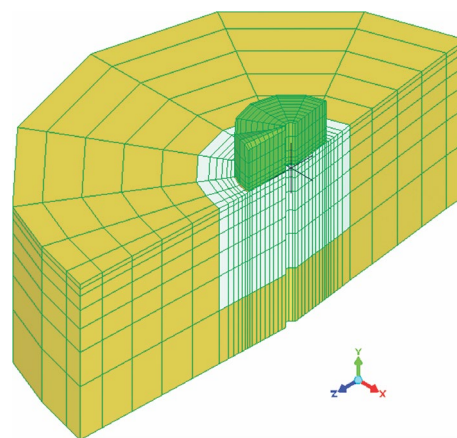


FIGURE 2 Finite element model of the tensile specimens. The components visible are: yellow, epoxy mould; white, dentin; green, ceramic rod. The cement is too thin to resolve. The black cross is at the origin of the axes with direction denoted in the inset. The apparent angle in YZ-plane is an artefact from checking the integrity of the model

The ceramic was then moved in along the axis and rotated in the face of the section to represent the measured cement thickness and its variation. The analysis was repeated for each of the fifteen ceramic-cement combinations with the average cement thickness and inclination found for each combination. Inclination angle was increased by a factor 1.57 over the measured mean value to account for randomness in the direction of the various cement thicknesses relative to the section examined by scanning electron microscopy.

Statistical calculations

The following models were chosen for the regression analysis: (i) Model 1: Cement thickness = $\alpha_0 + \alpha_1$ Ceramic + α_2 Cement + ε ; And (ii) Model 2: Peripheral cement thickness = $\beta_0 + \beta_1$ Ceramic + β_2 Cement + ε . ε = error term with random statistical noise. Regression analysis was performed using STATA version 16 (STATA Corp). The total sample size

in the study was $n = 150$ (5 cements x 3 ceramics x 10 in each group). A partial R-squared of 0.07 (Model 1) and 0.02 (Model 2) was observed for Ceramic. With a total sample size of 150 and significance level α of 5%, a power of $1 - \beta$ was 68% (Model 1) and 20% (Model 2) was reached. A partial R-squared of 0.23 (Model 1) and 0.37 (Model 2) was observed for Cement. With a total sample size of 150 and significance level α of 5%, a power of $1 - \beta$ was 99% (Model 1) and 100% (Model 2) was reached. Power calculation was performed with G*Power version 3.1.9.2. (gpower.hhu.de).

Box plots for cement thickness and peripheral cement thickness were made using ggplot package in R statistical computing (CRAN.org).

Microsoft Excel spreadsheet (Version 16.16.25, Microsoft Office 2018) was used for calculating the correlation between tensile bond strength and cement thickness, and the variation in cement thickness for each cement/ceramic combination.

RESULTS

The cement thicknesses are given in Figures 3 and 4. The mean cement thickness ranged from 20 to 40 μm . There was a tendency for thinner cement layers with zirconia specimens hot etched by KHF_2 and when using Multilink cement. A similar tendency was observed for peripheral cement thickness measurements.

Results of multiple linear regression analysis for cement thickness and peripheral cement thickness are given in Table 3. Combining all three ceramic types, a significantly thicker cement layer was found for Panavia than for the other types, except Variolink. The same was also observed for both cement thickness and peripheral cement thickness. The thinnest cement layers were observed for Multilink and Duo-Link, and for the Zir E specimens for most cements.

While most specimens showed some variation in cement thickness across the section, a difference of up to 90 μm from

TABLE 2 Elasticity data employed for each material

| Material | Elastic modulus (Isotropic, GPa) | Poisson ratio ^a | Source |
|----------------------------------|----------------------------------|----------------------------|--|
| Epoxy | 3.8 | 0.4 | Tzetzis et al. [27] |
| Dentine | 16 | 0.29 | Palamara et al. [28] Kinney et al. [29] |
| Cement | 6.6–10.4 | 0.43–0.61 | Barbon et al. [24] |
| Zirconia | >200* | 0.3 | Dental Direkt [30] |
| Lithium disilicate glass-ceramic | 95 | 0.3 | Ivoclar Vivadent [31] |

^aThe Poisson ratio is the relative amount by which a body that is stretched longitudinally decreases in a lateral dimension.

*Given the extreme difference between the elastic modulus of the cement and the materials to which it was directly bonded (zirconia and lithium disilicate glass-ceramic), the influence on the stress field in the cement was less than 2%.

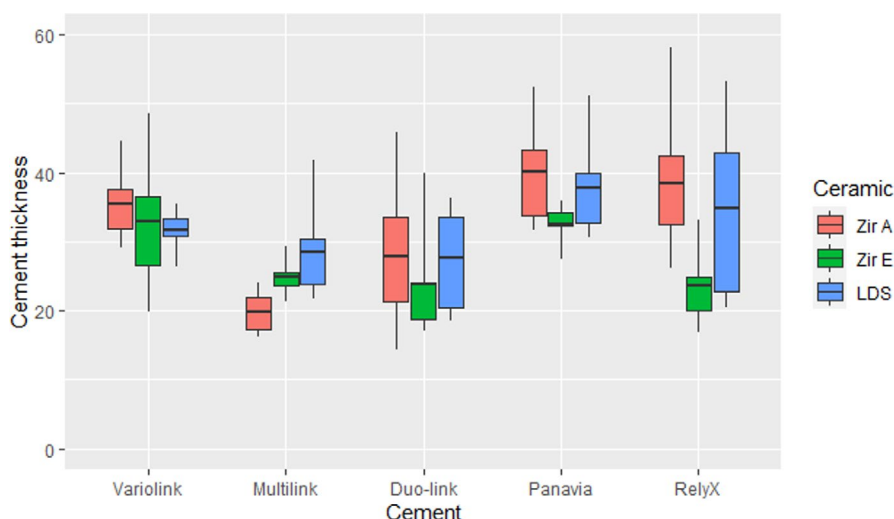


FIGURE 3 Cement thickness is defined as the thickness of the central point of the cement layer of each ceramic-cement combination. LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF_2 etched zirconia. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval

FIGURE 4 The peripheral thickness of the cement layer (called peripheral cement thickness) was derived from the regression parameters and located at the circumference of the rods. This is regarded as the thinnest cement layer. LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF₂ etched zirconia. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval

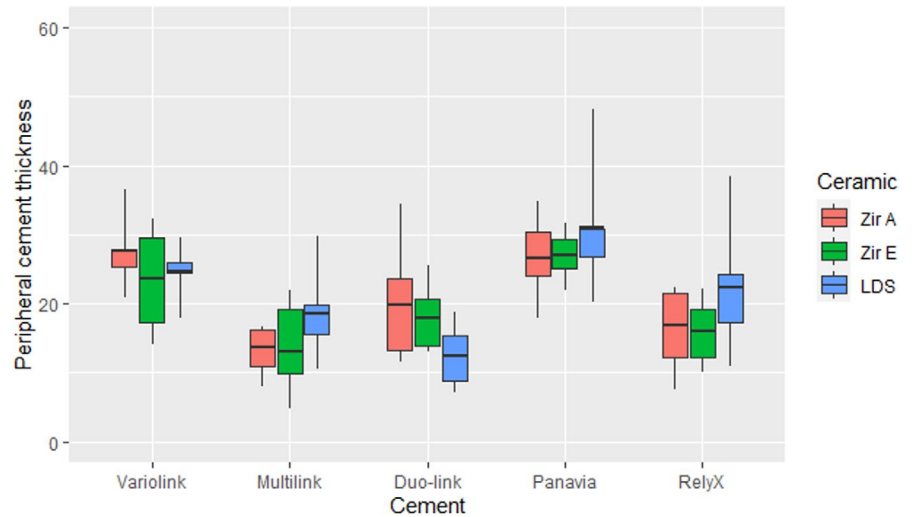


TABLE 3 Results of regression analysis of the effect of type of ceramic and type of cement on cement thickness and peripheral cement thickness, respectively. Zir A and Panavia are used as reference materials

| | Cement thickness Coefficient, (95% CI) | Peripheral cement thickness Coefficient, (95% CI) |
|------------------------|--|---|
| Ceramic | | |
| Zir A (reference) | 0 | 0 |
| Zir E | -4.83 (-8.15, -1.50) | -1.36 (-3.76, 1.04) |
| LDS | -0.30 (-3.81, 3.21) | 0.95 (-1.93, 3.83) |
| Cement | | |
| Panavia (reference) | 0 | 0 |
| Variolink | -3.46 (-7.01, 0.09) | -2.91 (-6.24, 0.41) |
| Multilink | -12.32 (-15.70, -8.93) | -13.05 (-16.40, -9.70) |
| Duo-link | -10.34 (-14.46, -6.21) | -11.47 (-15.18, -7.76) |
| RelyX | -4.50 (-9.25, -0.26) | -9.78 (-13.43, -6.14) |
| Constant | 38.50 (35.21, 41.80) | 28.33 (25.42, 31.24) |
| R-squared | 0.27 | 0.38 |
| Number of observations | 150 | 150 |

Abbreviations: Zir A, air borne particle abraded zirconia; Zir E, KHF₂ etched zirconia; LDS, hydrofluoric acid etched lithium disilicate.

one side to the other was found in several specimens. This corresponds to an inclination angle of 1.1 degree between the axis of the ceramic rod and the right angle to the dentin surface. However, the most observations of the inclination angle ranged between -0.3 and +0.3 degree (Figure 5).

The cement thickness measurements were compared to previously published data on tensile bond strength (Table 4) [17] using test specimens with an identical design and are presented in Figure 6.

Test groups with cement thickness of 25–35 μm, especially Multilink and Variolink, appeared to have the highest tensile bond strength, although this observation was not statistically significant. For test specimens with cohesive fractures in cement or combined fractures after tensile testing, a negative correlation (-0.5) with cement thickness was observed (data not shown).

The finite element analyses indicated that the tensile stress in the cement was concentrated at the periphery of the cement layer (Figure 7).

In specimens with varying cement thickness, the maximum computed tensile stress was at the thinnest edge of the cement layer. The analysis showed that the radial and lateral (hoop) stress components were large and tensile, regardless of the elastic modulus and Poisson ratio chosen for the cement. It was noted that the values obtained for the coarse 1292-element and the refined 21,168-element models agreed to within 10%.

All cements fulfilled the ISO requirement for cement film thickness; however, the largest cement thickness was observed for Variolink (Table 5).

DISCUSSION

The aim of the study was to measure cement thickness of dentin-ceramic test specimens and analyze the relation between the thickness and previously published tensile bond strength of similar test specimens [17]. A null hypothesis of no relation between cement thickness and tensile bond strength was tested and accepted. In addition, the cement film thickness of the used products was measured according to ISO standard 4049:2019 to investigate if a standardized

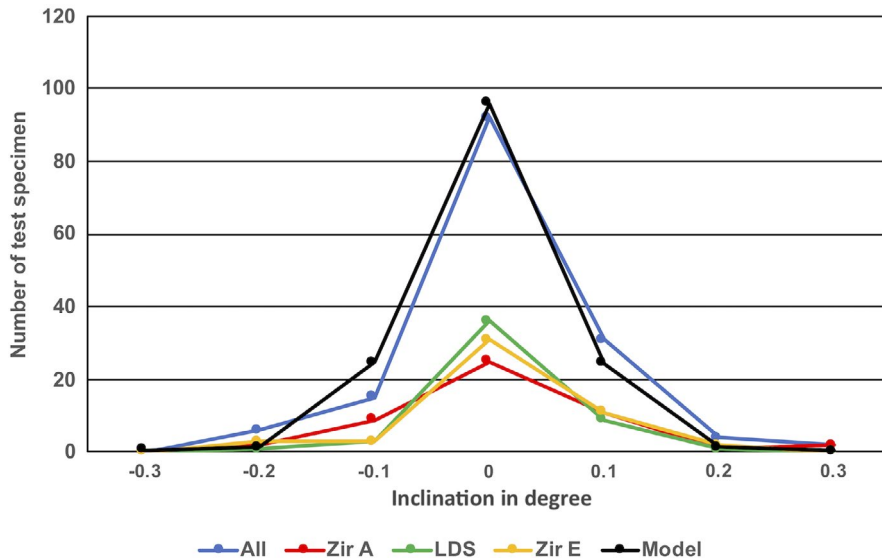


FIGURE 5 Distribution of the observed variation in cement thickness. The y-axis shows number of observations and the x-axis shows the degree of inclination for all ceramics combined (All) compared to a best-fitting normal distribution (Model) with standard deviation 0.28° . LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF₂ etched zirconia

TABLE 4 Mean tensile bond strength and standard deviation in MPa for ceramic rods cemented to dentin using five dual-setting resin-based cements, data taken from Sagen et al [17] with permission

| Cement | Zir A | Zir E | LDS |
|-----------|------------|------------|------------|
| Variolink | 14.6 (3.7) | 8.8 (2.6) | 11.4 (2.6) |
| Multilink | 13.3 (3.1) | 11.6 (3.4) | 7.1 (2.8) |
| Duo-Link | 6.4 (2.5) | 7.0 (1.5) | 4.9 (1.6) |
| Panavia | 5.2 (1.4) | 4.2 (2.2) | 3.6 (1.3) |
| RelyX | 8.6 (1.5) | 10.0 (2.2) | 9.5 (1.4) |

Abbreviations: LDS, hydrofluoric acid etched lithium disilicate; Zir A, air borne particle abraded zirconia; Zir E, KHF₂ etched zirconia.

method could foresee the results obtained when cementing ceramic rods to dentin.

Most of the observed failures in tensile testing were cohesive fractures in the cement, suggesting that the cement was the weakest link in the bonding of ceramics [17].

For each test unit, the cement thickness was measured on five evenly distributed points, giving 150 cement layer values for each cement. Linear regression analysis revealed that the cement thickness varied in all groups of test specimens. The inclination angle of the rod with respect to the dentine surface observed in this study raised questions as to whether this would be due to an asymmetry in the apparatus used to load the rod during setting of the cement. If such an asymmetry existed and was greater than any randomly oriented inclination angle generated, for example, by uneven but random application or hardening of the cement, the scatter plot of inclination angle values (Figure 5) would follow a cosine distribution falling to zero for an inclination angle imposed by the apparatus. Otherwise, the scatter plot would follow a normal distribution. Taken over all 150 measurements, the inclination angle was well described by a normal (Gaussian) distribution with a mean of zero and a standard deviation of

0.28° , although values up to 1.1° were observed. Any inclination angle due to a systematic misalignment of the apparatus is no greater than 0.1° .

The thickest cement layer was found for Panavia. The differences were significant when comparing Panavia to the other cements, except for Variolink. Panavia also had the lowest tensile bond strength in the previous published study [17]. The cement thickness was significantly lower when specimens were cemented to Zir E than to Zir A. This could be related to the smoother surface of Zir E, as shown by Sagen et al. [17].

The high number of cohesive fractures reported for Panavia in the former study [17] indicated that the cement was the weakest part of the test unit. Resin-based cements contain filler particles and the filler content of Panavia is 76% [10] which is the highest among the tested cements (Table 1). The high filler content is not likely to explain the inferior results because high inorganic filler contents (>75 wt%) have been associated with the favorable mechanical properties of resin-based composite material [11]. Still, an explanation might be related to the size and surface of particles. Lack of adhesion between the resin matrix and the particles, incomplete wetting of the surface of the particles, or unevenly distributed particles in the matrix may reduce the strength. The base and catalyst of Panavia are deposited on a mixing pad and mixed by hand for 20 s. Compared to auto-mixed cements, Panavia might have a greater risk of an inhomogeneous mixture, which affects both laboratory testing and clinical performance [7].

Properties of the cement, including viscosity, particle size, and applied force during cementation, influence the thickness of the cement. The peripheral cement thickness was on the average $10 \mu\text{m}$ smaller than the cement thickness (Figures 3 and 4) and represented the thinnest cement layer obtained when cementing ceramic rods to dentin. Determination of the

FIGURE 6 Cement thickness measured in the present study plotted against tensile force at break reported by Sagen et al. [17]. Points with same color represent results from ceramic rods with different surface treatment (LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF₂ etched zirconia)

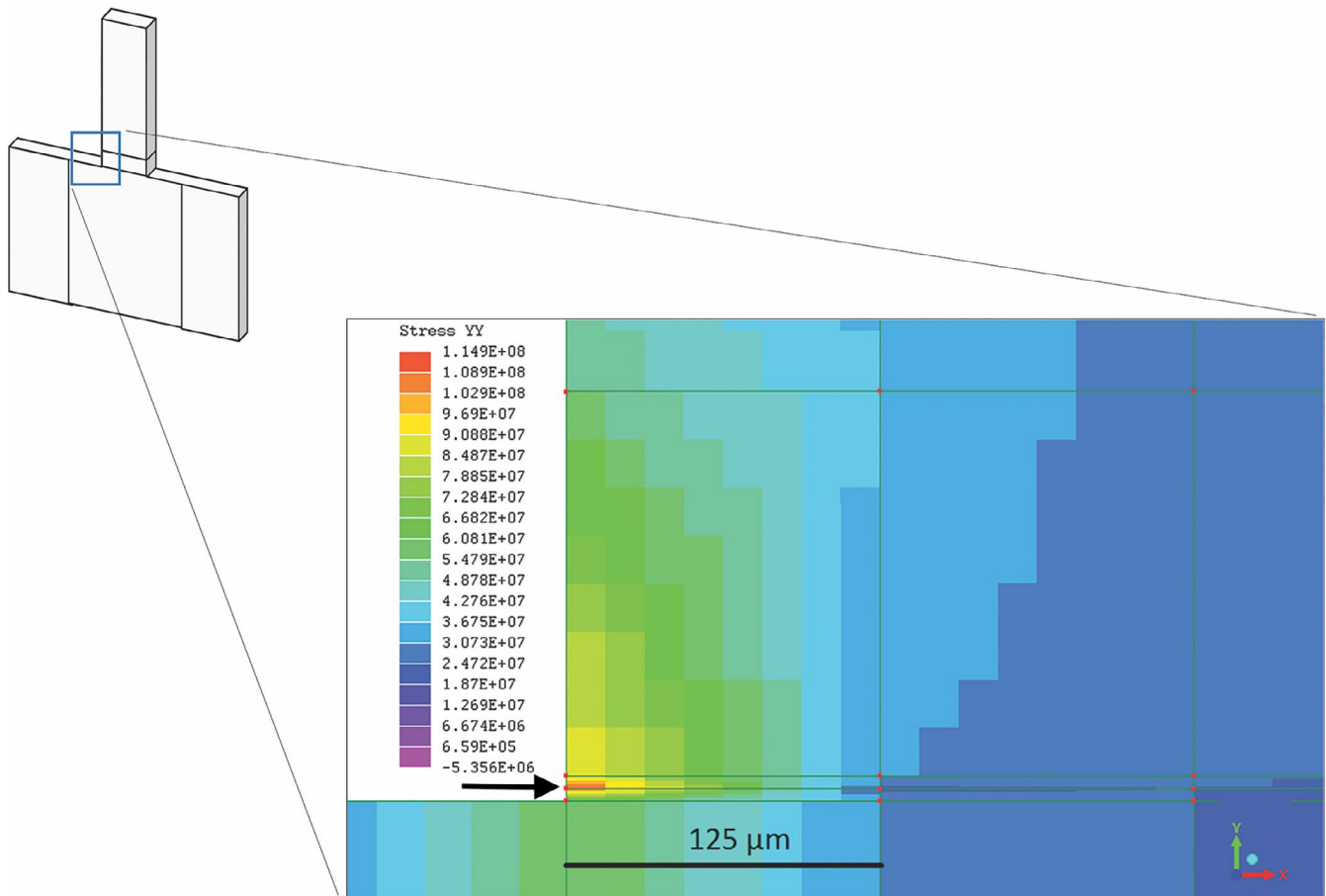
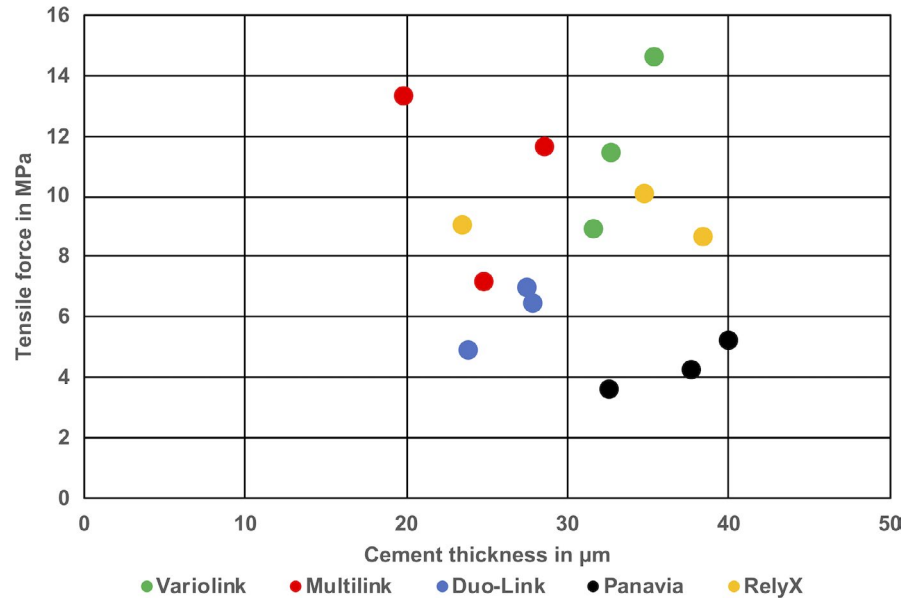


FIGURE 7 Presentation of finite element analysis of stress concentration. Detail of cement (two thin layers of elements extending across the image, black arrow), ceramic rod (above the cement layer), and dentin (below the cement) showing the location of the maximum stress. Color scale for stress (Pa) from violet indicating lowest value to red (highest value) in the vertical Y- direction

ISO cement film thickness should theoretically indicate the minimum expected cement thickness when cementing restorative material to dentin. This was not the case in the present study, except for one of the cements. The reasons for this

discrepancy could be differences in the applied load during polymerization and the fact that the cementing situation includes the use of bonding agents and primers that contribute to the thickness of the cement. Another explanation could

| Measurement | Variolink | Multilink | Duo-Link | Panavia | RelyX |
|-------------|-----------|-----------|----------|---------|-------|
| 1 | 12 | 8 | 12 | 6 | 7 |
| 2 | 11 | 3 | 4 | 11 | 5 |
| 3 | 21 | 6 | 1 | 5 | 19 |
| 4 | 21 | 7 | 4 | 4 | 5 |
| 5 | 22 | 7 | 5 | 10 | 6 |
| Median | 21 | 7 | 4 | 6 | 6 |

TABLE 5 ISO Cement film thickness in μm measured according to ISO 4049:2019 [16]

relate to the roughness of the cemented surfaces. The mean surface roughness of glass is much lower than that of the ceramic rods that were measured by Sagen et al. [17]. This may explain the differences observed between ISO cement film thickness and the measured cement thickness.

The mean cement thickness was in the range 20 to 40 μm (Figure 3). When comparing cement thickness with previously published data on tensile bond strength [17], there was a trend for a decrease in tensile bond when the cement thickness exceeded 35 μm (Figure 6). A study on the fracture strength of glass ceramic published by Rojpaibool and Leevailoj [23] showed a significant relation between high fracture load and a thinner cement layer. This indicates that a thin resin-based cement layer is favorable.

The finite element analysis (FEA) indicated that the site of greatest concentration of tensile stress was at the periphery of the cement layer, where it was thinnest because of the inclination of the ceramic rod. In line with this, Barbon et al. [24] observed that fractures started at the border of the specimens in micro tensile bond strength testing. For the ceramic-dentin test specimens, the three principal stresses at the circumference of the cement are all tensile (Figure 7), a situation that is much more likely to initiate fracture in a brittle material (such as hardened resin-composite) even when it exhibits some plasticity. Fractures often initiate in structural flaws in the cement layer. This could be due to the high filler content of Duo-Link and Panavia [9,10], as discussed earlier; the high number of cohesive fractures in the same cements [17] further substantiates that, at least for these cements, the cement was the weakest link in the test unit. Barbon et al. [24] showed that an increase in filler particle content resulted in a more viscous and stiffer resin-based cement and an increase in mixed and cohesive failures were observed. The particle content of RelyX is like that of Duo-Link and Panavia (Table 1). However, these particles are part of the matrix due to the glass ionomer similarity of RelyX [13] and they act differently from the fillers of Duo-Link and Panavia, as they are embedded in the resin matrix. Other possible contributions could be poor polymerization deep within the cement layer [8] and skewed coupling in the test machine so that there is a greater bending moment on the cement [18]. It was found that cement thickness in the range 25–35 μm could be related to the highest tensile bond strength of ceramic rods cemented to dentin. The ISO standard

4049:2019 sets the requirement for cement film thickness at a maximum of 50 μm . Most measurements of the cement thickness of the ceramic-dentin specimens were far below this value, showing that the ISO requirements were reasonable. However, the measurements of the peripheral cement thickness (Figure 4) obtained in cemented test specimens did not concur with the results of the ISO cement film thickness measurements (Table 5). This indicates that the ISO test method does not directly reflect a clinically relevant cementation procedure, especially when it comes to the applied load during setting.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

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