The apparent increase of the Young’s modulus in thin cement layers

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Summary Objectives. The bond of adhesive luting cements to the tooth tissues and restorative materials is expected to hinder their transverse contraction for the layer thickness applied in dental restorations. It was hypothesized that the hindering of the transverse deformation will influence the relation between stress and strain (the stiffness) in the direction perpendicular to the substrate surface. The aim of this study was to investigate the relation between cement layers with different ratio between bonded and free surface (C-factor) and the stiffness of these layers, i.e. an apparent increase of the Young’s modulus of the dental luting cement.

Methods. A commercial luting cement RelyX ARC (3M, St Paul, MN, USA) was used in this study. The ‘real’ Young’s modulus and the Poisson ratio were determined and these values were used in models with layers with different C-factors (0.5, 1.0, 2.0, 3.0, 4.0, 5.0 and 6.0) in a three-dimensional Finite Element Analysis program (FEMAP, E.S.P., Maryland Height, MO, USA). The apparent Young’s modulus was measured for layers with thicknesses of 0.5 mm (C = 6.0) and 6.0 mm (C = 0.5) and compared with the results of the Finite Element Analysis (FEA) analysis.

Results. The apparent Young’s modulus in the 0.5 mm layer was 20% higher than the apparent Young’s modulus in the 6.0 mm layer. This result was confirmed by the results of the F.E. analysis. For very thin layers the stiffness will be 25% higher than the Young’s modulus.

Significance. The hindering of the transverse contraction has to be taken into account studying the mechanical properties of dental luting cements because it influences the behavior of these luting cements in thin layers.

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Introduction

Despite the increased effort to prevent dental decay, many patients are still in need of prosthetic reconstructions. Analyses of failure of fixed restorations have shown caries to be their most frequent cause. Nevertheless, technically, its retention is the ‘Achilles heel’ of fixed prosthetic work. To fix the restoration a luting cement is used, which has to fill and seal the gap between tooth and restoration. Besides retention, a good seal of the tooth tissues is recognized as an important factor for the longevity of fixed dental restorations.

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cements, which show good adhesive properties, are becoming increasingly popular. Resin composite cements undergo polymerization contraction during setting\(^1,4\) and as a consequence of the adhesion to the restoration and the preparation, setting stresses arise in the cement.\(^5\) After adhesion is obtained its maintenance becomes an issue. It is not only that the developing setting polymerization stresses cannot exceed the bond strength or the cohesive strength of the materials involved but also the stressed cement has to withstand functional loading forces. As functional loading forces are unavoidable one should aim for low polymerization contraction stress development. Alster et al.\(^6,7\) studied the tensile strength and the polymerization contraction stress of thin resin composite layers. With decreasing layer thickness (increasing C-factor) the setting stress developed increases. Feilzer et al.\(^8\) even found spontaneous cohesive failure of resin-based restoratives tested at higher C-factors (C > 2.5) in a laboratory setup. Apparently, thin resin cement layers show different behavior compared with thicker resin-based restorative layers. However, the phenomenon of spontaneous cohesive failure was not found by others.\(^9\)

This may be explained by the compliance of the test setup used in these studies, as the influence of compliance on thin cement layers is relatively high.

In these studies a laboratory setup was used with a simple geometry existing of two parallel discs cemented together with a resin composite restorative or cement. For the prediction of the magnitude of these stresses clinically FEA may be a helpful tool. However, for a proper FEA, key properties such as Young’s modulus, yield point, and viscosity as a function of setting time, should be known. Braem et al.\(^10\) and Dauvillier et al.\(^11\) studied the development of Young’s modulus of resin composites during setting. A more simple approach is to calculate the relation between normal stress and strain with Hooke’s law,\(^12\) where the strain in the Z-direction in a triaxial stress state can be described as (Fig. 1)

\[
\varepsilon_z = \frac{\sigma_z}{E} - \nu \frac{\sigma_x}{E} - \nu \frac{\sigma_y}{E},
\]

or

\[
E = \frac{1}{\varepsilon_z} [\sigma_z - \nu (\sigma_x + \sigma_y)],
\]

where \(\nu\) is the Poisson ratio, \(\varepsilon\) is the elastic strain and \(E\) the Young’s modulus.

By uniaxial loading \(\sigma_x\) and \(\sigma_y\) are zero. The Young’s modulus can than be calculated with Hooke’s law \(E = \Delta \sigma_z/\Delta \varepsilon_z\); this equation is also used in most dental studies to calculate stresses in dental cements.

For thin dental luting cement layers, however, \(\sigma_x\) and \(\sigma_y\) cannot be considered zero. As a consequence of the adhesion of the cement layer to the restoration and the preparation an amount of strain will cause more stress due to the hindering of the transverse contraction in comparison with a not-hindered situation.

Therefore the ‘simple’ use of Hooke’s law in thin cement layers leads to an apparent increase of the Young’s modulus. No literature describes the apparent increase of the Young’s modulus for dental luting cements applied in thin layers.

Clearly the simple use of Hooke’s law for the calculation of stresses in thin cement layers will lead to an underestimation of stresses. This study aimed to investigate the relation between C-factor and stiffness of a luting cement layer by comparing laboratory test results with the results obtained by FEA.

**Materials and methods**

**Materials**

The material used was dual-curing luting cement RelyX ARC (batch AEAE, 3M, St Paul, MN, USA) a two-paste BisGMA-TEGDMA-based resin composite. According to the supplier Zirconia/silica filler is used to impart radiopacity, wear resistance and strength. Filler loading of the mixed cement is approximately 67.5% by mass. The average particle size for the filler is approximately 1.5 \(\mu\)m. The cement was handled and mixed according to the manufacturer’s instructions, no light cure was used.
General

To gather data for stiffness calculations, bonded luting cement discs (Ø6.0 mm) with thickness of 0.5 mm (C = 6.0, n = 5) and 6.0 mm (C = 0.5, n = 5) were, during their setting, exposed to stress/strain cycles in a universal testing machine. The 6.0 mm thick samples were considered as free of lateral constraint and were used to calculate the ‘real’ Young’s modulus and the Poisson ratio. These data were used in seven FEA models, with layers with C-factors of 0.5, 1.0, 2.0, 3.0, 4.0, 5.0, and 6.0, respectively. With the strain values caused by a fixed induced stress the apparent increase of the Young’s modulus was calculated for each model. The model was validated by comparing the experimentally found apparent increase of the Young’s modulus at C-factors 6.0 and 0.5.

Test setup

In a test setup in an automated universal testing machine (Hounsfield, Perrywood Business Park, Salfords, Redhill Surrey, UK) as described in detail by Alster et al.6 (Fig. 2) the luting cement was cured.

The surfaces of two steel cylinders (Ø6 mm) one connected to the moving crosshead with the load cell and one to the fixed base of the tensiometer were coated with a thin silica layer and subsequently silanized (Silicoater, Kulzer GmbH, Wehrheim, Germany) to ensure optimal bonding between steel cylinder and the freshly mixed samples inserted between the two cylinders.

Steel was chosen because the hindering of the transverse contraction with steel substrates will be close to the maximum.

Any axial displacement of the adhesive surfaces was determined with the aid an extensometer (LVDT type1304K, Millitron, Feinprüf Perthen GmbH, Göttingen, Germany) consisting of two inductive probes that were fixed as close as possible to these surfaces. In this way any possible measuring error caused by canting of the probe holder or the contact plane holder was excluded.

A data-acquisition console (Instrumat, Raamsdonksveer, The Netherlands) collected the data (time, load and displacement) simultaneously during the experiment at a sampling rate of 10/s.

The change in diameter under stress was registered with the aid of another extensometer (LVDT type 1318, Millitron, Feinprüf Perthen GmbH, Göttingen, Germany) consisting of two inductive probes fixed in a device, which was clamped to the cylinder in order to ensure the measurement of the diameter always at the same place halfway the cured composite cylinder.

Apparent increase of the Young’s modulus

To gather data for the calculation of the development of the Young’s modulus during the setting of the material and to establish the final value periodically, stress-strain cycles were performed. The samples were cured in the hindered condition (strain = 0) and every 200 s the obtained load was reduced to zero in 15 s, held at zero for 30 s, and in 15 s the condition of zero strain was reinstalled. To facilitate the recording of material response and the stress-strain cycles, the gearbox of the testing machine was modified to eliminate any play when the upward or downward motion of the crosshead was reversed and to enable extremely low crosshead speeds.

Determination of the Poisson-ratio

To obtain data for the calculation of the Poisson ratio stress-strain cycles (σ = 0 MPa – σ = 14.06 MPa) were performed on fully cured 6.0 mm samples at a crosshead speed of 200 μm/s during which the diameter and the change in length was registered continuously.

Compliance of the test setup

In this test setup two inductive probes were fixed as close as possible to the adhesive surfaces enabling, in the hindered condition, automatic compensation of any axial displacement of the adhesive surface,
to maintain the original sample height. Nevertheless, a small amount of compliance was inevitable, which may have a relatively large influence on the setting stress development in very thin cement layers.

In order to determine the compliance of the test setup the two steel cylinders were made out of one piece and the test setup was loaded with a cross-head speed of 200 µm/s in order to find the compliance of the test setup for every load registered. All displacement values measured were corrected for the compliance of the test setup.

All experiments were repeated five times and performed at room temperature (22 ± 1 °C).

**Finite element modeling**

The finite element modeling and post-processing were with FEMAP software (FEMAP 8.10, ESP, Maryland Height, MO, USA) while the analysis was done with CAEFEM software (CAEFEM 7.3, CAC, West Hills, CA, USA). The models consisted of two steel cylinders (L = 5 mm, Ø 6 mm) with composite layers in between with thicknesses, respectively, of 0.5, 0.6, 0.75, 1, 1.5, 3.0 and 6.0 mm. Each model consisted of approximately 25,000 brick solid elements. The distribution of the elements in the axial direction was chosen in such a way that the composite layer was three elements high for the 0.5, 0.6, 0.75, 1 and 1.5 mm layer, four elements high for the 3.0 mm layer and eight elements high for the 6.0 mm layer. The Young’s modulus and Poisson ratio used were for the steel, respectively, 190 GPa and 0.3413 and for the composite 4.738 GPa and 0.27. The materials were assumed to be homogeneous, linearly elastic and isotropic. All nodes in the bottom plane of the lower cylinder were assumed to be fixed, no translation or rotation was allowed in any direction. All the nodes in the top plane of the upper cylinder were uniformly loaded with sufficient load to introduce a stress in the Z-direction of 15 MPa in the steel cylinder.

**Calculation methods**

The normal stress was calculated by means of: $\sigma = F/A$, where $F$ is the load response and $A$ is the cross section of the sample.

The strain in the Z-direction was calculated from:

$$\varepsilon_z = (\Delta L_z - \Delta L_0)/L_0,$$  

where $\varepsilon_z$ is the elastic strain, $\Delta L_z$ is the displacement of the crosshead in the hindered situation during the cycle of the load to zero, $\Delta L_0$ is the compliance of the test setup and $L_0$ is the initial sample length.

The Young’s modulus according to the equation (2) is $E = 1/\varepsilon_z[\sigma_z - \nu(\sigma_x + \sigma_y)]$ (Fig. 1), where $\nu$ is the Poisson ratio, $\varepsilon$ is the elastic strain and $E$ the Young’s modulus.

The apparent Young’s modulus was calculated as $E_{\text{apparent}} = \Delta\sigma_z/\Delta\varepsilon_z$.

The apparent Young’s modulus ($E$) was calculated from the load/strain cycles up and down. The 6.0 mm thick samples were considered as free of lateral constraint. However, based on a trial run with FEA, the stiffness found was 3.7% higher than the Young’s modulus. To compensate for this model mismatch a 3.7% lower value of the Young's modulus was used in all FEA models.

The maximum value of the apparent Young’s modulus with the transverse shrinkage completely hindered can be calculated as follows.

The strain in the X- and Y-direction can be described as:

$$\varepsilon_x = \Delta\sigma_x/\varepsilon_x - \nu(\Delta\sigma_y)$$ (3)

$$\varepsilon_y = \Delta\sigma_y/\varepsilon_y - \nu(\Delta\sigma_x)$$ (4)

for $\varepsilon_x = 0$ and $\varepsilon_y = 0$ equation (3) and (4) can be written as:

$$\Delta\sigma_x = \nu(\Delta\sigma_y + \Delta\sigma_z)$$ (5)

$$\Delta\sigma_y = \nu(\Delta\sigma_x + \Delta\sigma_z)$$ (6)

$\Delta\sigma_x$ and $\Delta\sigma_y$ substituted in Eq. (2) gives $E_{\text{apparent}} = E_{\text{apparent}}/[1 - (2\nu^2/1 - \nu)]$, where $E_{\text{apparent}}$ is the real Young’s modulus and $\nu$ is Poisson's ratio.

The Poisson ratio was calculated from:

$$\nu = \varepsilon_{\text{rad}}/\varepsilon_z,$$  

where $\varepsilon_{\text{rad}}$ is the contraction of the diameter and $\varepsilon_z$ is the lengthening in the Z-direction.

**Results**

For the load range used in this study the stress-strain relation of the compliance of the test setup was a linear relationship. The compliance of the test setup increased with an increase of the tensile stress in the samples with 0.113 µm/MPa.

**Table 1** shows the results of the Poisson ratio determination. The axial strain and the associated radial contraction of the 6.0 mm thick sample at a load of 300 N (= 14.06 MPa) were used to calculate the Young’s modulus and the Poisson ratio.

In Fig. 3 the apparent Young’s modulus, obtained from the load/strain cycles, as a function of time is presented graphically for layers with C-factor 0.5 and 6.0 respectively. After 2h the Young’s modulus had reached the maximum value.
The stiffness of the layer with C-factor 0.5 was significantly lower ($p < 0.05$) than the stiffness of the layer with C-factor of 6.0. The load/strain cycles at the crosshead speed as tested did not show any significant visco-elastic behavior. These data did not differ significantly with the results of the FEA as presented in Table 2.

In Table 2 the apparent Young’s modulus and strain as calculated for different C-factors by FEA are presented. It can be clearly seen that the apparent Young’s modulus increases with increasing C-factor.

Fig. 4 shows the relation between the results of FEA and the test results of the apparent Young’s modulus as function of C-factor. The apparent Young’s modulus is presented here relative to the real Young’s modulus. The data found with the FEA were as expected the same as found experimentally for the 6.0 mm layer ($C = 0.5$) and did not differ significantly with the experimentally found results for the 0.5 mm layer ($C = 6$).

Discussion

This study shows clearly that a determination of the Poisson ratio can be carried out with a relatively simple test setup. In dental literature few papers are published in which measured Poisson ratio data were reported, while values for specific products are almost not available. Whiting et al.\textsuperscript{14} used an ultrasound method and found values between 0.23 and 0.32 for different dental composites. Chabrier et al.\textsuperscript{15} in a static compression test for dental composites found values between 0.40 and 0.44. The value of 0.27 for the Poisson-ratio of RelyX ARC found in this study, measured in tensile at slow loading rate, is in line with the data published by Whiting et al. The explanation of Chabrier et al. that the different loading rate situation compared to an ultrasound determination causes the difference is not confirmed by this study. The compliance of the test setup showed to have a linear relationship for the load range tested. This compliance has to be taken into account for stress–strain relations in thin layers. Even with the system in this study where compliance was minimized by mounting the two displacement transducers as close as possible to the adhesive surfaces of the test samples, a small compliance could still be determined, which will

<table>
<thead>
<tr>
<th>C-factor</th>
<th>Strain ($10^{-3}$)</th>
<th>$E_{\text{apparent}}$ (Gpa)</th>
<th>Increase (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>3.049</td>
<td>4.920</td>
<td>3.8</td>
</tr>
<tr>
<td>1.0</td>
<td>2.934</td>
<td>5.112</td>
<td>7.9</td>
</tr>
<tr>
<td>2.0</td>
<td>2.767</td>
<td>5.416</td>
<td>14.3</td>
</tr>
<tr>
<td>3.0</td>
<td>2.675</td>
<td>5.608</td>
<td>18.3</td>
</tr>
<tr>
<td>4.0</td>
<td>2.645</td>
<td>5.672</td>
<td>19.7</td>
</tr>
<tr>
<td>5.0</td>
<td>2.606</td>
<td>5.756</td>
<td>21.4</td>
</tr>
<tr>
<td>6.0</td>
<td>2.588</td>
<td>5.795</td>
<td>22.3</td>
</tr>
</tbody>
</table>

Table 2 The strain with a stress of 15 Mpa of material with Young’s modulus of 4.738 GPa and Poisson ratio of 0.27, the apparent Young’s modulus $E_{\text{apparent}}$ and the percentage of increase compared with 4.738 Gpa.

The apparent increase of the Young’s modulus in thin cement layers

Figure 3 Development of apparent Young’s modulus during the setting of the material.

![Figure 3](image-url)
have a significant influence on the stress-strain data output when testing thin layers.

Therefore, the apparent Young's modulus (Fig. 3) of the samples of layers with 0.5 and 6.0 mm thickness with a C-value of 6.0 and 0.5, respectively, are calculated with the stress and elastic strain data corrected for the compliance of the test setup. From Fig. 3 it can be seen that the Young's modulus increases most during the first 15 min, while it has nearly reached its final value after 20 min.

The apparent Young's modulus calculated for the 6.0 mm layer was lower than that for the 0.5 mm layer. From the results of the FEA (Fig. 4) it can be seen that the apparent Young's modulus increases quite rapidly with increasing C-factor and approaches a final value of approximately 1.25 times the Young's modulus in very thin layers, in line with the theoretical value for $C \gg 0$. Findings of for example Alster et al. who demonstrated a relation between layer thickness and contraction stress in thin resin composite layers were confounded by the error introduced by the apparent Young's modulus. However, for the layer thickness as used in that study the influence was relatively small.

Another way to determine a Young's modulus is by carrying out a three- or four-point bending test. These tests are not influenced by the hindered transverse contraction. The Young's modulus determined with these tests is therefore a real Young's modulus, which can be 80% of an apparent Young's modulus determined in a thin layer. The hindering of the transverse contraction has to be taken into account studying the mechanical properties of luting cements. Moreover, as a consequence, severe shear forces may develop at the adhesive interfaces of luting cements when they are loaded in tensile or compression. Stresses in the cement, like stresses due to bite forces on the cemented restoration, will increase the probability of bonding failure not only by compression forces but also by shear forces. This study indicates that FEA might be a good tool to study the real stresses occurring in the clinical situation. FEA programs calculate with the influence of the transverse contraction, while for the simple calculation of stresses using Hooke's law, one has to take the effect of neglecting the hindering of transverse stresses in the sample into account. This effect may be as large as 25% of underestimating the stresses.

It may be concluded that the hypothesis, that the hindering of the transverse deformation will influence the relation between stress and strain, the stiffness of the layer, i.e. the apparent Young's modulus of the material, in the direction perpendicular to the substrate surface in thin layers, is true.

References