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Core build-up designs

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CHAPTER 6

Shrinkage Stress and Bond Strength to Dentin for Compatible and Incompatible Combinations of Bonding Systems and Chemical and Light-cured Core Build-up Resin Composites

6.1 Abstract

Objective. Recent studies have shown that adhesives containing acidic monomers combined with composites can adversely effect the polymerization reaction producing low bond strengths. This phenomenon may also occur in making composite build-ups, jeopardizing one of the key factors for a successful core build-up restoration. The aim of this study was to investigate the shrinkage stress development and bond strength to dentin of core build-up resin composites combined with adhesives of various acidities. In addition the hypothesis was tested that light irradiation through chemical-cured composites during curing does not influence shrinkage stress or bond strength to dentin.

Materials and Methods. The chemical-cured (Clearfil Core) and light-cured (Clearfil Photo Core) core build-up resin composites were combined with two light-cured adhesives, Clearfil SE Bond (pH = 1.8) and One-Step Bond (pH = 4.3) and two dual-cured adhesives, Clearfil Photo Bond (pH = 2.5) and All-Bond 2 (pH = 6.1). Shrinkage stress development (at C = 3) and bond strength to dentin were determined for a period of 30 minutes in a universal testing machine where the opposing bonding surfaces were glass and dentin. To test the hypothesis, the combinations of the chemical-cured composites with the four bonding systems were also light irradiated for 40 seconds right at the start of curing.

Results. For all composite-adhesive combinations tested the adhesion to dentin resisted the developing polymerization shrinkage stresses. The use of adhesives showed a dramatic reduction in shrinkage stress at 30 minutes compared to the composites bonded without an adhesive. Only the chemical-cured composite combined with the light-cured adhesive SE Bond (pH = 1.8) showed for both shrinkage stress and bond strength significant lower values than the other combinations (app. 1 MPa vs. 3 MPa and app. 6 vs. 11 MPa respectively). Combined with the less acidic light-cured One-Step Bond (pH = 4.3) only shrinkage stress was significantly lower than the other combinations. The hypothesis was accepted for combinations of the chemical-cured composite with All-Bond 2 and One-Step Bond, but was not supported by combinations with Clearfil SE Bond or Clearfil Photo Bond, as a significant increase in shrinkage stress was found. The higher values found for bond strength were not significant.

Conclusions. Besides combinations of chemical-cured core build-up composites with light or dual-cured adhesives as recommended by the manufacturer, also combinations with adhesives of other manufacturers are compatible, provided that the pH is higher than approximately 4.3. Chemical-cured core build-up composites combined with light-cured adhesives with a pH lower than 1.8 lead to a significantly lower stress and bond strength compared to other combinations. Light irradiation during curing trough a combination of a chemical-cured composite and a low pH adhesive reactivates polymerization.
6.2 Introduction

Light-cured resin composites have largely superseded the use of chemical-cured composites in esthetic dental applications. They offer distinct advantages of improved storage stability, extended working time, increased degree of conversion, reduced air porosities caused by mixing, and enhanced physical properties [1-3]. However, chemical-cured composites still have important applications in contemporary restorative dentistry. The longer pre-gel phase of chemical-cured composites [4] has been adopted in the 'directed shrinkage technique' [5] for resin composite core build-up restorations. In this technique chemical-cured composites are used in bulk [6], which by their relatively slow setting rate are thought to provide flow to relieve the shrinkage stress developed during setting [7,8]. Also in areas that are not easily penetrable by light, chemical-cured resin composites are frequently used as core build-up materials or as luting agents for endodontic posts, crowns and bridges, inlays and onlays.

Another aspect is that chemical-cured materials have a more opaque, contrasting color, which allows the dentist better to identify the demarcation line of the core build-up, especially when it is located sub gingival. This is of importance, because the preparation for the crown restoration has to finish at least 1 mm beyond the core build-up finishing line to provide for an adequate zone of sound dentin for cementation. The identification of the demarcation line with the more translucent light-cured core build-up materials is a greater challenge to the clinician and often requires electro-surgery to enhance the visibility in this area.

During the last decade many different dentin bonding systems have been developed [9-11] to adhere resin composites to the tooth structure. Their reported versatility, ease of use and timesaving protocols have made dentists often willing to try the newest products to combine with the core build-up composites they commonly use in dental practice. However changing from one bonding system to another involves the risk that optimal bonding may be jeopardized, if a combination with a build-up composite is not compatible.

Besides the multi-step bonding systems where etchant, primer, and adhesive are applied in three separate steps, there are also simplified two-step bonding systems. These consist either of a combined etching-primer agent and an adhesive resin, or an etchant and a combined primer-adhesive resin. Even one-step bonding systems with the etchant, primer, and adhesive resin as a single component are now available. This large variety of bonding systems will possess great differences in chemical compositions and can easily lead to incompatibility with chemical or light-cured composites, if combinations are used other than recommended by the manufacturer.

It has been demonstrated that some simplified-step bonding systems were incompatible with chemical-cured composites [12], as no effective bonding could be achieved, while high bond
strengths were obtained with light-cured composites. This has been ascribed to the acidic properties that many of the adhesives of these simplified-step bonding systems possess, as they contain resin monomers with acidic functional groups to increase their etching capacity and/or hydrophilicity. When chemical-cured composites are brought in direct contact with these adhesives after they have been light cured, the basic tertiary amine activator in the composite surface layer becomes inactivated by an acid-base reaction with the acidic resin monomers in the unset oxygen inhibition layer covering the adhesive [13-17]. This will negatively effect the polymerization of the surface of the composite in contact with the adhesive and also the polymerization of the oxygen inhibition layer, which would normally set under acid-free conditions by virtue of the free radicals released in the chemical-cured composite. On the other hand light-cured composites will be less disturbed by the presence of acids in the inhibition layer, as placement of a light-cured composite is always followed by light initiation. Light initiation results in a considerably faster generation of free radicals compared to chemical-cured composites, which can better compete with the acid-base reaction. Both the contact surface of the composite and the inhibition layer benefit from this and will polymerize. Light initiation is not used for chemical-cured resin composites and on chemical grounds one can expect that an exposure to light will not help in better polymerization of the contact surface and inhibition layer.

The common way to study compatibility between bonding systems and resin composites is by determination of the bond strength to dentin [13-17]. Besides studying the bond strength to get information about the factors involved, shrinkage stress development determinations [18], can add important additional information to the subject, which is of interest in particular for core build-up restorations in the directed shrinkage technique. Shrinkage stress development informs about the rate of setting, which in turn provides information about the rates of the reactions taking place during setting [7].

The aim of this investigation was to determine the setting shrinkage stress development and the bond strength to dentin of a light-cured and a chemical-cured core build-up resin composite bonded with two multi-step and two simplified-step bonding systems for (1) compatible and (2) incompatible combinations. In addition the hypothesis was tested that light irradiation through a chemical-cured composite will neither affect shrinkage stress development nor bond strength.
6.3 Materials and Methods

**pH measurements**

The pH values of the various components of the bonding systems were determined with a pH meter Delta 350 (Mettler Toledo) and a combination pH surface electrode model 8135 (Orion Ross). The system was calibrated at pH 7.0 and pH 4.0. After every measurement the electrode was cleaned with acetone or ethanol depending on which bonding system was used. The measurements were performed at 23 °C in a darkroom with special red light on approximately 10 drops of each liquid and values were read after 15 seconds when the pH was stable.

**Bonding systems and core build-up resin composites**

Table 6.1 compiles the dentin bonding systems with their compositions and Table 6.2 the core build-up resin composites that were used in this study. Table 6.3 distinguishes the bonding systems to their type and surveys the pH values of the components used in the different steps. Table 6.4 shows the successive steps in each of the bonding procedures for bonding to dentin, as prescribed by the manufacturer and as they were used in the experiments.

**Setting shrinkage stress and tensile bond strength**

The setting shrinkage stress and tensile bond strength were determined in a universal testing machine (Instron) in a set-up where the core build-up resin composite was placed between a glass plate (of 4 mm thickness) and parallel to it the flat surface of a dentin cylinder, tightly cemented in a cylindrical hole drilled in a steel bolt head. The set-up was a modification of the one described previously by Dauvillier [19], where glass and steel were the parallel opposing surfaces. The dentin cylinders (diameter = 6 mm) were wet cut with a hollow diamond bur from the roots of central bovine incisors and wet ground at the bonding site with SiC paper up to grit 600 to create a smear layer.

Prior to bonding, the surface of the glass plate on the spot where the resin composite had to be adhered, was sandblasted with Al₂O₃ (50μm) until an even “frosted” surface developed. Remaining Al₂O₃ was removed by compressed air. After water spraying and drying, the glass plate was mounted to the stationary part of the framework of the tensilometer. One drop of Ceramic Primer (3M-ESPE) was applied to the surface and the solvent gently evaporated by an airflow. Finally a small amount of Scotchbond MP resin (3M-ESPE) was applied and blown out over the surface into a very thin layer and light cured for 20 seconds. The surface of the dentin was treated with one of the bonding systems from Table 6.1 according to the manufacturers
instructions (Table 6.4) and the bolt head immediately connected to the crosshead with the load-cell.

Table 6.1 Bonding systems components, compositions and curing type

<table>
<thead>
<tr>
<th>Bonding systems / curing type</th>
<th>Ingredients</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil Photo Bond / Dual-cured</td>
<td></td>
</tr>
</tbody>
</table>
*Etchant* 32% phosphoric acid and xanthan gum thickener.  
*Primer* Salicylic acid monomer.  
*Bonding Catalyst* Bisphenol A diglycidylmethacrylate (Bis-GMA), 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), 2-hydroxyethyl methacrylate (HEMA), hydrophobic aliphatic dimethacrylate, benzoyl peroxide and camphorquinone.  
*Bonding Universal* N, N-di-ethanol-p-toluidine, sodium benzene sulphonate and ethanol.  

| Clearfil SE Bond / Light-cured | 
*Primer* 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), 2-hydroxyethyl methacrylate (HEMA), hydrophilic dimethacrylate, camphorquinone, N, N-di-ethanol-p-toluidine and water.  
*Bonding* 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bisphenol A diglycidylmethacrylate (Bis-GMA), 2-hydroxyethyl methacrylate (HEMA), hydrophobic aliphatic dimethacrylate, camphorquinone (CQ), N, N-di-ethanol-p-toluidine and silanated colloidal silica.  

| All-Bond 2 / Dual-cured | 
*Etchant* 32% phosphoric acid and xanthan gum thickener.  
*Primer A* Na-N-tolylglycine glycidylmethacrylate, acetone, ethanol and water.  
*Primer B* Biphenyl dimethacrylate (BPDM), acetone, ethanol and photoinitiator.  
*D/E Resin* Bisphenol A diglycidylmethacrylate (Bis-GMA), urethane dimethacrylate (UDMA), photoinitiator (CQ) and amine activator.  
*Pre-bond Resin* 2-Hydroxyethyl methacrylate (HEMA), bisphenol A diglycidylmethacrylate (Bis-GMA), triethyleneglycol dimethacrylate (TEGDMA) and benzoyl peroxide.  

| One Step Bonding / Light-cured | 
*Etchant* 32% phosphoric acid and xanthan gum thickener.  
*Primer/Bonding* Biphenyl dimethacrylate (BDPM), bisphenol A diglycidylmethacrylate (Bis-GMA), 2-hydroxyethyl methacrylate (HEMA), amine, photoinitiator and acetone.  

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A sufficient amount of core build-up composite was applied onto the dentin surface and the crosshead lowered to a distance of 1 mm between the dentin surface and the glass plate. Excess of resin composite that was expressed was removed while shaping the specimen to the circumference of the dentin cylinder. The C-factor obtained in this way was \( C = 3 \). The C-factor is the ratio between the bonded surfaces and the free surface of the resin composite specimen = \( D/2h \), in which \( D \) is the diameter of the dentin cylinder (\( D = 6.0 \) mm) and \( h \) the height of the resin composite specimen (\( h = 1.0 \) mm).

Clearfil Photo Core was light-cured for 40 s from underneath the glass plate with an Elipar Highlight (3M-ESPE; 800 mW/cm\(^2\)), while Clearfil Core was either allowed to self cure or to self cure with an exposure to light for 40 s (from underneath the glass plate) to study the effect on the shrinkage stress and tensile strength. The latter experiments were to test the hypothesis. The computer started recording the shrinkage stress from the start of light curing Photo Core or 60 seconds after the start of mixing Clearfil Core and the stress development was measured during a period of 30 minutes. Throughout the measurement the axial contraction of the specimens was continuously counteracted by a feedback displacement of the crosshead (controlled by the computer) to keep the thickness of the specimen constant at 1.0 mm. At thirty minutes the set-up was loaded in tension until failure of the specimen to establish the 30 minutes bond strength to the dentin. For each bonding-composite combination eight measurements were performed (\( n = 8 \)) from which an average stress development curve and the average tensile bond strength to dentin were calculated.

In addition the setting shrinkage stress development of the two core build-up resin composites without the application of an adhesive was determined. The opposing bonding surfaces were glass (of 4 mm thickness) and a flat (machined) surface of a steel bolt head (diameter = 6 mm). Prior to bonding, the glass surface was treated as described previously and the flat (machined) surface of the steel bolt head was wet-ground on 600 grit SiC sandpaper and then sandblasted with \( \text{Al}_2\text{O}_3 \) (50μm), cleaned with compressed air, rinsed with water and acetone, and treated in a Silicoater (Heraeus Kulzer) to deposit a thin silica layer. A drop of fresh Silicoup (Heraeus Kulzer) was applied to silanize the surface. After drying, a small amount of Scotch Bond MP resin was blown out over the surface into a very thin layer and light cured for 20 s. The insertion of the composites and the C-factor was according to the description above.
**Bonding systems and chemical and light-cured core build-up composites**

**Table 6.2** Core build-up resin composites used in this study.

<table>
<thead>
<tr>
<th>Core build-up resin composite</th>
<th>Curing mode</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil Core</td>
<td>Chemical-cured</td>
<td>Kuraray Ltd. Japan</td>
</tr>
<tr>
<td>Clearfil Photo Core</td>
<td>Light-cured</td>
<td>Kuraray Ltd. Japan</td>
</tr>
</tbody>
</table>

**Table 6.3** Bonding systems and measured pH values of their components. Type 1 is a 3-step system with etchant, primer and adhesive as separate components; Type 2 is a 2-step system with an etchant and a combined primer/adhesive component; Type 3 is also a 2-step system, but with the etchant/primer combined and the adhesive as a separate component.

<table>
<thead>
<tr>
<th>Bonding system / Type</th>
<th>Components and their pH-values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photo Bond / Type 1 (Dual-cured)</td>
<td>Etchant 0.1* Primer 2.9 Adhesive 2.5 (mix)</td>
</tr>
<tr>
<td>SE Bond / Type 3 (Light-cured)</td>
<td>etchant and primer combined: 1.8 Adhesive 1.8</td>
</tr>
<tr>
<td>All-Bond 2 / Type 1 (Dual-cured)</td>
<td>Etchant 0.1* Primer 5.3 (mix) Adhesive 6.1</td>
</tr>
<tr>
<td>One-Step Bond / Type 2 (Light-cured)</td>
<td>Etchant 0.1* Primer and adhesive combined: 4.3</td>
</tr>
</tbody>
</table>

* Phosphoric acid

**Table 6.4** Successive steps in the bonding procedures for the bonding systems used to bond to dentin, indicating the time of application in seconds (s) and the number of layers applied.

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Bonding system</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Photo Bond</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>30 s</td>
</tr>
<tr>
<td>Rinsing with H₂O</td>
<td>20 s</td>
</tr>
<tr>
<td>Air drying (mode)</td>
<td>not dehydrated</td>
</tr>
<tr>
<td>Primer</td>
<td>1 layer x 5 s</td>
</tr>
<tr>
<td>Adhesive</td>
<td>1 layer</td>
</tr>
<tr>
<td>Light-curing</td>
<td>20 s</td>
</tr>
</tbody>
</table>
Chapter 6

**Figure 6.1** Schematical representation of the set-up in the tensiometer for measuring shrinkage stress and bond strength. Composite was bonded at the top to dentin with one of the adhesives and at the bottom to the silanized glass surface and either light-cured from below the glass (for the light-cured composite) or allowed to self-cure (for the chemical-cured composite). The axial contraction of the composites was continuously followed by the two probes (linear displacement transducers) and counteracted by a feedback displacement of the crosshead (controlled by the computer) to keep the thickness of the specimen constant at 1.0 mm.

**Statistical Analysis**

The data were statistically analyzed by a multiple analysis of variance (MANOVA), with the aid of the GLM subprogram of the SPSS package (Windows version 11.00). Effects with a P-value not exceeding .05 were considered significant. Whenever a main or interaction effect was significant on a multivariate level it was univariately examined next. Whenever called for, effects were further explored by means of one-way ANOVA and Tukey post-hoc tests or independent t-tests. The shrinkage stress and bond strength results were the dependent variables. In a first analysis the type of core build-up resin composite (Clearfil-Core or Photo-Core) and
bonding system (Photo Bond, SE Bond, All Bond and One Step Bond) were treated as a between subjects factor. In a second analysis, for the chemical-cured build-up resin composite Clearfil-Core only, the effect of light irradiation (without or with) and the bonding systems were treated as a between subjects factor.

6.4 Results

The results of the pH measurements on the various components of the bonding systems are incorporated in Table 6.3. During the 30 minutes measurement of the shrinkage stress no debonding occurred between dentin and resin composite. After tensile loading at 30 min after the start of curing all specimens fractured at the site where the composite was bonded to dentin. Figure 6.2 and Figure 6.3 respectively show the bar graphs of the 30 min shrinkage stress and bond strength to dentin. The graphical representation of the shrinkage stress developments for all composite-adhesive combinations and for the individual build-up resin composites (without adhesives) are all presented in Figure 6.4.

Effect of type of core build-up resin composite and bonding system (first analysis)

The results from the MANOVA with the type of core build-up resin composite and bonding system as the main effects, showed that these main effects were multivariately significant ($F = 27.179$, $P < 0.001$ and $F = 2.563$, $P = 0.023$). On a univariate level both main effects were significant for shrinkage stress ($F = 36.516$, $P < 0.001$ and $F = 4.030$, $P = 0.011$), but not significant for bond strength ($F = 1.098$, $P = 0.299$ and $F = 0.804$, $P = 0.497$). However, the interaction effect of the type of core build-up resin composite and bonding system was significant for shrinkage stress ($F = 13.992$, $P < 0.001$) as well for bond strength ($F = 4.665$, $P = 0.006$). The results of the one-way ANOVA and Tukey post-hoc tests and independent t-tests for determining differences between the type of bonding system and core build-up resin composite are presented in Table 6.5.
**Figure 6.2** Means of shrinkage stresses after 30 min for light-cured (Photo Core) and chemical-cured (Clearfil Core) core build-up resin composite combined with the four bonding systems studied. The combinations of the chemical-cured composite with light irradiation during curing are indicated as "+light".

**Effect of light irradiation through chemical-cured core build-up resin composite and type of bonding system (second analysis)**

The results from the MANOVA with the effect of light irradiation through the chemical-cured core build-up resin composite and the type of bonding system as the main effects, showed that these main effects were multivariately significant ($F = 3.316$, $P = 0.044$ and $F = 14.128$, $P < 0.001$). On a univariate level both main effects were significant for shrinkage stress ($F = 6.724$, $P = 0.012$ and $F = 28.900$, $P < 0.001$). For the bond strength, only one main effect, the type of bonding system, was significant ($F = 6.666$, $P = 0.001$); the effect of light irradiation was not significant ($F = 2.221$, $P = 0.142$). The interaction effect of the type of bonding system and the effect of light irradiation through the chemical-cured core build-up resin composite was significant for shrinkage stress ($F = 5.362$, $P = 0.003$), but not for bond strength ($F = 0.527$, $P = 0.666$). The results of the one-way ANOVA and Tukey post-hoc tests and independent t-tests for determining differences between the type of bonding system and the effect of light irradiation through the chemical-cured core build-up resin composite are presented in Table 6.6.
Bonding systems and chemical and light-cured core build-up composites

Figure 6.3 Means of bond strength to dentin after 30 minutes for light-cured (Photo Core) and chemical-cured (Clearfil Core) core build-up resin composite combined with the four bonding systems. The combinations of the chemical-cured composite with light irradiation during curing are indicated as "+light".

### Table 6.5
Means and standard deviations (in brackets) of shrinkage stress and bond strength to dentin at 30 minutes, for chemical and light-cured core build-up resin composite. Same superscript small letters indicate no significant differences in columns; same superscript capital letters indicate no significant differences in rows.

<table>
<thead>
<tr>
<th>Core material</th>
<th>Clearfil Core</th>
<th>Photo Core</th>
<th>Clearfil Core</th>
<th>Photo Core</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding system</td>
<td>Shrinkage Stress (MPa)</td>
<td>Bond Strength (MPa)</td>
<td>Shrinkage Stress (MPa)</td>
<td>Bond Strength (MPa)</td>
</tr>
<tr>
<td>Photo Bond</td>
<td>3.6 (1.1)^A</td>
<td>3.3 (0.7)^A</td>
<td>10.9 (2.8)^AD</td>
<td>10.4 (2.8)^AD</td>
</tr>
<tr>
<td>SE Bond</td>
<td>1.0 (0.2)^B</td>
<td>4.1 (0.7)^A</td>
<td>6.3 (1.9)^B</td>
<td>12.0 (2.9)^C</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>2.6 (0.8)^AB</td>
<td>3.1 (1.1)^B</td>
<td>11.2 (4.1)^B</td>
<td>8.8 (4.0)^AB</td>
</tr>
<tr>
<td>One-Step Bond</td>
<td>2.3 (0.6)^C</td>
<td>3.7 (0.6)^E</td>
<td>10.5 (3.8)^BC</td>
<td>11.0 (2.9)^AC</td>
</tr>
</tbody>
</table>

### Table 6.6
Means and standard deviations (in brackets) of shrinkage stress and bond strength to dentin at 30 minutes, for chemical-cured core build-up resin composite, without and with light irradiation. Same superscript small letters indicate no significant differences in columns; same superscript capital letters indicate no significant differences in rows.

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Shrinkage Stress (MPa)</th>
<th>Bond Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photo Bond</td>
<td>3.6 (1.1)^AA</td>
<td>4.4 (0.5)^AA</td>
</tr>
<tr>
<td>SE Bond</td>
<td>1.0 (0.2)^B</td>
<td>2.5 (0.6)^B</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>2.6 (0.8)^AB</td>
<td>2.3 (0.9)^AB</td>
</tr>
<tr>
<td>One-Step Bond</td>
<td>2.3 (0.6)^BC</td>
<td>2.2 (0.6)^BC</td>
</tr>
</tbody>
</table>

Without | With | Without | With

### Table 6.7
Means and standard deviations (in brackets) of shrinkage stress and bond strength to dentin at 30 minutes, for chemical-cured core build-up resin composite, without and with light irradiation. Same superscript small letters indicate no significant differences in columns; same superscript capital letters indicate no significant differences in rows.

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Shrinkage Stress (MPa)</th>
<th>Bond Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photo Bond</td>
<td>3.6 (1.1)^AA</td>
<td>4.4 (0.5)^AA</td>
</tr>
<tr>
<td>SE Bond</td>
<td>1.0 (0.2)^B</td>
<td>2.5 (0.6)^B</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>2.6 (0.8)^AB</td>
<td>2.3 (0.9)^AB</td>
</tr>
<tr>
<td>One-Step Bond</td>
<td>2.3 (0.6)^BC</td>
<td>2.2 (0.6)^BC</td>
</tr>
</tbody>
</table>

Without | With | Without | With

### Table 6.8
Means and standard deviations (in brackets) of shrinkage stress and bond strength to dentin at 30 minutes, for chemical-cured core build-up resin composite, without and with light irradiation. Same superscript small letters indicate no significant differences in columns; same superscript capital letters indicate no significant differences in rows.

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Shrinkage Stress (MPa)</th>
<th>Bond Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photo Bond</td>
<td>3.6 (1.1)^AA</td>
<td>4.4 (0.5)^AA</td>
</tr>
<tr>
<td>SE Bond</td>
<td>1.0 (0.2)^B</td>
<td>2.5 (0.6)^B</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>2.6 (0.8)^AB</td>
<td>2.3 (0.9)^AB</td>
</tr>
<tr>
<td>One-Step Bond</td>
<td>2.3 (0.6)^BC</td>
<td>2.2 (0.6)^BC</td>
</tr>
</tbody>
</table>

Without | With | Without | With

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Figure 6.4 Polymerization shrinkage stress development of core build-up resin composites combined with four types of bonding systems during a period of 30 minutes. (A) Light-cured Photo Core, (B) chemical-cured Clearfil Core, and (C) chemical-cured Clearfil Core with light irradiation through the composite. (D) Shrinkage stress of Photo Core and Clearfil Core without adhesives bonded to steel instead of dentin. Note that the scale of the vertical axis in D differs from A, B and C.
6.5 Discussion

The significance of the use of dentin as a substrate to bond at was to study whether the bond to dentin of Photo Core and Clearfil Core combined with the four bonding systems could at least resist the developing setting shrinkage stresses and if they did, to follow the shrinkage stress development during a 30 minutes period and to establish the tensile bond strength at the end of this period. The C-factor used in the experiments (C = 3) is an estimated average of situations the practitioner encounters in core build-up restorations when filling the entrance of the root canal or pulp chamber.

A factor of C = 3 corresponds to a situation where the build-up composite is placed in bulk for which 75% of the outer surface of the composite is bonded to tooth structure and the remaining surface is free (not bonded). None of the 96 specimens tested showed spontaneous failure and generated stress values after 30 minutes between 1.0 and 4.4 MPa. This is an encouraging outcome, in particular for light-cured composites such as Photo Core, as under clinical conditions the entrance of the root canal or pulp chamber are usually not filled in bulk for these materials, but in layers for which the stresses are expected to be even lower. After loading in tension, 30 minutes after the start of curing, all specimens fractured at the site where the composite was bonded to the dentin. This was not unexpected, as the adhesion of a composite to a silanized glass surface is always stronger than adhesion via elastic intermediate adhesives to any kind of substrate (in this case dentin).

Setting shrinkage stresses at 30 minutes were considerably higher when Photo Core or Clearfil Core was placed without a bonding layer (14.3 and 5.0 MPa respectively), as was done in the set-up where glass and metal were the bonding surfaces. This demonstrates the significance of the effect of a bonding layer in reducing the shrinkage stress. The factors that could have contributed to stress reduction are (1) the presence of the oxygen inhibition layer, (2) the elastic yielding of the adhesive and (3) the formation of voids in the adhesive layer.

(1) The oxygen inhibition layer covering a cured adhesive is always present as a result of the high reactivity of air oxygen with the starting radicals converting them into inactive peroxyradicals and manifests itself as an 'oily' substance. This may allow sliding of the composite during the first stages of setting, which will be beneficial in particular for light-cured composites in slowing down the high rate of stress development during light initiation.

(2) It has been recognized previously that an intermediate bonding layer between the cavity walls and the setting composite may act as an elastic buffer [20-22]. However the effectiveness depends on the thickness and an even distribution over the dentin surface, which in turn depends on the viscosity of the particular adhesive used and the number of coats applied [23].
(3) Formation of voids can occur as a result of the pulling action of the composite by its shrinkage on the adhesive layer, which may contain solvents (water, ethanol or acetone) that can evaporate under reduced pressure and may be enhanced by the presence of dentin tubules acting as tiny reservoirs for these solvents. A stress relieving effect of adhesives on shrinkage stress of composites has also been demonstrated previously, but the effect was only 50%, probably because the experiments were not performed with dentin [24]. Voids will lower the C-factor, as they increase the non-bonded surface and will consequently reduce the shrinkage stress [25]. The existence of voids may also explain that the tensile bond strength values were lower than those reported in the literature, where bond strength tests were performed on inverted truncated composite cones, which could polymerize towards the dentin surface without creating pulling stresses on the adhesive layer [26-28].

As shrinkage stress development of composites relates to the degree of conversion and in its turn to composite (cohesive) strength development, one can derive from Figure 6.4(D) that the strength of Clearfil Core at e.g. 10 minutes is only 40% of that at 30 minutes. According to the shape of the curve, which not yet leveled off at 30 minutes, the strength had still not reached its ultimate value at this time period. The leveling of the curves in Figure 6.4 (B and C) for Clearfil Core in conjunction with adhesives occurred much sooner at approximately 10 - 12 minutes. This is probably because the phenomena (1), (2) and (3) acting in the adhesive layer, as discussed earlier, will limit stress development in an early stage. It is reasonable to assume that shrinkage stress development for a composite-adhesive combination is related to bond strength development. The bond strength at 10 minutes of Clearfil Core combined with the adhesives may therefore be between 75-80% of the value at 30 minutes. Although at 10 minutes (relative to 30 minutes) the composite is still weak (40% strength) and the bond strength to dentin is not fully matured (75-80% value), clinical experience learns that trimming and finishing can be started safely at 10 minutes. An advantageous circumstance is that the shrinkage stress at the adhesive interfaces of a composite build-up and the tooth structure will also be lower at 10 minutes. It should be appreciated that lower stressed adhesive joints will better resist impact during trimming and finishing. However the strength and stress conditions for trimming and finishing will become more unfavorable at earlier time periods and maybe should be avoided.

For combinations of the light-cured composite Photo Core with the four bonding systems the shrinkage stresses at 30 minutes and the bond strengths differed not significantly from each other (Table 6.5). This shows that combinations of Photo Core with the two bonding systems Photo Bond and SE Bond of its own manufacturer are equally effective in bonding as combinations of Photo Core with the two bonding systems All-Bond 2 and One-Step Bond of another manufacture.
For the chemical-cured composites combined with the four bonding systems differences did occur at 30 minutes for stress and bond strength (Table 6.5). The lowest values for both the stress development and bond strength were recorded for the chemical-cured composite combined with the light-cured SE Bond, which may indicate the existence of an incompatibility for this combination. Recently it has been shown that incompatibilities can occur when chemical-cured composites are combined with adhesives containing acidic monomers [16,17,29]. The monomer used in the adhesive SE Bond is 10-Methacryloyloxydecyl dihydrogenphosphate (MDP), which is an acidic monomer producing a pH of 1.8 in the adhesive liquid. This monomer will therefore also be present in the oxygen inhibition layer after the adhesive has been light cured. When the chemical cured composite is brought into contact with it, the tertiary amine in the composite, necessary for the activation of the benzoylperoxide initiator, will be protonized and thereby be inactivated. This will affect the formation of starting radicals and thus the polymerization of the contact layer of the composite [9]. In addition starting radicals that did form in the composite contact layer will be converted into inactive peroxylradicals until all air oxygen in the inhibition layer is consumed.

The bond strength of the chemical-cured composite combined with the light-cured One-Step Bond, which is less acidic, seemed not to be affected (10.5 MPa vs. 11.0 MPa for the light-cured composite combined with One-Step Bond). This may be ascribed to the higher pH of 4.3 as compared to that of SE Bond (pH = 1.8), as less amine will be protonized in the composite at the contact surface. Why the present results on bond strength contrast those of Sanares [13], who found a difference of nearly 50% in the microtensile bond strength between a chemical and a light-cured composite bonded with One-Step Bond to dentin, is not clear.

Dual-cured adhesives with acidic monomers are more favorable to be combined with chemical-cured composites. The combination with the dual-cured Photo Bond, which like SE Bond also contains MDP, resulted in the same bond strength as the light-cured composite combined with this bonding system. The better performance must be ascribed to a larger amount of amine and the presence of benzoylperoxide in the inhibition layer, although also the incorporation of sodium benzene sulfinate in Photo Bond (Table 1) may have contributed. It has been suggested that this salt may further help the polymerization of the acidic monomers [30], probably because it suppresses protonization of the amine.

When a chemical-cured composite is placed on a dual or light-cured adhesive that is free of acidic monomers the situation is even more favorable. The adhesive of the dual-cured All-Bond 2 with a pH of 6.1 can be considered as neutral. Here, the amine is unaffected by an adverse reaction and only the competition remains between free radical formation (from the tertiary amine/benzoylperoxide redox) and free radical quenching by oxygen. The shrinkage stress at 30
min found for this combination and for the combination of the chemical-cured composite with One-Step Bonding was significantly lower than all others. At this point it is not possible to speculate on whether the lower stress for the chemical-cured composite combined with All-Bond 2 or One-Step Bond compared to a combination with Photo Bond, resulted from a difference in the stress relieving factors (1), (2), or (3) as discussed above, or from a difference in chemical conversion in the adhesive-composite contact area.

Based on the described chemical mechanisms about the causes for incomplete polymerization at the interface of adhesive and chemical-cured composite, one would not expect an influence on shrinkage stress development or bond strength by light irradiation through the chemical-cured composite. However the results of the experiments only partly confirmed this hypothesis. The combinations that were supportive were the chemical-cured composite combined with All-Bond 2 and One-Step Bond. For both combinations the stress developments and bond strengths did not change with an irradiation through the composite during curing. The stress curves for each pair of each combination almost coincided. This indicated that even heat from the light source transferred to the reacting system was not effective in accelerating the reaction. The chemical-cured composite combined with the light-cured SE Bond did show a significant increase in stress development at 30 minutes and although the increase of Photo Bond was not significant at 30 minutes (4.4 MPa compared to 3.6 MPa) the over-all increase in stress development was significant for all values up to approximately 25 minutes. The strength values at 30 minutes were not significantly increased, but a trend is visible (Figure 6.3).

The increase in stress development for these two bonding systems, which clearly shows a continuation of polymerization, is unexpected, but may find an explanation based on a prolonged activity of the acidic monomers on dentin. This explanation needs the assumption that after curing of the adhesive, the non-evaporated solvents (water or ethanol) still present in the polymerized primer-adhesive allow passage of the acidic monomers to the dentin surface. Because of the low pH of 1.8 (for SE Bond) and 2.5 (for Photo Bond) these acids will react at a moderate to fast rate with the dentin mineral and/or carbonate [31]. This lowers the concentration of acidic monomers in the oxygen inhibition layer, which drives the equilibrium of protonization of the tertiary amine to the left side of the equilibrium to release the amine. As discussed previously, a higher concentration of amine in the inhibition layer is essential for the polymerization in the contact layer between the light-cured acidic adhesive and the chemicalcured composite. The proposed mechanism is not likely to occur for light-cured adhesives like One-Step Bond with milder acidity and/or more volatile solvents as acetone.

The experimental set-up used in this study, where polymerization shrinkage stress development and tensile bond strength to dentin were measured on the same specimen, provided
important information about composite-adhesive compatibility. In future experiments with this set-up the morphology of fractured surfaces will be studied by SEM to establish more in detail the factors in the adhesive layer that have led to the dramatic reduction in shrinkage stress development.

6.6 Conclusions

The present study has shown that:

- Besides combinations of chemical-cured core build-up composites and light or dual-cured adhesives as recommended by the manufacturer, also combinations with adhesives of other manufacturers are compatible, provided that the pH is higher than approximately 4.3.
- It may occur that cross combinations of manufacturers products result in significantly lower stress developments, as seen for Kuraray's Clearfil Core combined with Bisco's All-Bond 2 and One-Step Bond compared to Kuraray's Photo Bond.
- Chemical-cured core build-up composites combined with light-cured adhesives with a pH lower than 1.8 showed inferior bond strength to dentin compared to other combinations.

6.7 References

Chapter 6


