Investigations into the factors affecting the shear bond strength of multiple component and single bottle dentin bonding systems to dentin

By

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A thesis submitted in conformity with the requirements for the degree of Master of Science

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Part 1 - Thermocycling and Long-Term Shear Bond Strength Study

Purpose: To compare the in vitro shear bond strengths (SBS) of two fourth generation (Scotchbond Multi-Purpose [SBMP] and All-Bond 2 [AB2]), and three single-bottle (Single Bond [SB], One Step [OS], and Prime and Bond 2.1 [PB]) dentin adhesives after thermocycling and long term storage. Materials and Methods: Flat bonding sites were polished with 600 grit paper on the labial surface of 300 decoronated bovine incisors mounted in acrylic resin. 60 teeth were assigned to each of the five adhesive systems. Teflon tape with a 4 mm hole in it was applied and the dentin surface etched with 35% phosphoric acid gel then rinsed. The adhesives were applied as directed by the manufacturer. Z100 composite resin was placed inside gelatin capsules, positioned over the dentin surface and visible light cured. Bonded specimens were divided into five groups each containing 12 samples: (1) stored in 37°C water for 24 hours, (2) stored in 37°C water for 7 days, (3) stored in 37°C water for 24 hours, (4) or 6.5 days before thermocycling for 500 cycles between 5°C and 55°C then returned to storage for a total of 7 days, (5) stored in 37°C water for 270 days. The samples were shear tested, the location of bond fracture was recorded, and the mean shear bond strengths subjected to statistical analysis. Results: The single bottle systems suffered statistically significant reductions in shear bond strength after 270 days as compared to the 24 hour control. The effect of thermocycling on the adhesive systems was variable. Both thermocycling parameters resulted in statistically significant reductions in SBS however, the SBS of OS increased after thermocycling at 24 hours. SB, PB, AB2 demonstrated non-significant reductions in SBS after thermocycling. The two thermocycling parameters produced similar SBS for all materials except OS.
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Part 2 - Dual Cure Bond Strength Study

**Purpose:** To evaluate the effect of two-step photopolymerization of Z100 composite resin and resin thickness on the shear bond strength (SBS) of Scotchbond Multi-Purpose and Single Bond to dentin. **Materials and Methods:** Flat bonding sites were exposed on the occlusal surface of 144 extracted human third molars and then polished with 600 grit paper. Half of the teeth were assigned to each adhesive system. Teflon tape with a 4 mm hole in it was applied and the dentin surface etched. The adhesives were applied as directed by the manufacturer. Polyvinyl chloride (PVC) clear mouthguard material with 4 mm holes punched in it were placed over the adhesive and Z100 composite resin was packed into the hole. Three different thicknesses of Z100 resin were produced (1.5 mm, 3.0 mm, and 4.5 mm) by varying the thickness of the PVC template. Gelatin capsules were inserted into the holes of the 3.0 mm and 4.5 mm templates. For each adhesive 24 teeth were bonded for each thickness. Half of these were cured for 40s at 700mW/cm² and half for 10s at 150mW/cm² then 30s at 700mW/cm². The teeth were stored in 37°C water for 7 days with the templates being removed after the first 24 hours. The teeth were shear tested, the location of bond fracture was recorded, and the mean shear bond strengths subjected to statistical analysis. **Results:** Thickness of Z100 resin and cure type did not significantly affect the bond of SBMP to dentin under any of the experimental conditions. At a thickness of 4.5 mm the bond of SB to dentin was significantly reduced with both types of curing. The percentage of purely adhesive fractures noted with SB increased as the thickness of Z100 composite resin increased.
Abstract

Part 3 - Microhardness Study

Purpose: To evaluate the effect on Knoop microhardness of dual-curing, material thickness, and storage in 37°C water for up to 7 days. Materials and Methods: Polyvinyl chloride (PVC) mouthguard material 1.5 mm, 3.0 mm, and 4.5 mm in thickness was used as templates. For each thickness 10 templates were prepared by punching 4 mm holes in the PVC material. A glass cover slip was placed under the template and then placed over a flat dentin surface. Z100 composite resin was packed into the mold so that it was flush with the surface of the template. For each thickness 5 samples were cured for 40s at 700mW/cm² and the other 5 were cured for 10s at 150mW/cm² and 30s at 700mW/cm². All samples were placed into 37°C water after curing. The Knoop microhardness was recorded at 4 points on the top and bottom of each sample at the following intervals: 2 hours, 24 hours, and 7 days. The mean Knoop microhardness was determined for each sample at each time period and then subjected to statistical analysis. Results: The method of curing and storage in 37°C water did not significantly affect the microhardness. A non-significant trend towards lower microhardness was noted for the top and bottom surfaces of the 4.5 mm samples. The results of this study should be verified using Rockwell or Vickers hardness tests since the Knoop test utilized in this study may not have been sufficiently sensitive.
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Introduction

Forty-five years ago Buonocore reported that adhesion of acrylic resins to enamel could be achieved by etching with 85% phosphoric acid (Buonocore, 1955). Soon afterward adhesion to enamel became a common practice in clinical dentistry. However, the strong bonds achieved with enamel could not be matched with dentin. In the mid-1980's clinically significant bond strengths to dentin became possible with the introduction of third generation dentin adhesives such as Scotchbond 2 (3M). The improved dentin adhesion was due to the use of solutions that modified the dentin surface prior to the application of the resin (Burke and McCaughey, 1995). This allowed for micromechanical retention of the resin whereas the previous attempts at dentin bonding focused on chemical bonds. Over the years improvements and modifications to the third generation products resulted in the development of a number of other systems which were later categorized as fourth and fifth generation adhesives.

The last few years have seen research and development of dental adhesives focusing upon simplifying the application technique. To this end a number of one-step, or fifth generation, dentin bonding agents have been developed which are said to retain the bond strengths of the multiple step systems. At present, these new systems have not been subjected to long-term studies.

As shown in Figure 1, a number of interrelated factors are involved in dentin bond strengths achieved in in vivo and in vitro studies (Miyazaki et al., 1996). The present study focuses on four of these parameters; thermocycling of
materials after bonding, time of storage of bonded specimens, the treatment of dentin prior to application, and the type of bonding agent.

The manufacturer's instructions supplied with adhesive and restorative resins include recommended curing protocols. These suggestions are based on the use of high intensity visible light since this provides a higher degree of conversion and more rapid polymerization. However, this rapid polymerization may result in greater polymerization shrinkage which has been reported to increase internal stress and microleakage (Sakaguchi and Berge, 1998). Recently, it has been suggested that composite resin material polymerized with low intensity light may have better marginal adaptation, however, compressive strength and other physical properties may be negatively affected (Unterbrink and Muessner, 1995). Mehl et al. (1997) have suggested that contraction stresses and microleakage may be reduced by allowing flow of the resin during setting by means of controlled polymerization. This is accomplished by briefly exposing the resin material to a low intensity light prior to complete polymerization with a higher intensity light. At present, it has not been established whether the use of a two-step curing protocol has any effect on the bond strength of polymeric materials to dentin.
Figure 1. Flow chart of interrelated parameters on dentin bond strengths. Adapted from Miyazaki et al., 1996.
Review Of The Literature

As outlined in the introduction, a large number of factors affect the adhesion of bonding systems to dentin. The following review will address the areas that have bearing on bond strength laboratory investigations. Additionally, the effect of light curing methods on the physical properties of restorative resins will be discussed.

Dentin Properties

The chemical composition of mature human dentin by weight is approximately 70% inorganic material, 20% organic material, and 10% water (Torneck, 1994). Its inorganic component is mainly Ca₁₀(PO₄)₆(OH)₂ (hydroxyapatite) and the organic portion is primarily Type I collagen with traces of glycoproteins, proteoglycans, phosphoproteins, and plasma proteins. The collagen is in the form of fibrils onto which the other organic molecules and apatite crystals are deposited. These fibrils give dentin an elastic quality that provides flexibility and prevents fracture of the overlying enamel.

The most distinctive characteristic of dentin is the presence of numerous tubules that run from the dentinoenamel junction to the dental pulp. The number of dentin tubules vary according to anatomic location. There are significantly less tubules in radicular compared to coronal dentin and the number of tubules per mm² is half at the dentinoenamel junction as it is at the pulpal surface. Additionally, physical processes such as aging, abrasion and caries alter the composition of the tubules by affecting the proportions of intertubular, peritubular and sclerotic dentin (Torneck, 1994). This variation in dentin structure has been
postulated to account for the large variations that are reported in laboratory evaluations of adhesion to dentin (Nakabayashi et al., 1991).

**Bovine Dentin**

The ideal situation for testing dentin bonding is living human dentin through an *in vivo* clinical trial (Rueggeberg, 1991; ISO/TC 106, 1991). The difficulties associated with *in vivo* bond strength studies, however, have led to the common practice of using extracted teeth as part of laboratory investigations (Rueggeberg, 1991). Additionally, the availability of human teeth as well as concerns regarding their potential as carriers of infection has led to the search for alternate substrates for adhesion testing (Rueggeberg, 1991). The most common substitute for human dental material is bovine teeth. Even though bovine dentin has become a common substrate for adhesion testing, only a few studies exist which directly compare bovine dentin with human dentin. Nakamichi *et al.* (1983) demonstrated that there were similar tensile bond strengths for enamel and superficial dentin in human and bovine teeth. In their study they found that bond strengths in bovine teeth decreased as the pulp chamber was approached and their results indicated that the superficial dentin demonstrated bond strengths as much as 10.7 times greater than that found by dentin near the pulp chamber. Additionally, they found that etched and unetched dentin of human and bovine teeth were morphologically similar. These investigators concluded that coronal bovine dentin is an acceptable substitute for human dentin. Tagami *et al.* (1990) found that bovine incisors possessed larger dentinal tubules and greater microporosity and were similar to human molar root dentin. Fowler *et al.* (1992)
found in their study of selected variables on adhesion testing that there was an overall tendency for bond strengths to be higher with bovine dentin as compared to human. Their results, however, were variable and not all of their samples followed this trend. They attributed some of this variation to differences in natural teeth, irrespective of whether they were bovine or human. Reeves et al. (1995) found in their in vitro study that microleakage around bonded human and bovine dentin was not significantly different. They concluded that bovine teeth can be used instead of human teeth for in vitro microleakage studies.

**The Smear Layer**

The presence and importance of the smear layer has been known for many years (Eick, et al. 1970; Pashley, 1984). It is composed of blood, saliva, bacteria, enamel, and dentin particles (Brannstrom, 1974). The morphology and composition of the smear layer can vary widely depending on the type of cutting instrument used, the area of dentin in which it was generated, and whether water was used or not during cavity preparation (Joynt et al., 1991). Smear material overlies the cut dentin and occludes the dentinal tubules. This led to the early belief that the smear layer served as a barrier that protected the pulp from noxious stimuli. However, any protective function is temporary because it can dissolve in oral fluids and it is usually contaminated with microorganisms (Nakabayashi and Pashley, 1998).

Early dentin bonding systems did not attempt to significantly alter the morphology or composition of the smear layer prior to bonding. Using the scanning electron microscope, Pashley (1991) found that the 5 to 7 MPa bond
strengths achieved with these systems actually represented the cohesive strength of the smear layer. Pashley (1992) later described several studies that were able to achieve bond strengths in the 7 to 10 MPa range after modifying the smear layer by treatments that involved either the simple application of water or by scrubbing of the dentin surface with a combination of 0.2% EDTA and 0.1% benzalkonium chloride (Tubulicid, Dental Therapeutics AB, Nacka, Sweden). The Tubulicid partially removed the smear layer while leaving most of the smear plugs intact. He concluded that there may be some value in continuing research into materials that bond to a reasonably intact smear layer since this will leave tubules occluded, perhaps resulting in reduced sensitivity and prevention of the decrease in bond strengths that are seen with over preparation of the dentin surface. However, as research into dental adhesion continued it appeared that the smear layer was actually an impediment to higher bond strengths and that the primer or resin must infiltrate the dentin substrate to achieve high bond strengths (Suh, 1991). The newer bonding systems require the complete removal of the smear layer.

**The Hybrid Layer**

Application of acid to the cut dentin removes the smear material and results in a demineralized dentinal surface with an exposed but unsupported meshwork of collagen fibres. Provided that this collagen meshwork maintains its integrity, resin monomers are able to penetrate these decalcified dental tissues and produce hybridized dentin. This resin/dentin interdiffused zone was first
described by Nakabayashi in 1982 utilizing 4-methacryloxyethyl trimellitate anhydride (4-META) (Nakabayashi and Pashley, 1998).

Van Meerbeek et al. (1993) examined the micromorphology of the hybrid layer under scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Three sublayers were observed within the hybrid layer. The upper layer lacked ultrastructural arrangement and the collagen appeared denatured by the acid conditioning. Underneath this layer, closely packed collagen fibrils with small interfibrillar spaces were arranged parallel to the dentin surface, which were presumed to restrict deeper resin impregnation into the interfibrillar spaces. As a result, resin impregnation primarily occurred at the upper denatured collagen layer with a decreasing gradient of intensity to the underlying collagen fibril arrangement and the unaltered dentin interface. The electron dense base layer demarcated the decalcified dentin zone from the deeper unaltered dentin. This layer consisted of incompletely dissolved hydroxyapatite crystals. These crystals had resisted the ethylene diamine tetra-acetic acid (EDTA) treatment required prior to TEM examination and the authors suggested that they were protected by resin encapsulation. Scanning electron microscope examination revealed that resin penetrates and intermingles with the exposed collagen fibers forming a transitional or “hybrid” layer of resin reinforced dentin (Miyazaki, 1996).

The hybrid layer varies in thickness depending on the bonding agent used and the depth and type of dentin pretreatment. Usually thicknesses of 3 to 10 um are achieved (Farah and Powers, 1995), however hybrid layers as thin as 1 um
were seen with the Gluma Bonding system (Inokoshi et al., 1993). It also appears that the thickness of the layer exerts little influence on the bond strength (Asmussen and Hansen, 1993).

The bonding area between the dentin surface and the restorative resin composite represents a gradual transition of materials within this thin layer. This allows for the possibility of continuous chemical bonding from the exposed organic component of the dentin to the primer to adhesive to resin (Miyazaki, 1996; Suh, 1991). It is likely that the resin hybrid structure and the penetration of resin into the opened tubules to form resin tags are the key-points in resin to dentin adhesion (Miyazaki et al., 1996; Titley et al., 1995). Therefore, any treatment of the dentin that improves the formation of these structures should enhance bond strengths.

**Thermocycling**

Thermocycling has been suggested as being an important component of bond strength testing. Nelson et al. (1952) demonstrated marginal percolation of fluid due to thermal changes for the first time (Shortall, 1982). This study was much publicized and prompted many workers to investigate the effect of temperature change on microleakage and bond strength. Marginal or ‘thermal’ percolation is thought to be due to differences in coefficients of thermal expansion between tooth structure and resin that lead to compression and tensile forces during thermocycling (Fitchie et al. 1993). It has been suggested by some authors that the forces exerted by polymerization shrinkage and thermal expansion exceed the bond strength of dentinal bonding agents (Fitchie et al.
1993). On the other hand some researchers question whether the effects of thermal percolation are of clinical significance in relation to composite restorations (Kidd et al., 1978).

The technical report developed at the ISO/TC 106 meeting in Trieste (1991) recommended that specimens in laboratory bond strength investigations be exposed to 500 cycles between 5 +/- 2°C and 55 +/- 2°C with a dwell time of at least 20 seconds and a transfer time between 5 and 10 seconds. These recommendations were re-affirmed in the Technical Report issued in 1994 (ISO TR 11405: 1994E). In spite of these efforts at standardization, there remains a great deal of variation in thermocycling protocols.

<table>
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<tr>
<th>Author et al. (Year)</th>
<th>No. of Cycles</th>
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<th>Dwell Time (s)</th>
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<td>5 - 55</td>
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Table 1. Thermocycling parameters in selected studies and whether a non-thermocycled control was included.

Studies which incorporate thermocycling in the shear testing protocols often do not include a non-thermocycled control. Therefore, little work is being done to investigate whether thermocycling samples through a reasonable
number of cycles has any significant effect on laboratory results. The few studies that do include a non-thermocycled experimental group show mixed results. Several studies are listed in Table 1 along with the conditions to which they exposed their samples.

Yap et al. (1996) investigated microleakage around class II composite resin restorations bonded with the Gluma Bonding System (Bayer Dental, Leverkusen, Germany) and Scotchbond Multi-Purpose (3M Corporation, St. Paul, MN, USA) after thermocycling human molars into which class II composite restorations had been placed. The preparations were designed so that the occlusal margins were unbeveled and in enamel whereas the gingival margin was in cementum. After thermocycling, no effect was noted on microleakage at the cervical margins, however Scotchbond Multi-Purpose showed a decrease of leakage at the occlusal margins.

Yap et al. (1997) investigated microleakage utilizing human molars which had a Z100 (3M Corporation Minnesota, USA) composite restoration placed over a glass ionomer base. After thermocycling, no effect on leakage was noted at the composite-dentin interface, however leakage at the enamel-composite interface was reduced.

Fitchie et al. (1993, 1995) placed class V restorations in bovine and human incisors. The incisal margin was beveled and located in enamel and the gingival margin was a butt joint terminating on cementum. The samples were thermocycled and the marginal leakage that was present after 24 hours and 60 days was recorded. Coefficients of thermal expansion were calculated for the
composite resin as well. They concluded that no correlation between microleakage and coefficients of thermal expansion of the composite resin could be established.

Carracho et al. (1991) used human molar teeth to investigate the effect of thermocycling on Scotchbond Dual Cure (3M), Scotchbond 2 (3M), and Mirage Bond (Chameleon Dental Products Inc.). The occlusal third of the tooth was ground down and dentin was exposed to which the materials were applied. Thermocycling was found to significantly reduce the shear bond strength of Scotchbond 2 and Scotchbond Dual Cure, but did not significantly affect Mirage Bond.

Rossomondo and Wendt (1995) suggested that shorter dwell times are more clinically relevant in microleakage studies utilizing thermocycling. They suggested thermal exposures of 10 seconds since this more closely resembled what would be expected in an oral environment. They also questioned the relevance of the in vitro thermocycling of composite resin materials. The coefficient of thermal expansion of tooth material differs widely from that of composite. The authors suggest however that thermocycling at clinically relevant dwell times may not be of consequence because of very slow rates of thermal diffusion through resin composites and unfilled resin materials. Craig et al. (1992) reported that the thermal conductivity of composite is approximately that of dentin and enamel, while amalgam is 20x more and gold is 300x more thermally conductive. Rossomando and Wendt placed class V preparations with unbeveled margins occlusally in enamel and gingivally in cementum. The preparations were
resin bonded with Liner F (Bisco), Universal Bond 3 (LD Caulk), Syntac (Vivadent) and restored with either amalgam or composite resin. No difference in the microleakage of non-thermocycled and thermocycled samples was noted.

**Acid Etching of the Dentin**

Bonding systems commonly used today achieve high bond strengths by removing the smear layer. This is accomplished by treatment with organic acids such as phosphoric, citric, maleic, and succinic acids (Douglas, 1989; Prati, 1993; Chan, 1994). Pashley et al. (1992) suggest that the removal of the smear layer and demineralization of the dentin matrix facilitates bonding through all or some of the following mechanisms:

- Removal of loose smear layer debris and exposure of dentin matrix
- Exposure of collagen fibrils and their epsilon-amino groups, which may catalyze HEMA polymerization.
- Exposure of intact collagen that serves as a scaffold for the creation of resin-collagen hybrid layer.

The pH and concentration of the acid dictates the depth of the demineralized layer of dentin. Chan (1994) and Titley et al. (1996) found that acid preparations commonly used in dentistry such as 10.0% maleic and 37.0% phosphoric produced a 1.5 to 4.0 μm depth of demineralization. Additionally, a significant amount of collagen was seen within tubules that had been widely demineralized producing a funneled appearance. However, they found that the adhesive resin was not able to completely fill the demineralized zone and substantial gaps remained. They found that the use of more dilute acids than
those provided commercially resulted in a smaller zone of demineralization, less deterioration of the physical properties of the dentin, and a more complete infiltration by the adhesive resin.

Other effects of acid etching considered by Pashley and others (1992) to be disadvantages are:

- Increase in dentin permeability
- Increase in dentin wetness
- Increased potential for pulpal irritation
- Potential for denaturation of the collagen

As a result of the potential problems associated with dentin conditioning the trend has been towards reducing the acid concentration and time of etch. Buonocore (1955) utilized a phosphoric acid concentration of 85%. Fusayama (1979) treated dentin with 40% phosphoric acid solution for 60 seconds to improve adhesion of the Clearfil Bond System-F. Kanca (1993) found that a 20 second application of 37% phosphoric acid completely removed the smear layer and exposed the tubule apertures. Chan (1994) found that a 15 second application of a 37% phosphoric acid aqueous solution resulted in removal of the smear layer, widening of the tubule orifices and demineralization of the superficial dentin. Additionally, Titeley et al. (1996) noted that there was minimal pH difference between a wide range of phosphoric acid concentrations (Table 2). Fourth and fifth generation materials presently in use commonly utilize 37% phosphoric gel etchant for 10 to 15 seconds (Farah and Powers, 1997). This
demonstrates an attempt to optimize the positive effects of dentin conditioning while minimizing the negative effects of aggressive etching.

<table>
<thead>
<tr>
<th>Phosphoric acid formulation</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>10% aqueous</td>
<td>1.42</td>
</tr>
<tr>
<td>10% gel</td>
<td>1.40</td>
</tr>
<tr>
<td>32% gel</td>
<td>1.09</td>
</tr>
<tr>
<td>35% gel</td>
<td>0.97</td>
</tr>
<tr>
<td>37% aqueous</td>
<td>1.14</td>
</tr>
<tr>
<td>40% gel</td>
<td>0.88</td>
</tr>
<tr>
<td>55% aqueous</td>
<td>0.88</td>
</tr>
</tbody>
</table>

*Table 2. The pH values of aqueous solutions and gel preparations of phosphoric acid. Adapted from Chan (1994) and Titley et al. (1996).*

**Dentin Priming**

Hydrophobic resins such as Bis-GMA (Bisphenol-glycidyl methacrylate) are not able to adequately diffuse into dentin treated with acid alone. Most modern dentin bonding systems utilize primers to circumvent this shortcoming. In general, dentin primers improve adhesion two different ways. Firstly, they treat the substrate to improve its wettability by the adherent, i.e. a hydrophilic substrate can be made more hydrophobic (Causton, 1982). Surface "wetting" is a concept that is commonly referred to when discussing primers. The extent to which an adhesive wets the surface of an adherend depends on the relative surface energies of the substrate and the liquid and on their intermolecular attraction (Craig et al., 1992). The degree of wetting may be determined by measuring the contact angle formed at the interface between the adhesive and the adherend. A liquid with a small contact angle is able to wet the surface more efficiently. A contact angle of zero degrees, therefore, indicates that the adhesive
spreads over the surface completely (Phillips, 1996). Secondly, active chemical
groups or atoms, capable of incorporation into the structure of a resin may be
attached to the surface, thus forming a chemical bond across the interface
(Causton, 1982).

After acid etching the demineralized dentin matrix will collapse if exposed
to drying (Titley et al., 1994). Primers maintain or recover the porosity of the
dentin and keep the demineralized dentin wet, which prevents their collapse
(Nakabayashi and Pashley, 1998). Also, they may be able to open or re-expand
the demineralized collagen network allowing deeper penetration of the resin
(Farah and Powers, 1995). Adhesive systems containing hydrophilic primers in
acetone or ethanol were found to produce their highest bond strengths when acid
conditioned dentin was left moist prior to bonding in a protocol referred to as “wet
bonding” (Gwinnett, 1992; Kanca, 1992; Tay et al., 1996). The improved
adhesion is related to the prevention of collagen network collapse that allows for
improved penetration of the primers into the demineralized dentin.

Primers are hydrophilic monomers of low viscosity usually in a highly
polar, low surface tension solvent such as acetone or ethanol (Suh and Cincione
1992). Different hydrophilic monomers are included in the commercially available
dentin bonding systems. The primers used in this study will be listed in the
description of the specific bonding systems later in this review. In general,
monomers such as HEMA (hydroxyethyl methacrylate), BPDM (biphenyl
dimethacrylate), and PMDM (pyromellitic diethylmethacrylate) are incorporated in
primers at concentrations of 15-55% (Van Meerbeek, 1992).
Classification of Dentin Bonding Systems

Dentin bonding systems have been classified either by their chronological development or by their chemical composition and surface treatment of the dentin substrate (Miyazaki, et al., 1996). To better understand the evolution of the dentin bonding systems they are often grouped into “generations”.

First generation agents were primitive and produced minimal shear bond strengths (< 3 MPa) (Suh, 1992). These early products utilized N-phenylglycine glycidyl methacrylate (NPG-GMA), cyanoacrylate, or glycerophosphoric acid dimethacrylate (GAD). NPG-GMA is typical of a bifunctional molecule or coupling agent since one end can bond into dentin and the other end can polymerize to the composite resin (Albers, 1996). Chemical bonding of the adhesive resin to constituents of the dentin was the proposed mechanism of action. For example, it was suggested that the NPG-GMA bonded to calcium and the cyanoacrylates adhered to collagen (Farrah and Powers, 1995). Intra-oral hydrolysis of the GAD and instability of the NPG-GMA in solution as well as the low bond strength were some of the problems that precluded their use in clinical practice (Miyazaki, 1996).

Second generation systems used polymerizable phosphates added to bisphenol glycidyl methacrylate (Bis-GMA) resins. Adhesion was based on the chemical bonding of phosphorus esters to calcium and isocyanates to collagen. Materials in this group such as Scotchbond (3M Corporation, St. Paul, MN) did
not use dentin primers or conditioners to facilitate penetration of the resin. However, Scotchbond did incorporate ethanol in the liquid that was mixed with the resin prior to application on the tooth surface. This lowered the viscosity of the adhesive resin and improved the wettability of the hydrophobic Bis-GMA resin (Albers, 1996). The bond strengths were found to be dictated by the cohesive strength of the smear layer and were usually in the 5 to 7 MPa range (Pashley et al., 1992).

In the late 1980’s the third generation materials appeared. Most of these agents contained a dentin primer or conditioner which dissolved the smear layer either totally or partially (Van Meerbeek et al., 1998). Scotchbond 2 (3M Corporation) utilized a primer containing 2.5% maleic acid, 55% HEMA and 40% water (Albers, 1996). This allowed for improved wetting, and deeper penetration of the adhesive resin into the conditioned dentin (Finger et al., 1994). The adhesive resin was water compatible and consisted of 62.5% Bis-GMA and 37.5% HEMA (Albers, 1996). Scotchbond 2 utilized a primer containing hydroxyethyl methacrylate (HEMA) which removed the outer dentin surface or smear layer. This dentin pretreatment resulted in micromechanical retention between dentin and resin. Bond strengths reported for third generation materials were as high as 18 MPa (Miyazaki, 1996). This represented a significant improvement over the earlier materials.

Fourth generation dentin adhesive systems appeared in the early 1990’s. Both the enamel and dentin are acid treated. This results in removal of the smear layer, demineralization of the surface dentin and exposure of the collagen fibrils.
The primer then penetrates this exposed collagen network and improves wetting. When the bonding resin is applied a "hybrid" layer of resin and collagen results. Once again bond strengths were significantly increased with most values over 20 MPa (Suh, 1991) and some reported values as high as 27 MPa (Miyazaki, 1996). Bond strengths had now approached what can be achieved with enamel bonding. When All-Bond 2 was initially introduced, Suh (1991) listed the ability to bond to materials other than enamel and dentin as additional evidence that a new (fourth) generation of materials had been developed.

Recently, a fifth generation of dentin bonding materials has been introduced. These systems combine the dentin primer with the adhesive resin into a single component and therefore, are also classified as "single-bottle" adhesives. The purpose of these materials is to simplify the adhesion process while maintaining the high bond strengths achieved with the fourth generation materials. Some researchers, however, are questioning the inference that fewer components directly translates to quicker and easier application (Fritz and Finger, 1999). At present, research is still required to substantiate the manufacturers' claims regarding the efficacy of these single bottle materials (Vargas et al., 1997). Further discussion of the fourth and fifth generation materials utilized in this study will be provided later in this literature review.

**Visible Light Resin Curing Systems**

The setting of Bis-GMA and other monomers is achieved through free radical polymerization. Free radicals can be generated in a number of different ways (Figure 2). Most modern restorative resins utilize a visible light curing
system and camphoroquinone (CQ) as the source of free radicals. A 468 +/- 20 nm wavelength (blue) light source excites CQ into a state that interacts with a tertiary amine such as N,N-dimethylaminoethylmethacrylate (DMAEMA) and free radicals are generated. When a free radical collides with the resin monomer’s carbon double bond, the free radical pairs with one of the electrons of the double bond leaving the other members of the pair free and reactive thus continuing the reaction (Albers, 1996).

**Chemical:**

Benzoyl Peroxide + 2% aromatic tertiary amine = Free Radical

**UV Light:**

0.1% Benzoin alkyl ether + 365 nm UV light source = Free Radical

**Visible Light:**

0.06% Camphoroquinone +
0.1% aromatic or 0.04% aliphatic tertiary amine +
468 +/- 20 nm. Visible light source = Free Radical

**Figure 2.** Chemical reactions that produce free radicals in three composite curing systems. Taken from: Tooth-Colored Restoratives 8th edition, Albers HF, Alto Books, Santa Rosa, CA, 1996.

Even though visible light cured materials offer many advantages there is the risk of insufficient conversion of the monomer into copolymer, and non-uniform polymerization shrinkage resulting in compromised physical properties. There are many factors which may negatively affect the degree of conversion (DC) including insufficient light intensity at the source, too short an irradiation time, and light absorption by the restorative material or the surroundings of the
restoration (Davidson-Kaban et al., 1997). However, it has also been shown that the more conversion there is in the light cured composite material, the greater the polymerization shrinkage (Ruyter, 1985). This problem draws attention to the fact that although it is desirable to maximize physical and mechanical properties it is also desirable to minimize polymerization contraction. Often, optimizing one side of this equation results in compromising the other (Sakaguchi and Berge, 1998).

High intensity lights provide higher values for degree of conversion and improve physical properties, so that the trend has been to construct units that provide higher intensity light. However, they also produce higher contraction strain rates during polymerization which may result in increased internal stress and microleakage (Sakaguchi and Berge, 1998). Composites cured with low intensity light have a better marginal adaptation but the physical properties such as compressive strength are negatively affected (Unterbrink and Muessner, 1995).

Some researchers suggest that a method of minimizing contraction is to allow flow of the composite resin during setting by means of controlled polymerization (Mehl et al., 1997; Sakaguchi and Berge, 1998). One method of achieving this is to prepolymerize at low light intensity followed by a final cure at a higher intensity.

Mehl et al. (1997) studied the effect of initially curing the composite restorative materials Tetric (Vivadent, Liechtenstein) and Charisma (Kulzer, Germany) with low intensity light followed by curing at 450 mW/mm². They varied
the initial light intensity, initial light curing time, and high intensity light cure time. Varying the light intensity was achieved by moving the light source a given distance away from the composite sample and the intensity on the top of the sample was determined by a radiometer. Samples of the cured composite resins were subjected to three point bending tests to determine the flexural modulus and to a microhardness tests. Additionally, class V restorations were placed in freshly extracted human molars, thermocycled and then examined for marginal gap formation and microleakage. From their results the authors concluded:

- "Initial cure with intensities as low as 225mW/cm² (50% of the maximum light intensity, 450mW/cm², used in the study) for 20 or 40 seconds do not decrease flexural strength and flexural modulus for either material."

- "Initial cure has no effect on microhardness"

- In the case of Tetric, the study demonstrated that initial cure at 56% and 70% of the maximum intensity (450mW/cm²) for 20 seconds followed by final cure at 100% for 40 seconds significantly reduced the marginal gap of the dentin–composite junction of class V cavities.

- "Softstart polymerization is a reliable method to combine better physical properties with increased marginal integrity of composite fillings at the dentin–composite junction."

Mehl and his colleagues found that an initial cure intensity that was too high did not allow enough flow to reduce internal stress, whereas a light intensity that was too low did not activate enough initiator molecules to start an adequate reaction. In both cases the resin material behaved as it would if exposed to high intensity light only. The authors also suggested that different composite materials would respond differently since the initiator concentrations and chemistry differ.
Sakaguchi and Berge (1998) studied the relationship between curing light energy density, degree of conversion and post-gel polymerization to test the hypothesis that the degree of contraction and conversion is related to the method by which light is delivered not solely on total light density. They found that the application of light at two intensities resulted in degree of conversion values that were not significantly different from those cured at either the higher or lower intensity for 40 seconds. However, the sample cured with two intensities showed a 21.8% reduction from the contraction strain that the authors had mathematically predicted. Another interesting finding was that the light source used in the study steadily diminished in intensity once activated. The light intensity at 40 seconds was 77.4% of the maximum recorded immediately after activation. This decay of the light intensity demonstrates that it is possible to have substantial error in calculating the true energy density if the intensity is only measured at the beginning or end of the curing period.

Polymerization force and degree of conversion with a two-step curing protocol was also investigated by Bouschlicher et al. (2000). The maximum shrinkage force during a 300 second period starting at the initiation of photocuring was recorded as was the degree of conversion. Forty seconds of curing at 800 mW/cm² was compared to 10 seconds at 100 mW/cm² + 30 seconds at 800 mW/cm². They found that mean maximum shrinkage force decreased slightly, but not significantly, with the two-step method and no effect on degree of conversion was noted.
Koran and Kurschner (1998) also investigated the effect of a two-phase light cure, this time on Pertac (ESPE, Inc., Seefeld, Germany) resin composite. The parameters investigated were surface hardness, shrinkage, viscosity, degree of polymerization and adhesion to a pretreated metal surface. They found that the two-step cure does not affect shrinkage, surface hardness, or residual monomer concentrations compared with the conventional continuous curing approach if the total irradiation is high enough to achieve complete polymerization. A lower viscosity was achieved and this allowed better material flow during the early stages of curing. Additionally, adhesion to stainless steel was strongest using the two-step approach with initial cure utilizing a light density of 150 mW/cm² for a 10 second period and then 30 seconds at 700 mW/cm². The least adhesive experimental group was continuous cure at 700 mW/cm² for 40 seconds. The authors qualified their results, however, by stating that further study utilizing adhesion to dental tissues was required to confirm this trend.

**Shear Bond Strength Testing**

Shear and tensile bond strength tests are commonly used to evaluate dentin bonding agents. These tests often produce results with large coefficients of variation (Van Noort et al., 1989; ISO/TC Technical Report, 1991; Miyazaki et al., 1996). This is due to the large number of factors that can influence the result of such tests (see Figure 1 – Flow chart of interrelated parameters on dentin bond strengths). The remainder of this discussion will centre on information that has direct bearing on this study.
In Retief’s (1991) discussion on laboratory adhesion tests he notes that both tensile and shear testing have been commonly utilized but shear bond strengths are currently considered more predictive. However, the forces exerted on restorations in vivo are very complex in nature so neither approach simulates the oral environment.

Van Noort et al. (1989) investigated the sensitivity of bond strengths to changes in testing conditions using finite element stress analysis. They found that there are two potential sources for scatter in measurement in nominal bond strength. First, there may be scatter due to differences in the stiffness of the composite resin used for the cylinder and variations in the point of load application. Secondly, small changes in the geometric arrangement and the mode of load application, will contribute to the variation in the data produced by different researchers. They also note that conventional tests to determine bond strength report the value of the nominal stress at failure, that is the fracture load per unit area of bonded surface. The nominal strength is actually controlled by the attainment of a critical stress locally at the most sensitive place, usually the edge of the bonded area.

Fowler et al. (1992) compared tensile and shear tests on bonded human and bovine dentin. They found no significant difference between the two modes of testing, except that the shear test produced more adhesive fractures. As a result, the authors suggested the use of shear test for adhesion testing since it appeared to be more likely to produce failure at the tooth-adhesive interface.
Their study also noted a slightly higher bond strength when bovine dentin was the substrate as opposed to human dentin.

As indicated earlier in the review of thermocycling parameters, the ISO (1991 and 1994) has attempted to standardize many of the aspects of bond strength testing. They suggested storage times of 24 hours in water at 37° C for short term testing and 180 days storage in water at 37° C for long term testing prior to application of shear or tensile forces. The raw data should be converted into stress units, i.e. force per unit area (MPa), for interpretation of results. In spite of the committee recommendations, a wide variety of storage protocols are utilized by laboratories.

Storage Media

Rueggeberg (1991), Miyazaki et al. (1996) and Titley et al. (1998) list several storage media that have been employed in bond strength studies (Table 3).

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
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</thead>
<tbody>
<tr>
<td>1.</td>
<td>&quot;Water&quot;, distilled water (24°C, 37°C, 60°C)</td>
</tr>
<tr>
<td>2.</td>
<td>30% sterile saline, saline, normal saline, saline at 37°C</td>
</tr>
<tr>
<td>3.</td>
<td>Distilled water and thymol</td>
</tr>
<tr>
<td>4.</td>
<td>Media containing formalin, chloramine, ethanol, physiological saline, thymol, sodium hypochlorite, methanol, glutaraldehyde and sodium azide.</td>
</tr>
</tbody>
</table>

**Table 3.** Storage solutions utilized in *in vitro* bond strength studies after application of restorative material (Rueggeberg, 1991; Miyazaki et al., 1996; Titley et al., 1998).
The effect of storage media utilized before bonding

The ISO reports (1991 and 1994) on guidance for the testing of adhesion of dental materials to tooth structure offered a number of recommendations on the handling of teeth prior to bonding. The teeth should be thoroughly washed in running water and then stored in distilled water or a 0.5% chloramine solution for at least one week. Thereafter, they should be stored in distilled water at 4°C and the water should be replaced periodically. No other chemical agents should be used since they may alter the tooth substance. The report also recommended use of teeth as soon as possible after extraction and no longer than six months post extraction.

Teeth from human or bovine sources are contaminated with bacteria and have the potential for the transmission of communicable diseases (Schulein, 1994). Therefore, it is necessary to disinfect these teeth prior to use. Additionally, investigators often attempt to chemically suppress bacterial growth in storage media utilized during bond strength studies. In Rueggeberg’s (1991) literature review a number of studies were described which looked at the effect of adding disinfecting chemicals to storage media. He concluded that with the exception of a few studies the researchers showed that the storage media does not effect the shear bond strengths of dentin bonding agents to extracted teeth.

In a recent study, Titley et al. (1998) investigated the effect of several storage media on shear bond strength utilizing Scotchbond Multi-Purpose (3M Corporation, St. Paul, MN, USA) on bovine dentin. The highest bond strengths
were achieved when the teeth used were either fresh (23.40 MPa) or frozen and thawed immediately prior to use (20.82 MPa). Bond strengths from fresh bovine teeth were found to be significantly higher than those attained from 0.05% thymol (15.83 MPa), 70.0% methanol (13.89 MPa), glutaraldehyde 2.00% (10.14 MPa), or teeth that were irradiated with gamma radiation (12.85 MPa). The authors concluded that their results support the recommendations of the ISO committee (1991 and 1994).

The effect of storage media utilized after bonding

Rueggeberg’s literature review (1991) noted that there are conflicting opinions regarding whether storage affects bond strength. Chan et al. (1985), Munksgaard and Asmussen (1984), and Zidan et al. (1987) found no change in dentin bond strength after storage for up to one year. Aquilino et al. (1987) reported decreases in bond strength of up to 45% after 11 months’ storage in water, showing a degradation of the composite bond surface. It should be noted, however, that these studies involved some of the earliest adhesive materials so that the results should be viewed with caution. Soderholm et al. (1984) reported that composites undergo both hydrolytic degradation in water and leaching of fillers.

Chan et al. (1997) investigated the effect of 180 day storage in distilled water on the shear bond strength of Scotchbond Multi-Purpose to demineralized bovine dentin. When phosphoric acid of various concentrations was used to condition the dentin, shear bond strengths at 180 days were significantly lower
than samples stored for only 24 hours. Interestingly, when maleic acid at concentrations 1% or greater was used instead of phosphoric acid the samples stored long term did not demonstrate a decrease in bond strength. In fact, the application of 10% maleic acid for 15 seconds then rinsing resulted in long term bond strength being significantly greater than short term. The authors speculate that the solubility of the reaction products produced by these acids may be responsible for the difference in long term storage results.

Carracho et al. (1991) studied the effect of storage in normal saline at 37°C on the bond strength of three dentin adhesives. Time of storage did not affect the shear bond strength of Scotchbond 2 and Scotchbond Dual Cure however, the bond strength of Mirage Bond (Chameleon Dental Products) was significantly improved after one month of storage. They postulated that water sorption over time may enhance contact between dentin and adhesive, which may result in greater adhesive strengths.

The effect of frequently changing the storage solutions in long term storage tests was investigated by Kitasako et al. (2000). Panavia 21 (Kuraray Co.), BISTITE (Tokuyama Co.), and MASA Bond (Experimental material, Sun Medical Co.) were applied to prepared bovine dentin surfaces and stored for one year in either water or phosphate buffered saline. Some of the stored samples had the solution changed daily while the remainder were not disturbed for the entire year. No other agents were added to the solutions. No statistical difference in shear bond strength was noted between the water and saline groups. However, the shear bond strength for the changed storage solution groups were
significantly lower than the unchanged solution groups. The authors made the following conclusions:

1. Hydrolysis may have been accelerated at the interface between the hybrid layer and dentin and between the hybrid layer and resin cement.

2. Changing the storage solution might induce the loss of calcium from the dentin, resulting in further exposure of the dentin collagen.

This paper demonstrates that the method of storage affects the bond strength but does not suggest which method is more appropriate. *In vivo* oral fluids are changed rapidly. However, saliva in an oral environment is a much more dynamic solution than those used in *in vitro* storage investigations. Therefore, neither changing the solution daily nor leaving it untouched represents an approximation of the oral environment.

Researchers in Japan have investigated the effects of very long term storage on bonded dentin. In Nakabayashi and Pashley's "Hybridization of Dental Hard Tissues" (1998), a 1987 study is described whereby bovine dentin is conditioned with a solution of 10% citric acid and 3% ferric chloride for 30 seconds, bonded with 4-METAMMA-TBB resin, then stored in water at 37°C for up to 5 years. A gradual reduction in bond strength from 18-10 MPa to 4 MPa was noted after 5 years. SEM examination demonstrated failure both between the top of the hybrid layer and the overlying resin and between the bottom of the hybrid layer and the underlying mineralized dentin. Additionally, an amorphous layer was noted adjacent to the hybridized dentin that may be the result of overetching. Dentin etched for 10 seconds does not have this defective zone. The authors suggest that the reduction in bond strength may be due to hydrolysis
of the dentinal collagen over many months. Lastly, Nakabayashi and Pashley (1998) suggest that this type of study must be repeated utilizing more current bonding procedures.

**Bonding Systems Used In This Study**

Five dental adhesive systems were used in this study. All-Bond 2 (Bisco Dental Products, Itasca, IL, USA) and Scotchbond Multi-Purpose (3M Dental Products, St. Paul, MN, USA) are commonly used fourth generation dentin adhesives. Prime and Bond 2.1 (L.D. Caulk-Dentsply, Milford, DE, USA), Single Bond (3M Dental Products), and One-Step (Bisco Dental Products) are representatives of the new single bottle fifth generation materials. The components of these products are listed in Table 4 (page 32).
<table>
<thead>
<tr>
<th>Product</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scotchbond Multi-Purpose</td>
<td>Primer: 55% HEMA, 13% polyalkenoic acid copolymer, 40% water</td>
</tr>
<tr>
<td></td>
<td>Adhesive: 62.5% Bis-GMA, 37.5% HEMA, &lt; 1% Photoinitiator</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>Primer A: 2.1% NTG-GMA, 67% acetone, 25% ethanol, 5% water</td>
</tr>
<tr>
<td></td>
<td>Primer B: 24% BPDM, 20% ethanol, photoinitiator, acetone</td>
</tr>
<tr>
<td></td>
<td>Adhesive: 60% Bis-GMA, 10% HEMA, 30% TEGDMA</td>
</tr>
<tr>
<td>Prime and Bond 2.1</td>
<td>UDMA, PENTA, bisphenol A dimethacrylate, acetone, cetylamine hydrofluoride, photoinitiator</td>
</tr>
<tr>
<td>Bisco One-Step</td>
<td>BPDM, Bis-GMA, HEMA, acetone, photoinitiator</td>
</tr>
<tr>
<td>3M Single Bond</td>
<td>Bis-GMA, HEMA, water, ethanol, polyalkenoic acid copolymer, photoinitiator</td>
</tr>
</tbody>
</table>

BPDM = Biphenyl dimethacrylate  
BisGMA = biphenol glycidyl methacrylate  
HEMA = 2-hydroxyethylmethacrylate  
NTG-GMA = N-tolyglycine-glycidyl methacrylate  
PENTA = dipentaerythritol penta acrylate monophosphate  
TEGDMA = tetraethylene glycol dimethacrylate  
UDMA = urethane dimethacrylate

Table 4. Components of the bonding systems used in this study.
Fourth Generation Adhesive Systems

Scotchbond Multi-Purpose

Scotchbond Multi-Purpose (SBMP) was introduced in 1992 and soon became one of the most popular adhesive systems in both clinical practice and in laboratory evaluations. For example, the 1994 IADR meeting in Seattle, Washington had 55 reports presented that pertained to the SBMP system (SBMP Dental Adhesive: Results published at the 1994 IADR). At present, hundreds of studies have been performed utilizing this product. This makes SBMP a logical choice as a control material in the evaluation of newer systems. In general, the bond strength to dentin for SBMP reflects the results shown in the previously mentioned IADR meeting where a range of 9.5 MPa to 21.5 MPa was presented.

SBMP consists of a three step application procedure of etching, priming, and then bonding. Until recently SBMP utilized 10% maleic acid as the etchant. However, 35% phosphoric acid is now supplied by the manufacturer in spite of evidence that maleic acid was a superior etchant for this product (Chan et al., 1997). The primer contains HEMA and a polyalkenoic acid co-polymer which resembles the modified polyacrylic acid found in some glass ionomer cements. The manufacturer claims that the addition of the copolymer aids in resisting the detrimental effects of moisture in a high relative humidity environment (SBMP Technical Product Profile, 1994; Fundingsland et al., 1992). The mechanism of this resistance to moisture was introduced by Peters et al. (1974) who suggested that the polyacrylic acid based systems had bonds which constantly form and
reform with the calcium ions. Apparently a dynamic equilibrium is formed at the interface in the presence of water which favours ion exchange reactions. Consequently moisture may not significantly affect the bonding procedure. Further confirmation of this is provided in a study by Burrow et al. (1995). These investigators demonstrated that environmental conditions such as that found in the oral cavity (30°C and 80% RH) did not adversely affect the tensile bond strength of SBMP on bovine dentin. Lastly, the manufacturers note that the adhesive is water compatible which enhances wetting.

**All-Bond 2**

All-Bond 2 was one of the first fourth generation dentin bonding systems to become available and differs from SBMP in a number of areas. Firstly, the system is provided with both 10% and 32% phosphoric acid semigel etchant. Both preparations are thickened with a polymer as opposed to silica or oxalate in an effort to minimize contamination of the etched dentin surface (Kanca, 1993). Additionally, the antibacterial agent benzalkonium chloride (BAC) is included in these etchants. Albers (1996) concedes that the BAC has demonstrated zones of bacterial inhibition in laboratory studies but questions whether it has any clinical significance.

Polymerization of the BPDM is initiated by mixing with NTG-GMA, which is not only a hydrophilic monomer but also a potent tertiary amine accelerator. Additionally, any self-curable material or resin placed on top of this BPDM-NTG-GMA layer will have their polymerization accelerated (All-Bond 2 Technical Manual, 1995). The mixed primer is both light-cured and self-cured, however
since the self-curing reaction is relatively slow, light curing for 10-20 seconds is recommended to increase the degree of conversion. Work performed by Bisco Dental Products has demonstrated that the self-cure properties of the mixed primers results in a Degree of Conversion (DC) of up to 35% in 10 minutes at room temperature. However, light-curing for 10 seconds after 3 minutes of self-curing instantly brings the DC up to 52% and eventually to 60% in 7 minutes. The manufacturer states that a faster initial rate of curing, as well as a higher overall DC, would be realized at 37°C from the combined effects of free radical initiation, faster polymerization propagation and reduced system viscosity at the higher temperature.

In house research by Bisco Corporation states that the resulting layer of primer resin on dentin is quite thin (approximately 5-6 microns). Normally, the curing of such a thin layer would be severely inhibited by the ambient oxygen. However, calculation of the FTIR (Fourier transform infrared spectroscopy) absorption spectra by the manufacturer before and after acetone rinse shows 75% cured and 25% uncured due to air inhibition. A still higher DC may be expected clinically if the primers were cured with a layer of bonding resin covering them as would be the case in restorative procedures (All-Bond 2 Technical Manual, 1995).

The adhesive resin supplied with the All-Bond system can also become dual curable with the addition of the Pre-Bond component. Pre-Bond is an unfilled resin composed of Bis-GMA, HEMA, and a peroxide. Bisco suggests that
this component can be added if the surface activity of the primed dentin needs to be reduced as in the use of a composite luting cement.

The adhesive strength of this product to dentin has been evaluated by a number of laboratories (Table 5, page 37). The reports exhibit a relatively wide range of values however, most were between 15 and 30 MPa depending on the type of substrate, method of application, and experimental conditions.
<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Dentin Treatment</th>
<th>Shear Bond Strength (mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wilder et al., 1998</td>
<td>Bovine dentin</td>
<td>15s etch, rinse, blot, 5 consecutive coats, then dry, LC 10s</td>
<td>16.3 ± 7.6 MPa</td>
</tr>
<tr>
<td>Hosoya et al., 1997</td>
<td>Permanent and primary bovine dentin</td>
<td>15s, rinse, air dried, 5 consecutive coats, dried, LC 20s</td>
<td>Primary: 30.77 ± 11.48 MPa to 35.46 ± 14.67 MPa Permanent: 13.85 ± 4.25 to 19.61 ± 6.53 MPa</td>
</tr>
<tr>
<td>Kanca, 1993</td>
<td>Buccal human molar dentin</td>
<td>20s etch (Bisco liquid), rinse, either dried completely or left moist, 5 consecutive coats, dried 5s, LC 20s</td>
<td>Moist: 36.1± 3.6 MPa Dry: 19.8± 2.9 MPa</td>
</tr>
<tr>
<td>Gwinnett, 1994</td>
<td>Human third molar dentin</td>
<td>20s etch, rinse, several different degrees of dentin moisture, applied primer for 10s, dried, adhesive, LC 20s</td>
<td>Wet: 24.20 ± 2.03 MPa Lightly dried: 19.83 ± 5.42 MPa Aggressively dried: 11.56 ± 4.93 MPa</td>
</tr>
<tr>
<td>Kanca, 1991</td>
<td>Human molar dentin</td>
<td>25-30s etch, rinse, either dried or left moist, 5 consecutive coats, dried 5s, LC 20s</td>
<td>Moist: 29.34 ± 4.09 MPa Dry: 16.11 ± 6.42 MPa</td>
</tr>
</tbody>
</table>

Table 5. Shear bond strength of All-Bond 2 reported in selected studies.
Fifth Generation (One-Bottle) Adhesive Systems

Prime and Bond 2.1

One of the first adhesive systems that combined the primer and bonding resins into a single component was Dyract PSA (Dentsply/DeTrey) which was later introduced into North America as Prime & Bond. This system used an acetone solvent with dipentaerythritolpentacrylate phosphoric acid ester (PENTA) as the primary adhesion promoter. This material may act as a low pH self-etching agent and have the ability to penetrate through the smear layer and onto the top of the intact dentin. Prime & Bond 2.1, which was introduced in the mid-1990's, has had elastomeric resins added to form a combination of relatively rigid and relatively flexible molecules compared to Dyract PSA. Also, cetylamine hydrofluoride was included and provides a source of fluoride ions (Barkmeier et al., 1999). As yet, there have not been a great number of studies investigating the clinical performance of this single-bottle system. Table 6 summarizes recent bond strength investigations utilizing Prime and Bond 2.1.

In the study performed by Barkmeier, et al. (1999) the effect of several different surface dentin conditions on shear bond strength were investigated. Interestingly, they found that acid conditioning of the dentin did not increase bond strength and that high bond strengths were achieved whether the surface dentin was left moist or not. The authors postulate that the ability of Prime & Bond 2.1 to bond to unetched, dry dentin is due to the low pH PENTA acting as a “self-etching” agent.
<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Dentin treatment</th>
<th>Shear Bond strength (mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Miyazaki et al., 1998</td>
<td>Bovine dentin</td>
<td>20s etch, rinse, dry, 30s application, dry, LC 10s (twice)</td>
<td>12.0 ± 2.6 MPa without TC.</td>
</tr>
<tr>
<td>Wilder et al., 1998</td>
<td>Bovine dentin</td>
<td>15s etch, rinse, dry, applied for 20s, dry, reapply adhesive, LC 10s</td>
<td>15.5 ± 3.0 MPa</td>
</tr>
<tr>
<td>Vargas et al., 1997</td>
<td>Flat human third molar dentin</td>
<td>15s etch, rinse, quick dry, applied for 20s, dry, reapply adhesive, LC 10s</td>
<td>22.27 ± 3.8 MPa</td>
</tr>
<tr>
<td>Perdigão et al., 1999</td>
<td>Bovine dentin</td>
<td>15s etch, rinse, blot, applied for 20s, dry, LC 10s, reapply, air-dry,</td>
<td>17.8 ± 4.1MPa</td>
</tr>
<tr>
<td>Barkmeier et al., 1999</td>
<td>Flat buccal dentin of human molars</td>
<td>Several different dentin treatment protocols</td>
<td>18.6 ±3.3 MPa to 21.3 ±4.2 MPa</td>
</tr>
</tbody>
</table>

**Table 6.** Shear bond strengths achieved in in vitro studies of Prime & Bond 2.1

**Single Bond**

Single Bond is the single bottle system manufactured by 3M. It is a solution of water, ethanol, HEMA, BisGMA, dimethacrylates, a novel photoinitiator system, and a methacrylate functional copolymer of polyacrylic and polyitaconic acids which had first been introduced with 3M's Vitrebond products.
This product has been only recently introduced and as a result only a small number of bond strength studies have been performed to date. Table 7 lists four recent shear bond strength studies utilizing Single Bond. Vargas et al. (1997) utilized human teeth in the study of 8 bonding systems including both Single Bond and Bisco One-Step. In this study Single Bond provided the strongest bond (22.27± 4.5 MPa) and was found to be significantly higher than the bond strength to Prime & Bond 2.1 (12.3 ± 3.8 MPa). Interestingly, they found that even the bond provided by SBMP (18.8 ± 8.3 MPa) was inferior to the single bottle 3M product, although the difference was not significant. These early results appear promising, however, the scarcity of literature on this material highlights the need for further study.

<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Dentin Treatment</th>
<th>Shear Bond Strength (Mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Swift and Bayne (1997)</td>
<td>Bovine dentin</td>
<td>15s etch, 5s rinse, two coats applied, air dry 5s, LC period not reported</td>
<td>19.2 ±4.3 to 23.2 ±2.7 MPa depending on the amount of moisture present on dentin</td>
</tr>
<tr>
<td>Miyazaki et al. (1998)</td>
<td>Bovine dentin</td>
<td>15s etch, rinse, two coats applied, dried, LC 10s</td>
<td>15.0 ±2.21 to 9.9 ±0.9 MPa</td>
</tr>
<tr>
<td>Price et al. (2000)</td>
<td>Human molar dentin</td>
<td>15s etch, rinse, two coats applied, blot dried, LC 10s</td>
<td>21.3±3.1 MPa (when used with 2mm Z100 resin)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>16.8±3.1 MPa (when used with 5mm Z100 resin)</td>
</tr>
<tr>
<td>Vargas et al. (1997)</td>
<td>Human third molar dentin (occlusal surface)</td>
<td>15s etch, rinse 10s, left moist, two coats applied, dried 5s, LC 10s</td>
<td>22.27 ±4.5 MPa</td>
</tr>
</tbody>
</table>

Table 7. Bond strength studies utilizing 3M Single Bond.
One Step

One Step is the single bottle product produced by Bisco and was introduced at approximately the same time as Single Bond. It contains BPDM, which is also found in primer B of All-Bond 2, as well as BisGMA and HEMA. Shear bond strength studies of this product are described in Table 8. Wilder et al. (1998) included Bisco One Step in their investigation of the shear bond strength of several fourth and fifth dentin bonding agents. The bond strength to bovine dentin (15.4 ±5.6 MPa) was the second lowest of the seven materials tested, however none of the materials demonstrated bond strengths statistically different from each other. This study is an interesting initial investigation, however only 100 bovine teeth were used for the entire study and none of the samples were thermocycled or stored for greater than 48 hours. Perdigão et al. (1999) compared the shear bond strength to bovine dentin of four new single bottle adhesives to One Step. After 24 hours storage and thermocycling for 500 cycles One Step produced the lowest shear bond strength. Once again, investigation of long term bond strength of this product has not been performed. The paucity of literature on the fifth generation products clearly highlights the need for further study.
<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Dentin Treatment</th>
<th>Shear Bond Strength (Mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vargas et al. (1997)</td>
<td>Human third molar dentin (occlusal surface)</td>
<td>15s etch, rinse, 10s, left moist, two coats applied, dried 10s, LC 10s</td>
<td>15.8 ±5.7 MPa</td>
</tr>
<tr>
<td>Swift and Bayne (1997)</td>
<td>Bovine dentin</td>
<td>15s etch, rinse, two coats applied, dried 10s, LC 10s</td>
<td>13.3 ±3.0 to 20.5 ±3.3 MPa depending on the amount of moisture present on dentin</td>
</tr>
</tbody>
</table>
| Wakefield et al. (1998) | Permanent human molar dentin | 15s, rinse, brief air dry, two coats applied, dried 5-10s, LC 10s, adhesive remaining on brush applied, air dried | Superficial dentin: 16.8±4.7 to 22.2±7.6 MPa  
Deep dentin: 12.1±2.6 to 13.1±6.4 MPa  |
| Perdigão et al. (1999) | Bovine dentin                  | 15s etch, rinse for 5s, blot, two coats applied, dried for 5s, LC 10s             | 14.8 ±4.1 MPa             |
| Miyazaki et al. (1998) | Bovine dentin                  | 15s etch, rinse, two coats applied, dried, LC 10s                                | 9.7 ±1.6 to 16.1 ±2.8 MPa depending on number of thermocycling cycles |

Table 8. Bond strength studies utilizing Bisco One Step.
Statement of the Problem

A great deal of published literature exists in the area of dentin adhesion however, the review of the literature suggests three areas where further investigation is required. Firstly, the long term durability of bonds to dentin achieved by one-bottle adhesive systems have not been investigated. Most studies report adhesive strengths obtained after a few hours or days after photopolymerization. A high initial bond strength is necessary to resist polymerization shrinkage of the restorative resin. However, it is essential that the bond remain intact over the long term if it is to be successful clinically. This study compares the shear bond strength obtained after long term storage in water of several single-bottle products with commonly used fourth generation systems. Secondly, the effect of two step curing protocols on the shear bond strength of adhesive systems is unclear. Given the recent commercial introduction of photopolymerization units capable of two step curing, further research in this area is necessary. Finally, further efforts are required to establish standardized laboratory protocols for the testing of newly introduced materials. Inter-investigator variation in laboratory procedure makes the comparison of results from different laboratories difficult. Previous work in this laboratory (Titley et al., 1994; Titley et al., 1998; Chan, 1994) has resulted in a reproducible method for the study of adhesion to dental tissues. The present study seeks to refine and expand this standardized testing protocol.
**Aims And Objectives**

**Part 1.a – Long Term Storage Study**

- To determine if storage in distilled water and thymol for 270 days at 37° C significantly affects the SBS of selected dentin bonding agents to bovine dentin as compared to short term storage controls.

- To determine if the SBS produced by fourth and fifth generation adhesive systems are comparable after 270 days of storage in distilled water and thymol at 37° C.

**Part 1.b – Thermocycling Study**

- To determine if thermocycling significantly affects the shear bond strength (SBS) of selected fourth and fifth generation dentin bonding agents to bovine dentin as compared to non-thermocycled controls.

- To determine if the SBS to bovine dentin provided by fourth and fifth generation dentin bonding systems is equally affected by a thermocycling protocol.

- To determine if thermocycling early (24 hours) or late (6.5 days) in a 7 day storage period results in significantly different SBS to bovine dentin.

**Part 2 – Dual Cure Bond Strength Study**

- To investigate whether the SBS of adhesive systems to dentin are different when the restorative resin is photo-polymerized by a single high intensity light source as compared to a dual cure “soft start” system.

- To determine if the fourth and fifth generation adhesive systems produced by 3M, Single Bond and Scotchbond Multi-Purpose, respond similarly to two step curing protocols.

- To compare the SBS of the 3M fifth generation system, Single Bond, to Scotchbond Multi-Purpose after 7 days storage in distilled water and thymol at 37° C.

**Part 3 – Microhardness Study**

- To demonstrate that the Knoop microhardness of Z100 composite resin (3M), which has been photo-polymerized by a dual cure system, is similar to the microhardness of resin cured by a single high intensity light source.
To investigate if material thickness of 1.5 mm, 3.0 mm, and 4.5 mm result in reduced Knoop microhardness when visible light cured by either a single or dual cure regimen.

To determine if the Knoop microhardness measured immediately after visible light curing is significantly different than the microhardness obtained from the samples after storage in distilled water with thymol at 37°C for 24 hours or 7 days.
Materials And Methods

1. Thermocycling and long-term bond strength studies

Tooth preparation

Freshly extracted bovine incisors were obtained ex abattoir and frozen in distilled water in accordance with a protocol developed in this laboratory (Titley et al., 1998). The teeth were thawed immediately prior to use. The teeth were decoronated with a band saw, the pulps removed from the crowns with a spoon excavator and a wet cotton pledget placed into the pulp chamber (Figure 3, page 57). The teeth were used within three months of harvesting.

Teeth used in the bond strength study were embedded in Auto Polymerizing Polymethyl Methacrylate (PMMA) (Esschem Co. Essington, PA) in the following manner. The crowns were affixed to a Plexiglas base with plasticene with the labial surface facing upwards and then placed into a mold 2.5 cm. in diameter and 2.0 cm. in depth. The PMMA was mixed and poured into the molds. The teeth were kept wet throughout the embedding process, and then stored in distilled water at 4°C until used. A small quantity of thymol was added to the water to inhibit bacterial growth (Rueggeberg, 1991).

Dentin surface preparation

The labial superficial dentin was exposed using a water irrigated grinding wheel with #180 and then #320 grit SiC paper. The final dentin surface was smoothed with #600 grit water irrigated SiC paper (Pashley et al., 1988; Titley et al., 1994). The embedded teeth were prepared using a jig that ensured that the test surface was parallel to the surface of the mounting material.
Bond Strength Studies

Twelve embedded samples were prepared for each experimental group. Teflon tape was used to limit the surface area of the prepared dentin. A hole 4 mm in diameter was punched into the tape using a leather punch and this was positioned over the dentin test surface (Figure 4, page 57). All materials were applied only within this exposed surface.

The prepared dentin surfaces were conditioned with 35% phosphoric acid gel (3M Corporation, St. Paul, MN) for 15 seconds then rinsed with distilled water for 30 seconds prior to application of an adhesive system. The surface of the dentin was blot dried with a Kim Wipe but left visibly moist prior to application of all adhesive systems (Kanca, 1992; Gwinnett, 1992).

Five adhesive systems were utilized in this study:

- All-Bond 2 (Bisco Dental Products, Itasca, IL)
- One Step (Bisco Dental Products, Itasca, IL)
- Scotchbond Multi-Purpose (3M Corporation, St. Paul MN)
- Single Bond (3M Corporation, St. Paul, MN)
- Prime and Bond 2.1 (Dentsply/Caulk, Milford, DE)

The bonding material was applied according to the manufacturers' instructions in accordance with the protocol shown in Table 9.

Once the adhesive had been applied to the dentin surface a # 4 gelatin capsule (Coni-Snap #4 Natural, Wiler Chemicals Ltd., London, Ontario) (surface area of 0.178 sq. cm., and 3-4 mm in height) was filled with Z100 composite resin, shade A3.5 (3M Corporation, St. Paul MN), and placed over the treated
area. The composite was then visible light cured (VLC) with a Visilux 2 model 5520 AA visible light curing unit (serial number 112669; 3M Corporation St. Paul MN) for 60 seconds from each side and from above the sample. The light output of the Visilux unit was monitored monthly with a “Cure Rite” light radiometer model No. 644726, serial No. 002097 (EFOS Inc., Williamsville, NY 14221).

Twelve teeth were bonded for each experimental group (Figure 5, page 58).

5 groups were bonded for each of the adhesive systems in the study. These teeth were placed in water with thymol at 37° C and exposed to the experimental conditions for that group. The experimental conditions were:

- Group 1 - Thermocycled after 24 hours of storage then placed in storage for 7 days in total before shear strength testing.
- Group 2 - Thermocycled after 6.5 days of storage and then immediately shear tested at 7 days.
- Group 3 - Stored in water for 7 days then shear tested.
- Group 4 - Stored in water for 24 hours then shear tested.
- Group 5 - Stored in water for 270 days then shear tested.

The water and thymol for the long-term (270 day) groups were changed every 2 weeks.
<table>
<thead>
<tr>
<th>Adhesives</th>
<th>Components</th>
<th>Method of Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scotchbond Multi-Purpose</td>
<td>Conditioner / Primer</td>
<td>Dentin etch 15 s</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>Rinse 30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Blot dry on paper towel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2 coats of primer</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 3-5 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Apply adhesive</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VLC 10 s</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>Conditioner / Primers A and B</td>
<td>Dentin etch 15 s</td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>Rinse 30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Blot dry on paper towel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A&amp;B mixed and 5 coats applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 5 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Adhesive applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VLC 10 s</td>
</tr>
<tr>
<td>All-Bond One Step</td>
<td>Conditioner / Primer / Adhesive</td>
<td>Dentin etch 15 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rinse 30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Blot dry on paper towel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2 coats applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 3-5 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Additional layer applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 3-5 s</td>
</tr>
<tr>
<td>Scotchbond Single Bond</td>
<td>Conditioner / Primer / Adhesive</td>
<td>Dentin etch 15 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rinse 30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Blot dry on paper towel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2 coats applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 3-5 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VLC 10 s</td>
</tr>
<tr>
<td>Prime &amp; Bond 2.1</td>
<td>Conditioner / Primer / Adhesive</td>
<td>Dentin etch 15 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rinse 30 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Blot dry on paper towel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Liquid applied for 20 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 5 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VLC 10 s</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Additional layer applied</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Air dry 3-5 s</td>
</tr>
</tbody>
</table>

Table 9. Method of application of the adhesive systems utilized within this study.

The thermocycling parameters utilized in this study are those suggested within the 1991 and 1994 ISO technical reports. The specimens were exposed to 500 cycles between 5±2°C and 55±2°C with a dwell time of 30 seconds in each
bath and a transfer time of 10 seconds utilizing a Haake N2 digital thermocycling machine (Haake Ltd., Serial Number 871836, Germany). The teeth were then replaced in water and thymol for the remainder of the 7 day storage period or immediately shear tested in the case of the 6.5 day specimens.

After the appropriate period the prepared teeth were shear tested to failure on an Instron Universal Testing Machine Model 4301 (Instron Corporation, Canton, MA.) (Figure 6, page 58). Each specimen was kept in water until testing. The polymethyl methacrylate bases were inserted into a fixed base and a loop of prestretched stainless steel wire was placed under the composite cylinder at the resin/dentin interface. The Instron machine was set at a chart speed of 10 cm/min and a crosshead speed of 0.5 cm/min with a load cell of 50 kg and then the samples were shear tested to failure. The peak force was recorded as Newtons and then divided by the surface area of the capsule and bonding interface (0.178 cm²) and the results expressed as megaPascals (MPa). Additionally, the type of bond failure was recorded as either purely adhesive or adhesive/cohesive if the fracture included dentin or composite as noted by examination under 25x light microscopy.

Statistical Analysis

Statistical analysis included calculation of the mean shear bond strengths (SBS) and the standard deviations for each group. A one-way analysis of variance (ANOVA) test was carried out to analyze the following:

- The effect of the type of material on SBS
- The effect of time on SBS
The effect of thermocycling on SBS

Two-way ANOVA was used for the following analyses:

- The effect of time and material on SBS
- The effect of thermocycling and material on SBS

Multiple pairwise comparisons were performed using Duncan’s Multiple Range test. Test of interaction between the effects of the factors was included in the ANOVA design. If a significant interaction occurred between factors, the main effects were retested at each level of the factors using the LSMEANS test. The statistical significance for all tests was set at a 5% level. The SAS System was used for the analysis of the data (prog GLM, version 6.23, 1996, Cary NC).

2. Dual-Cure bond strength study

Tooth and dentin preparation

This study utilized caries free human third molars which had been frozen in water immediately after extraction in accordance with a protocol developed in this laboratory (Titley et al., 1998). The teeth were thawed, approximately half of the root was removed and the teeth were notched at their cervical margins in order to enhance their retention in the embedding resin. Using plasticene the tooth was affixed to a plexiglas base with the occlusal surface facing upwards. They were then placed into a mold 2.5 cm. in diameter and 2.0 cm. in depth. The PMMA was mixed, poured into the molds and allowed to set. After removal from the molds the teeth were stored in distilled water at 4°C until used.

The occlusal surface was progressively ground using a water irrigated grinding wheel with a #180 and then #320 grit SiC paper until dentin was
exposed. The final dentin surface was polished with #600 grit water irrigated SiC paper immediately prior to use. The teeth were kept in distilled water prior to adhesive resin application.

**Resin Materials and Curing Light**

A fourth and a fifth generation single bottle adhesive system were used in this study. The former was Scotchbond Multi-Purpose (SBMP) and the latter Single Bond (SB). The resin composite was Z100, shade A3. All these products are produced by 3M Corporation. The curing light was the ESPE Elipar® Highlight (Figure 7) which can be programmed to render a high intensity cure (700 mW/cm² for 40 seconds) or a two step low intensity “soft start” (150 mW/cm² for 10 seconds) followed by a high intensity final cure (700 mW/cm² for 30 seconds).

**Thickness Template**

Polyvinyl chloride (PVC) mouth-guard material (Biostar, Scheu-Dental, Germany) was used to create templates 1.5, 3.0, and 4.5 mm thick. The 4.5 mm template was created by heating together sheets of 1.5 and 3.0 mm mouth-guard material. The sheets were then cut into pieces 3 cm x 3 cm. Using a leather punch which had been warmed in a Bunsen flame, a 4.0 mm diameter hole was punched into the centre of each template. The 3.0 and 4.5 mm templates were then lined with a cylinder cut from a #4 gelatin capsule and trimmed so that it was flush with the edges of the template (Figure 8).
Resin Application

The embedded teeth were removed from the distilled water and blown dry with compressed air. Teflon tape with a hole 4mm in diameter was applied over the dentin surface. The area of the dentin confined by the tape was etched with 35% phosphoric acid gel (3M Corporation, St. Paul, MN) for 15 seconds and then rinsed with distilled water for 30 seconds. The surface was blot dried but left moist. The adhesive was applied according to the manufacturers instructions and then VLC for 10 seconds at 700 mW/cm² with the curing tip 2-3 mm from the dentin surface. The PVC template was placed over the bonded dentin and filled with Z100 resin so that it was flush with the surface of the template (Figure 9). The curing tip was placed in contact with the resin surface and the Z100 was VLC according to the protocol shown in Figure 9 using the Elipar® Highlight (ESPE). The light intensity was verified to be within ±50 mW/cm² of the stated value using the “Cure Rite” light radiometer. Each group was comprised of 12 teeth.

Bonding System – SBMP and 3M Single Bond

- 1.5 mm - Dual Cure
- 3.0 mm - Single Cure
- 4.5 mm - Dual Cure

Figure 10. Flowchart of experimental protocol.

Note: Dual Cure:
- 10s-150mW/cm²
- 30s-700mW/cm²

Single Cure:
- 40s-700mW/cm²
The templates for the 1.5mm samples were removed immediately after curing. The 3.0 and 4.5 mm templates were removed 24 hours into the storage period during which time the gelatin lining had dissolved and the templates had become more pliable thereby allowing removal without placing stress on the resin-dentin bond. All the groups of teeth were stored in distilled water with thymol for 7 days at 37°C, and then the samples were shear tested to failure on the Instron Universal Testing Machine as described previously. The peak values were recorded in Newtons and then converted to megaPascals. The location of bond failure was recorded.

**Statistical Analysis**

The SAS system was utilized for analysis of these data. Mean shear bond strengths and standard deviations were calculated for each group. One-way ANOVA was carried out to analyze:

- The effect of material thickness on SBS
- The effect of curing type on SBS
- The effect of material type on SBS

Two-way ANOVA was used for the following analyses:

- The effect of material thickness and curing type on SBS
- The effect of curing type and material type on SBS
- The effect of material thickness and material type on SBS

Multiple pairwise comparisons were performed with Duncan's Multiple Range test. Test of interaction between the effects of the factors was included in the
ANOVA. Significant interactions were investigated using the LSMEANS test. The statistical significance for all tests was set at the 5% level.

3. Microhardness Examination of Single and Dual Cured Composite Resin

PVC templates 1.5, 3.0, and 4.5 mm in thickness were prepared and holes 4mm in diameter punched in them as described previously. For each thickness of material 10 templates were prepared. A No. 1 thickness glass cover slip (Ingram and Bell Ltd., Don Mills, Ontario) was placed underneath the PVC template and then placed on top of a flat human dentin surface. Z100 (3M Corporation) restorative resin, shade A3, was pressed into the mold and another glass coverslip was placed over the top surface of the composite material and vibrated until the coverslip was flush with the surface of the template. This was done to provide a flat surface for testing and in an effort to prevent the formation of an air-inhibited layer. For each thickness, 5 samples were single cured for 40 seconds at 700 mW/cm² and the other 5 were dual cured for 10 seconds at 150 mW/cm² then for 30 seconds at 700 mW/cm² using the Elipar Highlight (ESPE). The samples were immediately placed into distilled water with thymol after photopolymerization.

Using a Tukon 300 microhardness tester (Acco Industries Inc., Wilson Instrument Division, Bridgeport, CT) with a Knoop indenter and a 30 gram weight, the top and bottom surface microhardness of each specimen was determined at intervals of 2 hours, 24 hours, and 7 days. The samples were stored in distilled water with thymol at 37° C between test intervals. At each test
period four readings were obtained from each surface (8 readings for each specimen). Mean Knoop hardness numbers (KHNs) of the top and bottom of the composite resin were calculated for each sample.

The SAS system was utilized for analysis of the gathered data. The mean of the 4 microhardness measurements taken at each measurement period at both the top and bottom surfaces was calculated. Multiple pairwise comparisons of these microhardness means were performed with Duncan’s Multiple Range test. The statistical significance for all tests was set at the 5% level.
Figure 5. Embedded bovine incisor with composite resin cylinder applied to prepared dentin surface.
Figure 6. Instron shear testing machine
Figure 7. Espe Elipar® Highlight visible light curing unit.

Figure 8. PVC templates with gelatin capsules as used in the dual cure study.
Figure 9. PVC templates applied over the dentin and filled with Z100 composite resin.
Results

1a. Long Term Storage Results

All materials showed a decrease in bond strength at 270 days when compared to the 24 hour control. In fact, a reduction in SBS was usually seen after only 7 days with the exception of SBMP which actually improved its bond strength (Table 10).

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>24 Hours storage (Control)</th>
<th>7 Days storage</th>
<th>270 Days storage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scotchbond Multi-Purpose</td>
<td>11.5 ± 5.2</td>
<td>14.3 ± 4.6</td>
<td>8.0 ± 4.4**</td>
</tr>
<tr>
<td></td>
<td>n = 12</td>
<td>n = 12</td>
<td>n = 12</td>
</tr>
<tr>
<td>Single Bond</td>
<td>13.5 ± 4.9</td>
<td>9.5 ± 3.7</td>
<td>8.2± 5.2*</td>
</tr>
<tr>
<td></td>
<td>n = 12</td>
<td>n = 14</td>
<td>n = 12</td>
</tr>
<tr>
<td>Prime and Bond 2.1</td>
<td>7.7 ± 4.2</td>
<td>5.7 ± 2.7</td>
<td>3.2 ± 2.5*</td>
</tr>
<tr>
<td></td>
<td>n = 12</td>
<td>n = 12</td>
<td>n = 12</td>
</tr>
<tr>
<td>All-Bond 2</td>
<td>12.7 ± 6.0</td>
<td>9.6 ± 4.7</td>
<td>10.0 ± 4.6</td>
</tr>
<tr>
<td></td>
<td>n = 12</td>
<td>n = 24</td>
<td>n = 12</td>
</tr>
<tr>
<td>One Step</td>
<td>12.1 ± 4.5</td>
<td>8.1 ± 3.4</td>
<td>5.5 ± 4.3*</td>
</tr>
<tr>
<td></td>
<td>n = 13</td>
<td>n = 12</td>
<td>n = 12</td>
</tr>
</tbody>
</table>

Table 10. Short and long term shear bond strength of the tested materials (Mean ± SD) in MPa.
* Indicates significant difference from control (p < 0.05).
** Indicates significant difference between the 7 and 270 day storage periods (p < 0.05).

All the single bottle systems, Single Bond (p=0.005), One Step (p=0.0004) and Prime & Bond 2.1 (p=0.01), had statistically significant decreases in SBS at 270 days compared with the 24 hour control. The two fourth generation systems (SBMP and AB2) also showed reductions in bond strength after 270 days, but these were not statistically significant.

Scotchbond Multi-Purpose was the only system to demonstrate a significant increase in SBS at 7 days compared to 24 hours. When comparing
this higher SBS to the 270 day results it is noted that a significant drop has occurred over this time frame. It was the only material to show a significant decrease in SBS from 7 to 270 days storage.

The purpose of the study was not only to compare fourth and fifth generation materials as a group but also to compare the fourth generation products produced by 3M and Bisco with their single bottle counterparts. After 270 days the mean SBS of SBMP and SB were almost identical. Whereas the OS mean SBS was approximately 60% of the value obtained for AB2. SB was the only single bottle system that retained bond strengths at 270 days similar to those obtained with the fourth generation systems.
1b. Thermocycling Results

The shear bond strengths of the tested adhesive materials are listed in Table 11. Thermocycled results are presented with the non-thermocycled control.

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Control 7-Day</th>
<th>Thermocycled 24 Hours - Tested at 7 Days</th>
<th>Thermocycled 6.5 Days - Tested at 7 Days</th>
</tr>
</thead>
</table>
| Scotchbond Multi-
  Purpose            | 14.3 ± 4.6    | 9.8 ± 5.0* n = 12                        | 7.0 ± 3.6* n = 12                        |
| Single Bond          | 9.5 ± 3.7     | 10.5 ± 4.5 n = 13                        | 13.1 ± 4.2 n = 11                        |
| Prime and Bond 2.1   | 5.7 ± 2.7     | 4.2 ± 2.8 n = 12                         | 5.4 ± 3.8 n = 12                         |
| All-Bond 2           | 9.6 ± 4.7     | 7.9 ± 5.9 n = 12                         | 6.7 ± 3.3 n = 12                         |
| One Step             | 8.1 ± 3.4     | 13.4 ± 4.4* n = 12                       | 7.1 ± 3.5 n = 12                         |

* Indicates significant difference (p<0.05) compared to non-thermocycled control.

Table 11. Thermocycled and non-thermocycled shear bond strength results of the tested materials (Mean ± SD) in MPa at 7 days.

The effect of thermocycling on SBS was variable. Most thermocycled groups decreased in bond strength but of these only SBMP demonstrated a statistically significant reduction. Both thermocycled groups with this material were lower (p<0.02) compared to the 7 day non-thermocycled control. No other material suffered significant reductions with thermocycling. However, the SBS of One Step significantly increased after being thermocycled at 24 hours (p<0.005) prior to shear testing at 7 days. To verify that this was not an anomalous finding a second group was tested under the same conditions and a similar significant (p=0.005) increase in bond strength resulted. One Step was the only material
that demonstrated any significant increase in bond strength under either of the thermocycling conditions.

Prime & Bond 2.1 demonstrated the lowest bond strength in all experimental groups and, taken as a whole, P&B 2.1 was found to show significantly lower (p<0.05) bond strengths when compared to the other materials (Table 12). The other materials were not statistically different from each other.

<table>
<thead>
<tr>
<th>Bon Grouping</th>
<th>Mean</th>
<th>N</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>10.8</td>
<td>62</td>
<td>3MSB</td>
</tr>
<tr>
<td>A</td>
<td>10.1</td>
<td>60</td>
<td>SBMP</td>
</tr>
<tr>
<td>A</td>
<td>10.0</td>
<td>73</td>
<td>OS</td>
</tr>
<tr>
<td>A</td>
<td>9.4</td>
<td>72</td>
<td>AB2</td>
</tr>
<tr>
<td>B</td>
<td>5.3</td>
<td>60</td>
<td>P&amp;B2.1</td>
</tr>
</tbody>
</table>

Table 12. Bonferroni test for the effect of material type on SBS. Means with the same letter are not significantly different at p<0.05.

Thermocycling was performed either after 24 hours or 6.5 days of storage. When comparing the SBS obtained at 7 days it was shown, with the exception of One Step, that there were no statistically significant differences obtained between the two thermocycling parameters. In the case of One Step higher SBS was achieved with the 24 hour group (p<0.001).

Mode of fracture of the various groups is shown in Tables 13 through 17. It is difficult to detect any trends in this data except when looking at Prime & Bond 2.1. This material, almost exclusively, demonstrated adhesive bond failures. Even at 24 hours 75% of the bonds failed adhesively. These results support the finding that SBS for this material was consistently lower than those achieved with the other materials. Other trends noted in the mode of fracture data are as follows:
• Short term (24 hours) data tended to have less adhesive failures than samples which were stored for longer periods.

• SBMP tended to have the least number of adhesive failures, with none appearing until the samples were either thermocycled or stored for 270 days.

• Single Bond demonstrated a large increase in adhesive failures from 16.7% at 24 hours to 100% at 270 days. These tendencies were not noticed with the other materials.

<table>
<thead>
<tr>
<th>Failure</th>
<th>24 HRS</th>
<th>7 Days</th>
<th>TC 24/7</th>
<th>TC 6.5/7</th>
<th>270 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0</td>
<td>0</td>
<td>3 (25%)</td>
<td>5 (41.7%)</td>
<td>6 (50%)</td>
</tr>
<tr>
<td>A - C</td>
<td>12 (100%)</td>
<td>12 (100%)</td>
<td>9 (75%)</td>
<td>7 (58.3%)</td>
<td>6 (50%)</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 13. Mode of failure of SBMP groups.
A = Adhesive fracture
A - C = Mixed adhesive-cohesive fracture

<table>
<thead>
<tr>
<th>Failure</th>
<th>24 Hrs</th>
<th>7 Days</th>
<th>TC 24/7</th>
<th>TC 6.5/7</th>
<th>270 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2 (16.7%)</td>
<td>10 (71.4%)</td>
<td>5 (38.5%)</td>
<td>1 (9.1%)</td>
<td>12 (100%)</td>
</tr>
<tr>
<td>A - C</td>
<td>10 (83.3%)</td>
<td>4 (28.6%)</td>
<td>8 (61.5%)</td>
<td>10 (90.9%)</td>
<td>0</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>14</td>
<td>13</td>
<td>11</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 14. Mode of failure of Single Bond groups
A = Adhesive fracture
A - C = Mixed adhesive-cohesive fracture

<table>
<thead>
<tr>
<th>Failure</th>
<th>24 Hrs</th>
<th>7 Days</th>
<th>TC 24/7</th>
<th>TC 6.5/7</th>
<th>270 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>9 (75%)</td>
<td>11 (91.7%)</td>
<td>12 (100%)</td>
<td>10 (83.3%)</td>
<td>11 (91.7%)</td>
</tr>
<tr>
<td>A - C</td>
<td>3 (25%)</td>
<td>1 (8.3%)</td>
<td>0</td>
<td>2 (16.7%)</td>
<td>1 (8.3%)</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 15. Mode of failure of Prime and Bond 2.1 groups.
A = Adhesive fracture
A - C = Mixed adhesive-cohesive fracture
Table 16. Mode of failure of Bisco One Step groups.
A = Adhesive fracture
A – C = Mixed adhesive-cohesive fracture

<table>
<thead>
<tr>
<th>Failure</th>
<th>24 Hrs</th>
<th>7 Days</th>
<th>TC 24/7</th>
<th>TC 6.5/7</th>
<th>270 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5 (38.5%)</td>
<td>10 (83.3%)</td>
<td>10 (41.7%)</td>
<td>11 (91.7%)</td>
<td>10 (83.3%)</td>
</tr>
<tr>
<td>A – C</td>
<td>8 (61.5%)</td>
<td>2 (16.7%)</td>
<td>14 (58.3%)</td>
<td>1 (8.3%)</td>
<td>2 (16.7%)</td>
</tr>
<tr>
<td>Total</td>
<td>13</td>
<td>12</td>
<td>24</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 17. Mode of failure of All-Bond 2 groups.
A = Adhesive fracture
A – C = Mixed adhesive-cohesive fracture

<table>
<thead>
<tr>
<th>Failure</th>
<th>24 Hrs</th>
<th>7 Days</th>
<th>TC 24/7</th>
<th>TC 6.5/7</th>
<th>270 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>4 (25%)</td>
<td>16 (66.7%)</td>
<td>10 (83.3%)</td>
<td>9 (75%)</td>
<td>8 (66.7%)</td>
</tr>
<tr>
<td>A – C</td>
<td>8 (75%)</td>
<td>8 (33.3%)</td>
<td>2 (16.7%)</td>
<td>3 (25%)</td>
<td>4 (33.3%)</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>24</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

2. Results of the Dual Cure SBS Study

The effect of dual curing and thickness of restorative resin on the adhesive strength of Scotchbond Multi-Purpose and Single Bond to human dentin after 7 days was investigated. The method of curing (either single or dual) did not significantly affect any of the groups (Tables 18 and 19, and Figure 11). The thickness of the composite was significantly correlated with reduced SBS at the 4.5 mm level for Single Bond only. The SBS of the 4.5 mm Single Bond dual and single cure groups were significantly reduced (p<0.05) compared to their 1.5 mm counterparts. Except for the drop in bond strength of the fifth generation material
at 4.5 mm the SBS of Single Bond and Scotchbond Multi-Purpose remained consistent throughout the study.

<table>
<thead>
<tr>
<th>Curing Method</th>
<th>1.5 mm</th>
<th>3.0 mm</th>
<th>4.5 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dual Cure</td>
<td>16.0 ± 4.9</td>
<td>17.9 ± 4.7</td>
<td>16.2 ± 5.2</td>
</tr>
<tr>
<td>Single Cure</td>
<td>18.0 ± 6.3</td>
<td>17.3 ± 6.3</td>
<td>17.5 ± 5.3</td>
</tr>
</tbody>
</table>

**Table 18.** Shear bond strength of Scotchbond Multi-Purpose in MPa (Mean ±SD). No statistically significant differences among groups (p<0.05).

<table>
<thead>
<tr>
<th>Curing Method</th>
<th>1.5 mm</th>
<th>3.0 mm</th>
<th>4.5 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dual Cure</td>
<td>22.6 ± 5.6</td>
<td>21.9 ± 3.7</td>
<td>12.8 ± 5.8*</td>
</tr>
<tr>
<td>Single Cure</td>
<td>23.9 ± 3.7</td>
<td>23.2 ± 4.1</td>
<td>16.7 ± 5.0*</td>
</tr>
</tbody>
</table>

**Table 19.** Shear bond strength of Single Bond in MPa (Mean ±SD). *Indicates significant reductions in SBS compared to 1.5 mm and 3.0 mm groups at p < 0.05.

**Figure 11.** Mean shear bond strengths attained in this study.
The mode of fracture for the various groups is shown in Tables 20 and 21. For both materials the majority of fractures were of the mixed adhesive–cohesive variety. With Single Bond, a trend towards a greater percentage of adhesive fractures as the material's thickness increased is noted. With SBMP no trend is evident between thickness and fracture type. Finally, a large degree of similarity between the two curing types was noted in the percentage of adhesive fractures.

<table>
<thead>
<tr>
<th>Fracture</th>
<th>1.5 Dual</th>
<th>1.5 Single</th>
<th>3.0 Dual</th>
<th>3.0 Single</th>
<th>4.5 Dual</th>
<th>4.5 Single</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1 (8.33%)</td>
<td>0</td>
<td>2 (16.7%)</td>
<td>4 (33.3%)</td>
<td>5 (41.7%)</td>
<td>7 (58.3%)</td>
</tr>
<tr>
<td>A - C</td>
<td>11 (91.7%)</td>
<td>12 (100%)</td>
<td>10 (83.3%)</td>
<td>8 (66.7%)</td>
<td>7 (58.3%)</td>
<td>5 (41.7%)</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 20. Mode of fracture for **Single Bond**.
A = Adhesive fracture
A - C = Mixed adhesive-cohesive fracture

<table>
<thead>
<tr>
<th>Fracture</th>
<th>1.5 Dual</th>
<th>1.5 Single</th>
<th>3.0 Dual</th>
<th>3.0 Single</th>
<th>4.5 Dual</th>
<th>4.5 Single</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2 (16.7%)</td>
<td>2 (16.7%)</td>
<td>8 (66.7%)</td>
<td>7 (58.3%)</td>
<td>4 (33.3%)</td>
<td>3 (25%)</td>
</tr>
<tr>
<td>A - C</td>
<td>10 (83.3%)</td>
<td>10 (83.3%)</td>
<td>4 (33.3%)</td>
<td>5 (41.7%)</td>
<td>8 (66.7%)</td>
<td>9 (75%)</td>
</tr>
<tr>
<td>Total</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 21. Mode of fracture for **Scotchbond Multi-Purpose**.
A = Adhesive fracture
A - C = Mixed adhesive-cohesive fracture

### 3. Knop Microhardness Results

Tables 22 and 23 list the means and standard deviations of all groups.

The objectives of the study were to assess the effect of material thickness, cure
type and length of storage period on microhardness. These areas will be discussed separately.

<table>
<thead>
<tr>
<th>Group</th>
<th>2 Hours</th>
<th>24 Hours</th>
<th>7 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 Single</td>
<td>46.7 ±13.4</td>
<td>50.8 ±12.7</td>
<td>53.9 ±4.8</td>
</tr>
<tr>
<td>1.5 Dual</td>
<td>57.7 ±5.7</td>
<td>64.0 ±3.9</td>
<td>57.5 ±11.5</td>
</tr>
<tr>
<td>3.0 Single</td>
<td>52.2 ±7.4</td>
<td>46.6 ±8.8</td>
<td>42.4 ±9.6</td>
</tr>
<tr>
<td>3.0 Dual</td>
<td>34.4 ±8.1</td>
<td>45.9 ±9.0</td>
<td>45.6 ±7.4</td>
</tr>
<tr>
<td>4.5 Single</td>
<td>32.0 ±5.9</td>
<td>40.3 ±4.4</td>
<td>44.2 ±8.9</td>
</tr>
<tr>
<td>4.5 Dual</td>
<td>29.7 ±8.5</td>
<td>31.8 ±5.4</td>
<td>31.0 ±12.5</td>
</tr>
</tbody>
</table>

Table 22. Knoop microhardness (Mean ±SD) measured at the bottom of the samples.

<table>
<thead>
<tr>
<th>Group</th>
<th>2 Hours</th>
<th>24 Hours</th>
<th>7 Days</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 Single</td>
<td>43.7 ±2.6</td>
<td>39.9 ±11.3</td>
<td>41.3 ±8.9</td>
</tr>
<tr>
<td>1.5 Dual</td>
<td>57.0 ±4.7</td>
<td>44.3 ±7.7</td>
<td>42.3 ±7.0</td>
</tr>
<tr>
<td>3.0 Single</td>
<td>68.8 ±11.2</td>
<td>57.4 ±16.7</td>
<td>56.0 ±5.4</td>
</tr>
<tr>
<td>3.0 Dual</td>
<td>40.8 ±7.2</td>
<td>34.3 ±9.2</td>
<td>33.9 ±10.5</td>
</tr>
<tr>
<td>4.5 Single</td>
<td>22.5 ±4.1</td>
<td>22.6 ±10.4</td>
<td>34.4 ±8.0</td>
</tr>
<tr>
<td>4.5 Dual</td>
<td>27.7 ±14.6</td>
<td>39.2 ±8.8</td>
<td>38.6 ±6.7</td>
</tr>
</tbody>
</table>

Table 23. Knoop microhardness (Mean ±SD) measured at the top of the samples.

A. The effect of material thickness on microhardness.

When determining if the bulk of restorative material between the light source and the bottom composite surface adversely affected hardness, the top of each sample was used as the control. The single cured 4.5 mm samples had greater microhardness at the bottom of the sample as compared to the top, but this was only significant when tested at 24 hours (Figure 12, page 77).

Additionally, no effect was noted at all with the dual cured samples (Figure 13,
It appears that this thickness of material was insufficient to adversely affect microhardness.

The 3.0 mm and 1.5 mm thickness groups followed a similar pattern. It appeared to make little difference if the measurement was taken from the top or bottom of the sample. In fact only one group (Figure 14, page 78) demonstrated a significant drop in hardness after having been cured through a bulk of material (3 mm), whereas all others were either unchanged (Figures 15 and 16, pages 78 and 79) or harder (Figure 17, page 79).

The data obtained from the samples at the three time periods (2 hours, 24 hours, and 7 days) was also pooled and statistically analyzed (Table 24). Once again, the thicknesses used in the study did not appear to adversely affect the microhardness. Many of the groups showed greater hardness on the bottom of the resin sample though the microhardness was only significantly greater with the single cured 4.5 mm samples.

<table>
<thead>
<tr>
<th>Groups Compared</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 Single Top</td>
<td>41.6±8.0</td>
</tr>
<tr>
<td>1.5 Dual Top</td>
<td>47.9±9.1</td>
</tr>
<tr>
<td>3.0 Single Top</td>
<td>60.7±11.0</td>
</tr>
<tr>
<td>3.0 Dual Top</td>
<td>36.3±9.0</td>
</tr>
<tr>
<td>4.5 Single Top</td>
<td>26.5±9.3</td>
</tr>
<tr>
<td>4.5 Dual Top</td>
<td>35.2±11.2</td>
</tr>
<tr>
<td>1.5 Single Bottom</td>
<td>50.2±10.7</td>
</tr>
<tr>
<td>1.5 Dual Bottom</td>
<td>59.7±7.8</td>
</tr>
<tr>
<td>3.0 Single Bottom</td>
<td>47.1±8.7</td>
</tr>
<tr>
<td>3.0 Dual Bottom</td>
<td>42.0±9.4</td>
</tr>
<tr>
<td>4.5 Single Bottom</td>
<td>38.8±8.1</td>
</tr>
<tr>
<td>4.5 Dual Bottom</td>
<td>30.8±8.6</td>
</tr>
</tbody>
</table>

Table 24. Comparison of Knoop microhardness measured on top and bottom of resin samples. Data obtained at the three time periods have been pooled. * Significant difference (Bonferroni test; p<0.05).
B. The effect of time and cure type on hardness.

Table 25 shows the statistical comparison of microhardness between single and dual cured resin. For the most part, cure type did not have an effect on hardness at the bottom of the samples. In the case of the hardness measured on the surface closest to the light source though, 5 of the 9 mean microhardness values were significantly different. Interpretation of these results is difficult, however, since dual curing appeared to reduce hardness for the 3.0 mm samples while increasing hardness for some of the 1.5 and 4.5 mm samples. When the data for the 3 time periods are pooled, however, most of these differences disappear (Table 27, page 74). Only the top of the 3.0 mm samples demonstrated a significant reduction in hardness when they were cured with the dual setting. Cure type did not have a significant effect on any of the other surfaces.
### Top

<table>
<thead>
<tr>
<th>Groups Compared</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 Single-2 Hrs</td>
<td>57.0±4.7</td>
</tr>
<tr>
<td>1.5 Single-24 Hrs</td>
<td>44.3±7.7</td>
</tr>
<tr>
<td>1.5 Single -7 Days</td>
<td>42.3±7.0</td>
</tr>
<tr>
<td>3.0 Single-2 Hrs</td>
<td>40.8±7.2</td>
</tr>
<tr>
<td>3.0 Single-24 Hrs</td>
<td>34.3±9.2</td>
</tr>
<tr>
<td>3.0 Single-7 Days</td>
<td>33.9±10.5</td>
</tr>
<tr>
<td>4.5 Single-2 hrs</td>
<td>27.7±14.6</td>
</tr>
<tr>
<td>4.5 Single-24 Hrs</td>
<td>39.2±8.8</td>
</tr>
<tr>
<td>4.5 Single-7 Days</td>
<td>38.6±6.7</td>
</tr>
</tbody>
</table>

### Bottom

<table>
<thead>
<tr>
<th>Groups Compared</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5 Single-2 Hrs</td>
<td>57.7±5.7</td>
</tr>
<tr>
<td>1.5 Single-24 Hrs</td>
<td>64.0±3.9</td>
</tr>
<tr>
<td>1.5 Single-7 Days</td>
<td>57.5±11.5</td>
</tr>
<tr>
<td>3.0 Single-2 Hrs</td>
<td>34.4±8.1</td>
</tr>
<tr>
<td>3.0 Single-24 Hrs</td>
<td>45.9±9.0</td>
</tr>
<tr>
<td>3.0 Single-7 Days</td>
<td>45.6±7.4</td>
</tr>
<tr>
<td>4.5 Single-2 Hrs</td>
<td>29.7±8.5</td>
</tr>
<tr>
<td>4.5 Single-24 Hrs</td>
<td>31.8±5.4</td>
</tr>
<tr>
<td>4.5 Single-7 Days</td>
<td>31.0±12.5</td>
</tr>
</tbody>
</table>

Table 25. Comparison of mean KHN for single and dual cured Z100 resin for the top and bottom of the samples (Duncan test; p<0.05).
### Table 26. Comparison of mean KHNs obtained at 2 hours and 7 days (Duncan test; p<0.05).

<table>
<thead>
<tr>
<th>Groups Compared</th>
<th>Difference</th>
<th>2 Hours</th>
<th>7 Days</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Bottom</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.5 Single</td>
<td></td>
<td>43.7±2.6</td>
<td>41.3±8.9</td>
<td>No</td>
</tr>
<tr>
<td>1.5 Dual</td>
<td></td>
<td>57.0±4.7</td>
<td>42.3±7.0</td>
<td>Yes</td>
</tr>
<tr>
<td>3.0 Single</td>
<td></td>
<td>68.8±11.2</td>
<td>56.0±5.4</td>
<td>No</td>
</tr>
<tr>
<td>3.0 Dual</td>
<td></td>
<td>40.8±7.2</td>
<td>33.9±10.5</td>
<td>No</td>
</tr>
<tr>
<td>4.5 Single</td>
<td></td>
<td>22.5±4.1</td>
<td>34.4±8.0</td>
<td>No</td>
</tr>
<tr>
<td>4.5 Dual</td>
<td></td>
<td>27.7±14.6</td>
<td>38.6±6.7</td>
<td>No</td>
</tr>
<tr>
<td><strong>Top</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.5 Single</td>
<td></td>
<td>46.7±13.4</td>
<td>53.9±4.8</td>
<td>No</td>
</tr>
<tr>
<td>1.5 Dual</td>
<td></td>
<td>57.7±5.7</td>
<td>57.5±11.5</td>
<td>No</td>
</tr>
<tr>
<td>3.0 Single</td>
<td></td>
<td>52.2±7.4</td>
<td>42.4±9.6</td>
<td>No</td>
</tr>
<tr>
<td>3.0 Dual</td>
<td></td>
<td>34.4±8.1</td>
<td>45.6±7.4</td>
<td>No</td>
</tr>
<tr>
<td>4.5 Single</td>
<td></td>
<td>32.0±5.9</td>
<td>44.2±8.9</td>
<td>No</td>
</tr>
<tr>
<td>4.5 Dual</td>
<td></td>
<td>29.7±8.5</td>
<td>31.0±12.5</td>
<td>No</td>
</tr>
</tbody>
</table>

### Table 27. Comparison of mean KHNs of single and dual cured samples. Data for the three time periods have been pooled.

*Significant difference Bonferroni test; p<0.05.

Measurements were obtained initially at 2 hours, and again at 24 hours and 7 days. Table 26 displays the values obtained at 2 hours and 7 days. Except
for one of the 1.5 mm samples, storage in water for 7 days did not result in a significant reduction in surface hardness. Additionally, statistical comparison of the 24 hour group with the 2 hour and 7 day groups did not result in any significant differences. It appears that storage in water over the time frames utilized in the study did not have a significant effect on microhardness.

4. Comparison of Human and Bovine SBS

The methods and materials for the SBS studies using bovine dentin and human third molar dentin were similar. Therefore, it is possible to compare results obtained from these two types of dentin utilizing SB and SBMP. The studies with bovine material used resin that was approximately 3.0 mm in thickness and single cured. These results were compared to both single and dual cured human results obtained with a 3.0 mm resin thickness. Table 28 shows the results of statistical comparison of the bovine dentin SBS with human dentin SBS when resin thickness is 3.0 mm. In the case of both SB and SBMP the human dentin provided superior SBS after storage for 7 days. However, this increase was only statistically significant with SB where the SBS to human dentin was more than double that to bovine dentin. Additionally, SB had a trend towards less variation of SBS values when human material was used.

Table 29 compares the mode of fracture of the bovine and human studies. SBMP demonstrated a much higher incidence of purely adhesive fractures with the human material. SB had larger numbers of adhesive failures with the bovine
material, likely reflecting the much lower bond strengths obtained with the bovine dentin.

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Bovine Single Cured</th>
<th>Human Single Cured</th>
<th>Human Dual Cured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scotchbond Multi-Purpose</td>
<td>14.3 ± 4.6</td>
<td>17.3 ± 6.3</td>
<td>17.9 ± 4.7</td>
</tr>
<tr>
<td>Single Bond</td>
<td>9.47 ± 3.7</td>
<td>23.2 ± 4.1 *</td>
<td>21.9 ± 3.7 *</td>
</tr>
</tbody>
</table>

Table 28. SBS of bovine and human dentin after 7 days of storage (Mean ± SD) in MPa.

* Significant difference between bovine and human SBS (Bonferroni test p<0.05).

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Bovine Single Cured</th>
<th>Human Single Cured</th>
<th>Human Dual Cured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scotchbond Multi-Purpose</td>
<td>0%</td>
<td>58.3%</td>
<td>66.7%</td>
</tr>
<tr>
<td>Single Bond</td>
<td>71.4%</td>
<td>33.3%</td>
<td>16.7%</td>
</tr>
</tbody>
</table>

Table 29. Percentage of adhesive fractures obtained in the SBS studies listed in Table 28.
Figure 12. Single Cure – 4.5 mm Thickness. The 24 hour group is significantly different (Duncan test, p<0.05)

Figure 13. Dual Cure – 4.5 mm Thickness.
Figure 14. Single Cure – 3.0 mm Thickness. The 7 day group has significantly greater hardness on the top of the resin as compared to the bottom (Duncan test, p<0.05).

Figure 15. Dual Cure – 3.0 mm Thickness.
Figure 16. Single Cure – 1.5 mm Thickness.

Figure 17. Dual Cure – 1.5 mm Thickness.
The bottom surfaces of the 24 hour and 7 day groups are significantly harder than the corresponding top surfaces (Duncan test, p<0.05).
Discussion

Part 1a. Long-Term Bond Strength Study

Fourth generation systems such as Scotchbond Multi-Purpose and All-Bond 2 are capable of achieving significantly greater SBS over previously available products. A large number of in-vitro studies have been performed to characterize the performance of these products under laboratory conditions. While the vast majority of studies test bond strength after short periods of time, some investigators have looked at the effects of long term storage. In contrast, long term bond strength studies have not, as yet, occurred for newer products such as fifth generation adhesive systems. This lack of long-term data has not prevented their rapid introduction into the marketplace and use by dental practitioners.

Scotchbond Multi-Purpose and All-Bond 2

Scotchbond Multi-Purpose (SBMP) and All-Bond 2 (AB2) were selected as the fourth generation, multiple component, comparison materials for this study. These are logical control materials since they are produced by the same manufacturer as One Step (OS) and Single Bond (SB), have been used in clinical practice for many years, and have been extensively studied under laboratory conditions.

Utilizing similar laboratory techniques to those used in this study, Chan et al. (1997) studied the effect of conditioning dentin with different types and concentrations of acid on the long-term SBS of SBMP to bovine dentin. After conditioning with 37% phosphoric acid, bond strengths of 22.3±5.5 MPa at 24
hours and 14.9±1.7 MPa at 6 months were recorded. These are significantly greater than the 11.5±5.2 MPa (24 hours) and 7.96±4.4 MPa (270 days) achieved in the present study. One of the reasons for the lower SBS obtained in this study may be the use of the Teflon tape. Chan et al. (1997) did not use Teflon tape or any means other than the gelatin capsule to limit the spread of the agents applied to the dentin surface. Van Noort et al. (1991) demonstrated that an increase in tensile bond strength of more than 100% was possible when a flash of adhesive resin was present outside of the restorative resin – dentin interface. Therefore an extension of the adhesive resin beyond the interface will result in an artificially high value for the bond strength. The limitation of the exposed dentin surface area in the present study likely resulted in more accurate SBS measurements.

The highest bond strengths for SBMP (14.32±4.57 MPa) were recorded at 7 days. There is a paucity of literature reporting SBS results attained from intermediate storage periods such as the 7 day range used in this study. Al-Salehi and Burke (1997) reviewed the dentin bonding literature and found that only 4 out of the 50 published reports they examined contained bond strength data obtained after 7 days of storage. Therefore, the improvement in SBS over the first 7 days of storage cannot be independently confirmed. However, it is understood that the bond to dentin improves over several hours with most products due to continued polymerization. The SBMP Technical Product Profile (1992) graphically describes a greater than 200% (approaching 24 MPa) increase in adhesive strength to dentin after approximately 60 minutes post
application. It is possible that the bond strength continues to improve beyond the first 24 hours and represents the increase seen in this study.

All-Bond 2 results at 24 hours were similar to those reported by other researchers utilizing bovine dentin (Wilder et al., 1998 and Hosoya et al., 1997) and similar to SBMP. Additionally, AB2 and SBMP did not show a significant reduction in bond strength after 270 days. In fact, AB2 had the highest SBS at 270 days of all the materials tested.

The composition of All-Bond 2 differs from SBMP in a number of areas. In particular, All-Bond 2 primer contains minimal water (primer A contains 5% water with none in primer B) as compared to the 40% water in the SBMP primer. It is conceivable that the lower water content would result in an increased sensitivity to dry conditions. Tay et al. (1998) investigated the effect of various amounts of water within primer A. They found that prolonged desiccation often resulted in compromised hybridization when utilizing primer with only 5% water. AB2 primer prepared with 17% water for the purposes of their study allowed the material to be less sensitive to overdrying. However, the increased water content allowed for the formation of globules consistent with the “overwet” phenomenon when applied to moist and excessively wet dentin. AB2 and SBMP produced similar SBS at 24 hours and 270 days indicating that the different compositions of the two products appeared to perform equally well in this study.
One Bottle Adhesive systems

Prime & Bond 2.1

In this study the shear bond strengths of several fourth and fifth generation adhesive systems were examined after 270 days and compared to a 24 hour control for each material. With the exception of Prime & Bond 2.1, the bond strengths achieved at 24 hours were consistent with those achieved in other studies (Tables 30 and 32). It can be argued that Prime and Bond 2.1 and 2.0 are identical products for the purposes of bond strength studies. Their formulation is identical except for the addition of cetylamine hydrofluoride to 2.1 which provides a source of fluoride ions (Barkmeier et al., 1999). Therefore, values for both products are listed for comparison purposes in Table 31. This material's highest bond strength was attained at 24 hours (7.701 ±4.2 MPa) and was generally lower than results obtained by others with the exception of the results obtained by Lin et al. (1998).
<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Mean SBS(MPa)</th>
<th>Storage Period</th>
<th>Different from Present Study?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Miyazaki et al. (1998)</td>
<td>Bovine dentin (P&amp;B 2.0)</td>
<td>12.0 ±2.6</td>
<td>24 hours</td>
<td>Yes</td>
</tr>
<tr>
<td>Vargas et al. (1997)</td>
<td>Human third molar dentin (P&amp;B2.1)</td>
<td>22.27 ±3.8</td>
<td>24 hours then TC 300x</td>
<td>Yes</td>
</tr>
<tr>
<td>Prati et al. (1998)</td>
<td>Human molar dentin (P&amp;B2.0)</td>
<td>12.3 ±0.5</td>
<td>2 minutes</td>
<td>Yes</td>
</tr>
<tr>
<td>Barkmeier et al. (1999)</td>
<td>Human molar dentin</td>
<td>18.6 ±3.8 to 21.3±4.2 (P&amp;B2.1)</td>
<td>24 hours, TC 640 cycles</td>
<td>Yes</td>
</tr>
<tr>
<td>Wilder et al. (1998)</td>
<td>Bovine dentin (P&amp;B2.1)</td>
<td>15.5 ±3.0</td>
<td>48 hours</td>
<td>Yes</td>
</tr>
<tr>
<td>Lin et al. (1998)</td>
<td>Human Dentin</td>
<td>10.9 ±5.0</td>
<td>At least 24 hours</td>
<td>No</td>
</tr>
<tr>
<td>Abdalla and Davidson (1998)</td>
<td>Human molar dentin (P&amp;B2.1)</td>
<td>12.3 ±2.4</td>
<td>24 hours</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Table 30. Prime & Bond 2.1 and 2.0 shear bond strength studies and whether the results are statistically different than results achieved with this study.

At 24 hours the predominant fracture pattern for Prime & Bond 2.1 (75%) was at the adhesive layer. However, Abdalla and Davidson (1998) observed no fractures at the adhesive layer unless the dentin surface was contaminated with human blood prior to air-drying and application of the composite. In the present study contamination of the dentin surface did not occur prior to composite placement. In the study by Barkmeier et al. (1999) the percentage of cohesive failures in dentin ranged from 0% to 40%. This is more in line with what was seen in the present study.

Even though the initial SBS was low, bond strengths continued to drop over time and were significantly lower at 270 days (3.20±2.5 MPa). Additionally,
the SBS for Prime and Bond 2.1 was the lowest for all materials in every experimental condition. This trend has been observed in other studies. (Vargas et al., 1997; Miyazaki et al., 1998; Lin et al., 1998).

Barkmeier, Hammesfahr, and Latta (1999) reported the bond strength of Prime and Bond 2.0 and 2.1 to human enamel and dentin under a number of laboratory conditions. Prisma TPH Spectrum (Dentsply/Caulk) was applied to the dentin within a gelatin capsule, as was done in the present study. They noted that acid conditioning the dentin with 37% phosphoric acid did not improve the bond strength values. Also, when an acid conditioned dentin surface was air dried, bond strength was markedly reduced. Therefore, the protocol followed in this study of conditioning all dentin surfaces with 35% phosphoric acid may not have been optimal for this product. Additionally, every attempt was made to prevent desiccation of the dentin surfaces prior to application of the adhesive but some of the samples may have been dryer than was ideal. As was reported by Barkmeier, Hammesfahr, and Latta (1999) this combination of acid conditioning and excessive air-drying would result in a particularly large decrease in bond strength to dentin.

Prime and Bond 2.1 is acetone based (Table 31) and requires a moist surface for optimal dentin penetration (Kanca, 1992; Elhabashy and Swift, 1994). The acetone “chases” the water and helps replace the water by the primer on the dentin surface (Abdalla and Davidson, 1998). Wakefield and his colleagues (1998) have hypothesized that acetone may have a detrimental effect on the adhesive process.
<table>
<thead>
<tr>
<th>Product</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime &amp; Bond 2.1 (L.D. Caulk-Dentsply, Milford, DE, USA)</td>
<td>UDMA, PENTA, bisphenol A dimethacrylate, acetone, cetylamine hydrofluoride</td>
</tr>
<tr>
<td>One Step (Bisco Dental Products, Itasca, IL, USA)</td>
<td>BPDM, Bis-GMA, HEMA, acetone</td>
</tr>
<tr>
<td>Single Bond (3M Dental Products Division, St. Paul, MN, USA)</td>
<td>Bis-GMA, HEMA, water, ethanol, polyalkenoic acid copolymer</td>
</tr>
</tbody>
</table>

Table 31. Single bottle adhesive systems used in this study and their components. (Adapted from Vargas et al., 1997)

Acetone may be so hydrophilic that it is difficult to be completely volatilized. This may result in acetone remaining and compromising the polymerization process. Tay et al. (1996) observed that acetone-based single bottle adhesives were very susceptible to both over wet and dry conditions. In both laboratory and clinical usage, the exact amount of moisture adequate for optimal adhesion may be difficult to reliably reproduce.

If the use of acetone was responsible for the poor performance of Prime & Bond 2.1, then poor adhesion with other acetone-based products would be observed. However, the SBS of One Step, another acetone-containing product, was much higher under all experimental conditions. Therefore, inclusion of acetone is at best a partial explanation for the poor performance of Prime & Bond 2.1.

Table 31 shows that OS and SB contain Bis-GMA and HEMA whereas P&B 2.1 contains PENTA (a dipentaerythritol pentacrylate phosphonate ester). It has been proposed that the ether linkages of the phosphonated esters in PENTA...
may form hydrogen bonds with amide groups of dentin and complexation of calcium ions by the phosphonate moieties (Cebasso and Sahni, 1990). This interaction may be enhanced when the smear layer is not removed (Eliades, 1993). Instead, manufacturers of PENTA containing products (such as Prisma 2 and 3, Caulk/Dentsply) advocate smear layer modification to improve the attachment of the smear layer to dentin and to allow for better interaction of the bonding agent with the smear layer (Joynt et al., 1991). Therefore, the all-etch technique with phosphoric acid would not provide an ideal environment for PENTA containing products such as P&B 2.1.

**One Step and Single Bond**

The fifth generation systems from Bisco and 3M produced shear bond strengths at 24 hours that were similar to their fourth generation counterparts. These bond strengths were within the range of short-term values achieved by many other researchers who utilized bovine dentin as the adhesive substrate (Table 32). As was the case with Prime & Bond 2.1, the bond strengths after long term storage were significantly reduced for these products. In contrast, both fourth generation products did not demonstrate a statistically significant reduction in adhesion after 270 days.
<table>
<thead>
<tr>
<th>Researcher</th>
<th>Substrate</th>
<th>Material</th>
<th>SBS (MPa)</th>
<th>Different from present study?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Swift and Bayne (1997)</td>
<td>Bovine dentin</td>
<td>Single Bond</td>
<td>19.2±4.3 to 23.2±2.7</td>
<td>Yes</td>
</tr>
<tr>
<td></td>
<td></td>
<td>One Step</td>
<td>13.3±3.0 to 20.5±3.3</td>
<td>No</td>
</tr>
<tr>
<td>Wilder et al (1998)</td>
<td>Bovine dentin</td>
<td>One Step</td>
<td>15.4±5.6</td>
<td>No</td>
</tr>
<tr>
<td>Vargas et al. (1997)</td>
<td>Human molar</td>
<td>One Step</td>
<td>15.8±5.7</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>dentin</td>
<td>Single Bond</td>
<td>22.27±4.5</td>
<td>Yes</td>
</tr>
<tr>
<td>Prati et al. (1998)</td>
<td>Human molar</td>
<td>Single Bond</td>
<td>16.6±0.7</td>
<td>No</td>
</tr>
<tr>
<td>Miyazaki et al. (1998)</td>
<td>Bovine Dentin</td>
<td>One Step</td>
<td>16.1±2.8</td>
<td>Yes</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Single Bond</td>
<td>15.0±2.2</td>
<td>No</td>
</tr>
</tbody>
</table>

Table 32. Comparison of previously reported SBS values for 3M Single Bond and Bisco One Step.

The reduction in SBS over time is matched by an increase in adhesive bond failures (Tables 14 and 16, pages 66 and 67). Adhesive failures increased from 16.7% to 100% for SB and from 38.55% to 83.3% for OS. The increase in adhesive failures for the multi-step systems was much less pronounced.

The 3M products SBMP and SB are not acetone based and instead utilize ethanol and water as the solvents. The manufacturer claims that this allows these products to be relatively insensitive to the degree of dentin surface moisture present as compared to the acetone based products (Scotchbond Multi-Purpose Technical Product Profile). The Swift and Bayne study (1997) investigating the effect of various degrees of moisture on adhesion to dentin tends to support this claim. They found that even though SBMP and SB had the highest bond strengths and the lowest variation on wet dentin, they performed nearly as well on moist and overwet dentin. They hypothesized that the
insensitivity to various moisture conditions may be due to the presence of water in these products. If the dentin is too dry, the surface may be re-hydrated by the water within the product. Even if too much water is present on the dentin, the dilution of the resin does not appear to significantly affect the bond strength. In the present study 3M Single Bond achieved the highest bond strengths of the single bottle systems at 24 hours and 270 days, thus supporting the conclusions of Swift and Bayne.

**Part 1b. Thermocycling Study**

The effect of thermocycling on SBS was variable and most effects were not statistically significant. For all materials except One Step and SBMP, thermocycling at 24 hours or 6.5 days did not result in significantly different SBS when tested after 7 days of storage. The bond strength of OS significantly increased after thermocycling at 24 hours as compared to the 6.5 day thermocycled group and the non-thermocycled control. A second experimental group confirmed these findings. It is possible that OS polymerization is assisted by the heat present during the thermocycling process to a greater degree than the other materials. If so then perhaps OS would provide higher bond strengths *in vivo* (where the heat of the oral environment would enhance polymerization) than what was found in many *in vitro* studies.

Miyazaki *et al.* (1998) investigated the effect of 3,000, 10,000, and 30,000 cycles between 5° and 60°C on the bond strength of OS and SB to bovine incisor dentin. In all cases the thermocycling was performed after a 24-hour storage period in water at 37°C and then the bond was shear tested. At
3,000 cycles the SBS (OS – 13.9±2.9 MPa; SB – 11.6±1.7 MPa) were nearly identical to those achieved in this study. Even though OS was found to be resistant to large numbers of thermal cycles, Miyazaki et al. (1998) did not notice an increase in bond strength as was seen in this study. Perhaps this is due to the much larger number of cycles used in their investigation.

SBMP demonstrated significant reductions in SBS with both of the thermocycled groups. However, if the 24 hour group was used as the non-thermocycled control instead of the 7 day samples, then no significant decrease existed. P&B, SB, and AB2 did not demonstrate a significant change in SBS after thermocycling.

In general, thermocycling had relatively little effect on the type of failures produced when the samples were shear tested. However, SB demonstrated a large decrease in the number of adhesive failures after thermocycling. This material had a non-significant increase in SBS after thermocycling that is likely reflected in this reduction in purely adhesive fractures.

Gale and Darvell (1999) recently published an extensive review of thermocycling procedures used in the laboratory testing of dental restorations. They included 130 studies by 99 first authors selected from 25 journals. They noted that the regimens varied widely which made comparison of reports difficult. They concluded that “no definitive statement of a relevant regimen can be made and, in any case, the choice must be arbitrary to some degree because of wide variation”. However, the authors felt obligated to suggest a specific regimen in the interests of test standardization: 35°C, 15°C, 35°C and then 45°C, with a
corresponding dwell sequence of 28s, 2s, 28s, and 2s. Additionally, they suggested the adoption of 10,000 cycles since this would represent approximately a year of intra-oral service. In spite of these recommendations the authors' final conclusion was that thermal cycling "is an irrelevancy with spurious legitimacy, or is at best premature, because the validating preconditions have yet to be met". Attempts to standardize thermocycling parameters have been made by many laboratories and researchers prior to this article (Burger et al., 1992; Rossomando and Wendt, 1995; Tanaka et al., 1995; Yap et al., 1996; ISO/TC Dental Materials: Guidance on testing of adhesion to tooth structure, 1991). In most cases the protocol is established arbitrarily, as was ultimately done in the Gale and Darvell (1999) review.

Gale and Darvell state that thermocycling is likely a meaningless exercise. This opinion is supported by the work of Burger et al. (1992) who found that similar bond strengths were achieved when bonded samples were subjected to either 100 or 4,000 cycles. In spite of their contention that thermocycling is of little value, however, Gale and Darvell felt compelled to suggest a protocol which is very involved and time consuming.

The only way to determine whether thermocycling has an effect on dental materials is through the use of a non-thermocycled control as was done in the present study. Additionally, the protocol suggested within the 1991 and 1994 ISO Technical Reports was utilized due to the international recognition of the International Organization for Standardization as well as the simplicity and reasonable number of cycles suggested.
Two thermocycled experimental groups were utilized for each material. Samples were thermocycled at either 24 hours or at 6.5 days. Polymerization of the adhesive resin for an indeterminate period of time occurs after cessation of photopolymerization (Scotchbond Multi-Purpose Plus Dental Adhesive System Technical Product Profile, 1994). For this reason the 6.5 day protocol was included to insure that the polymerization of the adhesive resin was complete.

The purpose of the thermocycling study was to demonstrate that thermocycling procedures do not have a significant effect on SBS measurements performed in the laboratory. However, this was not conclusively shown since the materials responded variably to thermocycling. OS and SB appeared to be the most resistant to the presumed negative effects of thermal cycling. The bond strengths of both materials increased in some cases though only significantly in the case of OS when thermocycled at 24 hours. Additionally, the fact that one product performed differently depending on when in the storage cycle it was thermally stressed further complicates this issue. The results of this study suggest that standardization of thermal cycling parameters is essential, and in the absence of this, thermocycling test results from various laboratories are difficult if not impossible to interpret. In light of the importance of standardization, it is reasonable to suggest that all laboratories follow the ISO recommendations. This protocol is easy to apply, widely known, and independent of the biases that individual laboratories may hold.
Part 2. Dual Cure Bond Strength Study

This investigation had two primary objectives: 1. To determine whether two-step curing with 150 mW/cm² and 700 mW/cm² light intensities affected the SBS of SBMP and SB to human molar dentin. 2. To determine if increased composite thickness would enhance an effect on the SBS by dual-curing the composite restorative resin.

It is known that high intensity visible light produces a higher degree of conversion of resin materials (Sakaguchi and Berge, 1998; Davidson-Kaban et al., 1997), and greater depth of cure (Dunn et al., 1999). However, it is also understood that high intensity lights produce greater contraction strain during polymerization (Sakaguchi and Berge, 1998) due to increased velocity of the polymerization reaction (Kinomoto et al., 1999). The contraction strain can result in increased marginal gap formation (Unterbrink and Muessner, 1993) and microleakage (Sakaguchi and Berge, 1998) either immediately after placement of the resin or after thermal cycling. As a result, recent investigations have looked at allowing some flow of the resin early in the process of photopolymerization. This is accomplished by curing with a relatively low light intensity for a short time prior to exposure of the resin to conventional high intensity light for the remainder of the cycle (Sakaguchi and Berge, 1998; Koran and Kurschner, 1998; Davidson-Kaban et al., 1997; Mehl et al., 1997).

The Elipar® Highlight (ESPE America, Norristown, PA, USA) is a two-step visible light curing unit that has preset low and high light intensities that supposedly allow flow of the composite during initial cure. In the two-step setting
the unit emits light at approximately 150 mW/cm² for 10 seconds and then increases the intensity to 700 mW/cm² for either 30 or 50 seconds (Elipar® Highlight Product Monograph, 1997). This is one of the first widely available products that provides the practitioner with some degree of control over the speed of polymerization. In-house research by ESPE (Elipar® Highlight Product Monograph, 1997) has shown that the shrinkage rate of composite resin is reduced when the two-step setting is used. Recent investigations have shown that similar two-step protocols result in 20% less shrinkage stress than control (Feng and Suh, 1999). However, different composite resins appear to have different optimal two-step curing protocols (Wang and Suh, 1999).

While it is now understood that two-step curing has positive effects on marginal adaptation, microleakage, and contraction strain, researchers have not yet investigated whether these improvements affect adhesive strength to dentin. This study compared the Elipar® light’s 40 second two-step setting to the 40 second 700 mW/cm² single cure setting (control). Only 3M products were utilized in this study in order to allow for maximum product compatibility.

The method of curing did not significantly affect the SBS of any of the groups regardless of the type of bonding agent used or depth of thickness of the Z100 composite resin. Koran and Kurschner (1998) found that marginal adhesion of Pertac resin (ESPE, Inc., Seefeld, Germany) to pretreated metal surfaces were weakened by very fast polymerization and may be improved with a low intensity polymerization period. It should be noted that they used a push-out test design that would enhance the effects of improved marginal adaptation and not a
flat surface design such as was used in this study. They suggested that studies utilizing dental tissues were necessary to confirm this finding. The present investigation did not record improved adhesion using a conventional shear bond strength test and two-step polymerization.

Thickness of the Z100 composite resin was correlated with a reduction in SBS only when Single Bond was used with a 4.5 mm thickness of resin. At this level significant reductions in SBS were noted for both the one and two step curing groups bonded to dentin with Single Bond. It is interesting to note that Price et al. (2000) obtained almost identical results using a similar experimental protocol. In their experiment the SBS of SB used with a 5 mm thickness of Z100 (16.8 ± 3.1 MPa) was obtained after 24 hours and compared to the SBS obtained with a 2 mm thickness (21.8 ± 3.1). As in the present study, Price et al. (2000) obtained a statistically significant reduction in SBS with the greater resin thickness.

It is understood that the depth of cure is affected by thickness of resin material (Kamemizu et al., 1995) as well as other factors such as shade of the restorative material, length of light curing process, and light intensity. Manufacturer recommendations for Z100 composite are for 40 seconds visible light cure for thickness of material of approximately 2 mm (Z100 Product Monograph, 3M, St. Paul, MN). These recommendations are supported by evidence that significant reductions in degree of conversion occur near the 2 mm level (Rueggeberg et al., 1993). However, the results showed that when Z100 was used in conjunction with SBMP the SBS was unaffected by composite
thickness greatly in excess of that suggested by the manufacturer. It appears that the fourth generation adhesive system may be better able to maintain high bond strength when exposed to sub-optimal levels of light intensity.

In the present study flat, smooth dentin surfaces were used as the adhesion substrate. It has been shown that this design produces less stress to the resin-dentin bond as compared to what may be seen in a cavity preparation (Davidson et al., 1984). Feilzer et al. (1987) stated that as the ratio of the bonded to the unbonded surface area increased, the stress developed during curing greatly increased. The authors go on to state that cavity preparations have a "configuration factor" that would generate much more setting stress than would be seen when bonding to a flat surface. This is one more example of how in vitro testing differs from conditions found clinically.

**Suitability of Bovine Dentin in Adhesion Testing**

This study utilized the same basic experimental design as Part 1 which used bovine dentin as the adhesive substrate. Therefore it is possible to compare the 7 day results attained with bovine dentin with those recorded using human molar dentin. In the case of SBMP, bovine and human SBS were not significantly different though human bond strengths were generally higher. Single Bond SBS was significantly greater with human dentin until a 4.5 mm thickness of the composite was used. With the exception of the 4.5mm SB group all results were consistent with previously published reports using human dentin (Vargas et al., 1997; Cripe et al., 1998; Abdalla and Davidson, 1993).
The debate regarding the use of bovine teeth in bond strength evaluations continues almost two decades after Nakamichi, et al. (1983) published the report suggesting that bovine dental tissues may be an adequate substitute for human teeth in bond strength studies. They cautioned, however, that deeper bovine dentin yielded significantly lower values. Retief et al. (1990) found significantly lower SBS with bovine dentin when Scotchbond 2 and Silux was used in spite of deep penetration of the adhesive resin into the dentinal tubules. In contrast, Schilke et al. (1999) using another third generation bonding resin, Syntac (Vivadent, Liechtenstein), found no significant difference between human permanent dentin and bovine coronal dentin. The present study found a trend towards lower SBS with the bovine material. This may have been due to the dentin being ground deeper than what is ideal. This has the potential for greatly reducing SBS since the adhesive strength can be several times lower in deep bovine dentin as compared to superficial (Nakamichi, 1983). Additionally, Schilke et al. (1999) point out that the chemistry and structure of bovine teeth are not identical to human teeth and therefore it is logical that results would differ.

In many cases laboratory SBS evaluations look for trends, not absolute adhesive strength values. It is logical that evaluations using bovine tooth structure continue to have a place in the dental literature. Perhaps the SBS values from bovine dentin should not be directly compared with those attained with human material, but the trends that these values reveal remain valuable.
Appropriateness of the Shear Bond Strength Test

This study utilized shear bond strength as a method of estimating the effectiveness of the bond of the adhesive products to dentin. SBS testing continues to be one of the most common laboratory evaluations of adhesive systems. Al-Salehi and Burke (1997) found that 80% of the papers on the bond strengths of dentin bonding systems that they reviewed utilized the shear test. However, van Noort has suggested in the Journal of Dentistry’s 1996 and 1997 Dental Materials literature reviews that this is a crude process (Whitters et al., 1999; Strang et al., 1998) and alternate methods should be examined.

Alternate methods may include variations of the conventional shear test such as the “push-out” method that was adapted by Wakefield et al. (1998) from orthopedic biomaterials research. They claim that this method produces a more uniform shear stress at the composite-dentin interface when force is applied uniformly to the face of the composite specimen.

Tensile bond strength testing has been used by many laboratories over the last several years (Van Noort et al., 1991; Tanaka et al., 1995; Tam and Pilliar, 1994) and has recently been recommended by Nakabayashi et al. (1998). Nakabayashi and his colleagues (1998) claim that tensile testing is capable of showing defective resin infiltration of demineralized dentine in bonded interfaces more clearly than by conventional and/or ISO recommended methods. Additionally, the ISO has suggested a tensile testing protocol for use in bond strength measurements (ISO technical reports, 1991 and 1994). However, this method is seldom utilized.
Additionally, fracture toughness testing has been recommended as a means of assessing the fracture resistance of the dentin-composite interface (Tam and Pilliar, 1994; Ruse et al., 1996). The fracture toughness test is more likely to direct failures to occur along the bond plane and therefore, may provide a better measure of actual bond strength than the shear and tensile bond strength tests. Tam and Pilliar (1994) suggest that this type of test would be particularly useful for comparing the fracture resistance of bonded interfaces which would generally fail cohesively during other types of tests such as tensile bond strength testing.

In the 1997 Journal of Dentistry's literature review, van Noort (Whitters et al., 1999) suggests that microstructural investigations using TEM, confocal microscopy, or Atomic Force Microscopy (AFM) provide a better understanding of the underlying adhesion process. However, these microscopic evaluations are more time consuming, relatively expensive, and yield results that are also open to interpretation. Additionally, photomicrographs and microstructural investigations by one lab are not easily reproducible by another.

The shear bond strength test has its detractors but it remains arguably the most common method of adhesion testing. The large body of existing literature allows for ease of reproducibility and inter-investigator comparison. Since the products being tested were new to the marketplace it was necessary to employ a reliable testing method. This study further verified its results by using control products (SBMP and AB2) which had a number of SBS studies already in existence. This would not have been possible with other testing protocols.
**Microhardness Evaluation**

A number of hardness tests are used in dental materials testing. Most are based on the ability of a material to resist penetration by a point under a specified load. The principle difference between these tests is the shape of the indenter. The Brinell test measures the size of indentation produced by a ball shaped indenter. The Rockwell test is similar, however a metal cone may be used instead of a ball and the depth of indentation is measured instead of width. Vickers and Knoop tests use a pointed diamond indenter and the size of the impression made into the material is measured (Skinner’s Science of Dental Materials, 1991). The Knoop test was selected for this study because a number of small measurements could be taken over time without distorting the sample.

The surface microhardness results obtained in this study were generally inconclusive. A trend towards lower Knoop Hardness Numbers (KHNs) was noted with the 4.5 mm samples as compared to the 1.5 mm thickness, however, this reduction in microhardness was noted on the bottom and the top of the samples. Therefore, no effect due to thickness alone was evident even when a large bulk of material was present. This result is at odds with previous reports.

Kanca (1986) found that the shade and thickness of the restorative resin affected the hardness achieved after light curing. Grey shades resulted in lower hardness as compared to resin with light pigmentation. Additionally, resin thickness in excess of 2 mm resulted in lower hardness.

Davidson-Kaban et al. (1997) reported that regardless of the resin shade, and the intensity and exposure time of the curing unit, the Brinell Hardness at the
top of 1.5 mm samples of Silux-Plus or P-50 were invariably significantly higher than those obtained at the bottom. In this study a non-significant trend towards greater microhardness was noted at the bottom of the 1.5 mm samples. The Brinell test performed by Davidson-Kaban and her colleagues utilizes a hardened steel ball 1.0 mm in diameter and a 10 Kg load. After 30 seconds the width of the indentation was measured. This tests the surface hardness on a larger scale than the microhardness instrument used in this study. Perhaps a thin surface oxygen inhibited layer would have less of an effect on this more macroscopic type of measurement. Kamemizu et al. (1995) examined the depth of cure of a VLC composite resin by electron spin resonance spectroscopy (ESR). This method allowed for quantification of the polymer radicals generated after light irradiation and this was considered an indication of the degree of conversion. They found evidence of oxygen inhibition of free radical generation in the surface 0.5 mm of the material. This thin surface layer would have been where the “top” measurements would have been obtained with the Tukon microhardness tester. In addition, Knoop microhardness measurements are on such a small scale that some of them may have been affected by the filler particles. A measurement taken on a zirconia/silica filler particle is not a measure of the degree of curing of the resin. However, the filler particles in Z100 are only 0.6-0.7μm in size and therefore much smaller than the indentations made during Knoop measurements.

Additionally, Kamemizu and his colleagues (1995) noted an approximately 50% reduction in the number of free radicals generated at 2.0 mm as compared to 0.5-1.0 mm. More recently, Bouschlicher et al. (2000) have shown that a
significant decrease in degree of conversion occurs under 2mm of composite resin. Therefore, material thickness much less than the maximum used in the present investigation had an effect on degree of conversion. This compromise of the material on a molecular level, however, was not clearly evident in terms of the surface microhardness measured in this study. This indicates that for degree of conversion a threshold exists above which any reduction will not be noticed in compromised hardness.

This study also investigated the effect of dual curing on microhardness. The mean KHN’s on the bottom of the samples was generally unaffected by the type of cure. Many of the “top” means were affected by cure type, however the results were quite variable and difficult to interpret. Dual curing resulted in increased microhardness for the 3 mm samples and reduced microhardness for some of the 1.5 and 4.5 mm samples. Koran and Kurschner (1998) investigated the effect of sequential (two-step) versus continuous irradiation of a light-cured resin on a number of physical properties. Their results indicated that when the total dose of light intensity delivered to the resin composite material exceeded 17,000 mWs/cm² the overall surface hardness, as measured by the standard ball hardness test, of both the incident and opposite surfaces tended to remain constant. They concluded that dual curing did not have any effect on hardness as long as the total irradiation dose was adequate. The dual cure protocol utilized in the present study would have resulted in a 22500 mWs/cm² total irradiation dose. This may explain why trends in microhardness were not noted – the irradiation was more than adequate for full polymerization. Finally, Mehl et al. (1997)
studied the effect of a number of different initial light intensities on the Vickers microhardness of Tetric restorative resin. They did not detect any influence on material microhardness with respect to different initial cure conditions.

The choice of clear PVC material as the template in the dual cure and hardness studies may have been a confounding variable. It has been demonstrated that greater depth of cure is achieved when moulds of lighter color are used (Harrington and Wilson, 1993). This is due to light penetrating the mould and delivering a greater total light dose. In future studies, the use of a metal or opaque template could be used to maximize the effect material thickness has on hardness.

Lastly, this study attempted to detect any changes in microhardness that may occur after storage in water at physiological temperatures for 24 hours and 7 days as compared to the initial hardness. Ideally, the initial microhardness would have been recorded within a few minutes of VLC polymerization. However, due to the number of samples being prepared and tested, measurements were not recorded for up to 2 hours after the completion of visible light curing. When these 2 hour results were compared to the 1 and 7 day results virtually no difference in microhardness was noted. Only the top of the 1.5 mm dual cured samples showed a significant decrease in microhardness after 7 days. Since no other group demonstrated any significant difference it is likely that this is a statistical anomaly. Few reports are present in the literature regarding changes in surface hardness after initial polymerization. Ferracane (1985) demonstrated corresponding increases in KHN as degree of conversion increased for several
self-curing resins. He found that the increases in degree of conversion (DC) continued for several hours after curing was initiated. However, after the first 5-10 minutes the increase in DC was approximately linear with time through 24 hours. After this point, the DC and KHN had reached their maximal values. One must be cautious when presuming that these results obtained with self-curing resins would also be seen with VLC resins, however. Davidson-Kaban et al. (1995) compared surface hardness measured at 24 hours and 7 days to values obtained at 15 minutes. They reported a significant increase in hardness over time as compared to the initial measurement. As was the case with the depth of cure portion of the study their results differ from those obtained in the present investigation. One can speculate that the Z100 was more resistant to the experimental variables than the Silux-Plus and P-50 used by Davidson-Kaban and her colleagues. Additionally, the microhardness examination may not have been as sensitive to changes in hardness compared to the Brinell test. The electron spin resonance spectroscopy study of Kamemizu et al. (1995) investigated the effect of further visible light curing of a composite resin on free radical generation. The composite resin was initially visible light cured, after which some samples were further irradiated 3 minutes later while the remainder was further irradiated at 24 hours. They found that the amount of radicals regenerated at the second VLC decreased with increased storage time due to polymerization that occurred after removal of the light source. This demonstrated that further polymerization occurs for some time after the light source has been removed. However, if further cross-linking occurred during the course of storage
of the samples it did not result in increased microhardness in the present study. Further, much of the continued polymerization is known to occur within the first several minutes post-cure but the microhardness was not initially measured for up to 2 hours. As a result much of the post-cure polymerization may have already occurred. Davidson-Kaban et al. (1997) measured at 15 minutes and did find significant improvement in hardness at both 1 and 7 days.

The SBS study failed to demonstrate any significant change in bond strength at 7 days as compared to 24 hours. However, four of the adhesive materials demonstrated an increase in adhesive fractures when tested after 7 days of storage. One of the reasons that the microhardness study was undertaken was to investigate possible increases in hardness over this time frame. Any increase in hardness of the Z100 composite resin may reduce the number of fractures within the material and increase the number of purely adhesive fractures. The microhardness investigation showed that no appreciable change in hardness occurred over this time frame. However, only the surfaces of the material were tested, not the body of the composite cylinder, so the microhardness may not be representative. But when one considers that the top and bottom of the resin samples usually had a similar KHN it is reasonable to infer that the measured microhardness may reflect the internal hardness as well. This study was able to show that the increase in adhesive fractures after 7 days of storage was not likely due to “harder” Z100 resin preventing fracture within the composite button.
Conclusions

Part 1a – Long Term Bond Strength Study

Only the single bottle products suffered a significant reduction in SBS after 270 days storage in distilled water. In this study the single bottle systems, with the exception of Prime and Bond 2.1, generated high initial bond strengths but were more susceptible to the detrimental effects of long term water storage than the multiple component systems.

Part 1b – Thermocycling Study

The effect of thermocycling on the adhesive systems was variable thus making general conclusions difficult. In this study SBMP suffered a reduction in SBS whereas OS had an increase in bond strength if thermocycled early in the storage period. It appears that the effect of thermocycling is material specific and if the assumption is made that thermocycling has some clinical importance then it should remain part of a laboratory screening process.

Clinical Implications:

- The high bond strengths initially recorded were not maintained over time.
- The single-bottle systems produce a bond that is less durable over time.
- It is not possible to claim that the effect on bond strength observed with one material due to thermocycling will be seen with another. Depending on the adhesive system, thermocycling may have no effect, increase, or decrease bond strength.
Part 2 – Dual Cure Bond Strength Study

SBMP was less sensitive to composite resin thickness than the one bottle product (SB) and high bond strengths were obtained under even 4.5mm of restorative resin. Curing through 4.5 mm of Z100 resin resulted in a significant decrease in bond strength for SB.

Shear bond strengths obtained with SBMP and SB were higher with human dentin than bovine dentin.

Clinical Implications:

- The multiple component dentin adhesive was more resistant than the single-bottle system to the detrimental effects of curing through a bulk of restorative material.
- Dual curing of composite resin has no effect on the bond strength of dentin adhesive systems.
- Shear bond strength values obtained with bovine dentin should not be directly compared to values obtained with human dentin. However, trends observed with bovine material may be seen with the human material.
- The results of this study suggest that no more than 3.0 mm of resin composite should be cured at one time particularly when the bond is mediated by a single-bottle dentin adhesive.

Part 3 – Microhardness Study

In this pilot study the Knoop microhardness generally was not significantly reduced by thickness, cure type or storage for up to 7 days. It is likely that the Knoop microhardness test was not adequately sensitive for the hybrid Z100
composite resin and the filler particles interfered with accuracy. The results obtained in this study should be verified using Brinnell or Rockwell hardness tests. Additionally, opaque matrices should be used and the initial hardness value should be obtained within the first few minutes.

Clinical Implications:

- Visible light curing through much greater amounts of restorative resin than is recommended may still result in clinically acceptable hardness of material.
- Dual-curing and curing with a single high light intensity as described in this study results in composite resin that is equally hard.
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