

EFFECT OF DELAYED POLYMERIZATION TIME AND BRACKET
MANIPULATION ON ORTHODONTIC BRACKET BONDING

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ABSTRACT

This study examined the effect of bracket manipulation in combination with delayed polymerization times on orthodontic bracket shear bond strength and degree of resin composite conversion. Orthodontics brackets were bonded to extracted third molars in a simulated oral environment after a set period of delayed polymerization time and bracket manipulation. After curing the bracket adhesive, each bracket underwent shear bond strength testing followed by micro-Raman spectroscopy analysis to measure the degree of conversion of the resin composite. Results demonstrated the shear bond strength and the degree of conversion of ceramic brackets did not vary over time. However, with stainless steel brackets there was a significant effect ($p \leq 0.05$) of delay time on shear bond strength between the 0.5 min and 10 min bracket groups. In addition, stainless steel brackets showed significant differences related to degree of conversion over time between the 0.5 min and 5 min groups, in addition to the 0.5 min and 10 min groups. This investigation suggests that delaying bracket adhesive polymerization up to a period of 10 min then adjusting the orthodontic bracket may increase both shear bond strength and degree of conversion of stainless steel brackets while having no effect on ceramic brackets.

APPROVAL PAGE

The faculty listed below, appointed by the Dean of the school of Dentistry have examined a thesis titled “Effect of Delayed Polymerization Time and Bracket Manipulation on Orthodontic Bracket Bonding,” presented by Michael Ponikvar, candidate for the Master of Science degree, and certify that in their opinion it is worthy of acceptance.

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CHAPTER 1

INTRODUCTION

Orthodontic Brackets

Effective bonding of orthodontic brackets to tooth structure depends on bracket type, adhesive material and bonding protocol. In today's market there are several different brackets that can be purchased and used depending on doctor and patient objectives in addition to the clinical situation. The evolution of adhesive bonding has made great strides over the last 35 years (Graber 2005). When the field of orthodontics began it was important for practitioners to have a device that allowed for the controlled movement and manipulation of teeth. The orthodontic bracket was developed in order to align teeth in three dimensional space. The first brackets were attached to teeth by wrapping gold bands around them, crimping the overlapping metal and soldering the joint and a bracket to the structure to create a custom fit appliance (Hanson 1980). This was named the ribbon arch appliance and was the first of its kind yet modifications to this system quickly changed the bracket design. After much advancement, an increase in diversity and number of bracket designs quickly became available. Vertical and horizontal slots, single and twin winged brackets, prescription and self-ligating brackets became obtainable. Furthermore, different materials became accessible and while the first bracket was made of gold, different metal alloys have become popular as have many non-metal materials such as ceramic (Heravi and Bayani 2009; Tamizharasi 2010)

Stainless Steel Brackets

Stainless steel (SS) brackets, defined as a steel alloy with a minimum of 10.5% to

11% chromium content by mass are currently the most widely used brackets in orthodontic practices (Degarmo et al. 2012; Smith and Hashemi 2009). They exhibit clinical properties that make them superior to other materials in most categories such as strength and durability in addition to having accurately reproducible dimensions and low friction slot-wire interaction that provide exceptional slide mechanics. More so in the past, these brackets have been manufactured using the stamp technique whereby thin strips of metal are stamped into specific bracket dimensions (Zinelis et al. 2004). As wire technology improved, accuracy demands increased and a more precise method of bracket fabrication was required. In response brackets were formed by casting, which is used for making complex shapes that would be otherwise difficult or uneconomical to make by other methods. It is a process by which molten materials are poured into molds to produce brackets with highly precise internal slot dimensions (Proffit 2007). Despite precision, some low cost brackets are still manufactured using the stamp technique, but the majority of brackets are now made using the casting process. Alternatively, due to their superior esthetics, ceramic brackets have become popular although they have several drawbacks compared to their SS counterparts (Eliades and Brantley 2001).

Ceramic Brackets

Ceramic brackets came to the market in the mid 1980's (Gautam and Valiathan 2007). They are dimensionally stable, fairly durable, resist staining and are the most esthetic of all bracket materials. As a result these brackets received high acclaim and became immediately popular across North American until problems quickly surfaced (Proffit 2007). Fractures of brackets, friction within bracket slots, wear on teeth contacting a bracket, and

enamel damage from bracket removal soon became apparent (Saunders and Kusy 1994; Graber 2005). Many ceramic bracket designs have cycled through universal acceptance to obscurity as advances in technology addressed these shortcomings. Most currently, ceramic brackets are now made from alumina which is one of several forms of aluminum oxide. The two most common varieties are monocrystalline and polycrystalline (Proffit 2007). It is easier to manufacture a polycrystalline bracket due to its ability to mold and economic viability. However, its fabrication involves the fusing of ceramic grains causing structural imperfections at the grain boundaries and trace impurities (Gautam and Valiathan 2007). On the other hand monocrystalline brackets are manufactured using a different process that doesn't allow for imperfections and impurities. As a result monocrystalline brackets have superior optical properties and esthetics as they are clearer and more transparent than polycrystalline brackets (Gautam and Valiathan 2007).

Bracket Adhesives

There are two main categories of adhesives that are used for bracket bonding. They are acrylic and diacrylic resins, and they have slightly different intrinsic properties. The former, is composed of a methylmethacrylate monomer and ultrafine powder while diacrylic resins usually consist of bis-GMA (Graber 2005). The main difference between these two is how their chemical bonds begin to form. While linear polymers form in the acrylic, cross-linking polymerization is observed in the diacrylic resins. Since bond formation greatly contributes to the physical properties of these adhesives, greater strength, lower water absorption, and less polymerization shrinkage can be observed with the diacrylic materials (Omura et al. 1984; Rux et al. 1991).

Chemically-Cured Adhesives

These materials can also set in a variety of ways. The two most common adhesives are chemically cured and light cured, with the former being more popular. The chemically cured adhesive sets when the catalyst in the form of a liquid component (primer) is added to another component (paste). One component is added to the orthodontic bracket pad and the other to the desired tooth surface. When these components combine, a chemical reaction ensues and the material begins to set. Once the reaction is initiated the clinician has approximately 30-60 seconds of working time in order to precisely position the bracket before final setting (Brantley and Eliades 2001; Graber 2005). This process succeeded the previous two-paste system that was less efficient because it required more materials and time in addition to being more technically demanding.

Light-Cured Adhesives

Currently, the most prevalent technique is the light cured method (Keim et al. 2008); (Sakaguchi et al. 1992a). The evolution of adhesives to this type of system conveys several advantages. Not only does it allow for much longer working times as compared to the chemically cured system but it also provides clinicians the opportunity to cure adhesive on demand (Sakaguchi et al. 1992b; Sakaguchi et al. 1992c). These features lend themselves to other advantages in the private practice setting whereby staff members may place and position brackets before final manipulation while final curing is done by the orthodontist. Although delegation can be a major advantage in certain types of private practice, there are limited studies on the length of time that a bracket may be placed and exposed to ambient light before the curing process slowly initiates and ultimately affects the adhesive bonds.

Despite this fact, light cured adhesives are the most popular among orthodontists in North America (Graber 2005).

Bracket Bonding Procedure

Bonding is a seemingly straight forward process but simplicity cannot be confused with its ease of satisfactory completion. Inexperience or slight lapses in care can lead to less than ideal results as can a flaw in the multitudinous bonding procedures that are used across North America. Here we focus on direct bonding as opposed to indirect bonding as it is by far the more common technique (Milne et al. 1989). Ideally the steps involved in direct adhesive bracket bonding should include cleaning, etching, priming, and bonding.

Cleaning

Cleaning the tooth surface before bonding is an important step that involves the use of pumice, cotton roll, or an initial acid etch to remove plaque, excess debris and the organic pellicle that perpetually covers the tooth surface (Zachrisson 2007; Lill et al. 2008). Here moisture control is implemented as a safeguard to maintain a continuously dry field in order to prevent contamination of the tooth adhesive interface which will result in decreased bonding strengths (Proffit 2007). Often times devices such as lip retractors, saliva ejectors, cotton rolls, and dryangles are used in order to isolate the teeth by separating the soft tissues from them to eliminate the possibility of saliva encountering the desired bonding surface.

Etching

After these initial steps, it is most common to use either a two-step technique involving an etchant and primer before the addition of adhesive with a bracket or a one-step procedure that utilizes a self-etching primer. In the two step technique, after cleaning and

isolation, the teeth surfaces are treated with 37 % phosphoric acid (Legler et al. 1989). The conditioning solution or gel should be applied to the enamel surface for a period of 15 to 30 seconds. This technique results in significant penetration of the resin into micro porosities formed by the etchant and is the main factor behind long term strength and durability of the bond (Buonocore 1955; Glasspoole et al. 2001). The etchant is then rinsed off with copious amounts of water followed by removal of all moisture by drying the tooth surface until the classic etched and frosty glass appearance of enamel is achieved (Barry 1995; Lindauer et al. 1997; Ireland and Sherriff 2002).

Priming

A thin layer of primer is then applied uniformly to the etched surfaces of the tooth. It is suggested that the primer is then lightly air thinned for up to 2 seconds before brackets coated with adhesive are placed on the teeth. Due to their perceived efficiency and ease of use, self-etch priming is another popular technique used in lieu of a separate etching and priming step. The self-etching primers come in packets that contain three separate compartments. The first contains methacrylated phosphoric acid esters, photosensitizers, and stabilizers. The second include a combination of water and fluoride, while the third compartment contains micro brush used for material application. When the first two compartments are squeezed together, their contents mix and become activated. Further squeezing injects the mixture into the third compartment which contains the now wetted micro brush that is ready to be applied to the cleaned and isolated tooth surface. The self-etching primer is thoroughly rubbed on each tooth for at least 3 seconds before teeth are ready to receive brackets and adhesive (Unitek 2010a, b; Fleming et al. 2012; Unitek 2012).

In a final step brackets are placed on the height of contour of each tooth and positioned so the center line of the bracket is aligned with the long axis of the tooth before being finally cured into place. The brackets after curing are ready to transmit forces from orthodontic wires to the dentition by means of the adhesive-bracket interface.

Adhesive Polymerization

The final step in the bracket bonding procedure is light-curing of the bracket adhesive. Light cured adhesives begin to polymerize via photons, which are emitted from a light source and cause the activation of a catalyst (Strydom 2002). During the curing process a photo initiator works in conjunction with molecules of camphoroquinone that serve as light absorbers (Martin 2008). They exhibit maximal absorption when exposed to a wavelength of 470nm which correlates to a blue hue in the visible light spectrum (Abate et al. 2001). Until this point, halogen-based light-curing units have been the most widespread way of exposing adhesives to blue light. Now, it is the light emitting diodes (LED) that have become the most popular light source on the market due to durability and consistent performance in addition to their high intensity (Graber 2005). These lights are efficient and confer the highest degree of polymerization in relatively short amount of time (Henbest 2013).

Degree of Polymerization

The degree of polymerization is a key parameter or component in the light curing process and it is recommended that adhesives are fully cured before orthodontic forces are applied to them. Some companies recommend limited curing times such as 20 seconds for SS and ceramic brackets (Unitek 2012). Indeed, due to the difference in light transmission through these materials it has been suggested that bonding can be affected depending on the

bracket material. The chemical reaction occurred in the setting process of adhesive is a chain reaction and begins only at the perimeter of SS brackets as light cannot penetrate into the center of the adhesive through the bracket. As a result, because of opacity, it can take up to three days for adhesive on SS brackets to completely polymerize whereas the same degree of polymerization on ceramic bracket is almost immediate (Swartz 2007; Ozcan et al. 2008). Furthermore, bond failure patterns support this fact as the incidence of bond failure occurs more often between the SS brackets and adhesive than ceramic brackets, suggesting that incomplete polymerization might be the detrimental factor (Miyazaki et al. 1996; Graber 2005). For example, there are two broad categories of bond failure; adhesive failure and cohesive failure (Owens and Miller 2000). The first involves bond failures at the junction of enamel and the adhesive used to bond the bracket to the tooth. These types of breakdown often occur due to bonding procedure. Usually a step in a bonding procedure has been compromised or inadequately performed such as insufficient etch of enamel, lack of moisture control or deficient pellicle removal from the bonding surface. On the other hand, cohesive bond failures occur at the interface of the mesh padding of the bracket system and the adhesive. These failures frequently occur due to disruption in the polymerization process. Certainly, moving the bracket too much after it has been placed, using too little pressure when first applying the bracket, using SS brackets or excessively loading the bracket during the initial polymerization process which occurs under ambient light can cause increase in the incidence of cohesive bond failures (Brantley and Eliades 2001; Graber 2005)

Bond Strength

Bracket bond failure is a frustrating and a critical problem as it can negatively alter the integrity of an orthodontic appliance and cause a delay in overall treatment (Powers et al. 1997); (Powers and Sakaguchi 2006). In addition, when it occurs frequently it can have severe financial consequences to the practicing orthodontist as it results in loss of materials, increased chair time and often a lengthening of treatment time. As such many studies have investigated the factors affecting bond strength. Unfortunately, Mandall et al (2002) point out that there is a pronounced variety of controls and experimental designs in orthodontic bond strength investigations (Mandall et al. 2002). As a result the observed dissimilarity between the studies that have been previously investigated show marked clinical and statistical variation (Fox et al. 1994; Mandall et al. 2002; Lugato et al. 2009). For example, bond strength reported in one meta-analysis ranged from 3.5 to 27.8 MPa. This span grossly overlaps the scope of the clinically acceptable value of bond strength which is 6 to 8 MPa as suggested by Reynolds in his 1975 review of direct orthodontic bonding (Reynolds 1975). Also, the most commonly reported variables for in-vitro bond strength were adhesive type, crosshead speed, cleaning of enamel, etchant type, etching time, specimen storage time, storage solution of teeth before bonding, bracket type, total polymerization time, force location on bracket, photo-polymerization device, and blade design (Reynolds 1975; Reynolds and von Fraunhofer 1976a, b; Fox et al. 1994; Mandall et al. 2002; Finnema et al. 2010; Mansour et al. 2011). In a comprehensive and up to date review Finnema et al. (2010) examined 121 studies and found that of the variables previously mentioned only three were consistently shown to effect bond strength. They also concluded that many studies did not accurately record test circumstances, thereby significantly affected their potential outcomes.

Thus, only water storage of the bonded specimens, photo-polymerization time, and crosshead speed were identified as factors that affected orthodontic bond strengths. Furthermore, it was determined that storing the bonded teeth in tap water decreased bond strength by 10.7 MPa, while every successive second of light curing time improved bond strength by 0.077 MPa, and finally, bond strength increased by 1.3 MPa when crosshead speed was increased by 1mm per minute (Finnema et al. 2010). In order to achieve optimum bonding conditions during this current study, phosphate buffered saline (PBS) with sodium azide which more accurately represents the conditions in the oral environment will be used in lieu of tap water. Also, twenty seconds of light polymerization will be utilized because it is the time suggested by the Unitek bonding procedural guide and no optimum time for light curing has been scientifically determined (3M bonding guide). This most likely is associated with the change in light technology over the decades and lack of controls with previous investigations. Crosshead speed will be 1 mm per minute as it is the most universally accepted speed during in-vitro studies. In another study by Murfitt et al. (2006), 39 patients had a total of 661 brackets bonded to their teeth for a 12 month period. A variety of factors were analyzed such as patient gender, age, and tooth location in the dental arch and the number of manipulations of the bracket prior to curing. The results demonstrated that none of the factors significantly affected bond failures rate although it was noted that bracket that had been manipulated 4 or more times had a 100 % increase in bond failure rate (Murfitt et al. 2006). Indeed, it is suggested that once a bracket is placed on tooth structure, positioned, and excess adhesive is removed there should no longer be any further manipulation of the bracket in order to reduce the chances of bonding failure (Watts 2001; Graber 2005). Furthermore, Brantley and Eliades (2001) found that bond strength, time elapsed prior to curing, the amount of bracket

manipulation, and the ambient light may affect orthodontic bond failure rate (Brantley and Eliades 2001). In 2010, Gange examined the effects of bracket manipulation in two different groups. In the first he rotated orthodontic bracket approximately 5-10 degrees in a clockwise direction. In the second, the brackets were turned 10 degrees in a clockwise direction then twisted back to their original position. He concluded that there was no difference in shear strength between these groups (Gange 2010). Although there was no significant difference between these experimental groups, there was no control that could evaluate whether or not these groups had altered bond strengths compared to brackets that were not manipulated. Current literature points to the notion that too much or untimely bracket manipulation may cause decrement in orthodontic bond strengths although there is no research that has investigated how a specific degree of manipulation affects the bond strength (Brantley and Eliades 2001; Murfitt et al. 2006). Indeed as brackets with adhesive remain on tooth structure waiting to be cured, ambient light from normal clinical conditions start the curing process to a small degree (Martin 2008). Often times there is a delay between bracket placement and the final positioning and curing of the adhesive. Orthodontic auxiliaries may place the brackets and wait for the orthodontist to ultimately adjust the brackets before the adhesive is cured. Or perhaps due to bonding protocol many brackets are placed consecutively before curing takes place. Preliminary observational measurements have demonstrated that the delay, which varies greatly between different private practice settings, can range from 5 to 15 minutes (See Appendix A). During this time period, C=C double bonds in adhesive begin to be converted to C-C single bonds to form polymerized networks. Manipulating these brackets after a designated amount of time can cause detrimental effects in final cured adhesive structure and therefore the bond strength. With a delay in curing

under ambient light conditions, it is unknown how much decrement will occur in the bond strength of orthodontic brackets.

Problem Statement

No published research to date has described the effect of bracket manipulation after varying delay time, on orthodontic bond strength. More specifically, no research has investigated the degree of polymerization and change of shear bond strength that results due to such processes on ceramic and stainless steel brackets. The effect of bracket manipulation after varying delay time on orthodontic bonding is important because the adherence of orthodontic brackets to tooth structure provides the ability to control tooth movement. Often, brackets are placed on teeth and let to sit for lengthy times before they are manipulated and cured in their final positions. If the polymerization process has already begun to a significant degree, then the potential for decrease in final bond strengths may increase significantly and result in clinical bond failures and thus an increase in total treatment time. The purpose of this study is to examine whether degree of polymerization and final shear bond strength will be affected by manipulation of stainless steel and ceramic brackets, subsequent to initial placement and varying delay time under controlled lighting conditions.

Hypotheses

1. The shear bond strength of stainless steel versus ceramic brackets will vary as a function of bracket manipulation in combination with delay time.
2. The degree of polymerization of adhesive associated with bonded stainless steel versus ceramic brackets will vary as a function of bracket manipulation in combination with delay time.

3. There will be a correlation between degree of polymerization of the adhesive and bracket shear bond strength.

CHAPTER 2

MATERIALS AND METHODS

Tooth Specimen Collection

Maxillary first premolar teeth are often extracted in orthodontic patients and are used for in vitro investigations. However, due to their relative unavailability, 60 intact, maxillary 3rd molars from patients of oral surgery private practices in the Kansas City area were collected (AHSIRB Protocol # 13-04-NHSR). They were randomly divided into six treatment groups of ten; since each tooth was used once, every treatment group was comprised of 10 samples. The teeth were collected in containers and stored in 0.9% phosphate buffered saline¹ (PBS) with 0.002% sodium azide at 4°C. Teeth were rejected upon collection and examination if there were cracks, evidence of fluorosis, restorations, caries, abfraction lesions, or any unusual morphology. Debris and soft tissue remnants that remained from extraction procedures were removed from the teeth before disinfection, then they were once again stored in PBS solution with azide until the teeth were mounted.

Tooth Mounting

One tooth at one time was randomly selected and removed from refrigeration for testing. Each tooth was mounted in self-cured acrylic resin². With a mounting jig, the flattest part of the crown was oriented perpendicular to a plastic mounting ring³ filled with

¹ Dulbecco's Phosphate Buffered Saline, Sigma-Aldrich, 3050 Spruce St., St. Louis, MO 63103

² Biocryl #040-016, Great Lakes, 200 Cooper Ave., Tonawanda, NY 14150

³ Item #20-8180, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044

acrylic resin (Figure 1). Following one hour of setting time, the mounted teeth were removed from the jig and placed in PBS solution with azide until study commencement.

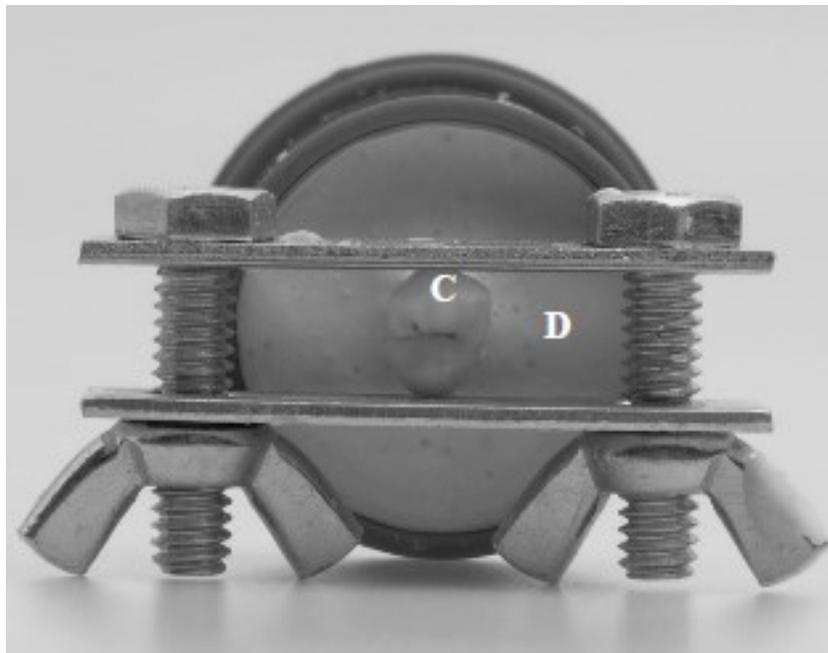


Figure 1. Mounting jig (A), plastic mounting ring (B), and maxillary tooth (C) fixed in self-cure acrylic resin (D). Profile view (top picture) and overhead view (bottom picture).

Bracket Bonding

Twin-wing orthodontic brackets, both SS⁴ and monocrystalline ceramic⁵, were used in this study. They are designed for maxillary first premolars with 0.018-inch slots in addition to concave bracket bases. Because third molars were used in this study and there are no orthodontic brackets specifically made for these variably-shaped teeth, a universal bracket, such as a maxillary first premolar bracket, was used to bond to the maxillary third molars. Universal maxillary first premolar brackets can be used interchangeably between first and second molars and were selected due to their ability to be used with various tooth surfaces and their size which most closely adapted to the surface contours of the third molars. The resin composite cement⁶ used was made up of 70-80% silane-treated quartz, 10-20% Bis-GMA, 5-10% Bisphenol A Bis (2-hydroxyethyl ether) dimethacrylate (Bis-EDMA), and 2% silane-treated silica.

In order to simulate clinical conditions, bracket bonding procedures were completed in an environmental chamber at 33°C (+/-2°) and 75% humidity (Plasmans et al. 1994). According to 3M Unitek bonding protocol the buccal surface of each tooth was polished mechanically with a slow speed hand piece using fluoride-free pumice⁷ and rubber cup for 5 seconds. The pumice was then rinsed off with an air/water spray for 5 seconds, followed by an air spray for 5 seconds. Once the tooth surface was properly prepared a self-etching primer⁸ was activated and applied to the tip of the primer brush. The primer was then rubbed on the center of the buccal surface of each tooth for 5 seconds. The primer brush was then

⁴ Master Series/MBT, American Orthodontics, 3524 Washington Ave., Sheboygan, WI 53081-1048

⁵ Radiance Plus™/MBT, American Orthodontics, 3524 Washington Ave., Sheboygan, WI 53081-1048

⁶ Transbond XT™, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

⁷ 1st & Final® pumice, Reliance Orthodontic Products, 1540 West Thorndale Ave, Itasca, IL 60143

⁸ Transbond Plus Self Etching Primer™, 3M Unitek, 2724 South Peck Road, Monrovia, CA 91016

placed back into the primer package and a gentle air burst was applied to the tooth for 2 seconds to produce a thin film. Cotton pliers were then used to pick up and load a premolar bracket with a uniform thickness of resin composite. The bracket was seated against the enamel surface, aligned with the pre-marked 10 degree offset, and depressed with a Hollenbeck carver⁹ to fully express any excess resin. The excess resin was then cleaned off around the bracket with the same carver. The bracket remained under 1, 200 lux lighting conditions, depending on the experimental group, until the following times elapsed (0.5min, 5 min, 10 min). Light intensity and delay times were based on observational measurements that were made in three orthodontic practices (See Appendix A).

After the appropriate time, the bracket was rotated, using the center of each bracket as the point of rotation, 10 degrees counterclockwise by aligning it with the long axis of the tooth. Each tooth was marked on the midpoint of the buccal cusp tip with a fine tip, black permanent marker. A protractor was then used to mark exactly 10° from the midpoint line, parallel to the long axis of the tooth. These two marks established the amount each bracket would be turned after the allotted time. The bracket adhesive was then cured with a curing light unit¹⁰ for a total of 20 seconds, 10 seconds from the gingival and 10 seconds from the occlusal according to manufacturer's specifications. Before each day of experimentation, the power density of the light curing device was checked with a radiometer¹¹ to ensure that the output being used was at least 400mW/cm².

⁹ Hollenbeck Carver, CVHL 1/2, Hu-Friedy, 3232 N. Rockwell, Chicago, IL 60618-5982

¹⁰ Ortholux™ LED Curing Light, 3M Unitek, 2724 South Peck Road, Monrovia, CA 91016

¹¹ OrthoLux LED radiometer, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

Shear Bond Strength Testing

Immediately after final curing of the orthodontic bracket, shear bond strength testing was performed using a universal testing machine¹² under ambient temperature and relative humidity conditions. A specimen holder with four locking screws was used to stabilize the mounted tooth and bracket on the universal testing machine platform (Figure 2). In order for load to be applied in vertical direction, parallel to the buccal surface of the tooth, the knife edge stainless steel rod attachment of the universal tester crosshead was positioned on the occlusal edge of the bonded bracket base (Figure 2). A crosshead speed of 1mm/min was used and the brackets were sheared off the enamel surface while maximum load in newtons (N) at debonding was recorded. Shear bond strength was calculated using the following equation:

$$\text{Shear bond strength (MPa)} = \text{Maximum compressive load (N)} / (\text{W} * \text{L})(\text{mm}^2)$$

where W= width of bracket base (mm), L = height of bracket base (mm). The bracket base area was 12.87 mm² and 10.73 mm² for ceramic and stainless steel brackets, respectively.

Representative load/extension graph data collected from the universal testing machine can be seen in Figure 3.

¹² Model 5967, Instron Corporation, 825 University Ave., Norwood MA 02062-2643

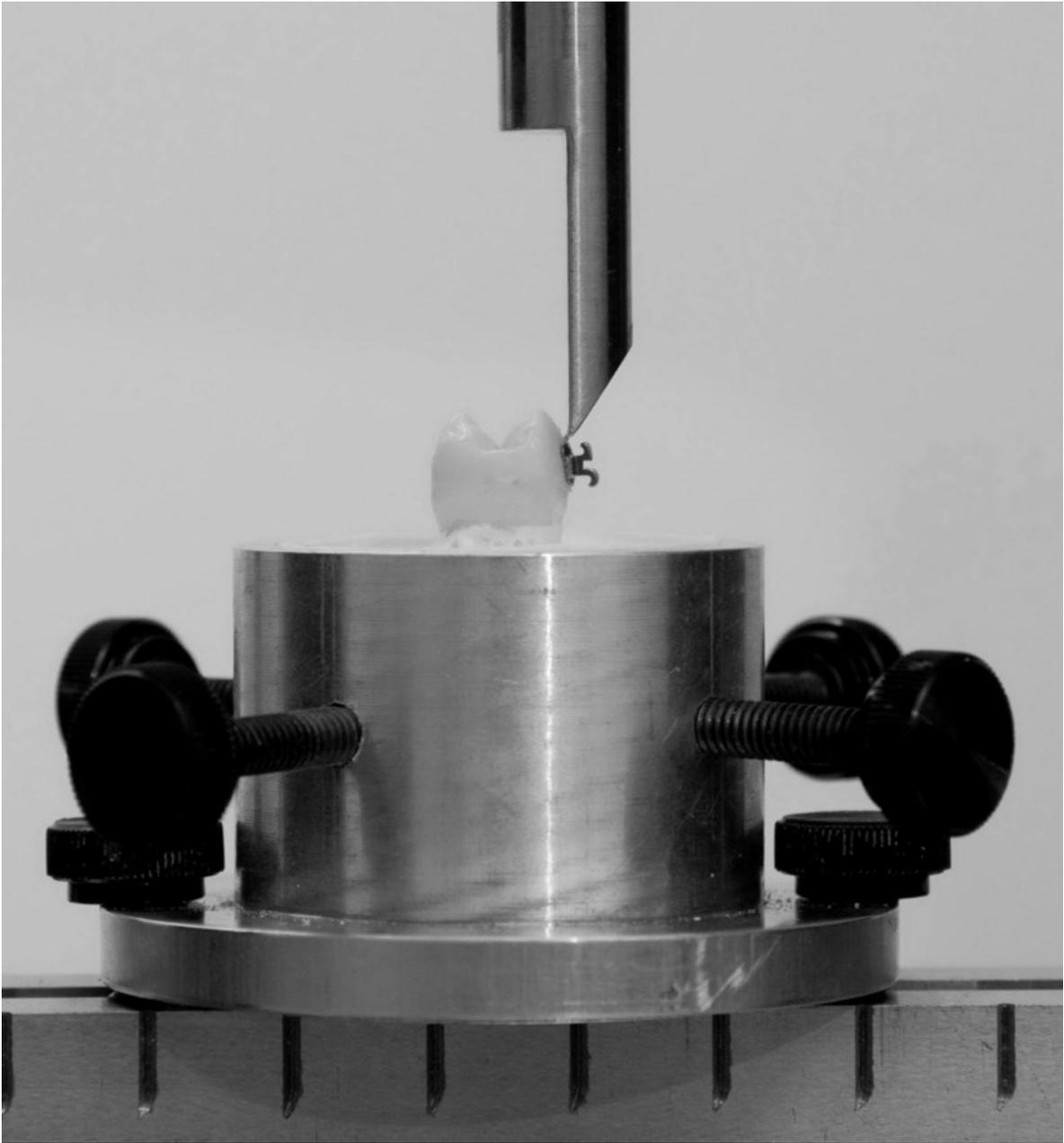


Figure 2. Specimen holder with locking screws used to stabilize the mounted tooth and bracket on the universal testing machine platform underneath the knife edge of the steel rod.

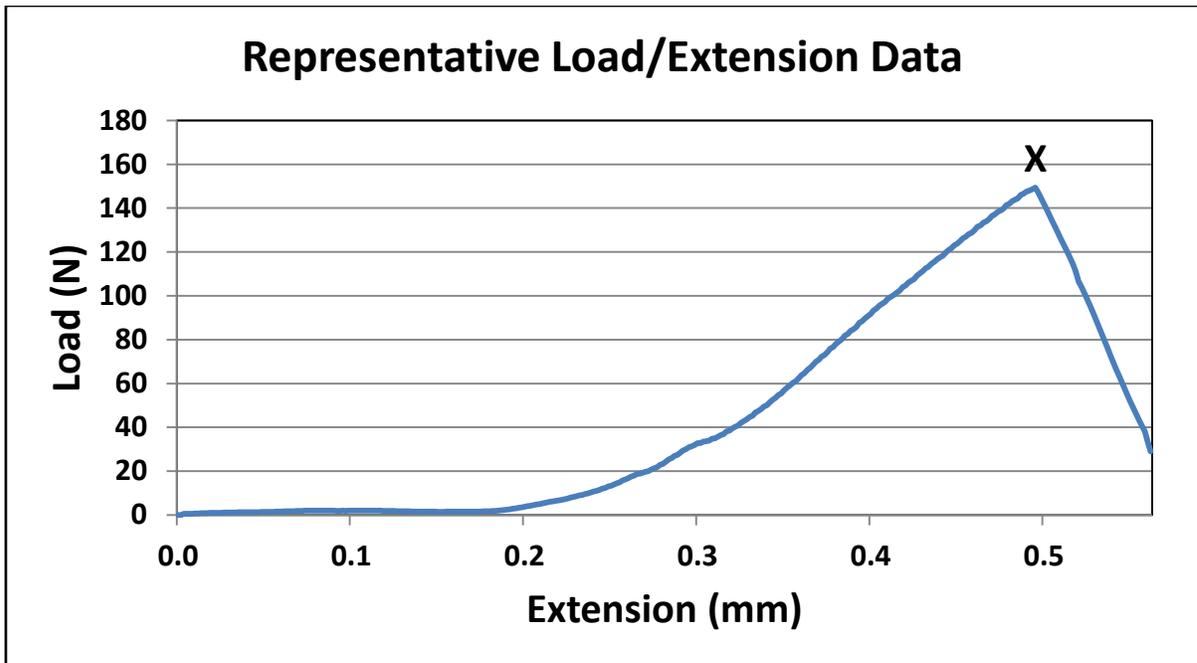


Figure 3. Representative load/extension graph collected during orthodontic shear bond strength testing. Maximum compressive load (as indicated by X) divided by bracket base area was used to calculate shear bond strength (MPa).

Degree of Conversion Measurements

Raman spectral collection was done immediately following shear bond strength testing and was finished within one hour after initial light activation for bracket bonding in order to minimize any dark cure effects, whereby polymerization can continue to occur subsequent to light activation in the absence of a continuous light source. If there was enough remaining cured adhesive on the base of the bracket, then point measurements were made in three separate locations. Conversely, if there was not a sufficient amount of cured adhesive on the bracket base, then point measurements were made on the residue on the enamel surface of the tooth. Micro-Raman¹³ analysis was performed using a He-Ne laser (wavelength of 632.8 nm) with spatial resolution of 1.5 μm . Spectra were collected in the region of 2000-440 cm^{-1} with a spectral resolution of 2.5 cm^{-1} .

Preceding each series of measurements, the spectrometer was calibrated internally using known lines of a silicon sample. A 60s span was used for spectral acquisition time, and two accumulations for a total of 120s per site was used. The laser was focused through a 50x objective lens. Spectral measurements of unpolymerized adhesive were made using the same instrumentation parameters.

In order to accurately calculate band heights, the Grams/AI fitting software¹⁴ was used. To establish a reference point an auto fit function of the software was applied over the entire range of collected spectra. Two-point baseline and maximum band height protocol were used to measure the band intensities.

¹³ HR800, Horiba Jobin, Yvon, 231 Rue deVille, Villeneuve, France 59650

¹⁴ GRAMS/AI v7.02, Galactic Industries Corp., 395 Main St., Salam, NH 03079

The degree of conversion was calculated as follows:

$$DC (\%) = 100 * [1 - (R^{\text{polymerized}} / R^{\text{unpolymerized}})]$$

here $R = \text{band height at } 1640 \text{ cm}^{-1} / \text{band height at } 1610 \text{ cm}^{-1}$. Figure 4 show representative data collected from micro-Raman spectroscopy analysis.

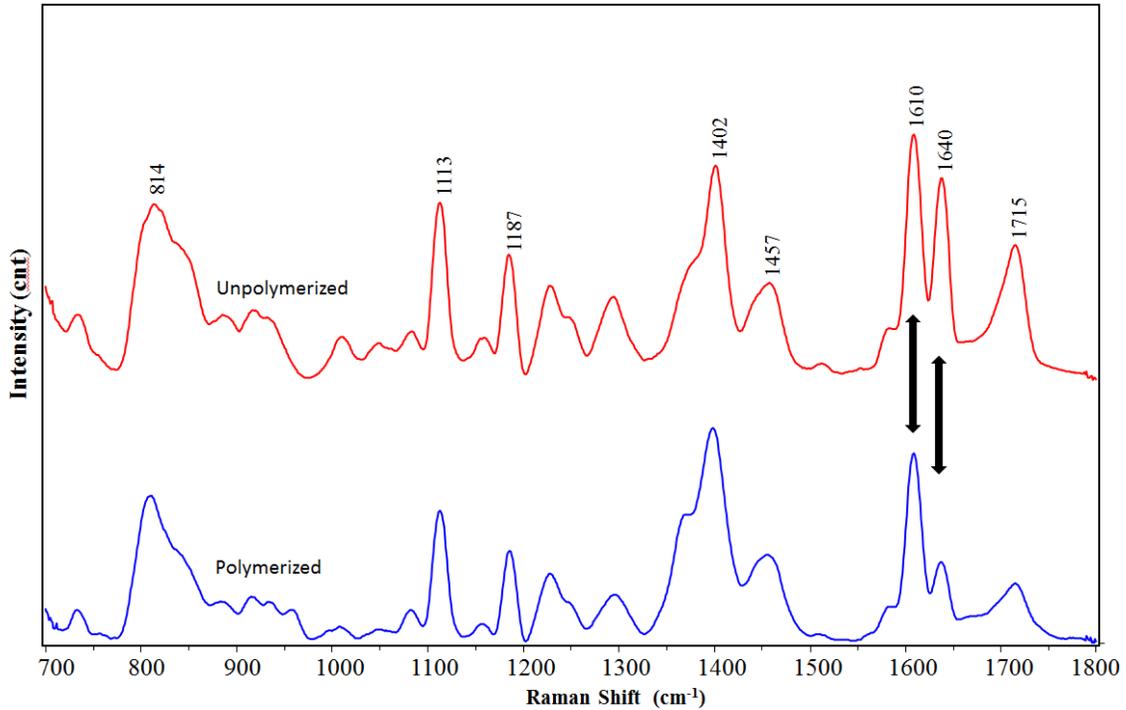


Figure 4. Representative Raman spectra for unpolymerized and polymerized resin composite. Peaks at 1610 cm^{-1} and 1640 cm^{-1} (arrows) were used for degree of conversion calculations.

Study Design and Sample Size

TABLE 1

RESEARCH DESIGN

| Bracket Type | Delay Time Prior to Manipulation and Light Cure | Shear Bond Strength (MPa) | Percentage Bond Strength Change from 30s (Baseline) | Degree of Conversion |
|----------------------------|--|------------------------------|--|-------------------------|
| Stainless Steel | 0.5 min | | - | |
| | 5 min | | | |
| | 10 min | | | |
| Monocrystalline Ceramic | 0.5 min | | - | |
| | 5 min | | | |
| | 10 min | | | |

*A convenience sample of 10 brackets was selected thereby resulting in 10 brackets/experimental group.

Data Analysis

Data was analyzed using the SPSS statistical software program¹⁵. Within each bracket type, shear bond strength and degree of conversion was evaluated quantitatively and was analyzed using descriptive statistics such as mean, standard deviation, and range. A one-factor (delay time) multivariate analysis of variance (MANOVA, $\alpha = 0.05$) was done for each bracket type to compare bond strength and degree of conversion as a function of delay time intervals before light curing. If significant differences were detected, a Tukey's post hoc ($\alpha = 0.05$) was used to determine where the differences existed.

To compare the effect of delay time on bond strength and degree conversion between the two types of brackets, the percent change in bond strength as a function of delay time (percent change at 5 and 10 min as compared to 30 sec) was compared using a 1-factor univariate ANOVA, while the degree conversion was directly compared between bracket types. Pearson correlations were also used to determine whether shear bond strength and degree conversion were correlated at each time point for each bracket type.

¹⁵ SPSS Version 20, 223 S. Wacker Dr., Chicago, IL 60606

CHAPTER 3

RESULTS

Shear Bond Strength Measurements

Sixty maxillary third molars were randomly assigned to one of six experimental groups. Orthodontic bracket bonding and shear bond strength testing was performed according to the protocol described in Chapter 2. Means and standard deviations (SD) of shear bond strength of SS and ceramic testing groups are presented in Figures 5 and 6 respectively.

Based on a 1-factor multivariate analysis of variance (MANOVA) within each bracket type, there were no significant differences between shear bond strength and delay times for ceramic brackets. However, with SS brackets there was a significant increase ($p \leq 0.05$) of shear bond strength over time. Based on Tukey's post-hoc analysis, the significant difference was identified between the 0.5 min and 10 min SS bracket groups. Based on these results, hypothesis one was not supported when considering ceramic brackets, but partially supported with SS brackets.

Degree of Conversion Measurements

Degree of conversion measurements were performed according to the protocol described in Chapter 2. Means and standard deviations (SD) of degree of conversion for SS and ceramic testing groups are presented in Figures 7 and 8 respectively.

Based on a 1-factor multivariate analysis of variance (MANOVA) within each bracket type, there were no significant differences between degree of conversion and delay times of ceramic brackets. Conversely, significant effects ($p \leq 0.05$) of delay time on degree

of conversion were identified in SS bracket groups. Degree of conversion increased across time but according to Tukey's post-hoc analysis only degree of conversion at a delay times at 0.5 min were significantly lower than at 5 min and 10 min, which were not different from each other. These results did not support hypothesis two for ceramic brackets, but partially substantiated hypothesis two for SS brackets.

Correlation between Shear Bond Strength and Degree of Conversion

Based on Pearson correlation analysis, there were no significant correlations between shear bond strength and degree of conversion of either orthodontic bracket group. The lack of correlation between shear bond strength and degree of conversion for all brackets did not support hypothesis three.

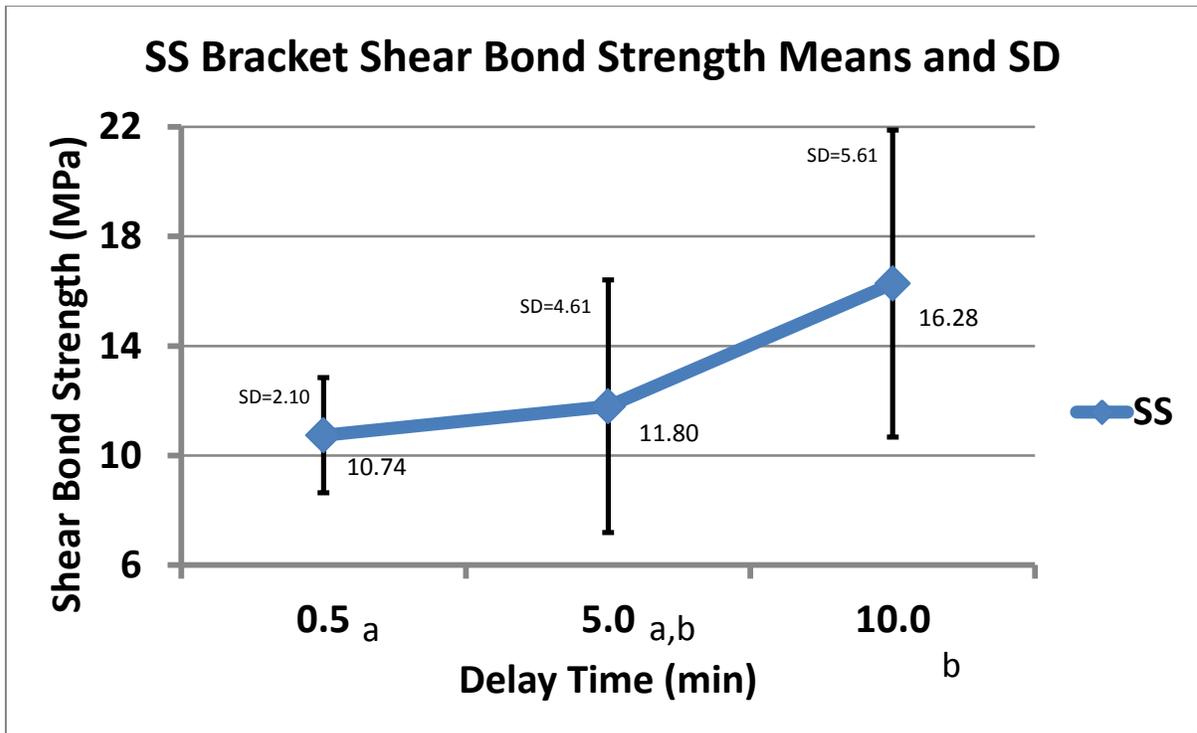


Figure 5. Mean and SD shear bond SS bracket strength values. N = 10/bracket type and delay time. There was a significant difference in shear bond strength as a function of time. As noted on graph, bond strength was significantly higher after 10 min as compared to 0.5 min (subsets are indicated by letters).

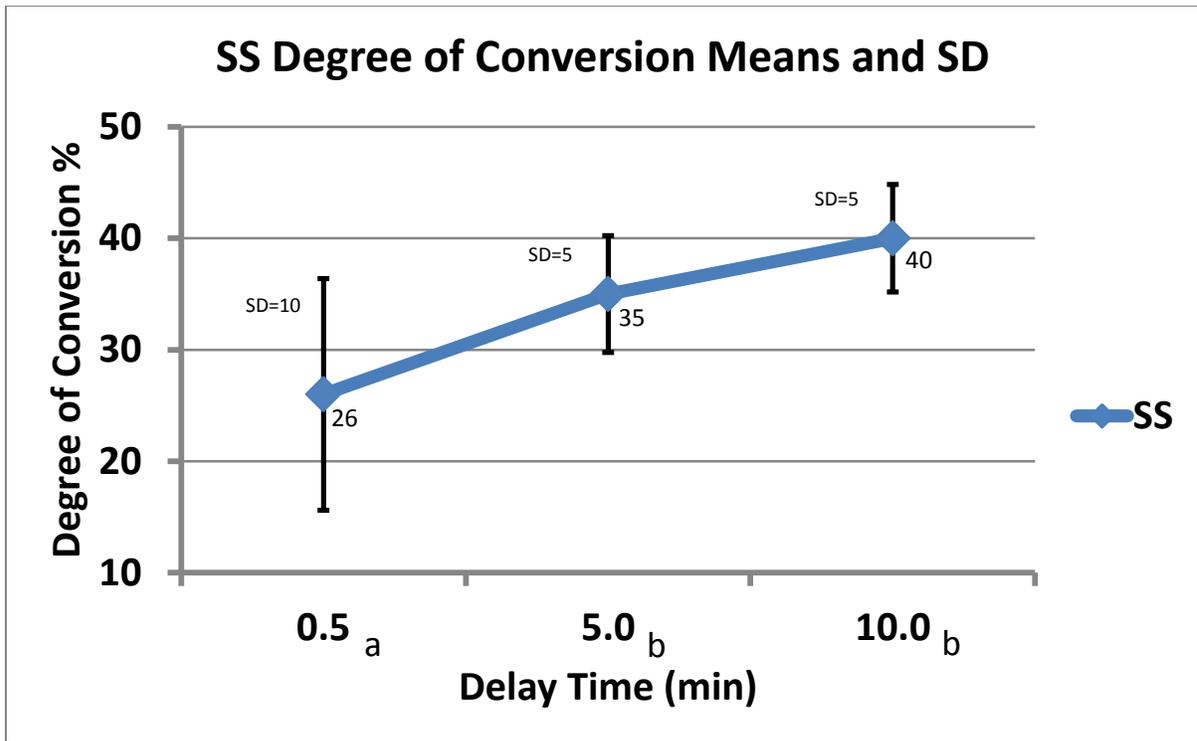


Figure 6. Mean and SD degree of conversion values of SS brackets. N = 10/bracket type and delay time. Degree conversion was significantly lower after 0.5 min as compared to after 5 and 10 min, which were not different from each other (subsets indicated by letters).

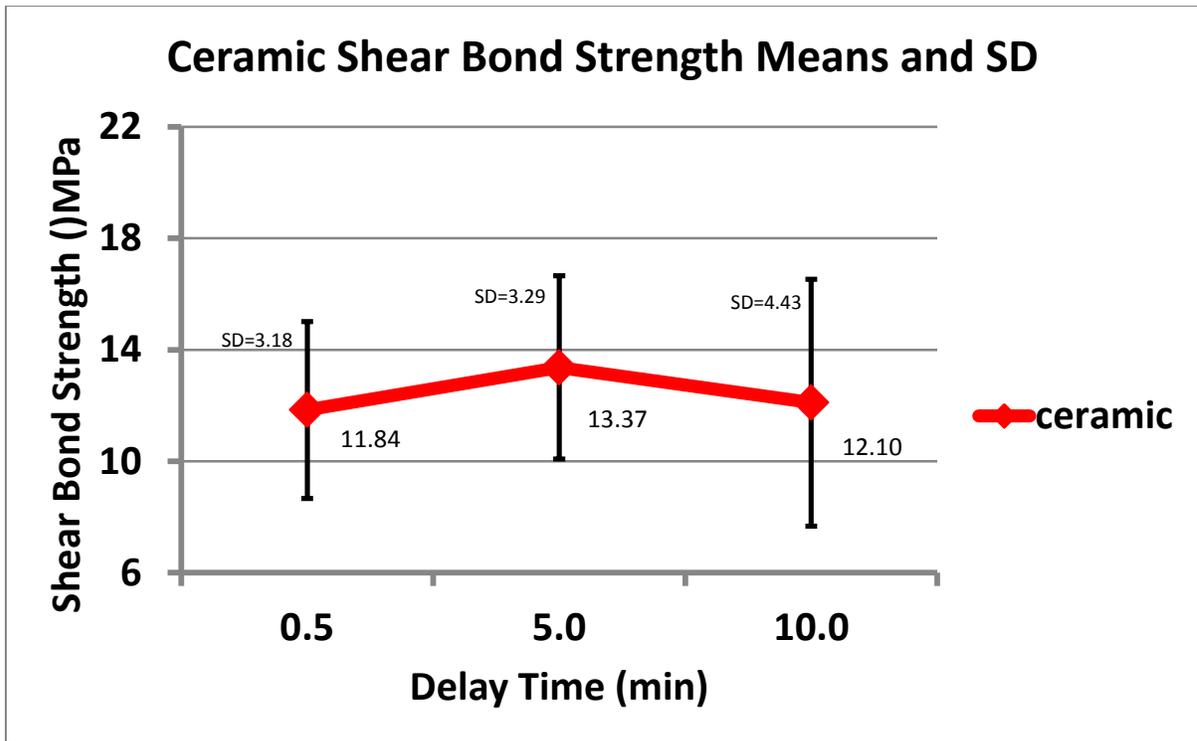


Figure 7. Mean and SD shear bond ceramic bracket strength values. N = 10/bracket type and delay time. There was no significant difference between shear bond strength across time.

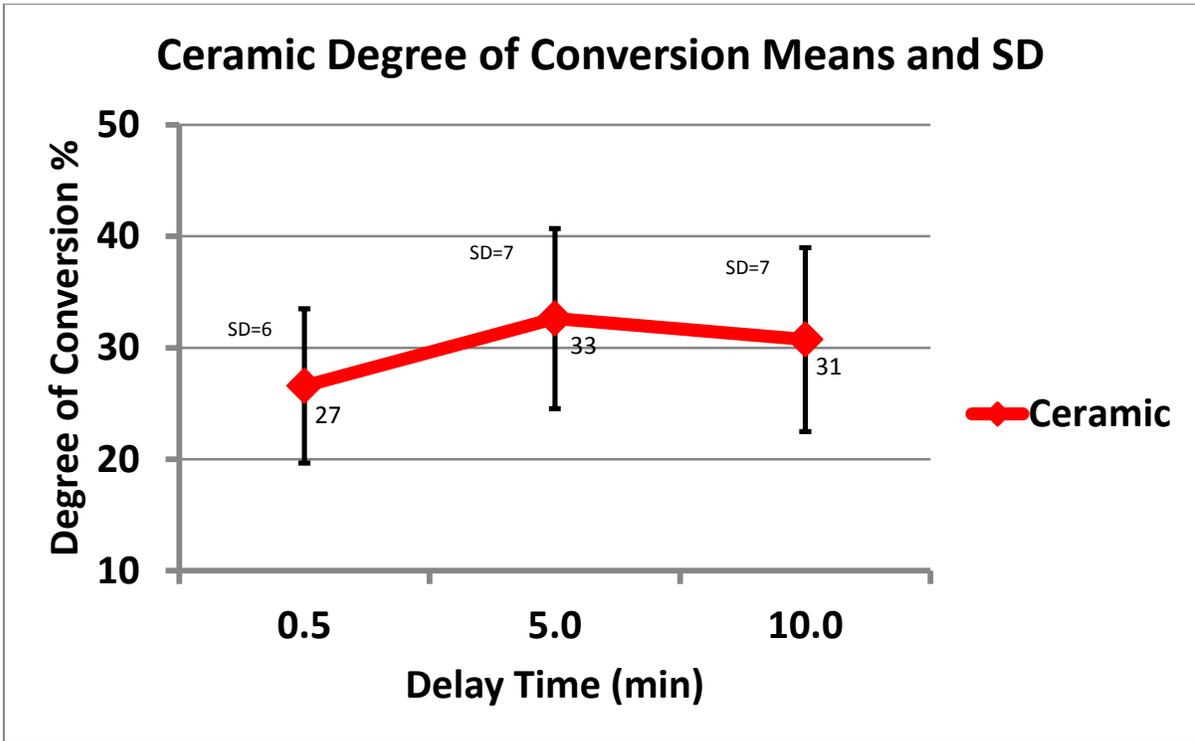


Figure 8. Mean and SD degree of conversion values of ceramic brackets. There was no significant difference in DC across time.

CHAPTER 4

DISCUSSION

The most ubiquitous adhesive used in orthodontics is a light-cure resin composite system and it is therefore pertinent to understand how it behaves when exposed to light sources that begin the curing process within the material (Sakaguchi et al. 1992a; Keim et al. 2008). To date there have been several studies in which the effects of light or bracket manipulation on degree of conversion and shear bond strength of composite resin have been evaluated. (Gange 2006; Murfitt et al. 2006; Shinya et al. 2009; Rachala and Yelampalli 2010); however, little has been done to examine their combined effects. Therefore this study investigates the effect of bracket manipulation after varying delay times on degree of polymerization and final shear bond strength under controlled lighting conditions.

Shear Bond Strength

The results of the current study showed that there was no difference in shear bond strengths over the range of delay times for ceramic brackets. As for the SS brackets, there was a significant effect of a 10 min delay time on bond strength before manipulation and curing. These results are in contrast to the first hypothesis which states that the shear bond strength of stainless steel versus ceramic brackets will vary as a function of bracket manipulation in combination with delay time. Although no previous research has examined the effect of bracket manipulation prior to curing during different delay times, this study demonstrates that after a given amount of time under ambient light, shear bond strength increases for SS brackets while it remains constant for ceramic brackets.

This finding is contrary to common notion that bond strength decreases over time as uncured resins are exposed to light, which starts the polymerizing/curing process. It is believed that if brackets are moved during this period, micro-fractures would occur throughout the cured adhesive that would compromise bond strength. In fact the shear bond strengths for SS brackets over time were even higher than strengths reported in literature for brackets bonded without delay times (Murray and Hobson 2003; Hajrassie and Khier 2007). Although it is prudent to cautiously evaluate data from different studies as different materials and experimental protocols could lead to an invalid comparison for shear bond strength numbers, it is uncertain why shear bond strength stayed the same across time for ceramics brackets but increased over time for SS brackets.

One possible explanation involves the heat which radiated from the light source and the relative thermal conductivity of the brackets used in this study. Research has shown that the thermal conductivity of metals is much higher than that of ceramics (Hirata 2009). The most classic example involves the silica fibers used to protect space shuttles when transitioning from orbit to the Earth's atmosphere. It exemplifies the extreme insulating properties of ceramics, whereby these materials can be heated to several thousand degree Fahrenheit and can subsequently be touched by hand within seconds (Callister and Rethwisch 2013). Due to these insulating properties it is fair to deduce the monocryalline brackets may have insulated the underlying adhesive and primer. Conversely, the base of SS brackets is completely metallic, thereby protecting the resin from light but possibly conducting its heat. Previous research has demonstrated that increased temperature of adhesive can increase the mechanical properties of the material including shear bond strength (Cantoro et al. 2008). The temperatures used to obtain significant results in the literature are likely

higher than those achieved in the current condition; however, it is possible that heat from the light source radiated into the resin beneath the metal bracket. Because of the conducting properties associated with SS brackets, the associated resin composite temperature could have risen slowly and eventually increased shear bond strength in the 10 min time delay group. With ceramic brackets, since they insulate the underlying material, the composite would be unaffected by the temperature.

Similarly, as temperature increases, the viscosity of the composites decreases as they become more flowable (Cantoro et al. 2008). In one study, an increase in the temperature of the resin resulted in a thicker hybrid smear layer and an increase in resin tag diffusion which would allow for better dentin tubule infiltration, as was exemplified using scanning electron microscope images (Cantoro et al. 2008). In addition it is reasonable to assume that the decrease in viscosity would also allow for better adaptation of the resin to the base of the brackets. Again, as temperature is presumed to have no effect on ceramic brackets, maximal resin adaptation to the bracket base and therefore maximum shear bond strength would occur by the first time delay group (0.5 min) and remain constant thereafter. The resin with the conductive SS brackets would warm more slowly and become less viscous as time increases, thereby exhibiting higher shear bond strengths in the longer delay time groups.

Furthermore, the bracket bases have different architecture. The ceramic brackets have a patented design called a Quad matte base, which has alumina particles only on the base's center (Empower 2012). This ultimately gives stronger bonding in the middle of the bracket pad while allowing for a weaker bonding area around the perimeter of the bracket. This design would likely not capitalize on the decreased viscosity of the resin as much as the bracket base for SS brackets. The SS bracket pad area was comprised of an etched foiled

with a mesh overlay, which produces a superior mechanical lock compared to ceramic brackets (Empower 2012). As a result, the SS bracket base would greatly benefit from reduced resin viscosity, which would occur during the longer delay time groups. As time increases and resin becomes more flowable, it can infiltrate into the mesh pockets of the base, thus increasing the shear bond strength properties.

Another possible explanation involves the hydrophilic component of the primer. Transbond self-etching primer is composed of several chemicals that mix together once all chambers of the package are combined. One of these components is water, which is used as a solvent, and comprises approximately 15-25% in volume (Unitek 2010b). The water is designed to evaporate after the bonding surface is wetted with the adhesive primer and is aided by air bursting the surface of the tooth (Unitek 2012). It is possible that the amount of evaporation for ceramic brackets did not change over time, due to constant temperatures of the associated resin. Since the ceramic bracket material shielded the composite from heat, evaporation rates would remain constantly low, resulting in similar water concentration of primer in all ceramic bracket delay time groups. On the other hand, since the primer under the SS brackets was potentially subject to more heat, due to conduction properties of metal, the evaporation rate of the water solvent is expected to be faster. Overall, the primer would become more concentrated as its composition would contain less water over time and shear bond strength would concurrently increase.

As delay time increases, additional etching may also be a process that is continuing to occur, which is related to several factors including amount of ambient light curing, interaction of primer with hydroxyapatite, and the elimination of surplus primer by way of air thinning with an air-water hand piece (Cinader 2010). All three factors act to neutralize the

etching process within the primer and when considering the light-transmitting ceramic brackets, excessive ambient light may play a role in halting the etching process at a level that does not continue past the first delay time group (Cinader 2010). Conversely, less light interacts with the primer under the opaque SS brackets, which may continue to etch for a long period of time. As this process continues for SS brackets, demineralization and primer infiltration occur simultaneously giving a deeper etching depth and primer penetration, which could increase bond strength during the 10 min delay time versus early delay time groups (Unitek 2010b).

Degree of Conversion

The second hypothesis of this study stated that the degree of polymerization of adhesive associated with bonded SS versus ceramic brackets will vary as a function of bracket manipulation in combination with delay time. It was found that degree of conversion did not significantly change during different delay times with ceramic brackets. However, when considering SS brackets, there were significant differences between the 0.5 min delay group in comparison with both the 5 min and 10 min groups. The paucity of research in this particular area does not offer any antecedent explanations for these significant differences. One possible explanation is that although the base of SS bracket is opaque and completely blocks light penetration, ambient light may still penetrate the surrounding tooth material. As a result, the entire crown can become illuminated, thereby reflecting light from the enamel to the neighboring orthodontic resin composite (Eliades and Brantley 2001). This process would slowly begin the curing process in the adhesive with SS brackets but would not be the case when using the translucent monocrystalline ceramic brackets. Here the ambient light

would penetrate through the bracket material and directly begin to cure the underlying resin composite. Indeed, in one study investigators examined the degree of conversion of Transbond XT after 20 s of light curing under different conditions (Shinya et al. 2009). One group of adhesive was directly light cured compared with a group that was light cured with an orthodontic bracket on top of it, and a third group with a glass fiber net added between the bracket and adhesive. They found the degrees of conversion for these groups to be 54.7, 37.0, and 44.1%, respectively. The author concluded that an increase of light transmission led to an increase in degree of conversion, which is corroborated by current theories in the literature. Here it is believed that the more total energy absorbed by a given resin will lead to a higher degree of conversion (Oesterle et al. 2001; Burgess et al. 2002). Furthermore, the total amount of energy can be delivered by light of different sources, combinations and exposure times (Miyazaki et al. 1996; Peutzfeldt and Asmussen 2005).

However, others do not agree that this relationship is simply linear, for example, investigators in another study tested the degree of conversion of resin composite receiving high and low amounts of total energy (Bang et al. 2004). Their results indicated that both groups were not significantly different even though one group received 100% more light energy than the other. This may suggest that materials have degree of conversion limit that does not continue to rise after a certain amount of light energy. In addition it is important to consider the kinetics of polymerization. In general, the speed with which energy is delivered to a resin system affects the polymerization process. When a given amount of energy is delivered in a short period of time with high intensity the polymerization process becomes subsequently inefficient. Degree of conversion is much higher for the same amount of energy if it is delivered over a longer period of time with lower intensity, resulting in longer

polymer chains, increased polymer network cross-linking, and molecules with higher molecular weight (Millar and Nicholson 2001). Therefore in respect to the current study, the slow increase in the total energy absorbed by the resin underneath the SS brackets may in fact lead to a higher degree of conversion over time as observed in the 10 min delay time group. The process is likely not as efficient in composite underneath ceramic brackets as it is directly exposed to light allowing for a rapid increase in total energy. The high influx of photon absorption through the monocrystalline structure could likely push the polymerization process of the resin to its limit in this environment leading to an early plateau of degree of conversion.

Furthermore as light energy totals accumulate slowly, so do resin composite temperature increases, which could be a factor in degree of conversion. One study investigated the degree of conversion on the surface of specimens between a room temperature and 60°C groups. In the first group the degree of conversion was reported at 55.5% after 30s of light polymerization, while 68.3% degree of conversion was observed in the higher temperature group (Lovell et al. 2001; Daronch et al. 2006; Awliya 2007; Prasanna et al. 2007). In another investigation, 10s of light polymerization was used to examine polymerization of the surface of the composite material at two different temperatures. It was found that the degree of conversion was 47.6% at room temperature and 65.4% at 60 °C (Lovell et al. 2001; Daronch et al. 2006; Awliya 2007; Prasanna et al. 2007). In the current study, resin composite temperatures may have remained constant, due to the insulating effects of the ceramic brackets, therefore showing no difference in conversion between time delay groups. On the other hand, the composite with the SS brackets may have had a slow rise in temperature as light and heat was transmitted by the metal bracket, thereby

increasing over time and concurrently allowing for a steady increase of degree of conversion over time. However, it is important to note that temperature of the adhesive was not measured in the current study, so this potential effect of increased temperature on either bracket bond strength or degree of conversion is based on speculation.

Correlation between Degree of Conversion and Bond Strength

Our third hypothesis stated that there would be a correlation between degree of polymerization of the adhesive and bracket shear bond strength. This postulate was based on a finding that a greater degree of conversion would be concomitant with increased physical properties such as flexural strength, resistance to fracture, microhardness, bond strength, resistance to wear, surface hardness, (Lovell et al. 2001; Awliya 2007; Cantoro et al. 2008; Sadek et al. 2008). However, the research conducted from these studies was not specifically on the shear bond strength of orthodontic composite resin. Furthermore, many different experimental protocols were used to examine the relationships between degree of conversion and the properties of the materials. Two recent studies have investigated the correlation between shear bond strength and degree of conversion of orthodontic composite resin with mixed results. One study found a moderately positive correlation between shear bond strength and degree of conversion as different curing units and different levels of total energy were used to polymerize resin composite (Henbest 2013). The second study examined the effect of pre-warming resin composite cement prior to bonding orthodontic brackets (Ries 2010). The results of this study indicated that there was no correlation between shear bond strength of orthodontic brackets and degree of conversion of composite resin. Similarly, the findings in the current investigation indicated no correlation between degree of conversion

and shear bond strength of either bracket group. The lack of association may be due to the use of adhesive primer. In orthodontic studies, resin composite is often bonded to enamel that needs to be etched and primed with an adhesive primer. Studies that examine resin composite often do not use a primer, thus leaving out a key factor that may change the chemical composition of the resin composite used in clinical based orthodontic investigations. When this primer is included in the bonding process it may interfere with the hybrid layer that it is involved in forming between the enamel and resin composite, thereby altering shear bond strength and its correlation with degree of conversion.

Study Limitations

In this study, maxillary third molars were used instead of premolars, which is what the base pads of the orthodontic brackets were designed for. Although, only well-formed third molars were selected for experimentation, they are the most variable teeth in the human dentition. The non-uniformity of the surface anatomy of these teeth could have affected bracket-tooth adaptability and therefore outcome metrics.

Crosshead speed used in this investigation was 1mm/min. Intraorally, the vertical forces created as teeth shear past one another are much faster than the experimental rate used, which was primarily utilized to compare data from this experiment to others in the literature using the same 1mm/min debond speed.

Saliva is another factor encountered in the intraoral environment that was not accurately represented in the experimental trials. Saliva can create a pellicle on the surface of the tooth that can create complications for bonding protocols if not removed in an adequate manner (Graber 2005). Although, a humid environment chamber was used during

bonding of all brackets, it only approximates the intraoral environment at best, while providing no saliva substitute.

The primer used in this experiment was a one-step etch and prime system that is very popular in private practice orthodontics. However, there are many other bonding systems available, including a two-step etch and prime system that could possibly have reacted differently under ambient light and time delayed bracket manipulations.

Also, teeth were collected and stored for a period of 4 months, a process that potentially altered the surface and integrity of the enamel bonding surface.

When mounting teeth in acrylic, although jigs were used, variation in three dimensional orientations of teeth could have contributed to inaccurate bracket placement. Inaccurate bonding of orthodontic brackets to the heights of contour of the tooth anatomy and its long axis are correlated to operator skill, even though a jig was also used for this process. Also, teeth that are not mounted completely perpendicular to the horizon and brackets not placed with extreme accuracy would compromise the ability to measure shear bond strength.

Clinical Significance

In terms of shear bond strength which is the most clinically significant factor assessed in this investigation, the results show that allowing brackets, either SS or ceramic, to sit on teeth under ambient light over extended periods of times before manipulation into final position, does not negatively affect bracket bond strength. In fact, bond strengths for SS brackets increase over time up to ten minutes, while those for ceramic brackets remained the same within a 10 min time period. These findings are important to the practitioner placing brackets as great lengths are often taken to prevent light from reaching the resin composite

associated with these brackets. Indeed, anecdotal evidence ubiquitously states that exposure to light before final manipulation of orthodontic brackets, especially with ceramic materials, is extremely detrimental to the bonding process and will result in increased bond failures and decrease practice efficiency. However, the findings of the current study do not support this wide spread subjective belief. The current results demonstrate that SS or ceramic brackets coated with adhesive and using a single-step etch and prime protocol, do not need to be shielded from ambient light. This is contingent upon the fact that light intensities do not exceed 1,200 lux, which is what can be expected when direct light from an operatory unit is turned away from the patient, during sunny environmental conditions in an office with many windows (See Appendix A).

Future Investigations

It was verified that shear bond strength of orthodontic brackets was not negatively impacted even after 10 min of time delay before final bracket manipulation, positioning and bonding. Future investigations could examine conditions similar to the current study but with longer delay times. The delays chosen would include time points past the optimal strength of the resin composite and even to the point of bond failure, thereby allowing the assessment of the total working range of the material which is often used in orthodontics. Furthermore, different bonding and bracket materials could be used in order to determine how they affect the shear bond strength outcomes. For example, there are multiple types of resin modified glass ionomers that release fluoride. Although they are currently not as ubiquitous as resin composite, it is still advantageous to examine the effects of ambient light on the performance of these materials. They can play a role in preventing caries and may become more popular

in the future as their bond strengths are engineered to become stronger, therefore ameliorating their most detrimental drawback. Also, there are different degrees of bracket clarity. While monocrystalline brackets are almost completely translucent, polycrystalline brackets, due to their multi-grain structure, appear frosty and would transmit a different amount of light, thereby leading to altered bond strengths when exposed to ambient light prior to curing. In addition, it has been postulated in this study that heat from ambient light sources may increase bond strengths, albeit showing a varying effect dependent on the type of bracket material used. A polycrystalline bracket would benefit from study as it would have to insulating properties of ceramic materials although would let less transmit through the base of the brackets, similar to SS brackets. This type of bracket could help delineate whether heat and light or a combination of the two is an important factor in bond strength and degree of conversion when different bracket materials are utilized. Also, to accurately examine any relationship between performance of different bracket types and heat, it would be advantageous to introduce an investigation that measures the temperature of not only the local environment but of the resin composite itself, perhaps using a temperature sensor such as a thermocouple.

CHAPTER 5

CONCLUSIONS

1. The shear bond strength of ceramic brackets did not vary as a function of bracket manipulation in combination with time delay. However, with SS brackets there was a significant effect ($p \leq 0.05$) of delay time on shear bond strength between the 0.5 min and 10 min bracket groups.
2. There were no significant differences between degree of conversion and delay times of ceramic brackets. Conversely with SS brackets, significant effects ($p \leq 0.05$) of delay time on degree of conversion were identified between the 0.5 min and 5min groups, in addition to the 0.5 min and 10 min groups.
3. There was no correlation between degree of polymerization of the adhesive and bracket shear bond strength.

LITERATURE CITED

- Abate PF, Zahra VN, Macchi RL. Effect of photopolymerization variables on composite hardness. *J Prosthet Dent* 2001;86:632-5.
- Awliya WY. The influence of temperature on the efficacy of polymerization of composite resin. *J Contemp Dent Pract* 2007;8:9-16.
- Bang HC, Lim BS, Yoon TH, Lee YK, Kim CW. Effect of plasma arc curing on polymerization shrinkage of orthodontic adhesive resins. *J Oral Rehabil* 2004;31:803-10.
- Barry GR. A clinical investigation of the effects of omission of pumice prophylaxis on band and bond failure. *Br J Orthod* 1995;22:245-8.
- Brantley WA, Eliades T. Instrumental techniques for study of orthodontic materials. In: *Orthodontic materials scientific and clinical aspects*. WA Brantley and T Eliades editors. Stuttgart: Thieme; 2001, pp. 49-76.
- Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955;34:849-53.
- Burgess JO, Walker RS, Porche CJ, Rappold AJ. Light curing--an update. *Compend Contin Educ Dent* 2002;23:889-92, 94, 96 passim; quiz 908.
- Callister WD, Rethwisch DG. Thermal properties. In: *Materials science and engineering: An introduction*. 9th ed. United States of America: Wiley; 2013, pp. 785-803.
- Cantoro A, Goracci C, Papacchini F, Mazzitelli C, Fadda GM, Ferrari M. Effect of pre-cure temperature on the bonding potential of self-etch and self-adhesive resin cements. *Dent Mater* 2008;24:577-83.
- Cinader. Chemical processes and performance comparisons of transbond plus self etching primer. Senior Project Development Engineer 2010;3M Unitek.
- Daronch M, Rueggeberg FA, Moss L, de Goes MF. Clinically relevant issues related to preheating composites. *J Esthet Restor Dent* 2006;18:340-50.
- Degarmo PE, Black JT, Kohser RA. Ferrous metals and alloys. In: *Materials and processes in manufacturing*. 11th ed. United States of America: John Wiley & Sons, Inc; 2012, pp. 152-82.
- Eliades G, Brantley WA. Instrumental techniques for study of orthodontic materials. In: *Orthodontic materials: Scientific and clinical aspects*. WA Brantley and T Eliades editors. New York: Thieme; 2001, pp. 49-76.
- Empower. Empower self ligating product guide. Sheboygan, WI: American Orthodontics; 2012.
- Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU. In-vitro orthodontic bond strength testing: A systematic review and meta-analysis. *Am J Orthod Dentofacial Orthop* 2010;137:615-22 e3.

- Fleming PS, Johal A, Pandis N. Self-etch primers and conventional acid-etch technique for orthodontic bonding: A systematic review and meta-analysis. *Am J Orthod Dentofacial Orthop* 2012;142:83-94.
- Fox NA, McCabe JF, Buckley JG. A critique of bond strength testing in orthodontics. *Br J Orthod* 1994;21:33-43.
- Gange P. Bonding in today's orthodontic practice. *J Clin Orthod* 2006;40:361-7.
- Gange P. Personal communication. Itasca, Illinois: Reliance Orthodontics Products Incorporated; 2010.
- Gautam P, Valiathan A. Ceramic brackets: In search of an ideal. *Trends Biomater. Artif. Organs* 2007;20(2):117-22.
- Glasspoole EA, Erickson RL, Davidson CL. Effect of enamel pretreatments on bond strength of compomer. *Dent Mater* 2001;17:402-8.
- Graber TM. Bonding in orthodontics. In: *Orthodontics: Current principles and techniques*. BU Zachrisson and T Buyukyilmaz editors. 5th ed. St. Louis: Elsevier Science; 2005, pp. 727-84.
- Hajrassie MK, Khier SE. In-vivo and in-vitro comparison of bond strengths of orthodontic brackets bonded to enamel and debonded at various times. *Am J Orthod Dentofacial Orthop* 2007;131:384-90.
- Hanson GH. The speed system: A report on the development of a new edgewise appliance. *Am J Orthod* 1980;78:243-65.
- Henbest N. Orthodontic bracket bond strength and resin composite adhesive degree of conversion associated with type of curing unit and total energy. Kansas City, MO: University of Missouri at Kansas City; 2013, pp. 1-18.
- Heravi F, Bayani S. Changes in shear bond strength of ceramic and stainless steel brackets with different visible light curing times and directions. *Aust Orthod J* 2009;25:153-7.
- Hirata Y. Thermal conduction model of metal and ceramics. *Ceramics International* 2009;35:3259-68.
- Ireland AJ, Sherriff M. The effect of pumicing on the in vivo use of a resin modified glass poly(alkenoate) cement and a conventional no-mix composite for bonding orthodontic brackets. *J Orthod* 2002;29:217-20; discussion 196.
- Keim RG, Gottlieb EL, Nelson AH, Vogels DS, 3rd. 2008 jco study of orthodontic diagnosis and treatment procedures, part 1: Results and trends. *J Clin Orthod* 2008;42:625-40.
- Legler LR, Retief DH, Bradley EL, Denys FR, Sadowsky PL. Effects of phosphoric acid concentration and etch duration on the shear bond strength of an orthodontic bonding resin to enamel. An in vitro study. *Am J Orthod Dentofacial Orthop* 1989;96:485-92.

- Lill DJ, Lindauer SJ, Tufekci E, Shroff B. Importance of pumice prophylaxis for bonding with self-etch primer. *Am J Orthod Dentofacial Orthop* 2008;133:423-6; quiz 76 e2.
- Lindauer SJ, Browning H, Shroff B, Marshall F, Anderson RH, Moon PC. Effect of pumice prophylaxis on the bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop* 1997;111:599-605.
- Lovell LG, Lu H, Elliott JE, Stansbury JW, Bowman CN. The effect of cure rate on the mechanical properties of dental resins. *Dent Mater* 2001;17:504-11.
- Lugato IC, Pignatta LM, Arantes Fde M, Santos EC. Comparison of the shear bond strengths of conventional mesh bases and sandblasted orthodontic bracket bases. *Braz Oral Res* 2009;23:407-14.
- Mandall NA, Millett DT, Mattick CR, Hickman J, Worthington HV, Macfarlane TV. Orthodontic adhesives: A systematic review. *J Orthod* 2002;29:205-10; discussion 195.
- Mansour AY, Drummond JL, Evans CA, Bakhsh Z. In vitro evaluation of self-etch bonding in orthodontics using cyclic fatigue. *Angle Orthod* 2011;81:783-7.
- Martin BD. Working time evaluation of light activated orthodontic adhesives. Baltimore, Maryland: University of Maryland; 2008, pp. 1-12.
- Millar BJ, Nicholson JW. Effect of curing with a plasma light on the properties of polymerizable dental restorative materials. *J Oral Rehabil* 2001;28:549-52.
- Milne JW, Andreasen GF, Jakobsen JR. Bond strength comparison: A simplified indirect technique versus direct placement of brackets. *Am J Orthod Dentofacial Orthop* 1989;96:8-15.
- Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength of light-cured composites. *Dent Mater* 1996;12:328-32.
- Murfitt PG, Quick AN, Swain MV, Herbison GP. A randomised clinical trial to investigate bond failure rates using a self-etching primer. *Eur J Orthod* 2006;28:444-9.
- Murray SD, Hobson RS. Comparison of in vivo and in vitro shear bond strength. *Am J Orthod Dentofacial Orthop* 2003;123:2-9.
- Oesterle LJ, Newman SM, Shellhart WC. Rapid curing of bonding composite with a xenon plasma arc light. *Am J Orthod Dentofacial Orthop* 2001;119:610-6.
- Omura I, Yamauchi J, Haruda I, Wade AT. Adhesive and mechanical properties of a new dental adhesive. *Journal of Dental Research* 1984;63:233.
- Owens SE, Jr., Miller BH. A comparison of shear bond strengths of three visible light-cured orthodontic adhesives. *Angle Orthod* 2000;70:352-6.

- Ozcan M, Finnema K, Ybema A. Evaluation of failure characteristics and bond strength after ceramic and polycarbonate bracket debonding: Effect of bracket base silanization. *Eur J Orthod* 2008;30:176-82.
- Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. *J Dent Res* 2005;84:659-62.
- Plasmans PJ, Creugers NH, Hermsen RJ, Vrijhoef MM. Intraoral humidity during operative procedures. *J Dent* 1994;22:89-91.
- Powers JM, Kim HB, Turner DS. Orthodontic adhesives and bond strength testing. *Semin Orthod* 1997;3:147-56.
- Powers JM, Sakaguchi RL. Resin composite restorative materials. In: Craig's restorative dental materials. JM Powers and RL Sakaguchi editors. 12th ed. St. Louis: Mosby; 2006, pp. 189-212.
- Prasanna N, Pallavi Reddy Y, Kavitha S, Lakshmi Narayanan L. Degree of conversion and residual stress of preheated and room-temperature composites. *Indian J Dent Res* 2007;18:173-6.
- Proffit WR. Contemporary orthodontic appliances. In: Contemporary orthodontics. 4th ed. St. Louis: Elsevier Science; 2007, pp. 395-431.
- Rachala MR, Yelampalli MR. Comparison of shear bond strength of orthodontic brackets bonded with light emitting diode (led). *Int J Orthod Milwaukee* 2010;21:31-5.
- Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod* 1975;2:171-8.
- Reynolds IR, von Fraunhofer JA. Direct bonding of orthodontic attachments to teeth: The relation of adhesive bond strength to gauze mesh size. *Br J Orthod* 1976a;3:91-5.
- Reynolds IR, von Fraunhofer JA. Direct bonding of orthodontic brackets--a comparative study of adhesives. *Br J Orthod* 1976b;3:143-6.
- Ries D. In vitro bond strength of orthodontic brackets and resin composite cement degree of conversion as a function of pre-cure temperature. Kansas City, MO: University of Missouri at Kansas City; 2010, pp. 1-13.
- Rux W, Cooley RL, Hicks JL. Evaluation of a phosphonate bis-gma resin as a bracket adhesive. *Quintessence Int* 1991;22:57-60.
- Sadek FT, Calheiros FC, Cardoso PE, Kawano Y, Tay F, Ferrari M. Early and 24-hour bond strength and degree of conversion of etch-and-rinse and self-etch adhesives. *Am J Dent* 2008;21:30-4.
- Sakaguchi RL, Cross M, Douglas WH. A simple model of crack propagation in dental restorations. *Dent Mater* 1992a;8:131-6.
- Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. *J Dent* 1992b;20:183-8.

- Sakaguchi RL, Peters MC, Nelson SR, Douglas WH, Poort HW. Effects of polymerization contraction in composite restorations. *J Dent* 1992c;20:178-82.
- Saunders CR, Kusy RP. Surface topography and frictional characteristics of ceramic brackets. *Am J Orthod Dentofacial Orthop* 1994;106:76-87.
- Shinya M, Shinya A, Lassila LV, Varrelä J, Vallittu PK. Enhanced degree of monomer conversion of orthodontic adhesives using a glass-fiber layer under the bracket. *Angle Orthod* 2009;79:546-50.
- Smith WF, Hashemi J. Engineering alloys. In: *Foundations of material science and engineering*. 5th ed.: McGraw-Hill; 2009, pp. 427-522.
- Strydom C. Curing lights--the effects of clinical factors on intensity and polymerisation. *SADJ* 2002;57:181-6.
- Swartz ML. Orthodontic bonding. *Orthod Select* 2007;16:1-4.
- Tamizharasi S. Evolution of orthodontic brackets. *J IADS* 2010;1:24-30.
- Unitek M. Material safety data sheet 3M unitek transbond plus self etching primer (712-090, 712-091). Monrovia, CA: 3M Unitek; 2010a.
- Unitek M. Apc plus adhesive coated appliance system: Faq. Monrovia, CA: 3M Unitek; 2010b.
- Unitek M. Bonding technique guide. Monrovia, CA: 3M Unitek; 2012.
- Watts DC. Orthodontic adhesive resins and composites: Principle of adhesion. In: *Orthodontic materials. Scientific and clinical aspects*. WA Brantley and G Eliades editors. 1st ed. New York; 2001, pp. 190-200.
- Zachrisson BU. Long-term experience with direct-bonded retainers: Update and clinical advice. *J Clin Orthod* 2007;41:728-37; quiz 49.
- Zinelis S, Annousaki O, Eliades T, Makou M. Elemental composition of brazing alloys in metallic orthodontic brackets. *Angle Orthod* 2004;74:394-9.

APPENDIX

DATA COLLECTION FOR LIGHTING CONDITIONS AND DELAY TIMES

Lighting conditions and delay time data was collected by visiting three separate private practice orthodontic offices on two different occasions. Ambient light condition data was collected using a lux meter¹⁶ during bright and dim environmental conditions in order to approximate a range of lighting conditions in a clinical setting (Table 2). Delay time was collected and determined by calculating the time that elapsed between the initial placement of orthodontic brackets and their subsequent repositioning before final light curing in a clinical setting during patient bracket bonding procedures (Table 3). Due to the observed measurements a light intensity of 1, 200 lux was selected because it is the upper limit of what could be expected during sunny environmental conditions in the intraoral environment. In addition, delay times of 5 and 10 min were chosen for experimentation as they approximate lower and upper limits of delay times seen in private practice orthodontic offices.

¹⁶ LX1010B Lux Meter, Sinometer, Sunshine Golf Building, 7008 Shennan Boulevard, Shenzhen, China

TABLE 1
LIGHTING CONDITIONS DATA COLLECTION

| Outside Lighting Conditions | Lux Meter Location | Distance from Outside Lighting (ft) | Light Intensity (Lux) |
|-----------------------------|--------------------|-------------------------------------|-----------------------|
| Sunny, No Clouds | Chairside | 10 | 6,700 |
| | Intraoral | 10 | 1,216 |
| | Chairside | 30 | 3,250 |
| | Intraoral | 30 | 640 |
| Overcast, Cloudy | Chairside | 10 | 1,060 |
| | Intraoral | 10 | 225 |
| | Chairside | 30 | 360 |
| | Intraoral | 30 | 80 |

TABLE 2
 DELAY TIME DATA COLLECTION

| Office Number | Delay Time |
|---------------|-------------|
| Office 1 | 13 min 4 s |
| | 12 min 36 s |
| | 10 min 22 s |
| Office 2 | 9 min 16 s |
| | 7 min 14 s |
| | 7 min 56 s |
| Office 3 | 6 min 6 s |
| | 6 min 18 s |
| | 9 min 45 s |

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