

**THE SHEAR/PEEL BOND STRENGTHS
OF ORTHODONTIC ATTACHMENTS TO
COMPOSITE RESIN**

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requirements for the degree of

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The Shear/Peel Bond Strengths of Orthodontic Attachments to Composite Resin

BY

Christopher James Woolaver

**A Thesis/Practicum submitted to the Faculty of Graduate Studies of The University
of Manitoba in partial fulfillment of the requirements of the degree
of
Master of Science**

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ABSTRACT

The objective of this study was to evaluate the shear/peel bond strengths of orthodontic attachments bonded to restorative composite resin using various adhesives and surface treatment protocols. The restorative resin Z100™ (3M, St. Paul, MN) was used in 120 cylindrical samples, polished with Sof-lex™ disks (3M), and stored in distilled water at 37°C for 7 days. Three surface treatments were then performed as follows: untreated (polished surface), acid-etched with 34% phosphoric acid for 15 seconds (3M/Unitek, Monrovia, CA), or air-abraded with 50µm aluminum oxide particles under 80 psi for 5 seconds using a Micro-Etcher (Danville Engineering, Danville, CA). 240 orthodontic buttons (GAC, Central Islip, NY) were bonded to each end of the samples. The two adhesives tested were Transbond™ XT (3M/Unitek) and Smartbond® (GAC). Samples were subsequently tested for shear/peel bond strength using a Zwick Materials Testing Device with a crosshead speed of 0.5mm/min. Samples were either tested after storage for 24 hours in distilled water at 37°C or after storage for 7 days in distilled water at 37°C and subsequent thermocycling of 500 cycles between 5°C and 55°C. Statistically significant ($p < 0.05$) differences were found in the shear/peel bond strengths, with air-abraded surfaces bonded with Transbond™ XT after 24 hours producing the highest value of 21.98 +/- 2.42 MPa. The lowest shear/peel bond strength was achieved in the thermocycled untreated/Smartbond® group, with a value of 6.88 +/- 1.29 MPa. All forty air-abraded samples bonded with Transbond™ XT experienced restorative resin fracture upon debonding. No other samples exhibited resin damage. It was concluded that orthodontic attachments can successfully be bonded to untreated, acid-etched, or air-abraded composite resin surfaces *in vitro* using either Transbond™ XT or Smartbond®, and mean shear/peel bond strengths ≥ 6 MPa can be achieved after 24 hours or after thermocycling. Air-abraded surfaces bonded with Transbond™ XT produced the highest shear/peel bond strength; however, this protocol also led to a high prevalence of resin fractures.

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CONTENTS

	Page Number
TITLE PAGE	i
COMMITTEE CERTIFICATION	ii
ABSTRACT	iii
ACKNOWLEDGEMENTS	iv
CONTENTS	v-viii
LIST OF FIGURES	ix-xi
LIST OF TABLES	xi

CHAPTER 1

INTRODUCTION

1.1	Foreword	2-4
1.2	Motivation for the Study	4-5
1.3	Purpose of the Study	5-6
1.4	Null Hypothesis	6

CHAPTER 2

LITERATURE REVIEW

2.1	Concept of Adhesion and Bonding	8-9
-----	---------------------------------	-----

2.2	Composite Resin	
2.2.1	Historical Background of Composite Resin	10-14
2.2.2	Aging and Water Sorption	14-16
2.2.3	Composite to Composite Bonding	16-19
2.2.4	Composite Resin Surface Treatments	19-21
2.3	Orthodontic Bonding	
2.3.1	Direct Bonding	22-23
2.3.2	Orthodontic Adhesives	23-25
2.3.3	Orthodontic Bonding to Various Materials	26-29
2.3.4	Bond Strength in Orthodontics	30-31

CHAPTER 3

MATERIALS AND METHODS

3.1	Materials Used in the Study	
3.1.1	Restorative Composite Resin	33-34
3.1.2	Composite Orthodontic Adhesive System	35-36
3.1.3	Cyanoacrylate Orthodontic Adhesive System	37-38
3.1.4	Orthodontic Attachments	38-39
3.2	Experimental Procedure	
3.2.1	Preparation of Composite Specimens	40-42
3.2.2	Composite Surface Treatments	43-44
3.2.3	Bonding of Orthodontic Attachments	44-46
3.2.4	Storage Conditions	46

3.2.5	Shear/Peel Bond Strength Testing	47-50
3.2.6	Evaluation of Fracture Sites	50-51
3.2.7	Statistical Analysis of Data	51
3.2.8	Preparation of S.E.M. Specimens	52

CHAPTER 4

RESULTS

4.1	Shear/Peel Bond Strengths	
4.1.1	24 Hour Shear/Peel Bond Strengths	54-56
4.1.2	Shear/Peel Bond Strengths After Thermocycling	56-58
4.1.3	Shear/Peel Bond Strengths After 24 Hour vs. Thermocycling	59-60
4.2	Modified Adhesive Remnant Index (ARI) Scores	61-66
4.3	Scanning Electron Microscopy Evaluation	66-74
4.4	Null Hypothesis	74

CHAPTER 5

DISCUSSION

5.1	Shear/Peel Bond Strengths	
5.1.1	24 Hour Shear/Peel Bond Strengths	76-82
5.1.2	Shear/Peel Bond Strengths After Thermocycling	82-85
5.2	Modified Adhesive Remnant Index (ARI) Scores	86-88

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1	Conclusions	90-91
6.2	Recommendations	92-93

APPENDICES	94-100
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REFERENCES	101-116
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LIST OF FIGURES

	Page Number
Figure 1: Fractured incisor	3
Figure 2: Composite resin repair	3
Figure 3: Cervical abrasion	3
Figure 4: Composite resin repair	3
Figure 5: Worn, fractured incisors	4
Figure 6: Composite veneers	4
Figure 7: Contact angle	9
Figure 8: Chemical structure of monomer in composite resins	11
Figure 9: Silane coupling agent	13
Figure 10: Z100™ restorative composite resin	33
Figure 11: Transbond™ XT light-cured orthodontic adhesive kit	35
Figure 12: Smartbond® orthodontic adhesive kit	37
Figure 13: Bondable orthodontic buttons	38
Figure 14: Z100™ sample, with mould	40
Figure 15: Ortholux™ light-curing unit	41
Figure 16: Sof-lex™ polishing discs	42
Figure 17: 500g attachment seating force	45
Figure 18: Bonding protocol diagram	47
Figure 19: Z100™ sample mounted in jig (diagram)	48
Figure 20: Shear blade applied parallel to interface (diagram)	48

Figure 21: Sample mounted in Bencor™ testing apparatus	49
Figure 22: Close-up of sample in Bencor™ apparatus	49
Figure 23: Zwick™ materials testing machine and computer	50
Figure 24: 24 hour mean shear/peel bond strengths graph	56
Figure 25: Mean shear/peel bond strengths of thermocycled samples graph	58
Figure 26: 24 hour vs. thermocycled samples mean shear/peel bond strengths graph	60
Figure 27: Debonded sample (20X) with ARI=0	61
Figure 28: Debonded sample (20X) with ARI=1	62
Figure 29: Debonded sample (20X) with ARI=2	62
Figure 30: Debonded sample (20X) with ARI=3	63
Figure 31: Debonded sample (20X) with ARI=4	64
Figure 32: ARI scores graph	65
Figure 33: SEM (300X) of polished resin surface	67
Figure 34: SEM (300X) of acid-etched resin surface	68
Figure 35: SEM (300X) of air-abraded resin surface	68
Figure 36: SEM (37X) of debonded sample with ARI=0	69
Figure 37: SEM (300X) of debonded sample with ARI=0	70
Figure 38: SEM (40X) of debonded sample with ARI=1	70
Figure 39: SEM (37X) of debonded sample with ARI=2	71
Figure 40: SEM (40X) of debonded sample with ARI=3	72
Figure 41: SEM (150X) of debonded sample with ARI=3	72
Figure 42: SEM (37X) of debonded sample with ARI=4	73

LIST OF TABLES

	Page Number
Table 1: 24 hour shear/peel bond strength descriptive statistics	55
Table 2: Shear/peel bond strength descriptive statistics of thermocycled samples	58
Table 3: Mean shear/peel bond strengths after 24 hour vs. thermocycling	60
Table 4: Mean modified ARI scores	65

CHAPTER 1

INTRODUCTION

	Page Number
1.1 Foreword	2-4
1.2 Motivation for the study	4-5
1.3 Purpose of the study	5-6
1.4 Null hypothesis	6

“For a long time it had seemed to me that life was about to begin – real life. But there was always some obstacle in the way, something to be got through first, some unfinished business, time still to be served, a debt to be paid. Then life would begin. At last it dawned on me that these obstacles were life.”

Sir Winston Churchill, 1945

CHAPTER 1

INTRODUCTION

1.1 FOREWORD

Adult patients contribute up to 24% of a private practice orthodontic case load (Gottlieb and Vogels, 1984). Considering today's epidemiological trends, it is likely that more adults will seek orthodontic treatment in the future (Gottlieb *et al.*, 1999). Many of these patients present with previously restored teeth. Numerous studies have established appropriate methods of bonding orthodontic attachments to materials other than enamel, including amalgam (Zachrisson and Büyükyilmaz, 1993; Zachrisson *et al.*, 1995; Büyükyilmaz and Zachrisson, 1996; Büyükyilmaz and Zachrisson, 1998; Sperber *et al.*, 1999), gold (Wood *et al.*, 1986; Zachrisson and Büyükyilmaz, 1993; Büyükyilmaz *et al.*, 1995), and porcelain (Johnson, 1980; Andreasen and Stieg, 1988; Kao *et al.*, 1988; Zachrisson *et al.*, 1996).

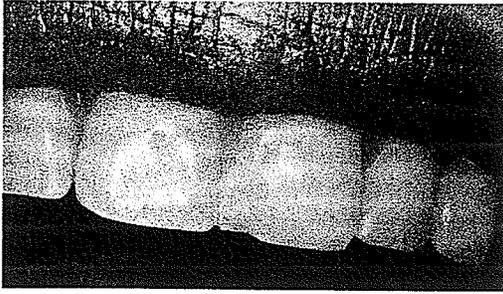


Figure 1. *Fractured incisor*

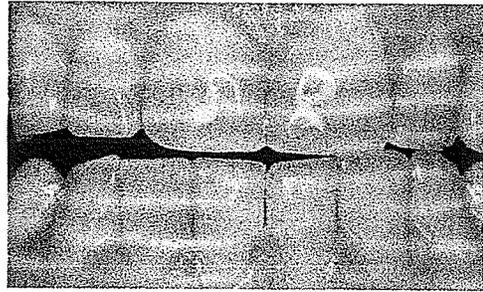


Figure 2. *Composite resin repair*

However, perhaps the most common restorative material that orthodontic attachments will have to be bonded to in the future is composite resin (Drake *et al.*, 1990). Composite resins are frequently used in restoring fractured anterior teeth (**Figures 1,2**), carious lesions and areas of abrasion (Goldstein, 1998) (**Figures 3,4**).

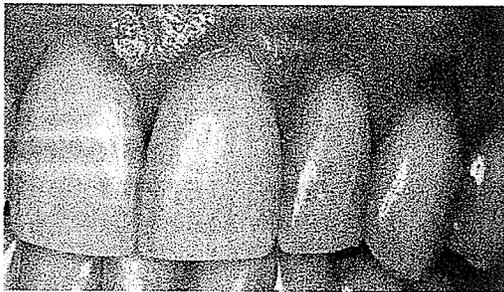


Figure 3. *Cervical abrasion*

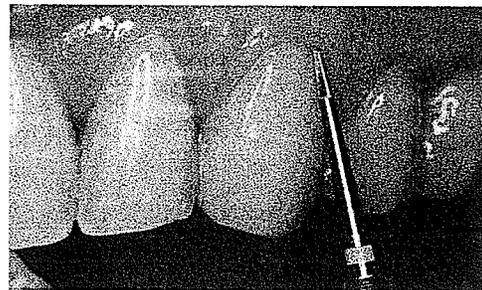


Figure 4. *Composite resin repair*

Composite veneers are also utilized for direct esthetic build-ups or as an alternative to porcelain (Goldstein, 1998) (Figures 5,6). When patients with these restorations seek orthodontic treatment, achieving an adequate immediate and long-term bond with orthodontic attachments is an obvious concern (Powers *et al.*, 1997). Unfortunately, the research and literature on orthodontic bonding to composite resin is limited (Lai *et al.*, 1999).

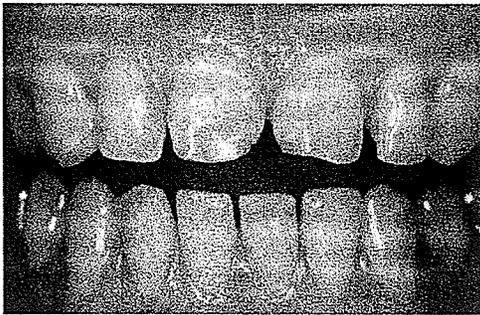


Figure 5. *Worn, fractured incisors*

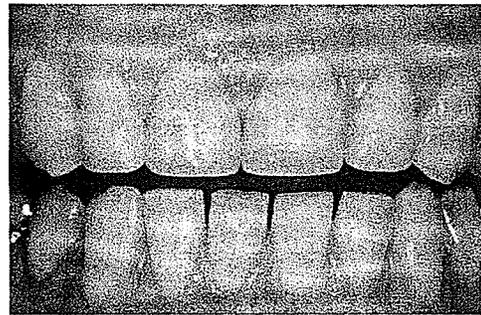


Figure 6. *Composite veneers*

1.2 MOTIVATION FOR THE STUDY

Bonding orthodontic attachments to composite restorations with a composite-based orthodontic adhesive is only known to have been reported a few times previously (Kao *et al.*, 1995; Chunchacheevachaloke and Tyas, 1997; Lai *et al.*, 1999). None of these studies investigated the effects of acid etching or air abrasion on the shear bond strength. Previous literature from the field of composite repair indicated that acid etching of the

composite surface did not significantly increase the bond strength (Boyer *et al.*, 1978), suggesting possible elimination of this clinical procedure. In its place, mechanical surface treatment, such as air-abrasion, appears to be more effective in increasing composite to composite bond strengths (Swift *et al.*, 1992; Turner and Meiers, 1993; Kupiec and Barkmeier, 1996; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997).

A new cyanoacrylate adhesive (Smartbond[®], Gestenco International, Göteborg, Sweden) has been developed for orthodontic bonding. The manufacturer claims the product provides sufficient bond strength to composite restorative materials. A previous cyanoacrylate adhesive was tested by Howells and Jones (1989). They found the material to have acceptable handling qualities (easily mixed, satisfactory viscosity, adequate working time), and comparable initial bond strength to a composite adhesive (124N vs. 132N). However, after storage in normal saline for 7 or 98 days, the hydrolysis of the polymerized material weakened the bond (to 6N), rendering it unsuitable for clinical use.

At present, no definitive protocol is known for bonding orthodontic attachments to restorative composite resin.

1.3 PURPOSE OF THE STUDY

1. To determine the effect of six different adhesive/surface treatment combinations on the shear/peel bond strength of orthodontic attachments to restorative resin after 24 hours and after thermocycling.

2. To determine the effect of thermocycling on the shear/peel bond strength of orthodontic attachments to restorative resin of the six experimental groups.
3. To evaluate the pattern of failure after debonding of orthodontic attachments to restorative resin.

1.4 NULL HYPOTHESIS

The null hypothesis states that there are no statistically significant differences in the shear/peel bond strength between the six adhesive/surface treatment groups. The null hypothesis also states that thermocycling has no effect on the shear/peel bond strengths.

CHAPTER 2

LITERATURE REVIEW

	Page Number
2.1 Concept of Adhesion and Bonding	8-9
2.2 Composite Resins	
2.2.1 Historical background of composite resin	10-14
2.2.2 Aging and water sorption	14-16
2.2.3 Composite to composite bonding	16-19
2.2.4 Composite resin surface treatments	19-21
2.3 Orthodontic Bonding	
2.3.1 Direct bonding	22-23
2.3.2 Orthodontic adhesives	23-25
2.3.3 Orthodontic bonding to various materials	26-29
2.3.4 Bond strength in orthodontics	30-31

CHAPTER 2

LITERATURE REVIEW

2.1 CONCEPT OF ADHESION AND BONDING

Mechanical bonding or *mechanical retention* are the simplest methods of achieving a strong attachment between two substances. The first attempts at retention in dentistry involved the placement of undercuts in materials to facilitate the “locking in” of subsequently placed materials.

The ultimate goal in dental bonding is to achieve true adhesion. When two substances are brought into intimate contact with each other, the molecules of one substance may be attracted to molecules of the other. Such an attractive force is known as *adhesion* when different molecules are involved and *cohesion* when like molecules are attracted. In dentistry, bonding is achieved by the application of a liquid material (the *adhesive*) to promote adhesion to a solid substrate (the *adherend*). For adhesion to occur, the adhesive and adherend surfaces must be attracted to one another at their interface. The surface of a material has greater energy than its interior. At the surface, the outermost atoms are not equally attracted in all directions by other atoms. While within the material, atoms held in

the solid lattice are equally attracted to each other. The increase in energy per unit area of surface is known as the *surface energy* or *surface tension* of a material. The greater the surface energy, the greater the potential for adhesion (Anusavice, 1996).

In addition to a high surface energy, the adhesive must be able to adequately wet the surface of the adherend. *Wetting* is the manifestation of the attractive forces between molecules of adhesive and adherend and may be defined as the process of obtaining molecular nearness or establishing interfacial contact. The extent to which an adhesive is attracted to the surface of an adherend can be determined by measuring the *contact angle* (**Figure 7**). The contact angle is the angle between the adhesive and the adherend at their interface. The smaller the contact angle, the more effective the adhesive is able to wet the surface of the adherend (Anusavice, 1996).

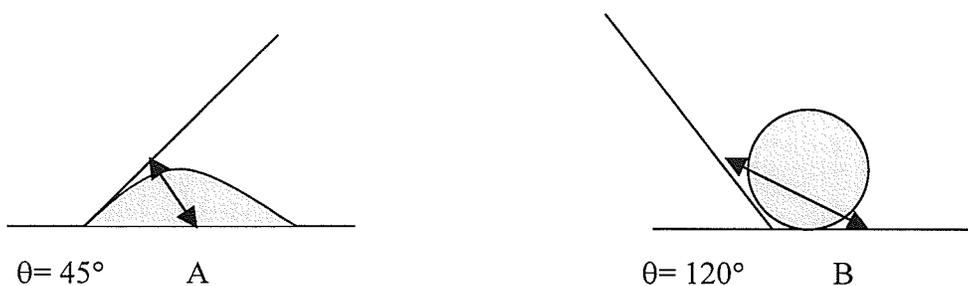


Figure 7. A, smaller contact angle allows the adhesive to spread further. B, larger contact angle allows for poor wetting of the surface.

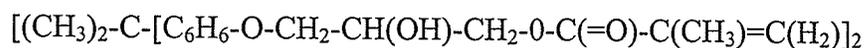
2.2 COMPOSITE RESINS

2.2.1 Historical Background of Composite Resin

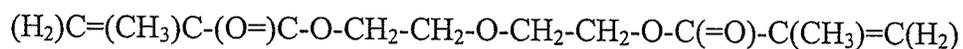
Synthetic resins have evolved as restorative materials principally because of their esthetic characteristics. The most widely used esthetic materials in dentistry today are composite resins (Gleich, 1999). The term “composite” means that it is a mechanical mixture of at least two different classes of materials that have limited mutual solubility. The combination of materials provides a product with properties superior or intermediate to those of the individual constituents (Anusavice, 1996). Dental composites are composed of synthetic polymers, inorganic fillers, molecules which promote the polymerization reaction, silane coupling agents to bond the inorganic filler particles to the polymer matrix, pigments, and small amounts of other additives to improve color stability (UV absorbers) and prevent premature polymerization (eg. hydroquinone) (Bayne *et al.*, 1994; Ferracane, 1995; Anusavice, 1996).

Development of dental composites began in the late 1950's and early 1960's when Dr. R.L. Bowen began experiments on reinforcing epoxy resins with filler particles (Bowen, 1963). For the past forty years, 80-90% of composites utilized the Bis-GMA(2.2-bis[4-(2-hydroxy-3-methacryloyloxy-propoxy)phenyl]propane) monomer developed by Dr. Bowen as the matrix-forming resin (Ruyter and Øysæd, 1987). Bis-GMA is extremely viscous at room temperature because of

the hydrogen bonding interactions that occur between the hydroxyl groups on the monomer molecules (Ferracane, 1995). Diluents of a more fluid resin such as TEGDMA (triethylene glycol dimethacrylate), which is also a crosslinking agent, are added to produce pastes of clinically usable consistencies (Ferracane, 1995) (**Figure 8**). Stannard (1993) reported that optimal properties are produced with a 1:1 ratio of Bis-GMA and TEGDMA.



Bis-GMA



TEGDMA

Figure 8. *Chemical Structure of Monomers in Dental Composites*

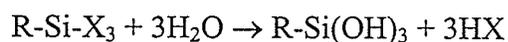
Inorganic filler particles are used to improve the strength (Ferracane *et al.*, 1987; Eversoll and Moore, 1988), increase stiffness (Braem *et al.*, 1989), change the coefficient of thermal expansion (Sodérholm, 1984; Brown, 1988), reduce polymerization shrinkage (Iga *et al.*, 1991), and increase the radiopacity (van Dijken *et al.*, 1989) of composite resins. Filler particles are most commonly produced by grinding or milling silicate particles containing oxides of barium, strontium, zinc, aluminum, or zirconium (Khan *et al.*, 1992). Composite resins have been classified according to their filler particulate size and percentage

(Anusavice, 1996). Microfilled materials have filler sizes of .04 μ m and filler loading of 35-60 wt%. Small particle-filled composites have large filler sizes of 1 to 5 μ m and increased loading of fillers up to 80-90 wt%. Hybrids are a third classification which combines .04 and 1 μ m particles in weight percentages in the 75-80 wt% range (Anusavice, 1996). Clinical Research Associates recommends hybrid resin use in Class 5 restorations where there is a high esthetic need (C.R.A., 1999). Their recommendation was based on an ordinal evaluation of 12 clinical characteristics such as colour match and surface smoothness. These are the surfaces to which orthodontic bonding will be generally required. Thus, it is likely that the type of composite encountered will be of the hybrid classification.

To achieve the optimal properties of the composite, coupling agents are used to chemically bond the filler particles to the resin matrix. Coupling agents displace adsorbed water and provide a strong chemical bond between the oxide groups on the glass filler surface and the polymer molecules of the resin (van Noort, 1994). Silane coupling agents have the general formula:



where R represents an organofunctional group and the X groups are hydrolysable groups bonded to the silane. The X units are hydrolysed and a tri-hydroxy-silanol is produced.



These silanols form hydrogen bonds with other hydroxyl groups on the glass surface. Water is removed upon drying in a condensation reaction to form a

covalent bond between the coupling agent and the glass. Finally, the organofunctional group, R, reacts with the polymer to link the two materials (van Noort, 1994) (**Figure 9**). The most common coupling agents are organosilanes such as 3-methacryloxypropyl-trimethoxy-silane (MPS) (Ferracane, 1995).

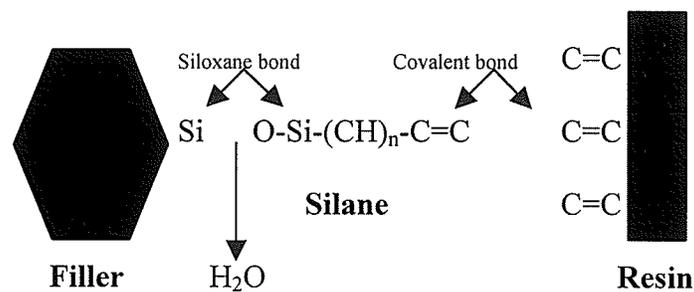


Figure 9. *Silane coupling agent,*
(after Ferracane, 1995)

The resins themselves polymerize by an addition mechanism initiated by free radicals (Anusavice, 1996). No byproducts are formed in addition polymerization reactions; the structure of the monomer is simply repeated numerous times. An external stimulus is used to activate the addition reaction (Anusavice, 1996). In self-cured resins the *induction* system is chemically activated by mixing two separate pastes. Typically, one paste contains benzoyl peroxide (the initiator) and the other paste contains an amine, dihydroxyethyl-p-toluidine (DHEPT) which works as an activator (Anusavice, 1996). The most popular composite resins are

now light-activated (C.R.A., 1999). The initiator in these products is camphoroquinone (CQ) which generates free radicals when exposed to blue light in the 470nm range of the electromagnetic spectrum (Ferracane, 1995). These products are popular because they are one-part systems, and working time is somewhat controlled by the operator.

2.2.2 Aging and Water Sorption

Composite resins are not inert materials. They have been shown to be soluble in water and in organic solvents such as ethanol (Craig and Payton, 1975; Pearson, 1979; Ferracane and Condon, 1990; Söderholm, 1990; Ferracane, 1994). The leaching of components has a potential impact on both the structural stability and biocompatibility of the material (Ferracane, 1994). Of course, the former is of concern to this study, as the structural stability of the composite may be a factor in the shear bond strength of orthodontic attachments.

It has been estimated by infrared spectroscopy that only 50-75% of the resin monomer double bonds are actually polymerized after light curing (Eliades *et al.*, 1987; Ferracane, 1990, 1994; Ferracane and Condon, 1992). As mentioned previously, the Bis-GMA and TEGDMA monomers polymerize by a free radical addition reaction involving substantial cross-linking. The cross-linking reaction produces a gel structure which severely reduces molecular mobility and greatly

slows the rate of polymerization (Ferracane, 1994). Most of the unreacted carbon-carbon double bonds are on molecules which have reacted at one end and are thus bound to the polymer chain and are not free to elute (Ferracane, 1994). However, the polymer matrix does contain a portion of totally free monomer molecules.

Studies have verified that virtually all of the components in composites may be leached into solution. Bis-GMA and TEGDMA have been identified in solution by many different sources (Inoue and Hayashi, 1982; Thompson *et al.*, 1982; Ferracane and Condon, 1990; Rathburn *et al.*, 1991; Tanaka *et al.*, 1991). Approximately 5-10% of the unbound monomer elutes into an aqueous solution. This equals about 2% of the weight of the resin component for most composites (Ferracane, 1994). Filler particles are also known to leach ions such as silicon, barium, strontium and sodium when stored in water (Söderholm, 1983, 1990; Söderholm *et al.*, 1984; Øysæd and Ruyter, 1986).

Fluoride has also been shown to be released from composite resins (Temin and Csuriz, 1988; Swift, 1989; Wiltshire and van Rensburg, 1995). The efficacy of such fluoride release as an anticariogenic agent is still under investigation (Swift, 1989).

The loss of components and subsequent water sorption is rapid during the initial soaking period and slows substantially within hours. Approximately 75% of the

elutable species are extracted within the first several hours (Pham and Ferracane, 1989; Ferracane and Condon, 1990; Wiltshire and van Rensburg, 1995).

Water uptake has been reported to be 2-3% of the weight of the composite (Fan *et al.*, 1985; Ferracane and Condon, 1990). Such water sorption may affect the mechanical properties of the composite and has been explained by filler-matrix debonding and hydrolytic degradation of the fillers (Söderholm *et al.*, 1984). Söderholm *et al.* (1984) hypothesized that the breakdown of the filler particles raised internal osmotic pressure within the composite structure and microcracks formed along the matrix-filler interfaces. These cracks propagated until they reached the surface of the composite, as evidenced in S.E.M. studies (Söderholm, 1984).

2.2.3 Composite To Composite Bonding

The majority of the literature on resin to resin bonding deals with the repair of previously placed composite resin restorations. The interfacial bond strength of composite to fresh (i.e., only a few minutes old) composite has been reported to be the same as the cohesive strength of the material (Lloyd *et al.*, 1980; Boyer *et al.*, 1984). Boyer *et al.* (1984) reported a transverse bond strength of between 53.2 and 109.4 MPa, depending on the type of composite resin. However, the bond strength to polished, untreated composite surfaces is in the range of 20% to 75%

of the cohesive strength of the respective substrate (Lloyd *et al.*, 1980; Vankerckhoven *et al.*, 1982; Chan and Boyer, 1983; Boyer *et al.*, 1984; Azarbal *et al.*, 1986; Pounder *et al.*, 1987; Kao *et al.*, 1988b; Puckett *et al.*, 1991). Absolute values for the cohesive and adhesive shear bond strengths reported in the above mentioned studies ranged from as low as 5.74 MPa to as high as 111.6 MPa depending on numerous factors such as substrate, surface treatments, adhesives, storage conditions, and bond strength testing methods.

There are three possible mechanisms of a “new” composite resin bonding to an “old” composite resin. Chemical bonds may form with the resin matrix (Brosh *et al.*, 1997), chemical bonds may form with the exposed filler particles (Brosh *et al.*, 1997), and micromechanical retention may be gained by penetration of resin monomer into undercuts on the restoration surface and perhaps even into microcracks in the matrix (Brosh *et al.*, 1997). This micromechanical bonding may be further enhanced by solvent bonding. Solvent bonding occurs as the methylmethacrylate monomer diffuses into the “old” resin, resulting in swelling of the set resin, and allowing penetration of the “new” resin (Powers *et al.*, 1997). Upon polymerization, the “new” resin is mechanically locked onto the surface of the “old” resin.

Chemical bonding between resin matrices is dependent upon the concentration and availability of unreacted methacrylate groups in the substrate resin (Vankerckhoven *et al.*, 1982; Puckett *et al.*, 1991; Turner and Meiers, 1993; Li,

1997). The concentration of such unreacted methacrylate groups decreases from 100% to approximately 50% as the resin polymerizes, so the potential for chemical bonding diminishes as the resin ages (Vankerckhoven *et al.*, 1982; Swift *et al.*, 1992). Also, when the restoration surface is polished, inorganic filler particles are exposed and the degree of unsaturated methacrylate groups is decreased to 25%. This limits the chemical bonding between resin matrices (Vankerckhoven *et al.*, 1982).

Chemical bonding to the filler particles would require the use of a silane coupling agent, much like the organosilane incorporated into individual composite materials as discussed in section 2.2.1. Silane coupling agents have been shown to improve the bond of composite to etched, sandblasted and roughened porcelain by up to 3 MPa (Stangel *et al.*, 1987; Andreasen and Stieg, 1988; Kao *et al.*, 1988a; Major *et al.*, 1995; Roulet *et al.*, 1995). However, silanes have failed to predictably increase the bond strength of new and old composites compared with dentin/enamel bonding agents (Azarbal *et al.*, 1986; Saunders, 1990; Sodérholm and Roberts, 1991; Swift *et al.*, 1994; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997). This may suggest that mechanical interlocking is the most significant factor contributing to repair strength, and thus, silanes are not recommended for clinical application of composite to composite bonding (Sodérholm and Roberts, 1991; Swift *et al.*, 1994; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997).

Numerous surface treatments and bonding agents have been advocated to improve the repair strength of composites. Most treatments attempt to increase the micromechanical bonding between the substrates (Brosh *et al.*, 1997). Wetting of the restoration surface by the repair material is a major factor controlling the repair bond strength as discussed in section 2.1. Unfilled resins improve the bonding of fresh material to polished composite surfaces by up to 6 to 25 MPa after 24 hours (Boyer *et al.*, 1984; Azarbal *et al.*, 1986; Puckett *et al.*, 1991; Swift *et al.*, 1992).

2.2.4 Composite Resin Surface Treatments

Many different surface treatment protocols have been recommended. Perhaps the most important procedure in composite to composite bonding is the roughening of the mature resin surface (Swift *et al.*, 1992; Turner and Meiers, 1993; Kupiec and Barkmeier, 1996). Turner and Meiers (1993) compared the shear bond strengths achieved with various surface treatments and with different adhesives. In a two-way ANOVA, they found the surface treatment to be highly significant ($p < 0.0001$) and the adhesive to be less significant ($p = 0.643$). Etching with 37% phosphoric acid, while vital in bonding to enamel (Abdullah and Rock, 1993; Johnston *et al.*, 1996; Olsen *et al.*, 1996; Powers *et al.*, 1997), appears to be relatively ineffective in composite bonding (Boyer *et al.*, 1978). Boyer *et al.*

(1997) reported no significant difference in tensile bond strength between resin layers with the use of 37% phosphoric acid. Hydrofluoric acid (HF) is used to etch porcelain surfaces for indirect restorations, intraoral repair, or orthodontic bonding (Zachrisson and Buyukyilmaz, 1993; Kern and Thompson, 1994; Major *et al.*, 1995). HF can etch the glass filler particles in hybrid and small particle composites. HF has been shown to cause some slight surface changes such as minor roughening (Kula *et al.*, 1983; Kula *et al.*, 1986); however, research does not reveal significant increases of bond strength with the use of HF for composite repair (Crumpler *et al.*, 1989; Swift *et al.*, 1992; Swift *et al.*, 1994; Brosh *et al.*, 1997). Mitchem *et al.* (1991) even recommended against the use of HF with hybrid composites as etching softens the resin surface. This change is likely as a result of the removal of the hard filler particles, as it has been shown that there is no change in hardness in unfilled resins after exposure to 15% HF for 6 months (Al-Jezairy and Williams, 1996).

Mechanical treatment of the composite surface may be accomplished with rotary instruments such as diamond or green carborundum stones or through the use of air abrasion with aluminum oxide particles. Brosh *et al.* (1997) described the surface treatment of composites with diamonds or green stones as providing “macro” retentive features which are controlled by the operator. Air abrasion resulted in “micro” retentive features that are under the control of the instrument. In the same study (Brosh *et al.*, 1997) the authors concluded that the “micro” retentive features demonstrated superior shear bond strengths over the “macro”

retentive features in combination with a bonding agent. Sandblasting increased the shear bond strength by 4.19 MPa and roughening with a diamond stone increased the bond strength by 0.64 MPa (Brosh *et al.*, 1997). Microetching with air abrasion is less invasive of the composite restoration than roughening with handpieces (Brosh *et al.*, 1997). Also, it is a relatively simple and quick procedure, and does not involve the use of strong acids intraorally. Recent studies have reported air abrasion as an effective means for surface preparation of aged composites (Swift *et al.*, 1992; Turner and Meiers, 1993; Kupiec and Barkmeier, 1996; Bouschlicher *et al.*, 1997).

2.3 ORTHODONTIC BONDING

2.3.1 Direct Bonding

Arguably, the most significant achievement in dentistry in the 20th century was the acid etch technique developed by Buonocore (1955). Utilizing various acidic mixtures, of which 30% to 40% phosphoric acid seems to be the most effective (Moin and Dogon, 1974; Retief, 1974; Legler *et al.*, 1990; Wang *et al.*, 1994; Olsen *et al.*, 1996), enamel surfaces are “etched,” a process in which the acid preferentially dissolves the centers or peripheries of the enamel rods. The etching time has also been debated and studied numerous times. Britton *et al.* (1990) compared bond strengths between 15- second and 60-second etch times. Their results indicated increased bond strengths in the 15-second group. Gorelick (1977) evaluated the effects of 60- and 90-second etching times, Barkmeier *et al.* (1985) compared 15- and 60-seconds of etching, and Beech and Jalaly (1980) evaluated 5-, 15-, 60-, and 120-second intervals. They all reported no decrease in bond strength as the result of shortened etching times. The most recent reviews on the subject of etching time (Olsen *et al.*, 1996) also concluded there was no significant effect on bond strength between 10- or 30-second etching intervals. Etched enamel allows a bonding agent of low viscosity to penetrate into the microscopic undercuts as evidenced by “resin tags” when seen under S.E.M. (Pahlavan *et al.*, 1976). Once polymerized, a micromechanical bond is established.

The acid etch technique provided the background for direct orthodontic bonding. Direct bonding eliminates the need for bands. This has several advantages such as enhanced ability for plaque removal by the patient (Zachrisson, 1976), minimizing soft tissue irritation and hyperplastic gingiva (Zachrisson, 1976), minimizing the danger of decalcification with loose bands (Zachrisson, 1976), elimination of the need for separation, absence of post-treatment band spaces, and improved esthetics during treatment. There is some controversy over who should get credit for the first direct orthodontic bonding. Most references, and the American Association of Orthodontists, seem to support the claim of George Newman (Newman, 1992). He reported a bonding technique using acrylic resins in 1965 (Newman, 1965). The early resins had a 15-minute setting time, which limited their acceptance in clinical practice (Retief and Sadowsky, 1975).

2.3.2 Orthodontic Adhesives

Currently, there are numerous materials available for direct orthodontic bonding. These include diacrylate composite resin-based products and glass-ionomer adhesives, which are available in either chemical or dual-cured systems (Powers, 1997), and cyanoacrylate systems (Örtendahl and Örtengren, 2000).

The composite resin adhesives are mostly based on the Bis-GMA resin (Bowen, 1963) modified to suitable viscosity for clinical use. The new chemically-cured resins have a reduced setting time of three to eight minutes (Wang and Meng, 1992; Mitchell, 1994; Lloyd and Scrimgeour, 1995). *In vitro* bond strengths do not appear to be affected by whether the composite is chemically-cured, light-cured, or dual-cured (Bradburn and Pender, 1992; Wang and Meng, 1992; Smith and Shivapuja, 1993; Whitlock *et al.*, 1994; Eliades *et al.*, 1995; Kao *et al.*, 1995; Chamda and Stein, 1996).

Glass ionomer materials are also now available as either chemically or light-cured systems for orthodontic bonding (Powers *et al.*, 1997). They have not replaced composite resins, as they have lower *in vitro* bond strengths to either etched or non-etched enamel (Rezk-Lega and Ogaard, 1991; Oen *et al.*, 1991; Wiltshire, 1994; Powers *et al.*, 1997). For example, Wiltshire (1994) recorded a mean shear bond strength of an orthodontic attachment bonded to etched enamel with a chemically-cured glass ionomer cement to be 5.5 MPa, and the comparative bond strength with a composite cement was 26 MPa.

A new group of resin-modified glass-ionomer (RMGI) cements do provide for increased bond strengths over conventional GI cements (Erickson and Glasspoole, 1994). Erickson and Glasspoole (1994) reported a shear bond strength of 20.5 MPa for a RMGI compared to 7.2 MPa for a conventional GI. Powers *et al.* (1997) also reported tensile bond strengths of 8 MPa to 25 MPa for five different

RMGI's to unetched enamel. However, a recent publication reported a low initial bond strength (after 30 minutes) for an orthodontic RMGI cement of 0.4 MPa vs. 5.2 MPa for a resin cement (Bishara *et al.*, 1999).

Recently, a cyanoacrylate adhesive system has been developed for direct orthodontic bonding. The manufacturer, Gestenco Int., claims adequate bonding to many surfaces including enamel, porcelain, and composite (Gestenco Int., 1999). Previous cyanoacrylate adhesives have failed to gain acceptance in the orthodontic community because of their poor durability in a wet environment. Crabb and Wilson (1971) studied three cyanoacrylate adhesives (Cyanodont, Eastman 910, and Permabond). After storage in 37° C saline for 24 hours, all of the bond strengths were reduced to near zero. More recently, Howells and Jones (1989) reported on another cyanoacrylate adhesive developed exclusively for orthodontic bonding. Again in their study, the material proved to be too susceptible to deterioration after storage in water. Bond strengths went from a mean of 124 N after one hour to 26 N after seven days and decreased to only 6 N after 98 days.

2.3.3 Orthodontic Bonding to Various Materials

Enamel:

The primary surface to which orthodontic brackets are directly bonded is enamel. The acid-etch procedure allowed for direct orthodontic bonding to be possible, and is discussed in section 2.3.1. Application of 30% to 40% phosphoric acid for at least 10 seconds seems to be the most effective in preparing enamel surfaces. Shear bond strengths of metal brackets bonded to etched, dry enamel with composite resin adhesives may attain values near 26 MPa (Wiltshire, 1994). Although glass ionomer cements can be bonded in a wet environment, the bond strengths are only 5-8 MPa (Chung *et al.*, 1999). Even when bonded dry, glass ionomers still produce lower bond strengths than composite resins (3-10 MPa vs. 26 MPa) (Rezk-Lega and Ogaard, 1991; Oen *et al.*, 1991; Wiltshire, 1994; Powers *et al.*, 1997, Chung *et al.*, 1999). The new RMGI cements are promising as they had similar shear bond strengths compared to a composite cement in a recent article (8.8 MPa vs. 10.4 MPa) (Bishara *et al.*, 1999).

Porcelain:

Bonding orthodontic attachments directly to porcelain or ceramic restorations has also been extensively studied. Major *et al.* (1995) compared adhesion promoters and recommended the use of a silanating agent. Use of a silane produced bond strengths of 6-14 MPa, whereas bond strengths without the primer ranged from 0.4 to 4 MPa (Whitlock *et al.*, 1994; Major *et al.*, 1995; Zachrisson *et al.*, 1996). Porcelain prepared with acidulated phosphate fluoride solutions produced low

bond strengths of less than 5 MPa (Barbosa *et al.*, 1995; Zachrisson *et al.*, 1996). Roughening of the porcelain surface seems to be generally contraindicated, as bond strengths become excessive and porcelain fractures occur upon debonding. Cochran *et al.* (1997) reported bond strengths of 28-39 MPa with sandblasting and silanization, and Barbosa *et al.* (1995) obtained bond strengths of 28-47 MPa with diamond bur surface preparation and silanization.

Gold:

Büyükyılmaz *et al.* (1995) compared sandblasting the gold surface to roughening with a diamond bur and found that sandblasting produced better bond strengths (20 MPa) than the diamond bur treatment (10 MPa). Superbond C & B™ (Sun Medical, Kyoto, Japan), a 4-META metal-bonding adhesive resin also produced superior bond strengths compared to a conventional composite resin (Büyükyılmaz *et al.*, 1995; Nollie *et al.*, 1997).

Amalgam:

Bond strengths of both conventional and 4-META adhesives to amalgam are generally low (3-6 MPa) (Zachrisson *et al.*, 1995). Sperber *et al.* (1999) recently sandblasted an amalgam surface and produced shear bond strengths similar to bonding to etched enamel with a resin cement (11.77 MPa vs. 10.76 MPa).

Composite Resin:

Bonding of orthodontic brackets to composite resin surfaces is only known to have been reported a few times. Newman *et al.* (1984) studied orthodontic bonding to a heat-cured composite resin (Isosit™, Vivadent Corp., Buffalo, N.Y.). They compared the shear bond strengths between brackets bonded with Concise™ (3M, St. Paul, MN) with or without silane application to brackets bonded to etched, natural teeth. No significant differences existed between the three groups, and their bond strengths were 1120-1300 lbs/in² (7.7 – 8.9 MPa).

Schwartz *et al.* (1990) studied the tensile bond strengths of metal brackets to resin substrates using three composite resin adhesives. They bonded to either untreated surfaces or surfaces treated with 37% orthophosphoric acid, reduced with a diamond bur, coated with silane agent, or coated with a dentine bonding agent. Though most of their results were not reported, they did report bond strengths of 4.3 +/- 2.0 MPa using Contacto on untreated resin surfaces. It was also reported that the use of Mono-Lok 2 and Unite produced tensile bond strengths of 10.5 +/- 3.2 MPa and 10.3 +/- 2.6 MPa.

Kao *et al.* (1995) compared the torsional bond strength of ceramic brackets bonded to composite resin veneer laminates and enamel. Silux Plus™ (3M, St. Paul, MN) veneers were fabricated and ceramic brackets were bonded with either a light-cured or chemically-cured composite adhesive. All samples were acid-etched before bonding and subsequently thermocycled. Torsional bond strengths

of between 30 MPa and 60 MPa were recorded for both the chemically- and light-cured adhesives.

Chunhacheevachaloke and Tyas (1997) also studied bonding to resin composite. Again, they compared two types of ceramic brackets and the effect of roughening the composite surface with a coarse Soflex™ disk (3M, St. Paul, MN). All samples were acid-etched and bonded with Transbond™ (3M/Unitek, Monrovia, CA). No significant differences were found with their shear bond strengths which ranged from 17.1 MPa to 19.2 MPa. Nineteen of the forty composite samples had cohesive failures upon debonding.

The most recent and thorough study of orthodontic bonding to composite resin was reported by Lai *et al.* (1999). They bonded metal, ceramic and polycarbonate brackets to Silux Plus™ (3M, St. Paul, MN) samples (roughened with Soflex™ discs) using either a light-cured resin modified glass ionomer cement, a chemical-cured composite, or a light-cured composite system. Half of the samples were tested after 24 hours and half were thermocycled. They concluded all groups to have clinically acceptable bond strengths (10.0 to 30.1 MPa) except the polycarbonate/chemically-cured group (3.58 MPa). 210 of 288 samples had damaged resin surfaces after debonding.

2.3.4 Bond Strength in Orthodontics

The bond strength of orthodontic attachments must be able to withstand both functional stresses (from occlusion and mastication) and operator stresses (from the orthodontic appliances) (Powers *et al.*, 1997). Newman *et al.* (1994) stated that "maximum strength is needed to compensate for the unfavorable, moist environment in which the polymer adhesive system operates, as well as variations in pH, thermal changes, impact forces from sticky, chewy, or hard foods, and sports accidents." However, the direct bonding of orthodontic attachments is a temporary procedure; after treatment, the attachments must be removed with minimal or no damage to the substrate, and this is best achieved with a low bond strength (Powers *et al.*, 1997).

Although it has been attempted, it is difficult to evaluate orthodontic bond strengths *in vivo* (Voss *et al.*, 1993). Laboratory testing allows for improved standardization of testing procedures and the use of more sensitive equipment (Retief, 1991).

Different types of bond strengths are reported in the literature including shear, shear/peel, tensile, and torque (Ostertag *et al.*, 1991; Powers *et al.*, 1997). In shear bond strength testing, the debonding force is applied directly and parallel to the junction of the bracket and adhesive (Fox *et al.*, 1994). True shear bond strength is impossible to determine practically. Using a 3D finite element analysis,

Thomas *et al.* (1999) found the tensile and compressive stresses exceeded the shear component. The term shear/peel is used in the literature to reflect this phenomenon (Katona, 1994). Most studies reporting shear bond strength are actually testing the shear/peel bond strength (Katona, 1997). In tensile strength testing, the debonding force is applied perpendicularly to the substrate surface. Up to 15% of the stresses are in fact shear and compressive in nature, again directing the results to a tensile/peel force (Thomas *et al.*, 1999). A final method of bond strength testing is torsion loading, in which the attachment is "twisted" off. This method is less favoured because most mechanical testing machines cannot perform it (Katona, 1997). Also, the results of a torsion test are reported in N/m and cannot be directly compared to tests of shear, tensile, or peel strength which are reported in MPa (Katona, 1997).

A bond strength is only relevant if it can be correlated clinically. The absolute value for clinically adequate shear bond strength most often quoted is from the work of Reynolds (Reynolds, 1975; Reynolds and von Fraunhofer, 1976). They recommend a bond strength of 60-80 kg/cm² (5.9-7.9 MPa). However, the original paper does not state any scientific method for calculating this value. Just as important as the minimum recommended bond strength is the maximum recommended bond strength. Retief's work (1974a) must also be considered when accessing bond strengths, as he demonstrated enamel fractures on specimens with bond strengths as low as 9.7 MPa.

CHAPTER 3

MATERIALS AND METHODS

	Page Number
3.1 Materials Used in the Study	
3.1.1 Restorative composite resin	33-34
3.1.2 Composite orthodontic adhesive system	35-36
3.1.3 Cyanoacrylate orthodontic adhesive system	37-38
3.1.4 Orthodontic attachments	38-39
3.2 Experimental Method	
3.2.1 Preparation of composite specimens	40-42
3.2.2 Composite surface treatments	43-44
3.2.3 Bonding of orthodontic attachments	44-46
3.2.4 Storage conditions	46
3.2.5 Shear/peel bond strength testing	47-50
3.2.6 Evaluation of fracture sites	50-51
3.2.7 Statistical analysis	51
3.2.8 Preparation of S.E.M. specimens	52

CHAPTER 3

MATERIALS AND METHODS

3.1 MATERIALS USED IN THE STUDY

3.1.1 Restorative Composite Resin

Material	Manufacturer	Batch Number
Z100™ Shade A3	3M Dental Products St. Paul, MN, USA	8UR 5904A3

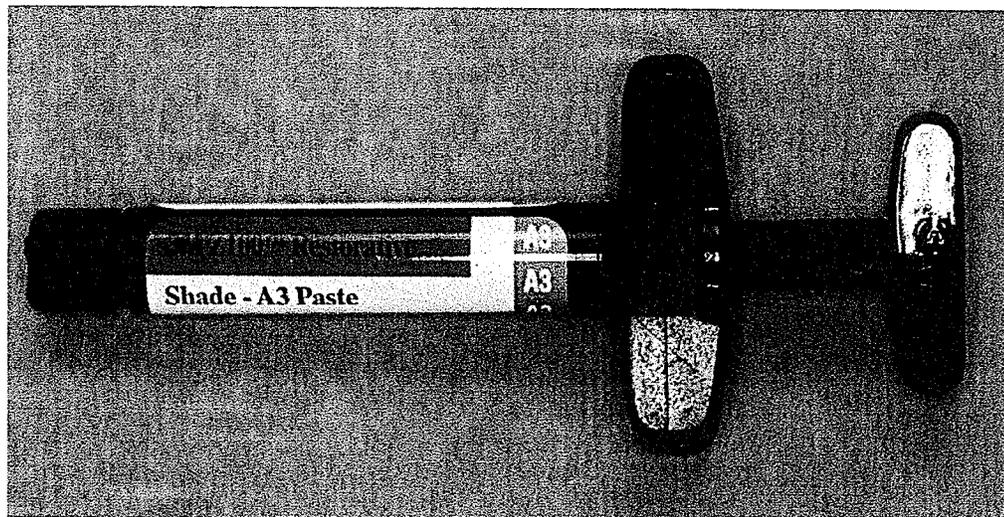


Figure 10: *Z100™ restorative composite resin*

Z100™ is a heavily filled, light-cured hybrid dental restorative material. Its resin composition is Bis-GMA and TEGDMA (refer to 2.2.1), which accounts for 15% by weight of the product (Ferracane, 1994). Thus, the remaining 85% of the material are filler particles. Z100™ uses zirconia silica fillers produced by a synthetic sol-gel process. The sol-gel process mixes a metal carboxylate and a metal oxide sol together to form a gel by dehydration. After heat treating, the gel is ground to produce the filler particles. Z100™ filler particles are rounded, and are in a wide distribution of sizes (average 0.5-0.7µm, largest 4µm) (Ferracane, 1995).

Recently, Clinical Research Associates evaluated seven popular restorative resins in twelve categories relating to clinical use and performance of the materials. Z100™ was the clinician's first choice in 6 of 12 characteristics, and was the highest rated material in their study (C.R.A., 1993). In a follow-up study, Clinical Research Associates concluded that the "currently best suited material" for Class 5 restorations where there is a high esthetic need and low caries potential is a hybrid resin such as Z100™ (C.R.A., 1999). Class 5 restorations cover the facial surfaces of the teeth, and since orthodontic attachments are most commonly bonded to the facial surfaces, a material like Z100™ would likely be encountered.

3.1.2 Composite Orthodontic Adhesive System

Material	Manufacturer	Batch Number
Transbond™ XT light-cured adhesive paste and primer; Tooth conditioning gel	3M Unitek, Monrovia, CA, USA	Adhesive: 9AD, 0002-02 Primer: 9E

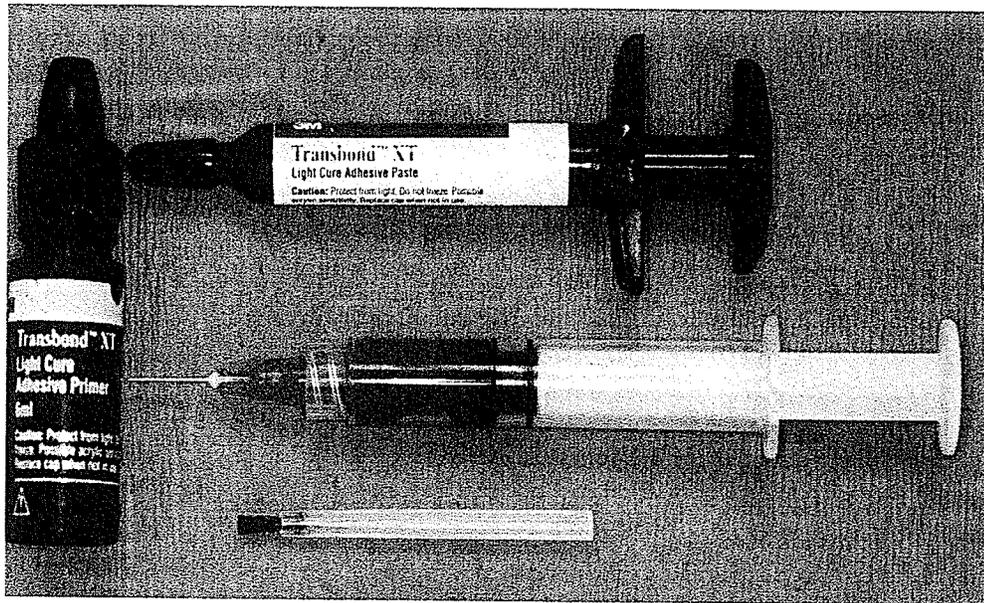


Figure 11: *Transbond™ XT light-cured orthodontic adhesive paste, Transbond™ XT light-cured adhesive primer, tooth conditioning gel.*

Transbond™ XT adhesive paste is a light-cured, hybrid composite resin. The resin base is Bis-GMA and TEGDMA in a 1:1 ratio, and the filler particles are a hybrid

of silica with an average size of 3 μ m (Bradburn and Pender, 1992; Manufacturer Safety Data Sheets 11938, 1993). Filler loading is approximately 82 wt%. A diketone and an organic amine are added as photoinitiators. Transbond™ XT light-cured primer is an unfilled resin of 50% Bis-GMA and 50% TEGDMA (M.S.D.S. 11939, 1993; Lai *et al.*, 1999). The tooth conditioning gel is 34% phosphoric acid in an amorphous silicate gel.

Advantages of visible light-cured orthodontic adhesives are the high early bond strength (Eliades *et al.*, 1991), minimal extent of oxygen inhibition (Lekka *et al.*, 1989), and extended working time. Light-cured orthodontic adhesives (and specifically Transbond™ XT) have been shown to have adequate, if not superior, immediate and long-term bond strengths as compared to chemically cured adhesives (Bradburn and Pender, 1992; Wang and Meng, 1992; Eliades *et al.*, 1995; Chamda and Stein, 1996). Also, Transbond™ XT appears to be the industry standard, as it is commonly used as the control or experimental adhesive in recent published studies (Wang and Meng, 1992; Eliades *et al.*, 1995; Chamda and Stein, 1996; Damon *et al.*, 1997; Weinberger *et al.*, 1997).

3.1.3 Cyanoacrylate Orthodontic Adhesive System

Materials	Manufacturer	Batch Number
Smartbond® Instant Adhesive	Gestenco International,	Adhesive: 98K
Smartbond® Etching Gel	Göteborg, Sweden	Etch: GE 5192

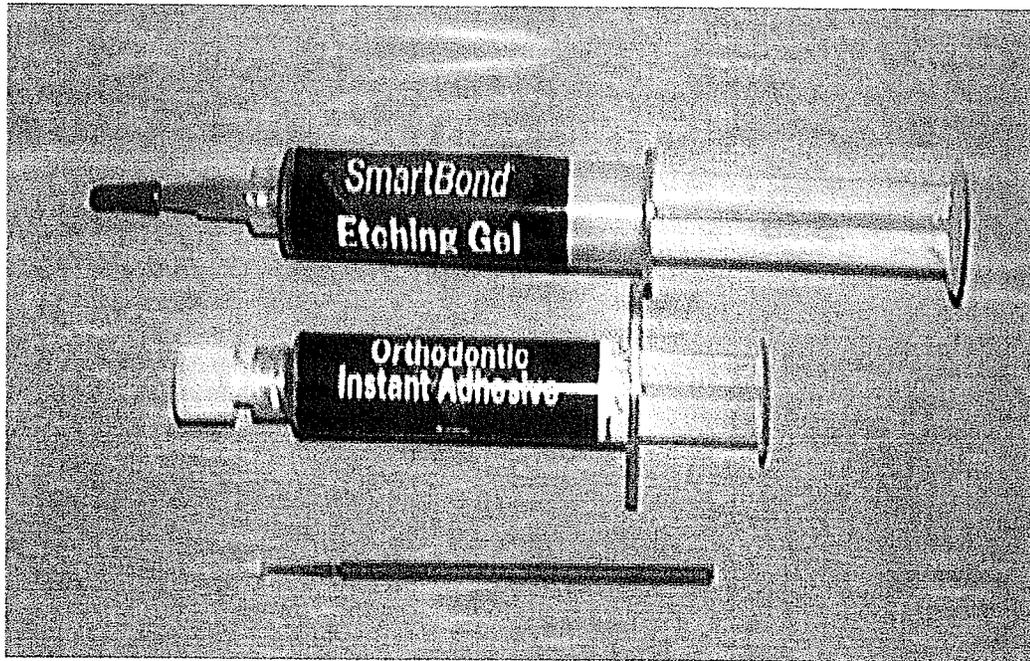


Figure 12: *Smartbond® instant orthodontic adhesive paste,
Smartbond® etching gel*

Smartbond® is a cyanoacrylate ester. Its composition is 85-90% ethyl cyanoacrylate, 5-10% poly (methylmethacrylate), 5-10% amorphous silica, and 0.1-0.5% hydroquinone (M.S.D.S. 52-175-99, 1997). The etching gel is 37% phosphoric acid in an amorphous silicate gel.

The manufacturers of Smartbond® are promoting it for the following reasons:

- ◆ true “one-step,” no light, no primers
- ◆ wet bonding
- ◆ can be used with metal, plastic and ceramic brackets
- ◆ bonds to composite and porcelain materials

It is the final claim that is being tested in this study. No other orthodontic adhesive systems are known to specifically claim to bond with composite resin.

3.1.4 Orthodontic Attachments

Material	Manufacturer	Batch Number
Lingual buttons, flat, bondable	GAC International, Central Islip, NY	#30-000-00

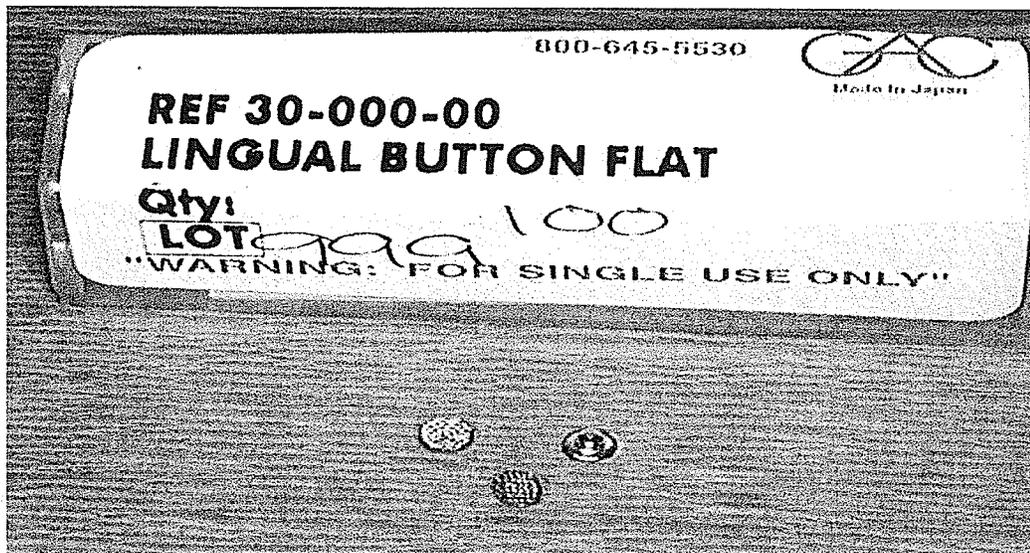


Figure 13: Bondable orthodontic buttons

Round, flat-based bondable orthodontic buttons (#30-000-00, GAC International, Central Islip, NY) (**Figure 13**) were chosen as the orthodontic attachments for this study. Having a flat base eliminated the possible variable of a curved base affecting bond strengths, and made surface area determination easier. The diameter of 20 buttons was measured three times each with digital micrometer calipers (Mitutoyo Digital Micrometer, Tokyo, Japan). The nominal base area was calculated using the formula: $A = \pi r^2$ (**Appendix #1**). The buttons were found to have a surface area of 9.18mm^2 .

3.2 EXPERIMENTAL METHOD

3.2.1 Preparation of Composite Specimens

One hundred and twenty composite resin specimens were fabricated using a 5mm section of a plastic 1mL disposable syringe as a mould (**Figure 14**).

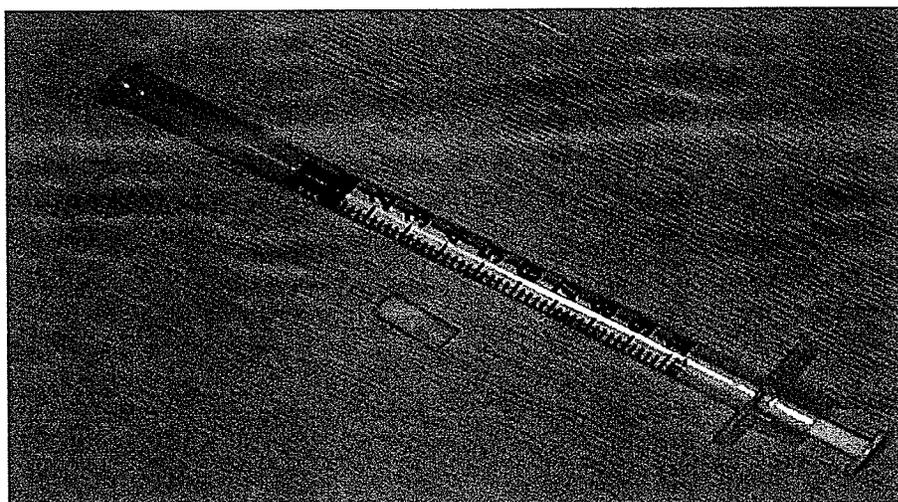


Figure 14: *Z100™ sample, 1mL insulin syringe (mould)*

The mould was placed on a glass microscope slide and then the restorative composite resin (Z-100™, 3M Dental Products, St. Paul, MN) was packed directly into the mould from its dispensing tube. Care was taken to begin packing at the bottom of the mould to prevent developing voids in the samples, and it was packed with a dental spatula. The resin was covered with another glass microscope slide and compressed with a 500g weight, again, eliminating possible voids (Swift *et al.*, 1992). The top surface was light-cured with an Ortholux™ (3M/Unitek, Monrovia, CA) light-curing unit (**Figure 15**) for 20 seconds from a

distance of 1mm. The glass slides were flipped over and the other side was cured for an additional 20 seconds in a similar manner. Finally, the resin in the middle of the mould was light-cured for 20 more seconds. After removal from the plastic moulds, all specimens were light-cured an additional 5 minutes in a Triad Light-curing Unit (Dentsply Int., York, PN), to ensure complete and adequate polymerization.

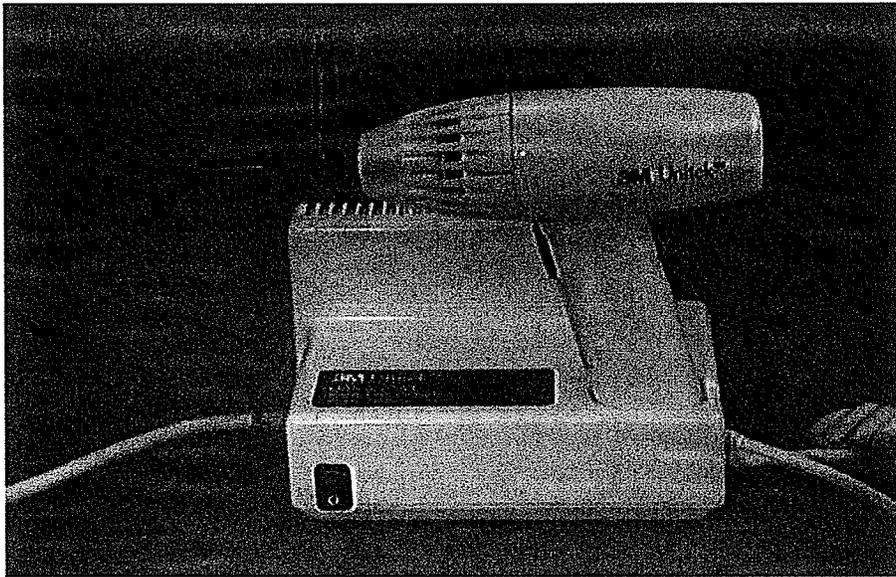


Figure 15: *Ortholux™ light-curing unit*

All 120 specimens were polished in the same manner to simulate the conditions of bonding to finished restorations. Fine (600 grit) and super-fine (1200 grit) Sof-lex discs (3M Dental Products, St. Paul, MN) were utilized on each specimen with a slow speed handpiece by running each disc over the composite surface for five seconds (Berastegui *et al.*, 1992; Jeffries *et al.*, 1992). New Sof-lex discs were

used on every third sample. The Sof-lex finishing system (**Figure 16**) was designed by 3M to polish the surface of its composite resins. It is a series of four aluminum oxide coated abrasive discs that decrease in particle size and abrasiveness as one progresses from coarse (150 grit), through medium (360 grit), fine (600 grit) and super-fine (1200 grit). Previous research indicates that the Sof-lex disc system is highly effective, and the average surface roughness appears to be sufficiently low to produce a highly polished surface that is light reflective when visually inspected (Berastegui *et al.*, 1992; Jeffries *et al.*, 1992).

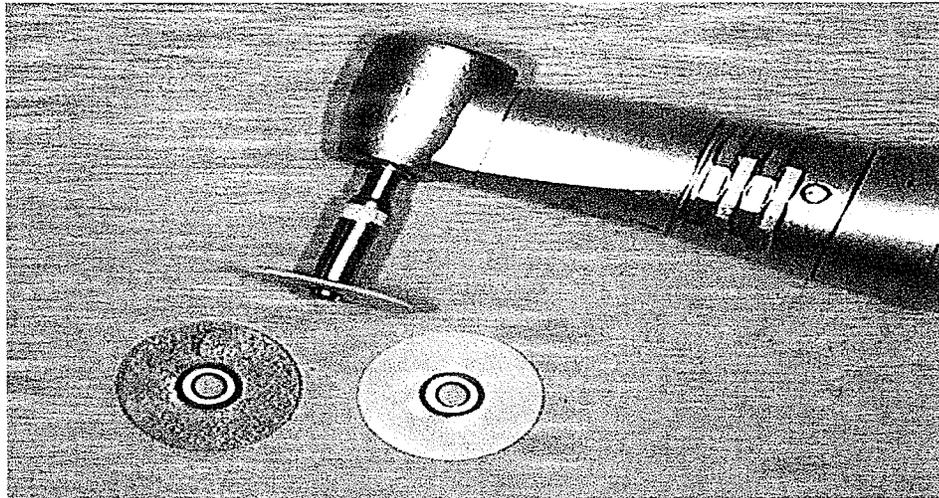


Figure 16: *Sof-lex polishing discs*

Each sample was washed and dried using a triplex air-water syringe. After polishing, the samples were placed in distilled water at 37° C for 7 days. This allowed adequate water sorption, to simulate an “old” composite restoration (Hirasawa *et al.*, 1983; ISO 4049, 1988; Suliman *et al.*, 1993).

3.2.2 Composite Surface Treatments

The 120 samples were randomly divided into three groups of 40. One group of 40 did not have any further surface treatment performed, but retained its polished surface. Before bonding the orthodontic attachment, the sample surface was just cleaned by polishing with the super-fine (1200 grit) Sof-lex disc again for five seconds. This ensured no residue, such as a biofilm, remained from storage in the distilled water for 7 days.

The second group of 40 samples had the composite surface acid etched with 34% phosphoric acid gel (Tooth Conditioner Gel, 3M/Unitek, Monrovia, CA) (Moin and Dogon, 1974; Retief, 1974; Legler *et al.*, 1990; Wang *et al.*, 1994; Olsen *et al.*, 1996) for 20 seconds (Gorelick, 1977; Beech and Jalaly, 1980; Barkmeier *et al.*, 1985; Britton *et al.*, 1990; Wang *et al.*, 1994; Olsen *et al.*, 1996), continuously agitating with an applicator brush (Baharav *et al.*, 1987; Baharav *et al.*, 1988). The acid was rinsed from the resin surface with water from the triplex syringe for 20 seconds, and dried with air from the triplex syringe for 10 seconds.

The third group of 40 samples had the composite surface air abraded with 50 μm aluminum oxide particles under 80 psi for 5 seconds using a Micro Etcher (Danville Engineering, Danville, CA) at a distance of 5mm with the abrasive particles directed at right angles to the composite surface (Swift *et al.*, 1992; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997). The air-abraded surfaces were

rinsed with water from the triplex syringe for 10 seconds and dried with air from the syringe for 10 seconds.

3.2.3 Bonding of Orthodontic Attachments

Half of the composite specimens from each of the surface treatment groups (20 samples each) were randomly chosen to have the orthodontic buttons bonded with Transbond™ XT (3M/Unitek, Monrovia, CA) according to the manufacturer's instructions as follows. Transbond™ XT Light Cured Adhesive Primer (3M/Unitek, Monrovia, CA) was applied to the composite surface in a thin layer. Transbond™ XT adhesive paste was applied to the entire button base, and the button was seated firmly, with a uniform 500g force measured with a Bencor™ Multi-T testing device (Driesen *et al.*, 1989), on the resin sample surface (**Figure 17**).

Excess adhesive was removed with an explorer, and the bond was light-cured for 40 seconds (Wang, 1992) with the light held directly above, but not touching, the orthodontic attachment. Two buttons were bonded onto opposite ends of each composite sample (Zachrisson *et al.*, 1996), totaling 40 orthodontic attachments bonded with Transbond™ XT per surface treatment group.

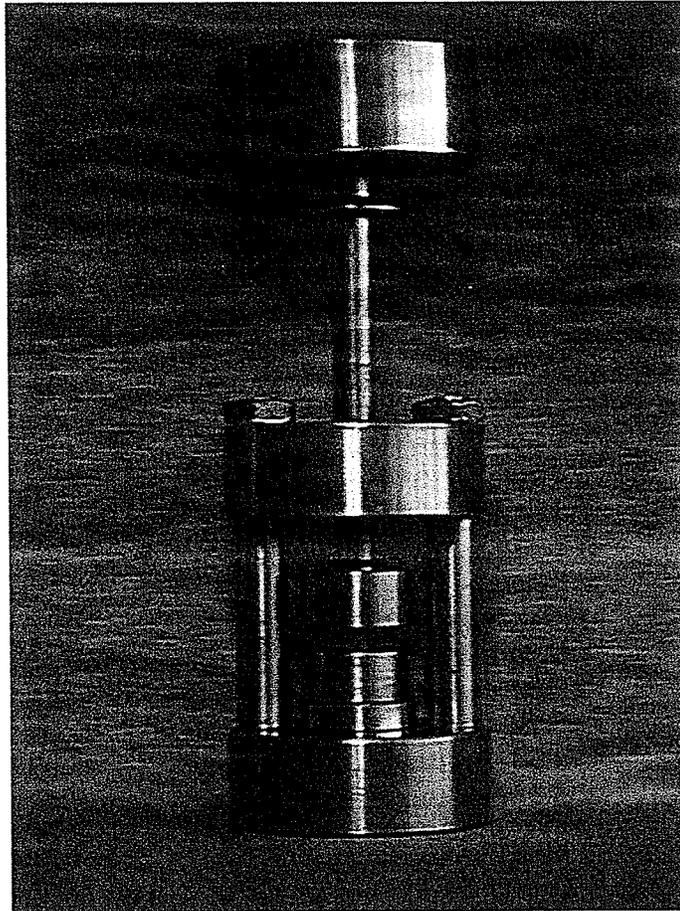


Figure 17: *500g attachment seating force*

The other half of the composite specimens from each of the surface treatment groups (20 samples each) had the orthodontic attachments bonded with Smartbond[®] (Gestenco International, Göteborg, Sweden) according to the manufacturer's instructions as follows. The sample surface was moistened with distilled water using an applicator tip. The adhesive was applied sparingly to the attachment base and the button was seated with a similar, uniform 500g force.

Excess adhesive was removed with an explorer, and the bond was allowed to set undisturbed for 5 minutes. Two buttons were bonded onto opposite ends of each composite sample (Zachrisson *et al.*, 1996), totaling 40 orthodontic attachments bonded with Smartbond[®] per surface treatment group.

3.2.4 Storage Conditions

After orthodontic bonding, all samples were stored in distilled water at 37°C for 24 hours (ISO, 1994). Half of the samples (10 restorative composite samples with 20 bonded orthodontic attachments) from each of the six groups (three surface conditions, two adhesives) were randomly chosen for thermocycling. The samples were thermocycled 500 times between 5°C and 55°C water baths with a 30 second dwell time in each bath and a transfer time of 10 seconds between baths (Darbyshire *et al.*, 1988; Moore and Vann, 1988; ISO, 1994; Al-Salehi and Burke, 1997). After thermocycling, these samples were again stored in distilled water at 37° C for 7 days. The thermocycling provided mechanical stresses to the bonds and the additional storage in water for 7 days saturated the bonding materials.

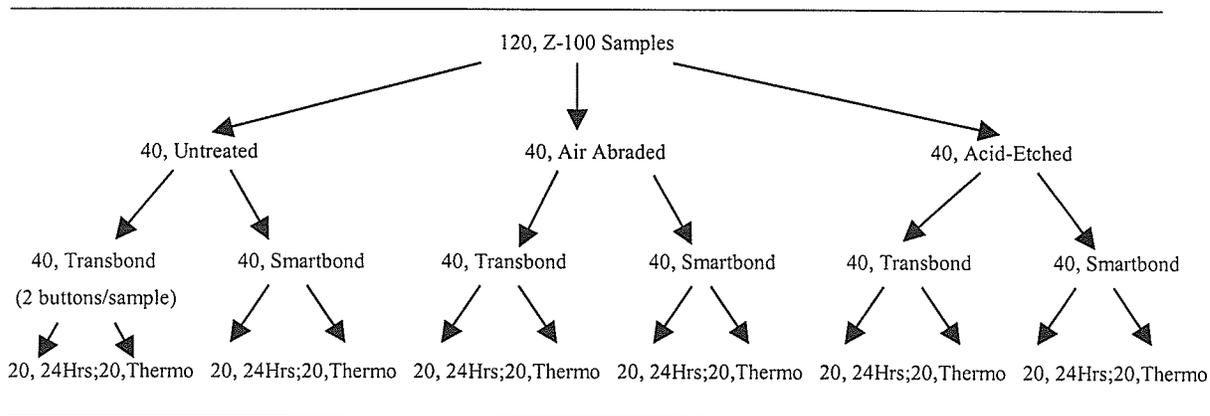


Figure 18: Bonding protocol diagram

3.2.5 Shear/Peel Bond Strength Testing

After storage for 24 hours or thermocycling, the samples were mounted horizontally and secured with a stabilizing screw into a jig (**Figure 19**) in a Bencor™ Multi-T testing apparatus (Driesen, *et al.*, 1989) in a Zwick Universal Testing Machine Model #1445(Zwick GmbH & Co., Ulm, Germany) (**Figures 21,22,23**). A straight edge chisel applied a shear/peel force at a rate of 0.5 mm/minute (ISO, 1994). The load was applied directly and parallel to the attachment/adhesive/resin interface until failure (**Figure 20**). By inputting the surface area of the base of the orthodontic button, the bond strength was provided in MPa by the Zwick computer (**Figure 23**). All debonding procedures were carried out by one operator.

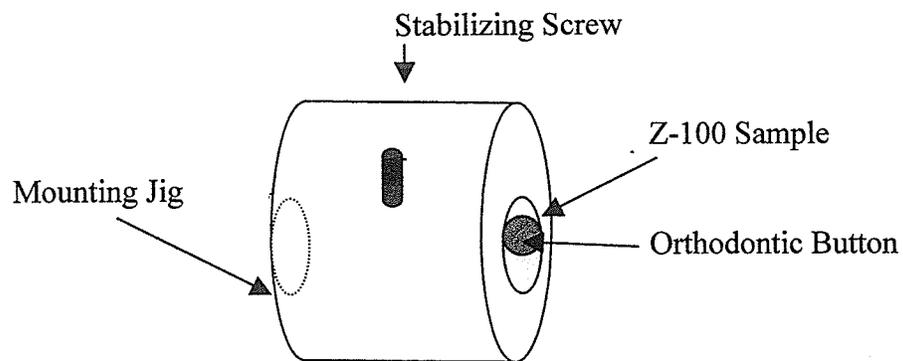


Figure 19: *Z100™ sample mounted in jig*

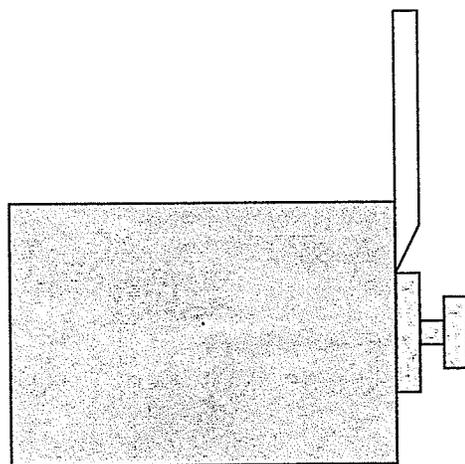


Figure 20: *Shear blade applied directly and parallel to attachment/adhesive/resin interface*

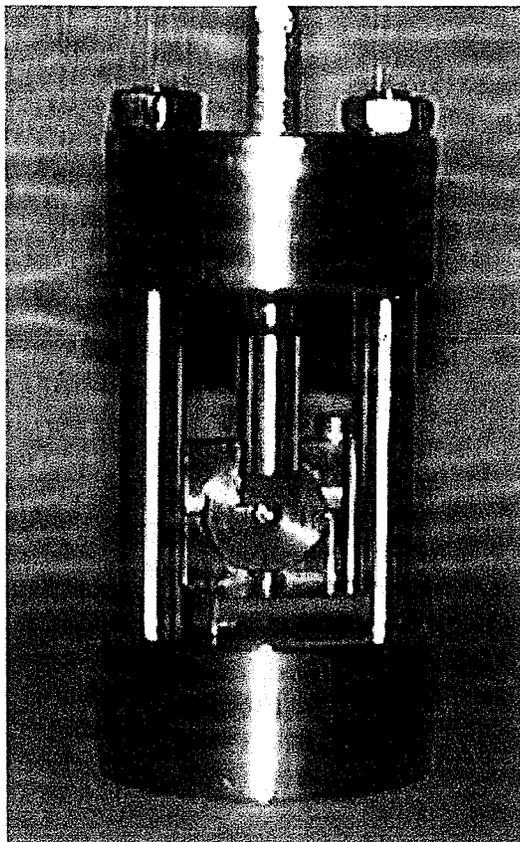


Figure 21: *Sample mounted in Bencor™ testing apparatus*

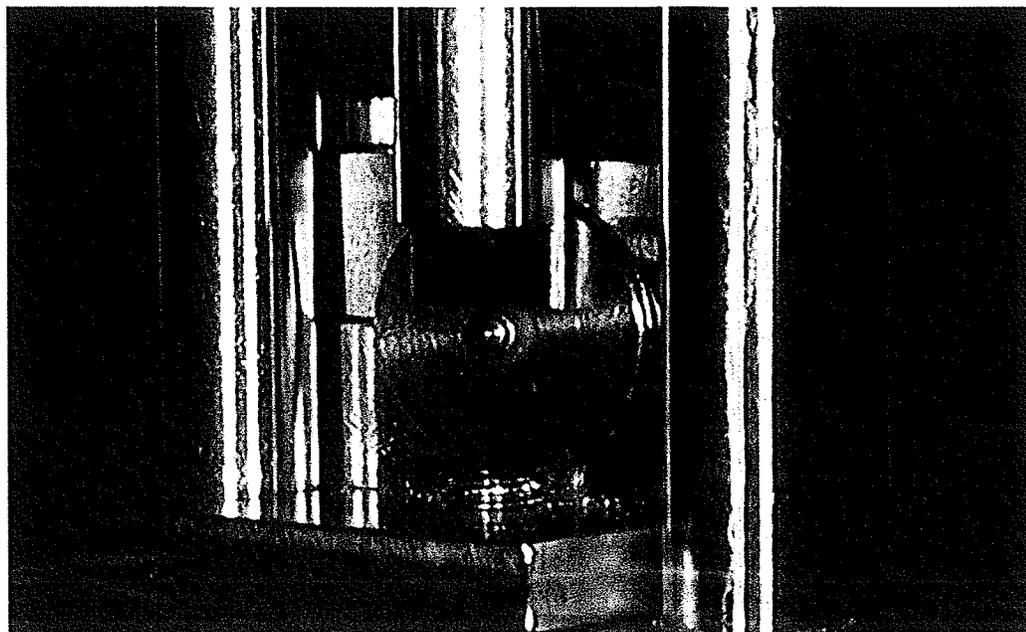


Figure 22: *Close-up of sample mounted in Bencor™ testing apparatus*



Figure 23: *ZwickTM materials testing machine and computer*

3.2.6 Evaluation of Fracture Sites

Each debonded button and composite surface was examined under a light-optical stereomicroscope (Nikon SM2-10, Tokyo, Japan) at 10x magnification by two independent evaluators. Intra-operator error was evaluated by rescoring 10 randomly chosen samples 5 times each with at least a three day interval between evaluations. The location of failure during debonding was classified according to a modified Adhesive Remnant Index (ARI) of Årtun and Bergland (1984).

Score 0= no adhesive left on composite surface

Score 1= less than half of the adhesive left on the composite surface

Score 2= more than half of the adhesive left on the composite surface

Score 3= all of the adhesive left on the composite surface with distinct
impression of bracket mesh

Score 4=composite resin fracture

3.2.7 Statistical Analysis

The results of the shear/peel bond strength testing after 24 hours and after thermocycling were analyzed using a two-tailed analysis of variance (ANOVA) at the 5% level of significance to determine if significant differences existed between the mean bond strengths of the six adhesive/surface treatment groups (Hassard, 1991). The means were then compared using Tukey's multiple range test to determine where the significant differences were.

The 24 hour and thermocycled bond strengths for each adhesive/surface treatment group were compared to each other using a repeated measures ANOVA at the 5% level of significance to determine if significant differences existed between the bond strengths measured at different times on the individual resin samples. A least squares mean analysis was used to determine where the significant differences were (Hassard, 1991).

3.2.8 Preparation of SEM specimens

Additional representative samples of the various surface treatments were prepared and did not have orthodontic attachments bonded. These were sputter coated with gold/platinum in a SEM Autocoating Unit (Hummer V, Technics, Tokyo, Japan). Each specimen was then evaluated in a scanning electron microscope (Model JSM 35C, JEOL, Tokyo, Japan) taken at 12kV under various magnifications between 100X and 350X for surface characterization. Representative samples of each ARI type after debonding were also coated in the same manner and evaluated in a scanning electron microscope (Model JSM 5900 LV, Tokyo, Japan) taken at 12 kV under various magnifications between 37X and 300X for surface characterization.

CHAPTER 4

RESULTS

	Page Number
4.1 Shear/Peel Bond Strengths	
4.1.1 24 hour shear/peel bond strengths	54-56
4.1.2 Shear/peel bond strengths of thermocycled samples	56-58
4.1.3 Shear/peel bond strengths after 24 hour vs. thermocycling	59-60
4.2 Modified Adhesive Remnant Index (ARI) Scores	61-66
4.3 Scanning Electron Microscopy Evaluation	66-74
4.4 Null Hypothesis	74

CHAPTER 4

RESULTS

4.1 SHEAR/PEEL BOND STRENGTHS

4.1.1 Twenty-four Hour Shear/Peel Bond Strengths

The highest mean bond strength was obtained by the air-abraded samples bonded with Transbond™ XT (21.98 ± 2.42 MPa). Their mean bond strength was significantly higher than the mean bond strengths of all other surface treatment/adhesive groups after 24 hours ($p < 0.001$).

All samples bonded with Smartbond® had mean bond strengths of similar statistical magnitude. No significant difference existed ($p > 0.05$) between untreated samples (11.88 ± 2.40 MPa), acid-etched samples (13.34 ± 1.69 MPa), and air-abraded samples (13.73 ± 2.08 MPa).

The lowest mean bond strengths were obtained using Transbond™ XT on untreated surfaces (9.69 ± 2.44 MPa) and acid-etched surfaces (8.13 ± 3.32 MPa).

No significant difference existed between these groups ($p>0.05$), but their mean bond strengths were significantly lower than all other groups after 24 hours ($p<0.01$).

The individual shear/peel bond strengths of each specimen, as well as mean shear/peel bond strengths, maximum, minimum, standard deviations, and coefficients of variance for each group are listed in **Appendix 2**.

The comparative data for the mean shear/peel bond strengths, standard deviations, and coefficients of variance are listed in **Table 1** and **Figure 24**.

	N	Mean (MPa)	Std. Deviation (MPa)	Coefficient of Variance (%)
Transbond/untreated	20	9.69	2.44	25.18
Transbond/acid-etched	20	8.13	3.32	40.64
Transbond/air-abraded	20	21.98	2.42	11.01
Smartbond/untreated	20	11.88	2.4	20.2
Smartbond/acid-etched	20	13.34	1.69	12.67
Smartbond/air-abraded	20	13.73	2.08	15.15

Table 1. *Twenty-four hour shear/peel bond strength descriptive statistics*

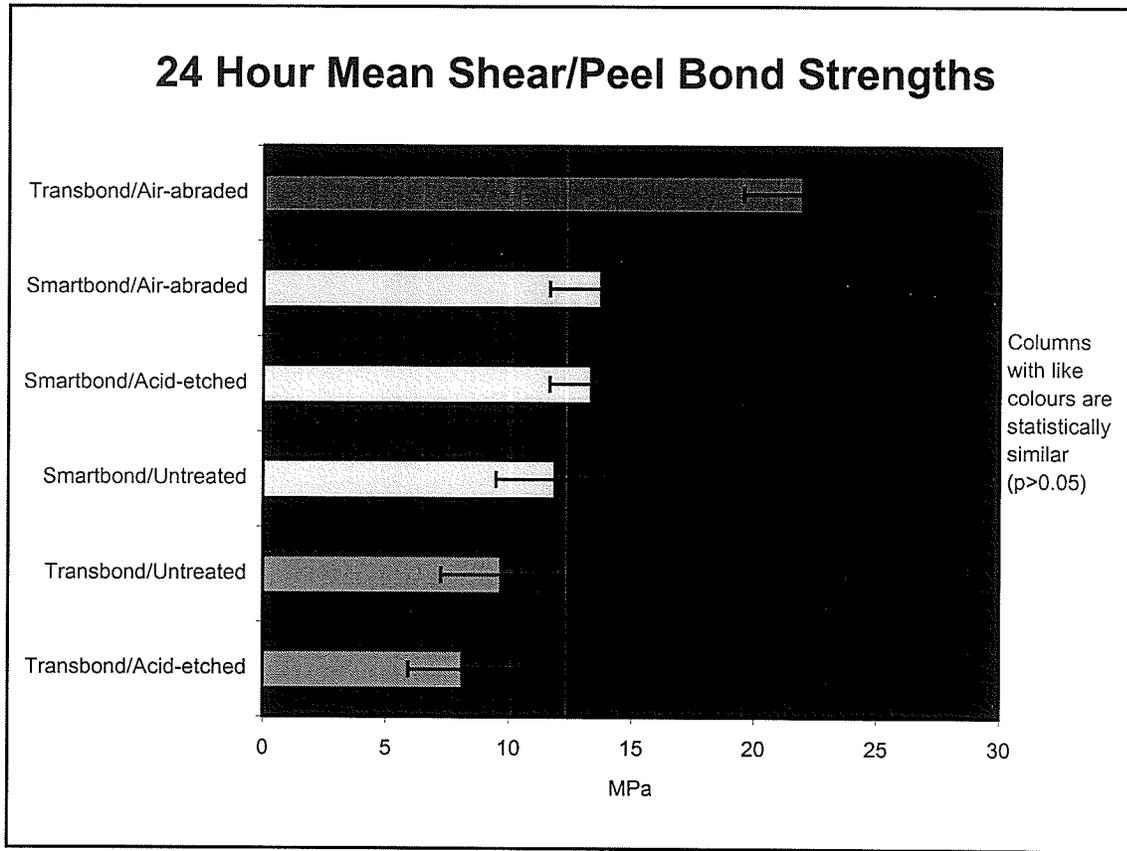


Figure 24. *Twenty-four hour mean shear/peel bond strength graph. Horizontal bars indicate standard deviations.*

4.1.2 Shear/Peel Bond Strengths of Thermocycled Samples

The highest mean bond strengths were obtained by air-abraded samples bonded with Transbond™ XT (21.85 ± 2.27 MPa). Their mean bond strength was significantly higher than the mean bond strength of all other surface treatment/adhesive groups after 24 hours ($p < 0.001$).

Acid-etched samples bonded with Smartbond[®] (12.41 ± 1.56 MPa) and air-abraded samples bonded with Smartbond[®] (11.32 ± 3.22 MPa) had mean bond strengths of similar statistical magnitude. No significant difference existed between these two groups ($p > 0.05$).

The lowest mean shear/peel bond strengths were obtained in the untreated/Transbond[™] XT group (7.43 ± 2.22 MPa), the acid-etched/Transbond[™] XT group (7.10 ± 3.10 MPa), and the untreated/Smartbond[®] group (6.88 ± 1.29 MPa). No significant differences existed between these three groups ($p > 0.05$), but their mean shear bond strengths were significantly lower than all other groups after thermocycling ($p < 0.001$).

The individual shear/peel bond strengths of each specimen, as well as mean shear bond strengths, maximum, minimum, standard deviations, and coefficients of variance for each group are listed in **Appendix 2**.

The comparative data for the mean shear/peel bond strengths, standard deviations, and coefficients of variance are listed in **Table 2** and **Figure 25**.

	N	Mean (MPa)	Std. Deviation (MPa)	Coefficient of Variance (%)
Transbond/Untreated	20	7.43	2.22	29.88
Transbond/Acid-etched	20	7.09	3.09	43.66
Transbond/Air-abraded	20	21.85	2.27	10.39
Smartbond/Untreated	20	6.88	1.29	18.75
Smartbond/Acid-etched	20	12.41	1.56	12.57
Smartbond/Air-abraded	20	11.32	3.22	28.45

Table 2. *Shear/peel bond strength descriptive statistics for thermocycled samples*

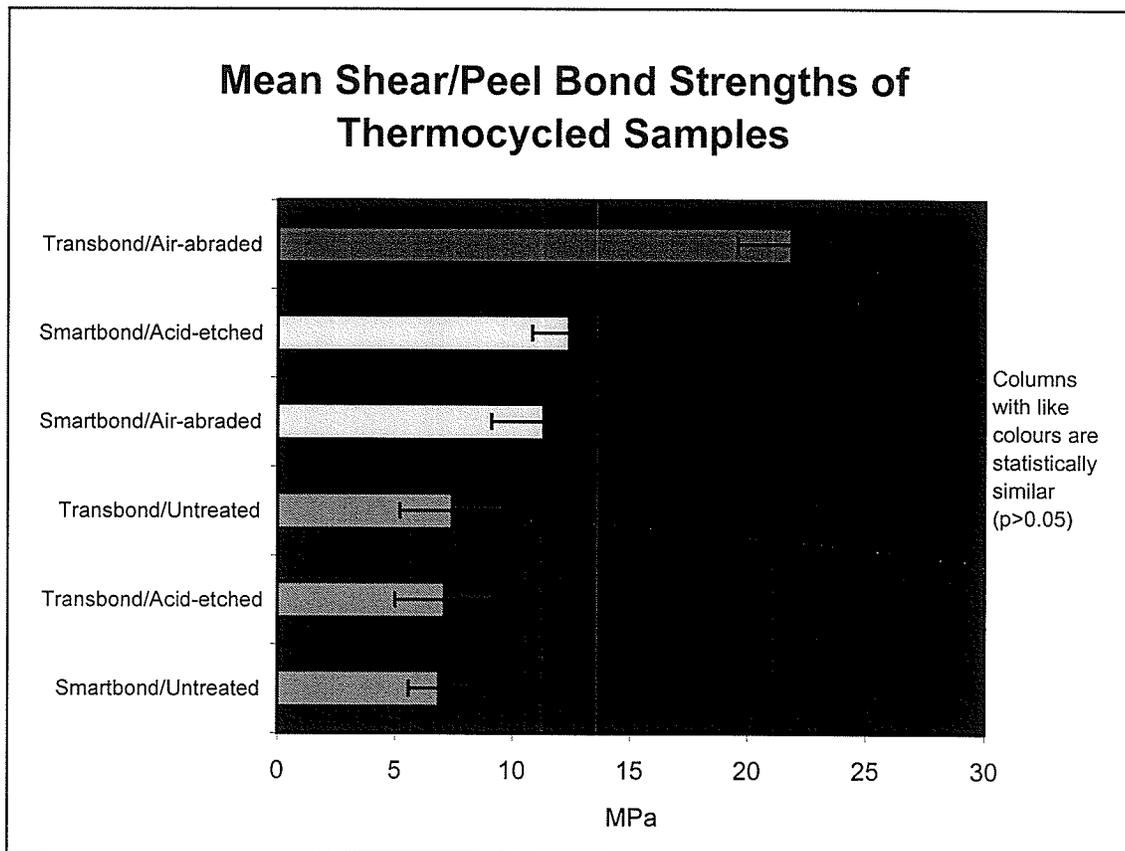


Figure 25. *Mean shear/peel bond strengths of thermocycled samples graph.*

Horizontal bars indicate standard deviations.

4.1.3 Bond Strengths After Twenty-four Hours vs. Thermocycling

A repeated measures ANOVA and paired t-tests were done for each group at the 5% level of confidence. The mean shear/peel bond strengths measured after 24 hours and after thermocycling were found to be significantly different between air-abraded samples bonded with Smartbond[®] (13.73 MPa vs. 11.32 MPa) ($p < 0.05$), untreated samples bonded with Smartbond[®] (11.88 MPa vs. 6.88 MPa) ($p < 0.001$), and untreated samples bonded with Transbond[™] XT (9.69 MPa vs. 7.43 MPa) ($p < 0.05$). In all three cases, the bond strength decreased with thermocycling.

There were no significant differences in the mean shear/peel bond strength measured after 24 hours or after thermocycling for the other three surface treatment/adhesive groups ($p > 0.05$).

The comparative data for the mean shear/peel bond strengths, standard deviations, and coefficients of variation are listed in **Table 3** and **Figure 26**.

	24 Hours (MPa)	Thermocycled (MPa)
Transbond/untreated	9.69	7.43
Transbond/acid-etched	8.13	7.09
Transbond/air-abraded	21.98	21.85
Smartbond/untreated	11.88	6.88
Smartbond/acid-etched	13.34	12.41
Smartbond/air-abraded	13.73	11.32

Table 3. Mean shear/peel bond strengths after twenty-four hours vs. thermocycling.

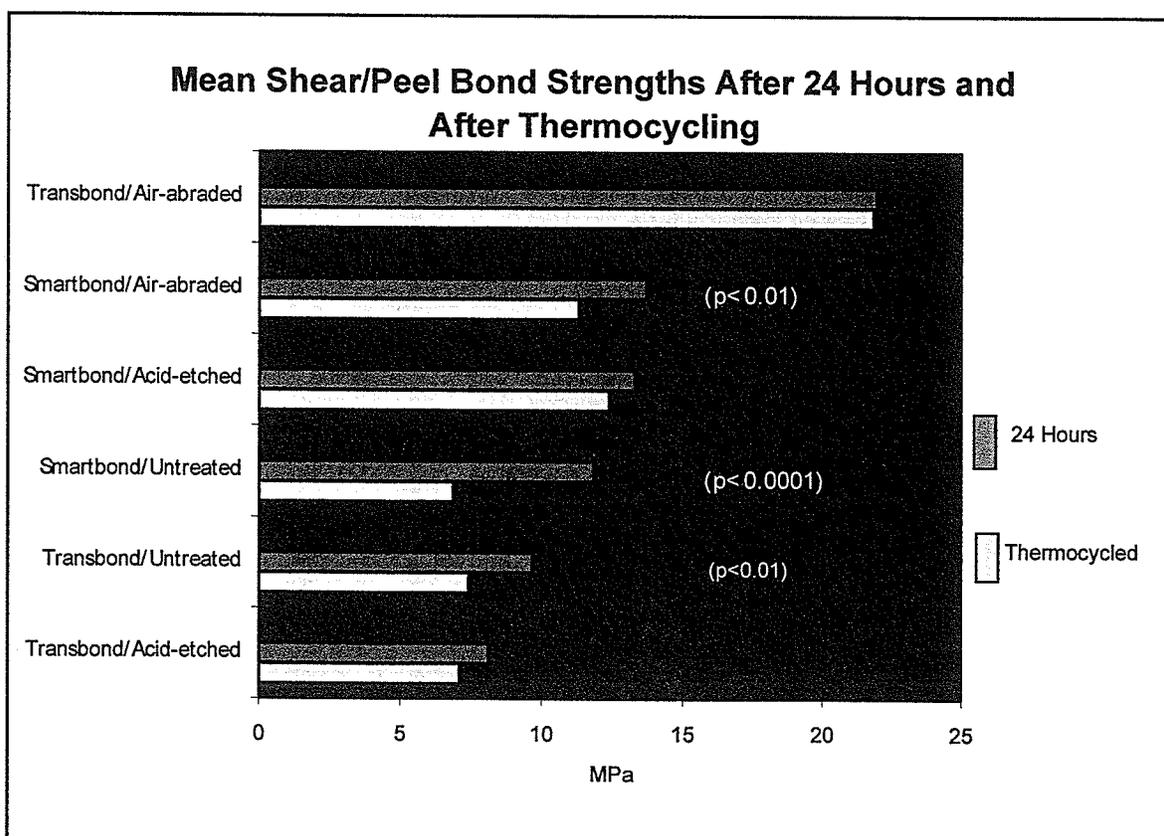


Figure 26. Mean shear/peel bond strength graph of twenty-four hours vs. thermocycled samples

4.2 MODIFIED ADHESIVE REMNANT INDEX (ARI) SCORES

The most common type of debond was one in which no adhesive remained on the composite resin surface and no damage was done to the resin surface (ARI =0) (Figure 27). This failure pattern was observed in 80 (33%) of the samples. All of the samples displaying this failure pattern were bonded with Transbond™ XT to either untreated or acid-etched surfaces.

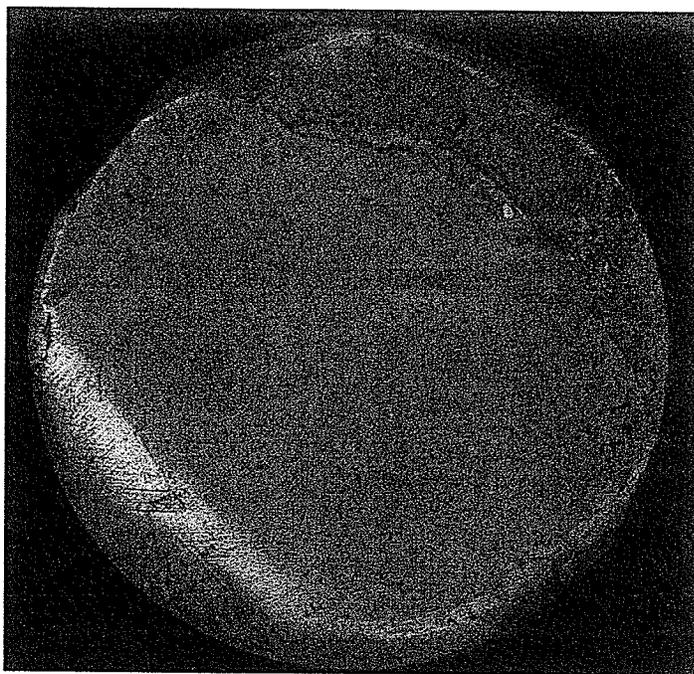


Figure 27. *Debonded sample (20X) with an ARI score of zero (no adhesive remaining on the resin surface)*

Various amounts of adhesive remnants remained on the resin surfaces (ARI scores of 1,2, or 3) of 120 (50%) of the samples. All of these failure patterns were observed in samples bonded with Smartbond® under all of the three surface treatments (Figures 28, 29, 30).

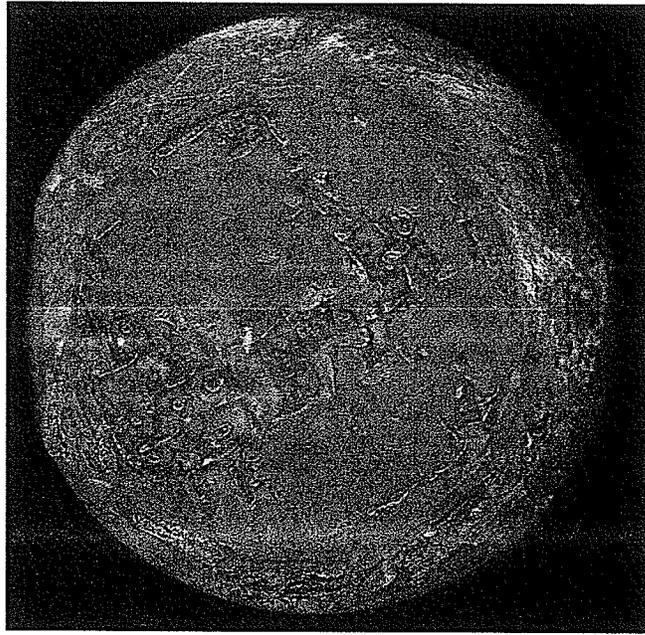


Figure 28. *Debonded sample (20X) with an ARI score of one (less than half of the adhesive remaining on the resin surface)*

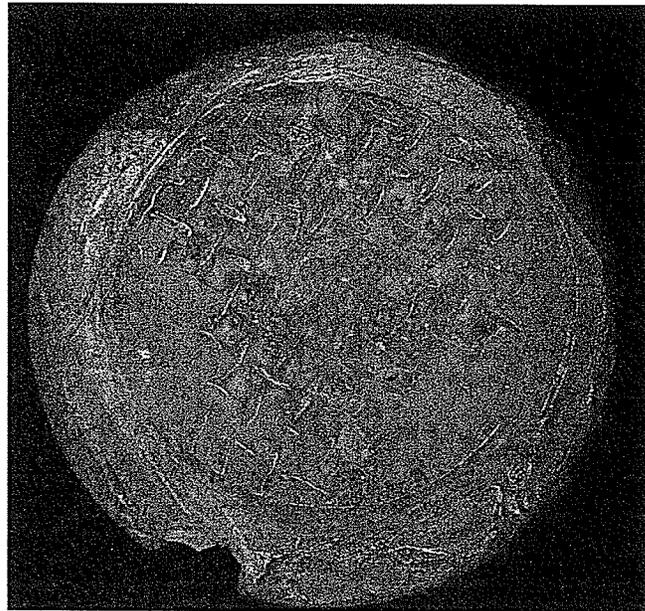


Figure 29. *Debonded sample (20X) with an ARI score of two (more than half of the adhesive remaining on the resin surface)*

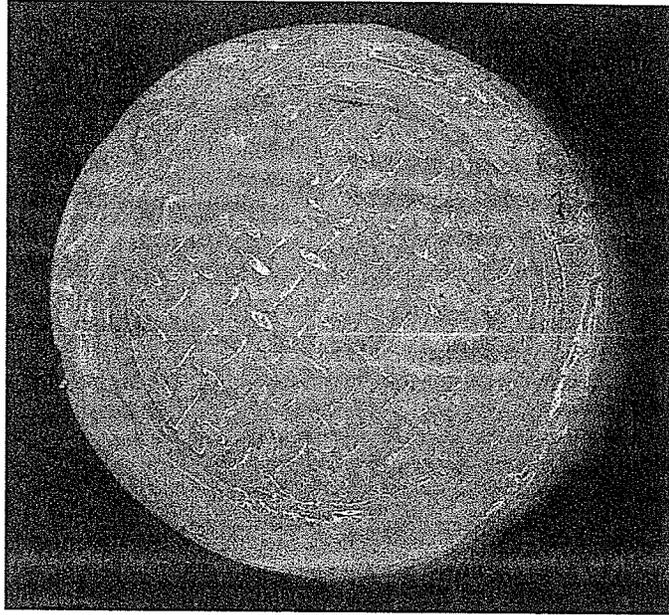


Figure 30. *Debonded sample (20X) with an ARI score of three (all of the adhesive remaining on the resin surface with a distinct impression of the attachment mesh)*

Composite resin fractures (ARI score=4) occurred in 40 (17%) of the samples. All of these resin fractures were in samples bonded with Transbond™ XT to air-abraded surfaces (**Figure 31**).

A Chi squared analysis was used to compare the incidence of composite resin fracture between the groups at 24 hours and after thermocycling. It was determined that significant differences existed, with samples bonded using Transbond™ XT to air-abraded surfaces had an increased incidence of composite

resin fracture (100%) over all other groups ($p < 0.0001$). All other groups had statistically similar composite resin fracture incidences of 0% ($p > 0.05$).

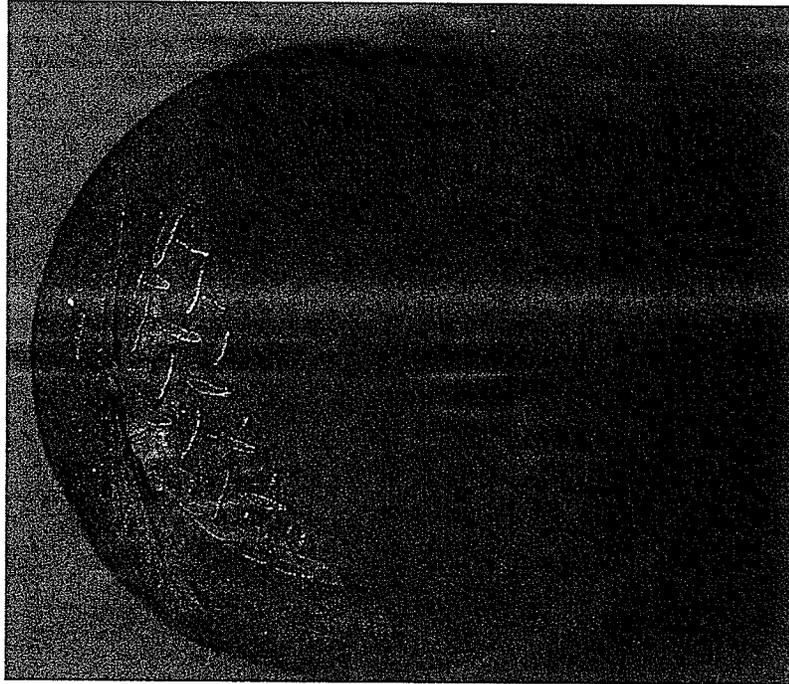


Figure 31. *Debonded sample with an ARI score of four (composite resin fracture)*

The adhesive remnant index score for each sample is listed in **Appendix 2**.

Comparative data of the mean ARI scores for the six adhesive/surface treatment groups after twenty-four hours and after thermocycling are listed in **Table 4**. An overall graphic representation of the ARI scores is given in **Figure 32**.

	24 hours	Thermocycled
Transbond/Untreated	0	0
Transbond/Acid-etched	0	0
Transbond/Air-abraded	4	4
Smartbond/Untreated	1.95	1.85
Smartbond/Acid-etched	1.8	1.8
Smartbond/Air-abraded	2.95	2.8

Table 4. Mean modified adhesive remnant index scores

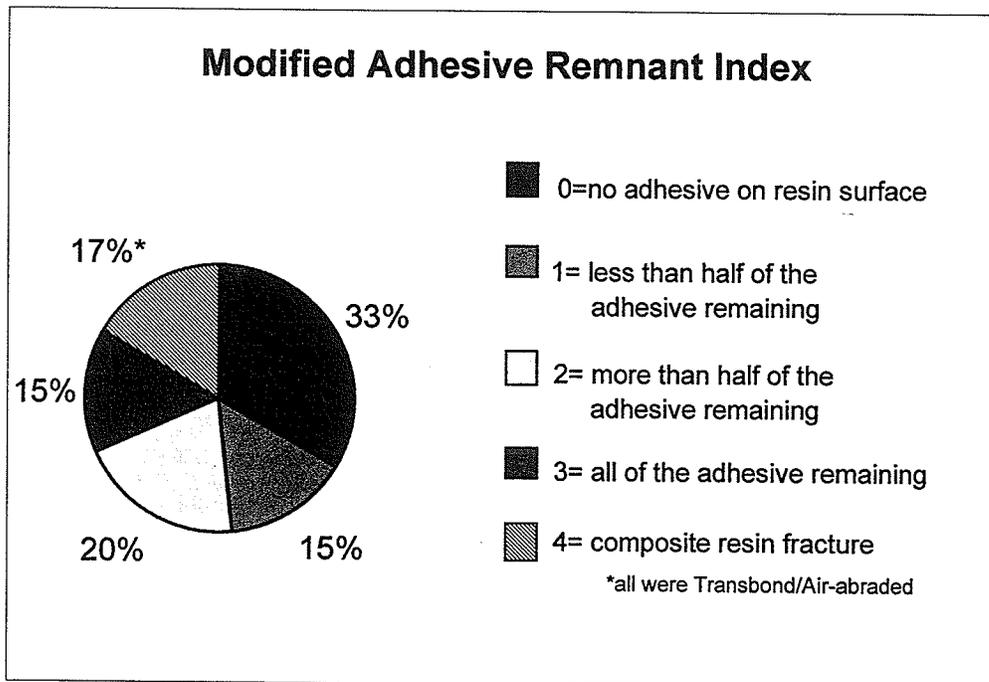


Figure 32. Graphic representation of ARI scores

Evaluation of samples under 20X stereomicroscope for ARI scores also revealed the presence of minor bubbles in the remaining adhesive in six samples. These small bubbles occurred with the use of Smartbond[®] under the various surface treatment conditions. It may be that Smartbond[®] is more susceptible to bubbles because the adhesive must be “dabbed” on with an applicator. This process may incorporate bubbles into the adhesive. The bubbles never appeared to exceed five percent of the bonding area under visual examination. In all six instances, the shear/peel bond strengths for these samples closely matched the mean of their respective group, and none were beyond the standard deviation. As the minor bubbles did not appear to affect the shear/peel bond strength values, these samples were used in the data analysis.

4.3 SCANNING ELECTRON MICROSCOPY EVALUATION

Visual evaluation of scanning electron micrographs taken of a polished composite resin sample (300X) (Figure 33) and an acid-etched sample (300X) (Figure 34) representative of control surfaces, revealed smooth surfaces with minimal irregularities. These surfaces would provide limited micromechanical retention.

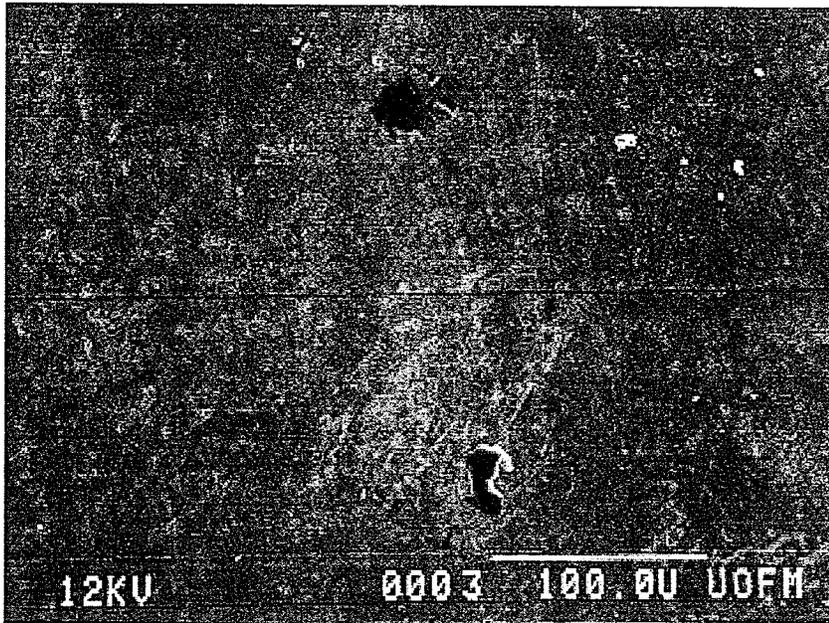


Figure 33. SEM of a polished composite resin surface (300X)

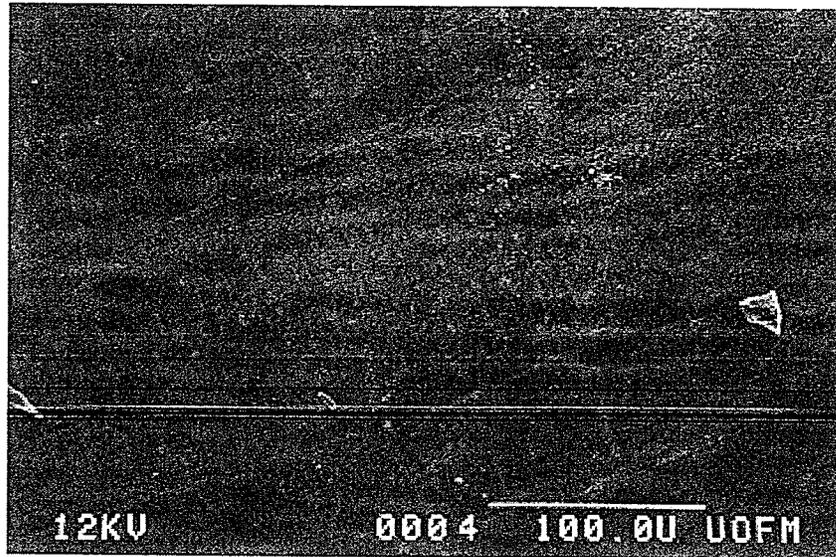


Figure 34. SEM of an *acid-etched composite resin surface (300X)*

The scanning electron micrograph of an air-abraded control sample (300X) (Figure 35) revealed a highly irregular surface with numerous undercuts that would allow for micromechanical retention.

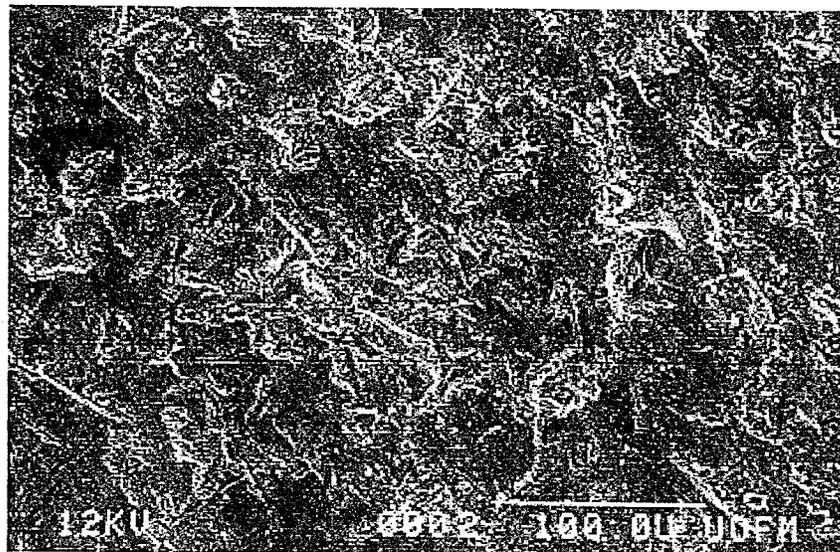


Figure 35. SEM of an *air-abraded composite resin surface (300X)*

Scanning electron micrographs were also done on representative samples, following debonding, of each type of modified ARI score. Samples displaying an ARI score of zero (**Figures 36,37**) revealed smooth surfaces with minimal irregularities. No restorative resin damage was apparent.

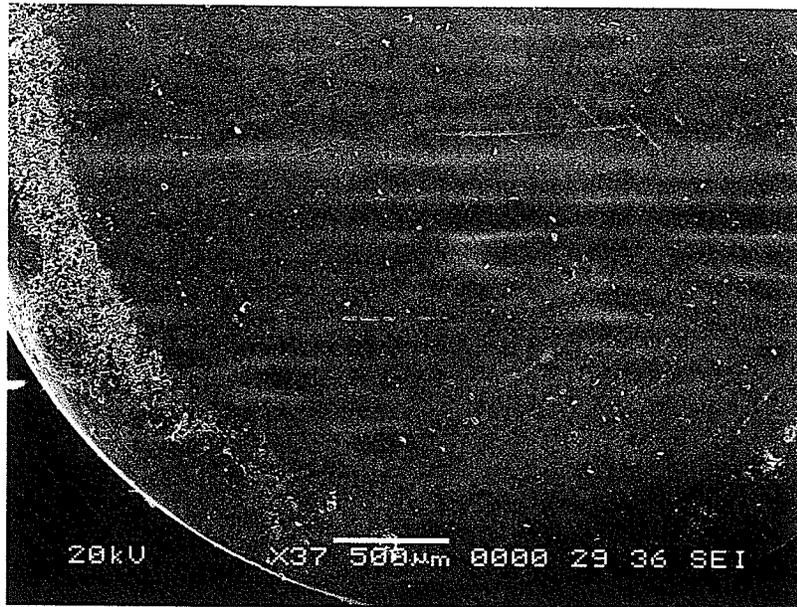


Figure 36. SEM (37X) of a debonded sample with an ARI=0

Scanning electron micrographs of samples with ARI scores of one or two revealed surfaces with varying amounts of adhesive remaining on the set resin (**Figures 38,39**). No restorative resin damage was apparent.

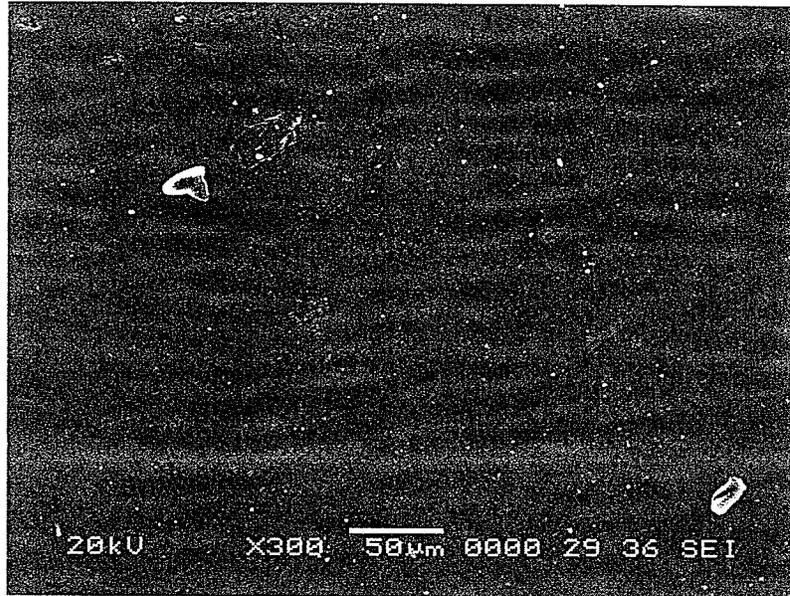


Figure 37. SEM (300X) of a debonded sample with an ARI=0.

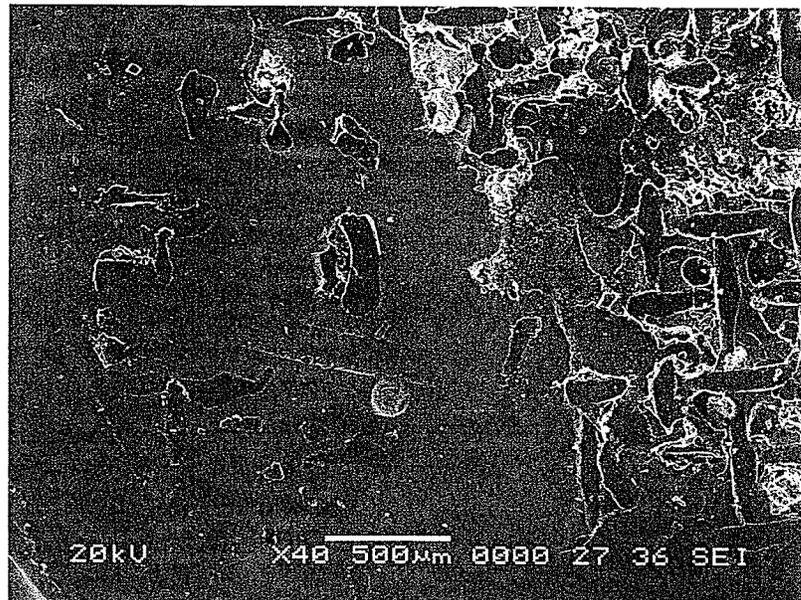


Figure 38. SEM (40X) of a debonded sample with an ARI=1.

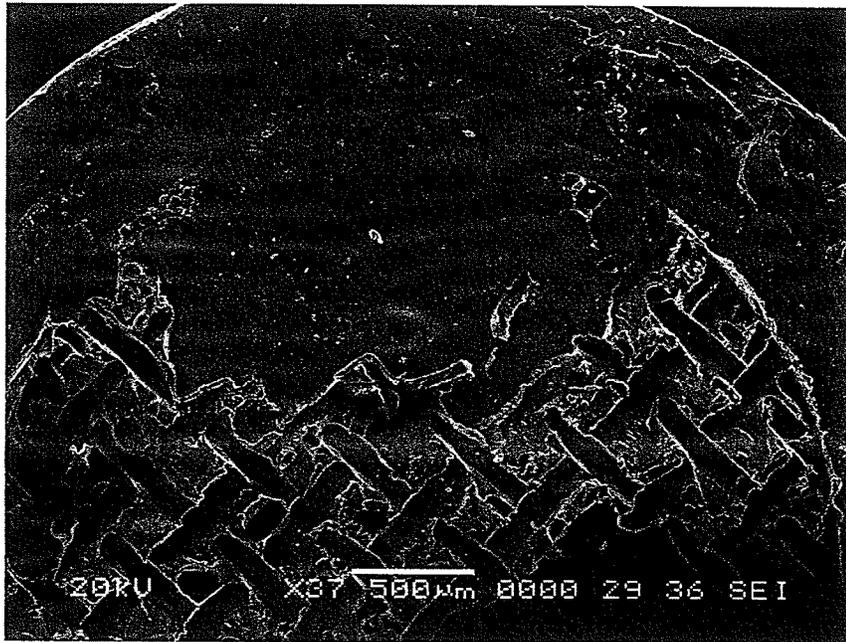


Figure 39. SEM (37X) of a debonded sample with an ARI= 2.

Scanning electron micrographs of samples with ARI scores of three revealed surfaces with all of the adhesive remaining on the set resin (Figures 40,41). The adhesive left a distinct impression of the attachment base mesh. No restorative resin damage was apparent.

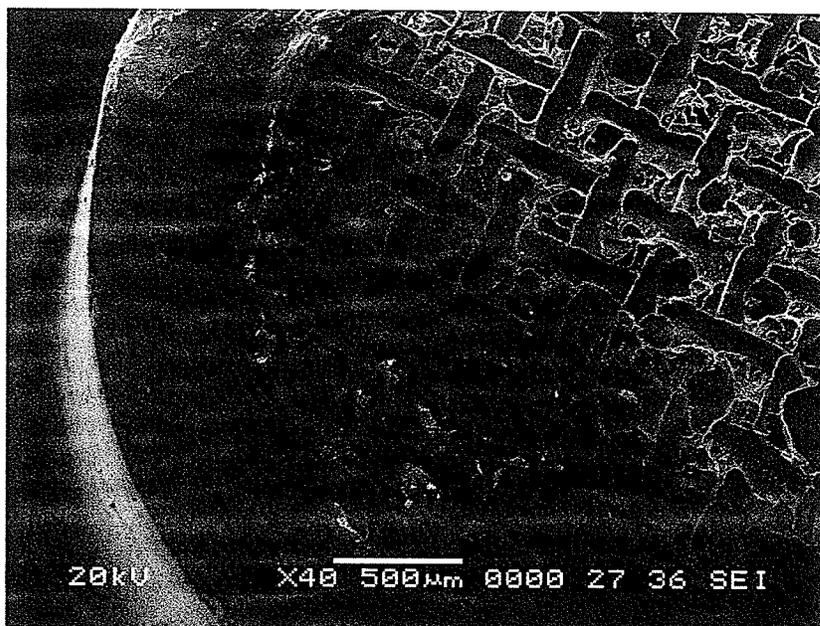


Figure 40. SEM (40X) of a debonded sample with an ARI=3.

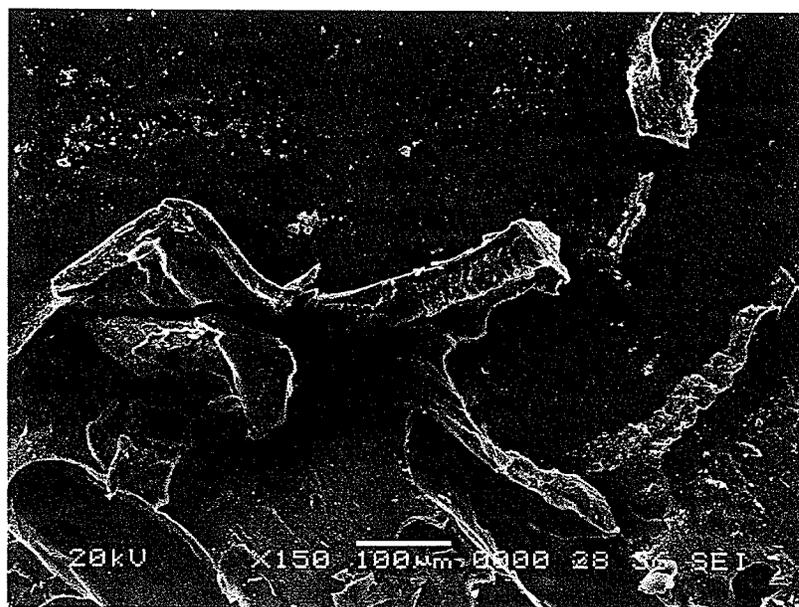


Figure 41. SEM (150X) of a debonded sample with an ARI=3.

Scanning electron micrographs of samples that experienced restorative resin fracture (ARI=4) (Figures 42,43) revealed varying amounts of adhesive remaining next to sharp edges of fracture lines. Focus was lost as the fractured surface quickly fell away from the field of view. Varying amounts of set resin fractured from the cylindrical samples, and in all instances, the orthodontic attachments remained adhered to the fractured segments.



Figure 42. SEM (37X) of debonded sample with an ARI=4.

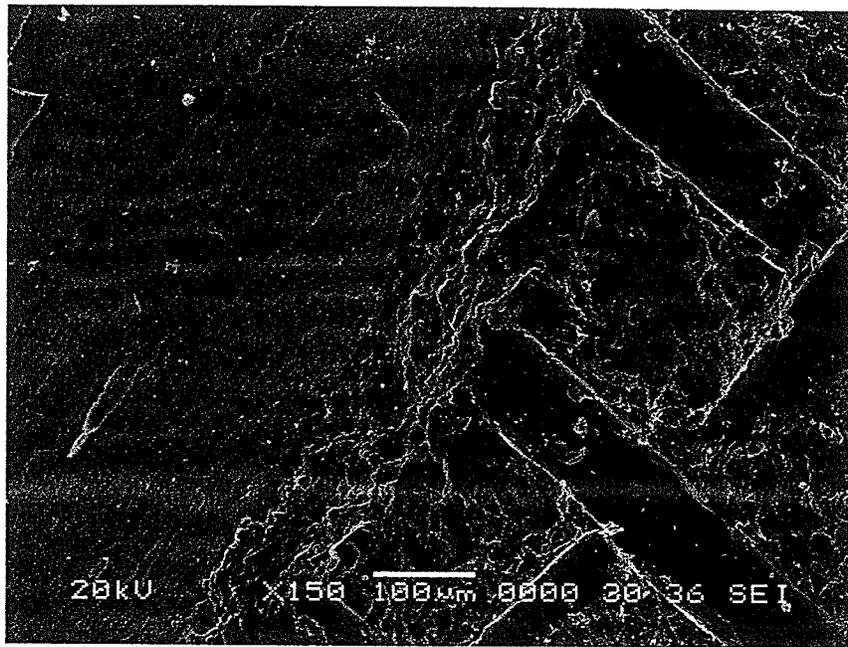


Figure 43. SEM (150X) of a debonded sample with an ARI=4.

4.4 NULL HYPOTHESIS

Statistically significant differences were found in the shear/peel bond strengths between the different adhesive/surface treatment groups. Statistically significant differences in shear/peel bond strength were also found between the groups for samples tested after twenty-four hours and thermocycled samples. The null hypothesis is therefore rejected. It is evident that adhesive/surface treatment and thermocycling have a significant effect on the shear/peel bond strength of orthodontic attachments to composite resin surfaces.

CHAPTER 5

DISCUSSION

	Page Number
5.1 Shear/Peel Bond Strengths	
5.1.1 24 hour shear/peel bond strengths	76-82
5.1.2 Shear/peel bond strengths of thermocycled samples	82-85
5.2 Modified Adhesive Remnant Index (ARI) Scores	86-88

CHAPTER 5

DISCUSSION

5.1 SHEAR/PEEL BOND STRENGTHS

5.1.1 Twenty-Four Hour Shear/Peel Bond Strengths

Statistically significant differences were found in the mean shear/peel bond strengths between the six surface preparation/adhesive groups after twenty-four hours ($p < 0.05$). The highest mean bond strengths were obtained when attachments were bonded to air-abraded surfaces with Transbond™ XT. All three surface preparations using Smartbond® were of similar magnitude ($p > 0.05$), but their mean bond strengths were lower than the air-abraded/Transbond™ XT group ($p < 0.05$). The lowest mean bond strengths were obtained in the untreated and acid-etched surface groups bonded with Transbond™ XT ($p < 0.05$). Accordingly, for Transbond™ XT, surface preparation of the resin substrate resulted in significant differences in the 24 hour bond strength values. However, for Smartbond®, 24 hour shear/peel bond strengths were unaffected by differences in the substrate surface.

No standardized protocol exists for bond strength testing in orthodontics (Fox *et al.*, 1993; Eliades and Brantley, 2000). As a result, most reported studies have numerous variables that differ from each other. In 66 papers reporting bonding to enamel, Fox *et al.* (1993) found nine different types of teeth were used, including human, bovine and baboon. In comparing the limited data on orthodontic bonding to composite resin, the differences in substrate must also be considered. Schwarz *et al.* (1990) used microfilled and hybrid resins, while Kao *et al.* (1995), Chunchacheevachaloke and Tyas (1997), and Lai *et al.* (1999) only used microfilled resin substrates. In the present study, a hybrid resin was chosen because it will more likely be encountered clinically in the future (C.R.A., 1999). These substrate differences may have an effect on the bond strength values, due to the differences in filler material and loading, type of resin, and surface physical characteristics.

Storage prior to debonding is another source of error in comparing studies. Schwartz *et al.* (1990) did not report their storage protocol. Kao *et al.* (1995) thermocycled their samples 500 times between 4°C and 55°C water baths and then stored the samples in 37°C water for seven days. Chunchacheevachaloke and Tyas (1997) and Lai *et al.* (1999) both stored their samples for 24 hours in 37°C water. Thermocycling is done to mechanically “stress” the bond (ISO, 1994), and there may be differences in bond strengths between 24 hours of storage and seven days of storage because of water saturation.

The method and direction of debonding may be a major source of inter-study error. As previously mentioned, shear, shear/peel, tensile, and torsional bond strengths have all been reported. Bond strength values may differ depending on whether the debonding instrument was placed at the adhesive interface, the bracket base, or on a bracket tie-wing. The crosshead speed of the loading apparatus may affect the bond strengths. Chunhacheevachaloke and Tyas (1997) used 5mm/min, Lai *et al.* (1999) used 1mm/min, and the present study used 0.5mm/min. It is possible that viscoelastic behaviour of the adhesives may manifest itself at lower crosshead speeds (Eliades and Brantley, 2000).

The orthodontic attachments used may provide a source of error. Some studies used ceramic brackets (of which there are numerous materials) (Lai *et al.*, 1999), and others used different forms of metallic orthodontic attachments. Different attachments may have dissimilar bases and retentive features (Lai *et al.*, 1999). The orthodontic adhesive used in bonding may provide disparities in bond strengths as well. Manufacturers keep the exact formulation of their adhesives secret for proprietary reasons, and the differences between products may have profound effects on the chemical or mechanical bonding properties (Kao *et al.*, 1995; Powers *et al.*, 1997; Lai *et al.*, 1999; Örtendahl and Örtengren, 2000). These numerous factors make direct comparisons between bond strength studies difficult, and comparisons can only be interpreted in a very broad, general manner.

Chunhacheevachaloke and Tyas (1997) bonded ceramic brackets to roughened and unroughened microfilled composite resin surfaces using Transbond™ XT. They reported no significant differences in the shear bond strengths of their experimental groups after twenty-four hours ($p>0.05$), and mean bond strengths ranged from 17.1 +/- 5.73 MPa to 19.2 +/- 6.75 MPa. The values reported are similar to the mean shear/peel bond strength obtained in the air-abraded/Transbond™ XT group in the present study (21.98 +/- 2.42 MPa).

Lai *et al.* (1999) bonded metal brackets to roughened, microfilled composite resin surfaces using Transbond™ XT. After twenty-four hours, their mean shear/peel bond strength was 14.5 +/- 8.16 MPa. This value is much lower than that obtained in the present study. A possible explanation may be their use of a microfilled restorative resin, compared to the use of a hybrid resin in the present study. No studies could be found in the English literature which report on the bond strengths of resins to both microfilled and hybrid resins.

A literature search failed to uncover any publications regarding the use of Smartbond® on resin surfaces. Örtendahl and Örtengren (2000) bonded stainless steel brackets to acid-etched enamel with Smartbond® and obtained shear bond strengths of 22 MPa after twenty-four hours. This is much higher than the bond strengths to composite resin obtained with Smartbond® after twenty-four hours in

the present study. This suggests that Smartbond® may induce significantly higher bond strengths to enamel than to composite resin.

Some of the results of the present study were as expected, while others were not anticipated. It was anticipated that untreated and acid-etched surfaces would provide similar bond strengths, and air-abraded surfaces would increase the bond strength. These expectations were based on previous results from the field of restorative composite resin repair (Boyer *et al.*, 1978; Swift *et al.*, 1992; Turner and Meiers, 1993; Kupiec and Barkmeier, 1996; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997). The efficacy of Smartbond® could not be anticipated, as no previous benchmark had been established for cyanoacrylate orthodontic adhesives.

The results did follow expectations in that acid-etching failed to significantly increase the shear/peel bond strengths over untreated surfaces with either adhesive. This suggests that acid-etching of composite resin surfaces may not be necessary prior to orthodontic bonding. Acid-etching of enamel has proven to be an essential process, as the acid preferentially dissolves the centers or peripheries of the enamel rods, providing undercuts in the tooth surface for mechanical adhesion (Wang *et al.*, 1994; Olsen *et al.*, 1996). However, acid-etching does little, if anything, to alter surfaces of composite resin (Brosh *et al.*, 1997). This was supported by the control scanning electron micrographs of acid-etched samples, which revealed smooth surfaces with minimal irregularities. The

untreated and acid-etched surfaces appeared the same under scanning electron micrograph examination.

Air-abrasion of the substrate surfaces significantly increased the shear/peel bond strengths when using Transbond™ XT. This too follows expectations as numerous reports have advocated the use of air-abrasion for surface preparation in composite repair (Swift *et al.*, 1992; Turner and Meiers, 1993; Kupiec and Barkmeier, 1996; Bouschlicher *et al.*, 1997; Brosh *et al.*, 1997). Air-abrasion was shown on the scanning electron micrographs to provide a highly irregular surface with numerous undercuts that would allow for improved micromechanical retention. Interestingly, air-abrasion did not significantly affect the shear/peel bond strengths with the use of Smartbond®. It may be that the cyanoacrylate adhesive is too viscous to penetrate into the microscopic undercuts created with air-abrasion. Smartbond® appeared to be much more viscous than Transbond™ XT primer during handling. Thus, the bond strength values would remain unchanged.

From the present study, any of the six non-thermocycled surface preparation/adhesive groups could potentially meet the clinically adequate shear/peel bond strength of 5.9 to 7.9 MPa, in accordance with recommendations by Reynolds (1975). Smartbond® provided increased shear/peel bond strengths after 24 hours over Transbond™ XT unless air-abrasion was utilized. The air-

abraded group bonded with Transbond™ XT did, however, produce significantly higher bond strengths after twenty-four hours than all other groups ($p < 0.05$).

5.1.2 Shear/Peel Bond Strengths of Thermocycled Samples

Statistically significant differences were found in the mean shear/peel bond strengths between the six surface preparation/adhesive groups after thermocycling ($p < 0.05$). The highest mean bond strengths were obtained when attachments were bonded to air-abraded surfaces with Transbond™ XT. Acid-etch and air-abraded surfaces bonded with Smartbond® were of similar magnitude ($p > 0.05$), but their mean bond strengths were lower than the air-abraded/Transbond™ XT group ($p < 0.05$). The lowest mean bond strengths were obtained in the untreated and acid-etched surface groups bonded with Transbond™ XT ($p < 0.05$) and the untreated group bonded with Smartbond®.

The paper by Lai *et al.* (1999) is the only known study reporting on the shear/peel bond strength of orthodontic attachments to composite resin after thermocycling. They reported a mean bond strength of 20.0 +/- 10.5 MPa between stainless steel brackets and a roughened microfilled composite resin (Silux Plus™, 3M, St. Paul, MN) after thermocycling 500 times between 5°C and 55°C. This value is similar to the mean shear/peel bond strength obtained after thermocycling for the air-abraded/Transbond™ XT group in the present study (21.85 +/- 2.27 MPa).

A literature review failed to identify, for comparison, any studies of the bond strength of Smartbond[®] after thermocycling.

From the present study, it appears that any of the six surface preparation/adhesive groups may potentially provide clinically adequate shear/peel bond strength after thermocycling as recommended by Reynolds (1975). The air-abraded group bonded with Transbond[™] XT produced significantly higher bond strengths after thermocycling than all other groups ($p < 0.05$).

Significantly lower mean shear/peel bond strengths were found after thermocycling the following three groups: untreated/Transbond[™] XT, untreated/Smartbond[®], and air-abraded/Smartbond[®]. This is in agreement with Lai *et al.* (1999) who reported a significantly lower bond strength between stainless steel brackets and resin surfaces after thermocycling. Numerous other articles on orthodontic bonding to enamel, amalgam, and porcelain also report decreases in bond strength after thermocycling (Bishara *et al.*, 1998; Bourke and Rock, 1999; Sperber *et al.*, 1999). Zachrisson *et al.* (1996) discussed how the adversarial forces of polymerization shrinkage, thermal expansion coefficient mismatch, and hygroscopic expansion of composite resin challenges the orthodontic bonds during thermocycling. These factors are involved in the decrease in bond strengths commonly found after thermocycling. Another possible explanation for the thermocycling affecting Smartbond[®] samples more

than Transbond™ XT samples is that the cyanoacrylates have been shown to hydrolyze upon exposure to water (Buonocore, 1973). The thermocycled samples were stored for 7 days in water to allow water sorption to occur prior to thermocycling, and perhaps the cyanoacrylate decomposed slightly, decreasing the shear/peel bond strengths.

It is interesting to note that thermocycling did not have a significant effect on the shear/peel bond strength of the following groups: air-abraded/Transbond™ XT, and both acid-etched groups. Every sample in the air-abraded/Transbond™ XT group experienced restorative composite resin fracture. Thus, the mean value of 21.85 +/- 2.27 MPa may actually be the cohesive fracture strength of the restorative resin, not the shear/peel bond strength between the resin and the orthodontic attachments. It is entirely possible that the thermocycling did decrease the bond strength, but it was still higher than the cohesive strength of the restorative resin.

Of greater interest is how the process of acid-etching seemed to alleviate some of the negative effects of thermocycling on the bond strength. Neither group that had the resin surface acid-etched experienced a significant decrease in shear/peel bond strengths. No similar studies are known to exist that corroborate this finding, but we can speculate why these results were obtained. It has been shown by scanning electron micrograph that acid-etching does little, if anything, to physically affect the surface of composite resin. However, perhaps there were chemical changes

produced by the acid-etch, which allowed for increased tolerance to thermocycling. It may be that the acidulation provides a better surface for bonding. Perhaps the acid-etch modifies the surface chemistry of the set resin in such a way as to improve wetting and subsequent solvent bonding.

It should also be noted that *in vitro* data cannot be accurately correlated to *in vivo* situations. Zachrisson (2000) lists three reasons why laboratory data must be interpreted with caution: “(1) a continually increasing tensile or shear load applied to bonded brackets in the laboratory is not representative of the force applications that occur clinically; (2) the type of debonding force in machines is not the same as the force applied in clinical debonding; and (3) the complex oral environment with variations in temperature, stresses, humidity, acidity, and plaque is not reproducible in the laboratory.” Clinically, orthodontic forces are not continually increasing. Clinical forces are highest immediately upon application, and then experience different rates of decay depending upon the appliance utilized. In debonding an attachment clinically, torsional forces are often applied to the bracket tie-wings. This deforms the bracket base, which allows for easier debonding. In the present study, a shear/peel force was applied to the attachment/adhesive/resin interface, not to bracket tie-wings. It is easier to standardize samples in this manner to evaluate the adhesive and not the deformation of the attachment. The third point of Zachrisson’s is self-explanatory. Nevertheless, rigorous *in vitro* testing provides comparative baseline data which can be useful in biomaterials selection for the clinical situation.

5.2 MODIFIED ADHESIVE REMNANT INDEX (ARI) SCORES

The evaluation of adhesive remnant indices can be interpreted in different ways. There is no definitive protocol for an “appropriate” ARI score, as several different scoring systems appear in the literature (Årtun and Bergland, 1984; Kao *et al.*, 1995; Lai *et al.*, 1999). Having minimal adhesive remaining on the tooth or restoration after debonding makes clean-up more efficient, but it exposes the tooth or restoration to higher incidences of damage (Årtun and Bergland, 1984; Powers *et al.*, 1997; Bourke and Rock, 1999). The most relevant data is that of tooth or restoration damage.

In the present study, restorative composite resin damage was only found with one type of surface preparation/adhesive combination. Every sample that was air-abraded and bonded with Transbond™ XT experienced cohesive resin fracture upon debonding (ARI=4). These groups also had significantly higher ($p<0.0001$) shear/peel bond strengths than all other groups. No other samples revealed any resin substrate damage. Thermocycling had no significant effect on ARI scores.

One possible explanation is that perhaps the Transbond™ XT primer was able to penetrate the micromechanical undercuts produced with air-abrasion, and once cured, locked the attachment to the resin substrate with more strength than the cohesive strength of the resin itself. As discussed earlier, perhaps the cyanoacrylate was unable to penetrate into these undercuts as efficiently as the

primer. Another theory on the resin fractures may be that the air-abraded surfaces were weakened from the formation of microcracks. Upon application of the debonding force, the microcracks may have penetrated into the set resin, resulting in cohesive failure. Thus, in this theory, it was actually a decrease in set resin surface strength, not an increase in shear/peel adhesive bond strength that led to the cohesive failures. Smartbond[®], possibly because of its greater viscosity, did not penetrate into the irregular, air-abraded composite. As a result, the bonds were still weaker than the cohesive strength of the weakened, air-abraded resin surfaces. The potentially superior solvent bonding of Smartbond[®] would have been masked by its inability to penetrate the air-abraded microcracks.

Chunhacheevachaloke and Tyas (1997) reported 55% of their samples bonded to roughened resin surfaces experienced resin fracture. Lai *et al.* (1999) later reported that 73% of their roughened resin samples experienced damage upon debonding.

None of the untreated or acid-etched samples bonded with Transbond[™] XT showed any adhesive remaining on the resin surfaces (ARI=0). These groups had the lowest mean shear/peel bond strengths as well. All of the Smartbond[®] samples had various amounts of adhesive remaining on the resin surfaces after debonding (ARI=1, 2, or 3). Smartbond[®] samples had higher shear/peel bond strengths than the untreated or acid-etched Transbond[™] XT groups. It appears that Transbond[™] XT was less effective at bonding to resin surfaces unless they were air-abraded. A

possible theory explaining this result may be that Smartbond[®] was more effective at solvent bonding than Transbond[™] XT. With superior solvent bonding, Smartbond[®] could more effectively bond to the smooth surfaces of the untreated and acid-etched samples. However, the use of Transbond[™] XT without air-abrasion may alleviate having to clinically remove excess adhesive and refinishing of the resin restorations after debonding. With the use of Smartbond,[®] more cleanup and restoration refinishing may be expected.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

	Page Number
6.1 Conclusions	90-91
6.2 Recommendations	92-93

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

From the present study, the following conclusions can be made:

- Orthodontic attachments can be successfully bonded *in vitro* to Z100™ composite resin surfaces that are either untreated, acid-etched, or air-abraded using either Transbond™ XT or Smartbond® adhesive, and mean shear/peel bond strengths ≥ 6 MPa can be achieved after 24 hours or after thermocycling.
- Statistically significant differences ($p < 0.05$) exist between the shear/peel bond strengths of the different surface treatment/adhesive combinations after 24 hours and after thermocycling, with the air-abraded/Transbond™ XT group providing the highest bond strengths.
- Thermocycling resulted in significantly ($p < 0.01$) lower shear/peel bond strengths in the air-abraded/Smartbond® group and both of the untreated surface groups.

- The use of Transbond™ XT on air-abraded surfaces resulted in restorative resin fracture 100% of the time, whereas none of the other samples experienced this type of fracture.

Therefore, the null hypothesis, which stated that there are no statistically significant differences in the shear/peel bond strength between the six adhesive/surface treatment groups and that thermocycling has no effect on the shear/peel bond strengths is rejected.

6.2 RECOMMENDATIONS

From the results of this study and an investigation of the literature, the following recommendations are made:

1. The protocol for bonding orthodontic attachments to composite resin surfaces:
 - Acid conditioning should be utilized, as it appears to have a positive affect on the resin surface, which limits adhesive degradation.
 - The use Transbond™ XT in combination with an air-abrasion procedure is not recommended, as substrate resin fracture may occur upon debonding.
 - Both Transbond™ XT or Smartbond® adhesives are suitable bonding agents as they may provide adequate shear/peel bond strengths of at least 5.9 MPa to 7.9 MPa, as suggested by Reynolds (1975).
 - The use of Transbond™ XT may limit adhesive cleanup and resin refinishing upon debonding.
2. The bond strength of orthodontic attachments to composite resin after long-term storage (>6 months) needs further investigation, as no studies are known to exist which address this issue at present.

3. The appropriate finishing, polishing, and repair techniques for composite resin surfaces damaged during the debonding or clean-up procedures requires further investigation.
4. *In vivo* clinical trials need to be undertaken to validate the reliability of *in vitro* data.

APPENDIX #1

Calculation of Bonding Base Area of Lingual Buttons

	DiameterMeasurement	Measurement #2	Measurement #3
Button # 1	3.42mm	3.42	3.42
Button #2	3.42	3.43	3.42
Button #3	3.43	3.43	3.42
Button #4	3.43	3.42	3.42
Button #5	3.43	3.42	3.43
Button #6	3.42	3.42	3.41
Button #7	3.43	3.42	3.43
Button #8	3.42	3.42	3.42
Button #9	3.44	3.43	3.43
Button #10	3.42	3.42	3.41
Button #11	3.42	3.42	3.43
Button #12	3.42	3.41	3.42
Button #13	3.42	3.42	3.42
Button #14	3.42	3.42	3.41
Button #15	3.42	3.43	3.42
Button #16	3.42	3.42	3.43
Button #17	3.42	3.42	3.42
Button #18	3.41	3.42	3.42
Button #19	3.42	3.42	3.41
Button #20	3.41	3.42	3.42

Sum 205.28mm
 /60 measurements
 Average Diameter 3.4213mm
 Area= $\pi(\text{radius})^2$
Average Area of Buttons= 9.18mm

APPENDIX #2

Shear/Peel Bond Strengths and ARI Scores of Untreated Samples Bonded with Transbond

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	6.13	8.68	0	0
2	9.64	8.22	0	0
3	10.12	14.65	0	0
4	10.09	6.31	0	0
5	6.89	5.15	0	0
6	7.43	5.27	0	0
7	16.67	4.95	0	0
8	10.24	8.76	0	0
9	7.49	6.24	0	0
10	11.28	6.87	0	0
11	9.54	7.47	0	0
12	8.88	4.7	0	0
13	9.91	9.34	0	0
14	7.05	7.1	0	0
15	7.37	7.31	0	0
16	11.96	6.68	0	0
17	9.73	6.01	0	0
18	12.91	8.54	0	0
19	11.22	9.25	0	0
20	9.25	7.1	0	0

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	9.69	7.43	0	0
Standard Error	0.546510461	0.49614461	0	0
Standard Deviation	2.444069083	2.218826148	0	0
Sample Variance	5.973473684	4.923189474	0	0
Range	10.54	9.95	0	0
Minimum	6.13	4.7	0	0
Maximum	16.67	14.65	0	0

**Shear/Peel Bond Strengths and ARI Scores of Untreated
Samples Bonded with Smartbond**

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	15.02	6.26	2	1
2	10.24	7.37	2	1
3	13.27	8.15	2	1
4	10.63	4.75	2	1
5	12.97	6.29	2	1
6	11.56	6.87	2	1
7	6.28	4.94	2	1
8	9.16	7	2	1
9	14.78	5.2	1	1
10	11.75	6.38	2	1
11	14.76	7.28	3	1
12	11.75	7.47	2	1
13	13.72	8.76	2	1
14	12.79	7.31	2	1
15	12.63	7.1	1	1
16	10.52	9.54	2	1
17	7.51	7.38	2	1
18	14.4	8.42	2	1
19	10.41	6.01	2	1
20	13.38	5.16	2	1

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	11.8765	6.882	1.95	1
Standard Error	0.536348902	0.288649852	0.088108784	0
Standard Deviation	2.398625209	1.29088138	0.394034463	0
Sample Variance	5.753402895	1.666374737	0.155263158	0
Range	8.74	4.79	2	0
Minimum	6.28	4.75	1	1
Maximum	15.02	9.54	3	1

**Shear/Peel Bond Strengths and ARI Scores of Air-Abraded
Samples Bonded With Transbond**

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	23.35	23.85	4	4
2	22.23	22.48	4	4
3	21.15	21.01	4	4
4	22.51	20.36	4	4
5	17.32	25.05	4	4
6	22.79	24.1	4	4
7	20.34	19.86	4	4
8	22.26	22.51	4	4
9	25.37	18.75	4	4
10	23.05	22.73	4	4
11	18.34	24.3	4	4
12	20.9	21.15	4	4
13	21.01	22.53	4	4
14	22.48	22.79	4	4
15	22.79	19.43	4	4
16	17.78	23.95	4	4
17	24.69	20.78	4	4
18	20.1	15.62	4	4
19	24.87	22.26	4	4
20	26.17	23.46	4	4

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	21.975	21.8485	4	4
Standard Error	0.541380059	0.508423919	0	0
Standard Deviation	2.421125228	2.273740889	0	0
Sample Variance	5.861847368	5.169897632	0	0
Range	8.85	9.43	0	0
Minimum	17.32	15.62	4	4
Maximum	26.17	25.05	4	4

**Shear Bond Strengths and ARI Scores of Air Abraded
Samples Bonded With Smartbond**

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	15.46	15.98	3	3
2	11.04	12.69	2	3
3	15.5	7.97	3	2
4	15.8	9.51	3	2
5	13.41	12.16	3	3
6	10.32	4.73	3	2
7	13.7	6.29	3	3
8	17.27	10.27	3	3
9	10.49	8.98	3	3
10	15.78	14.39	3	3
11	16.2	6.61	3	2
12	14.13	11.67	3	3
13	12.79	12.81	3	3
14	9.89	15.45	3	3
15	13.09	9.89	3	3
16	15.32	14.96	3	3
17	13.4	13.22	3	3
18	14.18	12.94	3	3
19	13.72	11.28	3	3
20	13.1	14.53	3	3

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	13.7295	11.3165	2.95	2.8
Standard Error	0.464945087	0.719917128	0.05	0.091766294
Standard Deviatio	2.079297642	3.219567272	0.223607	0.410391341
Sample Variance	4.323478684	10.36561342	0.05	0.168421053
Range	7.38	11.25	1	1
Minimum	9.89	4.73	2	2
Maximum	17.27	15.98	3	3

**Shear/Peel Bond Strengths and ARI Scores of Acid-Etched
Samples Bonded With Transbond**

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	9.95	5.46	0	0
2	19.75	3.82	0	0
3	7.32	4.42	0	0
4	5.37	6.78	0	0
5	10.97	6.87	0	0
6	6.64	2.95	0	0
7	8.21	5.51	0	0
8	11.28	12.79	0	0
9	7.31	6.75	0	0
10	6.09	6.99	0	0
11	9.16	15.2	0	0
12	5.08	6.03	0	0
13	8.22	7.37	0	0
14	5.75	10.41	0	0
15	8.45	2.78	0	0
16	8.78	7.49	0	0
17	6.01	9.54	0	0
18	4.54	9.06	0	0
19	7.22	6.08	0	0
20	6.43	5.71	0	0

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	8.1265	7.1005	0	0
Standard Error	0.742187831	0.692845192	0	0
Standard Deviation	3.319164884	3.098497896	0	0
Sample Variance	11.01685553	9.600689211	0	0
Range	15.21	12.42	0	0
Minimum	4.54	2.78	0	0
Maximum	19.75	15.2	0	0

**Shear Bond Strengths and ARI Scores of Acid Etched
Samples Bonded With Smartbond**

Sample #	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
1	13.7	11.28	2	2
2	13.09	11.38	2	1
3	15.83	15.85	2	2
4	14.44	11.39	2	2
5	13.34	13.91	2	1
6	13.88	12.63	2	2
7	13.06	14.25	1	2
8	14.6	13.16	2	1
9	11.56	12.05	2	2
10	12.97	11.22	2	1
11	13.27	11.96	1	1
12	11.49	10.76	2	1
13	13.99	9.17	2	1
14	15.28	12.53	2	1
15	15.61	14.8	2	1
16	11.86	11.67	2	2
17	11.49	12.67	1	2
18	13.72	13.48	2	2
19	14.76	12.91	2	1
20	8.78	11.03	1	2

Descriptive Statistics

	Shear Bond Strength (MPa)		ARI	
	24 Hrs	Thermocycled	24 Hrs	Thermocycled
Mean	13.336	12.405	1.8	1.5
Standard Error	0.377660666	0.348179854	0.091766	0.114707867
Standard Deviation	1.688949845	1.557107643	0.410391	0.512989176
Sample Variance	2.852551579	2.424584211	0.168421	0.263157895
Range	7.05	6.68	1	1
Minimum	8.78	9.17	1	1
Maximum	15.83	15.85	2	2

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