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Bond Strength of the Ceramic Orthodontic Bracket-Adhesive Interface

Moon Woo Limb
Loyola University Chicago

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**BOND STRENGTH OF THE CERAMIC
ORTHODONTIC BRACKET-ADHESIVE INTERFACE**

by

Moon Woo Limb, D.D.S.

**A Thesis Submitted to the Faculty of the Graduate School
of Loyola University of Chicago in Partial Fulfillment
of the Requirements for the Degree of
Master of Science**

June

1988

DEDICATED TO

My Parents

For their unconditional love and sacrifice
which made an impossible dream
a reality

My love, Mie

For filling my life with love and happiness

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VITA

Moon Woo Limb, the son of Kap Joon Limb and Suk Youn Ra, was born February 23, 1960, in Taegu, Korea.

His elementary education was obtained in the public schools of Seoul, Korea. His secondary education was completed in 1978 at Serramonte High School, Daly City, California.

In March, 1979, Mr. Limb entered Yonsei University College of Liberal Arts and Science in Seoul, Korea, for pre-dental education, completing it in February, 1981.

In March, 1981, Mr. Limb entered Yonsei University College of Dentistry in Seoul, Korea, receiving the degree, Doctor of Dental Surgery, in February, 1985.

Upon completion of his dental education, he worked as a dentist in a public hospital and a private clinic.

Dr. Limb began graduate studies in the Department of Oral Biology and postgraduate studies in the Department of Orthodontics at Loyola University, School of Dentistry in Maywood, Illinois, in July, 1986. In May, 1988, he received the Certificate of Specialty in Orthodontics.

TABLE OF CONTENTS

| | Page |
|---|------|
| ACKNOWLEDGMENTS..... | ii |
| VITA..... | iv |
| LIST OF TABLES..... | vi |
| LIST OF ILLUSTRATIONS..... | vii |
| CONTENTS FOR APPENDIX..... | x |
| Chapter | |
| I. INTRODUCTION..... | 1 |
| II. REVIEW OF THE LITERATURE..... | 3 |
| Historical Background..... | 3 |
| Bond Strength of Metal Brackets..... | 7 |
| Bond Strength of Recycled Metal Brackets..... | 19 |
| Bond Strength of Plastic Brackets..... | 21 |
| Bond Strength of Ceramic Brackets..... | 25 |
| III. METHODS AND MATERIALS..... | 28 |
| IV. RESULTS..... | 40 |
| V. DISCUSSION..... | 49 |
| SUMMARY AND CONCLUSION..... | 72 |
| REFERENCES..... | 75 |
| APPENDIX..... | 81 |

LIST OF TABLES

| Table | Page |
|---|------|
| 1. Product, Catalog Number, and Manufacturer of the Brackets Tested..... | 29 |
| 2. Product, Batch Number, and Manufacturer of Direct Bonding Adhesives Used..... | 30 |
| 3. Product, Type of Retention, and Nominal Area of the Bracket Bases Tested..... | 41 |
| 4. Mean Tensile Bond Force and Bond Strength (psi) for each Bracket/Adhesive Combinations Tested..... | 42 |
| 5. Mean Tensile Bond Force and Bond Strength (kg./cm. ²) for each Bracket/Adhesive Combinations Tested..... | 43 |
| 6. Data for Tensile Bond Force for Each Bracket/Adhesive Combinations Tested..... | 44 |
| 7. Student T-Test for Tensile Bond Strength..... | 82 |
| 8. Load at Cohesive Failure of Ceramic Brackets..... | 84 |

LIST OF ILLUSTRATIONS

| Figure | Page |
|--|------|
| 1. Plastic cylinder with special mounting jig in place..... | 32 |
| 2. Special loading jig and the hook, which would be mounted on the upper part of Instron machine..... | 32 |
| 3. Special assembly built on a ceramic bracket that had fragile wings..... | 36 |
| 4. The apparatus for testing tensile bond strength of the bracket-adhesive interface..... | 38 |
| 5. Mean tensile force of bracket/adhesive combinations tested..... | 45 |
| 6. Mean tensile bond strength of bracket/adhesive combinations tested..... | 46 |
| 7. SEM photograph of STARFIRE with its smooth surfaces..... | 51 |
| 8. SEM photograph of a base of STARFIRE with its four retention grooves..... | 51 |
| 9. Further enlarged photograph of the base of STARFIRE showing indications of what seems to be a coupling agent..... | 52 |
| 10. SEM photograph of a base of STARFIRE debonded from ACHIEVE demonstrating mostly cohesive failure of the adhesive..... | 52 |
| 11. SEM photograph of STARFIRE exhibiting cohesive failure of the bracket. The whole neck portion of the bracket has been fractured..... | 55 |
| 12. SEM photograph of internal structure of ALLURE III revealing polycrystalline formation..... | 55 |

13. SEM photograph of inner structure of TRANSCEND showing polycrystalline structure..... 56

14. SEM photograph of inner structure of INTRIGUE demonstrating polycrystalline formation..... 56

15. SEM photograph of ALLURE III showing smooth facial surface..... 57

16. Further enlarged photograph of a corner where the slot was ground into the facial surface of ALLURE III. The contrast between the coarse surface of the slot and the smooth facial surface was evident..... 57

17. SEM photograph of a base of ALLURE III with its six square-shaped indentations..... 59

18. SEM photograph of debonded surface of ALLURE III with indentations filled with ACCUBOND and a few air bubbles..... 59

19. SEM photograph of TRANSCEND..... 61

20. Further enlarged photograph of the slot of TRANSCEND showing the roughness of the slot and much smoother lateral surface of the bracket..... 61

21. SEM photograph of a base of TRANSCEND. The base was much smoother than the rest of the surfaces.. 64

22. SEM photograph of the base of TRANSCEND debonded from CONCISE. The smoothness of the new base had disappeared..... 64

23. SEM photograph of the base of TRANSCEND debonded from DYNA-BOND PLUS. The part of the base that had adhesive failure was still smooth like the unused base..... 65

24. Further enlarged photograph of the base of TRANSCEND debonded from DYNA-BOND PLUS. The slight gap between the cement and the base indicated an incomplete adhesive failure..... 65

25. SEM photograph of INTRIGUE..... 68

| | | |
|-----|--|----|
| 26. | Further enlarged photograph of INTRIGUE showing countless micro porosities..... | 68 |
| 27. | SEM photograph of a base of INTRIGUE revealing polycrystalline structure under high magnification..... | 69 |
| 28. | SEM photograph of the base of INTRIGUE debonded from CONTROL displaying both adhesive and cohesive failures..... | 69 |
| 29. | Further enlarged photograph of the base of INTRIGUE at the junction between adhesive and cohesive failures..... | 70 |
| 30. | SEM photograph of a base of the metal bracket debonded from CONCISE..... | 70 |

CONTENTS FOR APPENDIX

| Table | Page |
|--|------|
| 7. Student T-Test for Tensile Bond Strength..... | 82 |
| 8. Load at Cohesive Failure of Ceramic Brackets..... | 84 |

CHAPTER I

INTRODUCTION

Esthetics is one of the major objectives of orthodontic treatment. Through orthodontic correction, malaligned teeth assume a more normal relationship to each other and to the rest of the craniofacial structures. However, during the treatment, patients have to live with the undesirable look of the metal orthodontic appliances.

This condition has improved greatly with the advancement of dental adhesives. Small metal brackets are able to be attached directly to the teeth rather than to the bands first and then cemented to the teeth as units. Early studies (Mizrahi, 1972; Dijkman & Retief, 1972; Lee et al., 1974; Reynolds & von Fraunhofer, 1976, 1977; Low & Fraunhofer, 1976; Johnson et al., 1976; Moin & Dogon, 1977) have shown that this bracket-bond system is strong enough to withstand orthodontic forces.

Different kinds of clear plastic brackets have also been developed. Although they are much more aesthetic than the metal brackets, staining, discoloration, and distortion under load were big disadvantages (Miura, 1972; Cohl et al., 1972; Dobrin et al., 1975; Reynolds & von Fraunhofer, 1977;

Moser et al., 1979; Buzzitta et al., 1982; Pulido & Powers, 1987)

During the last few years, manufacturers have marketed a series of tooth colored and clear ceramic brackets that are a significant aesthetic improvement. In addition to their superior aesthetic qualities, some brands claim to produce bond strength that is comparable to or greater than the metal bracket-adhesive system. However, being fairly new in the market, insufficient data documenting bond strength exists. Nevertheless, the demands for these brackets by clinicians are already exceeding supplies.

The purpose of this study is to investigate the tensile adhesive bond strength of these new ceramic brackets using bonding adhesives recommended and distributed by the respective manufacturers and one bis-GMA adhesive. To be used as a control, one type of mesh-backed metal bracket will also be tested. Clinically, this study should aid orthodontists in selection of ceramic brackets, and academically, provide a base for future studies.

CHAPTER II

REVIEW OF THE LITERATURE

HISTORICAL BACKGROUND OF DIRECT BONDING

Direct bonding of orthodontic attachments to tooth surface received serious consideration after Buonocore's (1955) demonstration of increased adhesion on tooth surface produced by 85 per cent phosphoric acid pretreatment. Before then, cementing metal bands with attached brackets to teeth with zinc phosphate cements was the only accepted method by which brackets could be attached to teeth. Researchers and orthodontic clinicians have been interested in the development of methods by which brackets can be directly affixed to the teeth. Direct bonding of orthodontic attachments to enamel without etching have been attempted by Sadler (1958) using four dental cements and two general purpose adhesives, but all nine materials were unsuccessful.

A bandless system has several advantages over band system (Newman, 1965; Cohl et al., 1972; Reynolds, 1975); they are as follows:

1. Improvement of aesthetic qualities.

2. Ease of manipulation and decreased patient discomfort.
3. Elimination of the need of separation of adjacent teeth.
4. Improved oral hygiene at the gingival margin.
5. Decreased soft tissue irritation.
6. Reduced risk of decalcification which may occur under bands.
7. Easier detection and treatment of caries.
8. Elimination of post-treatment band spaces.
9. Facilitation of more exact mechanical positioning of brackets.
10. Facilitation of the application of attachments to partially erupted teeth.

Newman (1965) was one of the first to report direct bonding of orthodontic attachments to the tooth surface. In vivo, he used an epoxy resin (diglycidyl ether of bisphenol A with a polyamide curing agent) on rabbit teeth after etching with 40 per cent phosphoric acid for 60 seconds. Although bond strength improved with surface pretreatment with phosphoric acids the cure time of 15 minutes to 30 minutes for the epoxy resin was too long.

In a subsequent study, Newman et al. (1968) were able to shorten the cure time to approximately 5 minutes using modified acrylic resins. Newman (1969, 1971) published further articles describing the use of acrylics as a

satisfactory adhesive. In all his studies, Newman used plastic attachments because of their aesthetic quality as well as their quality to readily bond to adhesives. Metal brackets needed extensive surface preparation for them to bond with the adhesives.

Several other cements were also tested for their feasibility as a direct bonding orthodontic adhesives. Mitchell (1967) was successful in limited clinical trial of black copper cement with gold direct attachments. However, his efforts with an epoxy resin had failed. Smith (1968) introduced zinc poly-acrylate (carboxylate) cement, and with Mizrahi (1969, 1971) tested its bond strength with orthodontic lingual buttons. They found that the bond strength was superior to that of certain existing dental cements.

The usage of a unique bonding system of methacrylate with catalyst TBB (tri-n-butyl borane) was described by Miura, Nakagawa and Masuhara (1971). It was devised for plastic brackets and found to be effective. Diacrylate resins became available in the early 1970's and comprise many of the current adhesives (e.g. CONCISE, 3M). These materials have been widely accepted by dentistry. By careful selection of filler concentrations and particle size, these materials have been used as pit and fissure sealants, anterior restorative materials, occlusal restorative materials for posterior teeth, and bonding

agents. In orthodontics, the unfilled or lightly filled materials are known as sealants (bonding agents or adhesion promoters). Sealants may be applied to the etched enamel surface prior to the use of an adhesive, which enhances adhesion to the enamel surface.

BOND STRENGTH OF METAL BRACKETS

For more than two decades, orthodontic brackets and attachments for direct bonding systems were mainly made of stainless steel or polycarbonate. Of the two, stainless steel brackets are by far the most widely used mainly due to their durability and strength. Since the bonding adhesive does not bond to metals, various types of bases were designed to improve the mechanical retention of the attachments to the adhesives.

Perforated base and curled lip base were among the first types of the bases designed, and their adhesive strength was first tested in vitro by Lee and his colleagues (1974). Depending on the types of adhesives, the 24 hour adhesive strength was in the range of 5 to 16 pounds for the perforated bases and 8 to 23 pounds for the curled lip bases. However, with all adhesive systems used in the test, the deterioration of adhesive strength to metal occurred rapidly as time passed.

Brackets with the retentive lip bases were also tested in vitro for their shear strength by Johnson, Hembree, and Weber (1976) with seven direct-bonding materials. A total of 210 stainless steel brackets with retentive lip bases were bonded to bovine incisors and then

stored in a 30 per cent saline solution until tested. The mean shear strength [sic]¹ values of 0.42 to 30.17 pounds at 1 day, 2.98 to 30.64 pounds at 1 month, and 2.86 to 31.87 pounds at 3 months were reported.

Welding wire gauze to the base of orthodontic bracket was also used to create retention. Gauzes of various mesh sizes have been used for this purpose. Mizrahi (1972) used British Standard 100 mesh gauze while Dijkman and Retief (1972) used 60 mesh gauze. Adhesive bond strength of gauzes with different mesh sizes were compared by Reynolds and von Fraunhofer (1976) with the orthodontic buttons using three filled diacrylate resins. They concluded that when metal attachments are used for direct bonding, the use of coarse mesh gauzes is advised for mechanical retention; possessing a wire diameter not less than 150 μm (with a matching aperture of approximately 250 μm). They found not only that fine gauze wires do not permit adequate hold strength between button and gauze, but that gauze may also distort on loading.

In the following year, Reynolds and von Fraunhofer (1977) presented another study comparing four types of orthodontic attachment, and their recommended adhesives. They have demonstrated that mesh-base metal brackets do, indeed, provide superior bond strength when compared to

¹ Authors quoted "strength" as pounds incorrectly, whereas the term strength is defined as load/area.

perforated metal-base brackets, which had the lowest bond strength. A polymer-coated metal attachment showed slightly greater bond strength than the perforated base system, but the mean strength achieved was some 40 per cent less than that obtained with the gauze-backed brackets, thus disproving the concept that providing a "chemically favorable" retention aid for a metallic attachment is good in practice as well as in theory.

Superior bond strength of mesh-backed brackets was also demonstrated by Moin and Dogon (1977). They used a highly filled diacrylate enamel bond system and found that the bond strength of mesh-backed brackets doubled the value compared to that of metal-perforated brackets. The mean value of bond strength [sic] was between 30 to 35 pounds with perforated backings and between 60 and 70 pounds with meshed backings.

With the improvement of the bracket design, the cohesive strength of bonding materials came to play a more crucial role in determining ultimate bond strength, this prompted the development of the bonding adhesives. Over the years, many studies were done to compare the bond strength of various bonding adhesives. Low and von Fraunhofer (1976) compared the retentive capacity of mesh-base brackets with various composite restorative materials. Using a tensile test technique, they found that all the composites tested provided adequate bond strength. They stated that weakness

in the attachment is not at the tooth-adhesive interface, but at the mesh-adhesive junction. They also found that mesh-base bracket provides superior bond strength when compared to perforated metal-base bracket.

Faust et al. (1978) presented a similar study investigating the tensile bond strength of thirteen direct bonding orthodontic adhesives. Bond strength with metal brackets ranged from 270 to 757 pounds per square inch (psi), with most failures occurring at the adhesive-bracket interface. After cleaning and re-etching of the teeth, brackets were rebonded with each cement; values of rebond strength ranged from 180 to 680 psi. They found that differences in bond strength among cements were more dramatic than differences between bond and rebond strength.

Thanos, Munholland, and Caputo (1979) also investigated the bond strength of mesh-base and perforated metal-base brackets using different adhesive systems. The bond strength was determined by means of tension, shear, and torsion tests. After statistically analyzing the data they drew the conclusion that mesh-base brackets were more retentive than the perforated metal-base brackets in tension, while perforated metal-base brackets were more retentive in shear.

As years passed, the variety of orthodontic brackets and bases increased. A study was carried out by Dickinson and Powers (1980) who evaluated fourteen direct-bonding

orthodontic bases with two bonding adhesives using plastic cylinders and human teeth as substrates. They concluded, contrary to the findings of Reynolds and von Fraunhofer (1976), that bond strength was independent of nominal area and mesh size for the bases tested. Instead, they found that the process of spot-welding of the brackets to the bases decreased the nominal area available for retention and also, this may produce an area of stress concentration which can initiate the fracture of the adhesive at the adhesive-base interface. They believed that inadequate spot-welding may even lead to separation of the bracket from the base.

In the same year, Lopez (1980) investigated the retentive shear strengths of sixteen designs of commercially available stainless steel attachment bases with edgewise bracket. Contrary to findings by Thanos et al. (1979) he concluded that solid bases with perforation around the periphery of the base generally had lower mean shear strengths than the other base designs. He also found that smaller foil-mesh bases could be used without sacrificing significant shear strength.

Maijer and Smith (1981) examined the retention variables that exist between seven commercially available bracket bases. Shear strength data and comparison of the scanning electron microscope observations of bracket bases before testing and bond-fracture surfaces after testing led

to the following conclusions:

- (1) Weld spots reduce the retentive area.
- (2) Weld spurs could be responsible for lower bond strengths in some foil-mesh samples.
- (3) Weld spots on the edges of attachment bases should be avoided to prevent a poor marginal resin-mesh seal.
- (4) Bracket bases should be designed to prevent air entrapment under the base; photo etched steel brackets did not allow air to escape easily thus produced large number of air voids on bonding surfaces.
- (5) The best resin penetration and bond strength were obtained with a fine mesh bracket base of the woven mesh type - lightly filled resin gives superior results with this type of mesh base.

Buzzitta, Hallgren, and Powers (1982) evaluated in vitro the tensile bond strength and failure location of three types of brackets (polycarbonate, stainless steel, and ceramic) using natural teeth and plastic as substrates. He found that for the metal brackets a highly-filled diacrylate cement gave the highest bond strength, between 0.87 kg./mm.² and 1.33 kg./mm.², while unfilled cement gave the lowest bond strength, between 0.56 kg./mm.² and 0.79 kg./mm.². He also noticed that with the stainless steel brackets bond failure occurred at the bracket-cement interface.

A study was presented by Hansen and his associates (1983) to test the theory that a special porous metal powder coating can provide better mechanical keying than mesh by virtue of its greater surface area and intricate network of microscopic void. Identical brackets were laser-welded to an equal number of conventional foil-mesh and powder-coated bases of identical shape and peripheral dimensions. The experimental base material was found to provide significantly greater tensile bond strength at the metal/adhesive interface. The mean bond strengths of the foil-mesh was 0.352 kg./mm.^2 and the powder-coated foil was 0.662 kg./mm.^2 . Both values appeared very low compare to tensile bond strength of other studies, but authors claim that they are due to difference in testing method as well as in cement used.

Bond strength studies of various orthodontic adhesives continued in the 1980's. Alexandre et al. (1981) evaluated shear bond strength of three orthodontic adhesives and found no significant differences 1 day after placement. However, the bond strength was perceived to increase for some products after 27 days. The interface was also studied to determine the mode of failure. In all cases bond failure occurred as mixed adhesive-cohesive phenomena, either enamel adhesive, bracket adhesive, or combination of the two.

Schulz and his associates (1985) investigated bond strengths of three resin systems used to bond orthodontic

wires directly to teeth and compared these values with those found for directly bonded orthodontic brackets. Shear and tensile strengths were measured at 30 minutes and at 48 hours on 120 human teeth with orthodontic wires directly bonded to the teeth and the other 120 teeth with directly bonded mesh-base metal brackets. They found at 30 minutes brackets were significantly stronger than embedded wires, and one adhesive was significantly stronger than others. However, all significant differences between any of the three resin systems using either bonded brackets or wires disappeared at 48 hours.

Oral environment is constantly subjected to temperature fluctuations. The effects of this phenomena on the bond strengths of bonding resins to etched enamel have been evaluated through the process of temperature cycling (Lee, Swartz, & Culp, 1969; Bishara, Khowassah, & Oesterle, 1975). The effect of temperature cycling on the tensile and shear strengths of bonded and rebonded orthodontic attachments was investigated by Jassem, Retief, and Jamison (1981). The samples were subjected to 500 temperature cycles between 5°C. and 55°C.. The result was that the temperature cycling adversely affected on tensile bond and rebond strengths. However, tensile versus shear and bond versus rebond strengths for similarly prepared specimens were not significantly different.

The effects of sealing resins on bond strength in the

direct bonding of orthodontic attachments were also investigated. Results by Reynolds and von Fraunhofer (1976) indicated that these resins did not enhance bond strength. Faust et al. (1978) found that they even reduced the bond strength of bonding resins that involved a one-step procedure. Jassem, Retief, and Jamison (1981) also concluded that the sealing resin had no effect on the tensile and shear bond and rebond strength.

To enhance bonding of adhesives to metal brackets, several commercial surface treatments became available. Their effect on tensile bond strength was examined by Siomka and Powers (1985) using three types of direct-bonding metal bases. The five commercial surface treatments were: etching, silanation, surface activation, etching plus silanation, and etching plus surface activation. Non-treatment was used as a control. The bases were either mesh, photo-etched, and grooved, and were loaded with a no-mix adhesive to plastic substrates. The highest bond strength was that of grooved base with no treatment. Etching improved the bond strength of the grooved bracket by 56 per cent, while silanation improved the bond strength of the mesh bracket by 28 per cent. However, none of the treatments were effective in increasing the bond strength of the photo-etched bracket.

There is a higher failure rate clinically among bonded brackets on posterior teeth than on anterior teeth

(Gorelick, 1977; Zachrisson, 1977). The higher masticatory forces generated in the posterior regions of the mouth, and the differences in enamel micromorphology as shown by different etching pattern for the posterior teeth (Gailil & Wright, 1979; Arakawa, Takahashi, & Sebata, 1979) were considered as possible causes. Knoll, Gwinnett, and Wolf (1986) undertook a study, in vitro, to determine the maximum shear strength of brackets bonded to anterior and posterior teeth. Brackets were bonded to two groups comprising 12 incisors and 12 molar teeth. Results of the study showed higher bond strengths, statistically significant, for the brackets bonded to anterior teeth. It was concluded that differences in etching patterns do not necessarily affect shear bond strength and the predominantly weak link in bonding chain was at the bracket-resin interface. The authors speculated from the basis of this observation that the lower values for molar teeth may relate to adaptation of the bracket and nonuniform resin thickness.

The usage of light cured resin for orthodontic bonding were suggested by Cohl et al. (1972). Since transillumination was essential for curing of the adhesive, clear plastic brackets were used. A clinical study using an ultraviolet-sensitive adhesive system with the perforated metal brackets were reported by Garn (1976). He also used the plastic brackets and found that the majority of bond failures of both types of brackets involved both cohesive

and adhesive bracket interface failures.

The feasibility of a light cured resin as an orthodontic bonding adhesives were further examined, because of its advantage of providing sufficient working time for accurate placement of lingual brackets. Lingual brackets were used with a system of orthodontic treatment, which places brackets on the lingual surface of the tooth - rather than the buccal or labial surface of the tooth - to further enhance the esthetics of the treatment. Andreason et al. (1984) compared the shear strengths of mesh-backed metal brackets with a light cured microfilled composite resin (HELIOSIT) and an autopolymerizing composite resin (CONCISE). A significant difference ($p < 0.01$) was found between the bond strength of CONCISE and HELIOSIT activated for 20 seconds, but no significant difference was found between CONCISE and HELIOSIT activated for 40 seconds.

A similar study was conducted by King et al. (1987). The tensile and shear strengths of direct bonded, mesh backed stainless steel, lingual orthodontic brackets were evaluated by means of chemically cured composite resins and transilluminated light cured composite resins using bovine teeth as substrates. The results of this investigation showed that the bond strengths of the orthodontic brackets bonded with light cured composite resins were significantly less ($p < 0.05$) than the bond strengths of the orthodontic brackets cemented with traditional adhesives and orthodontic

composite resins. Nevertheless, authors believe the bond strengths achieved with the light cured composite resins should be adequate to withstand the forces of mastication and orthodontic movement. The mean tensile bond strength for the three light cured composite resins ranged from 129 kg./cm.² to 141 kg./cm.² while for the other two chemically cured composite resins ranged were 147 kg./cm.² and 158 kg./cm.². The mean shear bond strengths ranged from 49 kg./cm.² to 57 kg./cm.² for the light cured composite resins and 61 kg./cm.² and 66 kg./cm.² for two chemically cured composite resins.

BOND STRENGTH OF RECYCLED METAL BRACKETS

In an effort to minimize waste and cost to the orthodontist and ultimately to the patient, several processes for removal and refinishing of used direct-bond brackets exist on the orthodontic market. Buchman (1980) examined recycled brackets for changes in base torque angle and slot width, and concluded that the amount of changes is of little clinical relevance. A number of studies also, have been undertaken to determine whether there are any changes in the retentive capacity of metal brackets after being commercially recycled.

Mascia and Chen (1982) used 120 human incisor teeth and bonded them with several different brands of direct-bonding brackets and tested for retention prior to and after recycling of the brackets by two different commercially available methods. Measurements of shearing strengths were performed to observe any possible changes in the retentive properties of the brackets. A decrease in retentive strength was noted in all types of recycled brackets. One type of bracket showed a statistically significant change in strength, depending on the process used in recycling, while the other brackets did not show any difference between the two processes.

However, McClea and Wallbridge (1986) found no significant differences in tensile bond strength as well as in shear bond strength between either commercially or domestically recycled bases, and new bases when they compared bond strength of new and recycled orthodontic metal brackets. Mean tensile bond strength [sic] of new orthodontic brackets were 5.95 kg. while brackets recycled commercially and domestically were 5.53 kg. and 5.25 kg., respectively. All tensile failures occurred at the resin-mesh interface.

The effects of four rebonding procedures on tensile bond strength of four filled diacrylate orthodontic adhesives were evaluated by Wright and Powers (1985). The four rebonding procedures that were examined were thermal reconditioning, chemical reconditioning, removal of residual adhesive with a green stone, and grinding the mesh-base with a green stone. The results indicated that the initial bond strengths for the no-mix adhesive and both two-paste system were significantly greater than the tensile bond strengths for any rebonding condition. Different rebonding conditions reduced tensile bond strength to differing degrees. The initial bond strength for the visible, light-cured adhesive was not significantly different from three of the four rebonding conditions and was lower than the initial bond strength of the other three adhesives.

BOND STRENGTH OF PLASTIC BRACKETS

In addition to metal brackets, there have been several studies concerning bond strength of plastic or polycarbonate brackets. These brackets were first introduced in 1963, and because of their aesthetically pleasing white or clear features, they were readily accepted especially for adult patients who needed only simple anterior teeth movement. However, there were certain limitations to the usage of these brackets due to their weakness as a material (Miura, 1972; Cohl et al., 1972; Reynolds & von Fraunhofer, 1977; Moser et al., 1979; Buzzitta et al., 1982; Pulido & Powers, 1987), and their wear and distortions to certain orthodontic mechanics (Dobrin, Kamel, & Musich, 1975). Nonetheless, they were widely used, and studies were conducted to examine their bond strength with various adhesive systems.

The effect of water immersion on shear strength of plastic brackets bonded to the enamel surface of extracted teeth was evaluated by Miura (1972). He used his unique bonding system of methacrylate with catalyst TBB (tri-n-butyl borane). He kept these bracketed teeth in water for six months at 37°C. with a load of 1 kg./cm.², and then subjected them to mechanical stress with a shear testing

instrument. He found that bond strength was decreased about 20 per cent after immersion in the water.

An ultraviolet sensitive adhesive was investigated in bonding of clear plastic orthodontic brackets by Cohl, Green, and Eick (1972). Bond strength was tested in tension and in shear at both 24 hours and 30 days. The mean shear strength were 706 psi (49.6 kg./cm.²) and 821 psi (55.7 kg./cm.²), respectively, at 1 day and 30 days. The mean tensile strength was 508 psi at 30 days as compared to 448 psi at 1 day. The weakest links in the bonding system were found to be the bracket-adhesive interface and the bracket itself.

Reynolds and von Fraunhofer (1977) compared bond strength of one polycarbonate bracket with three metal brackets using adhesives recommended for each of the brackets. The greatest bond strengths, as expressed by the tensile load to failure, were found with the polycarbonate-acrylic resin adhesive system. However, authors noted that these bond strengths exceeded the strengths of the bracket themselves and special techniques were necessary to test these brackets. They further stated that due to this low strength of the polycarbonate bracket, usefulness and general applicability are limited for these attachments.

The bond strength in shear of four resin cements intended for bonding polycarbonate brackets to the tooth surface were evaluated by Moser, Marshall, and Green (1979).

One hundred four polycarbonate brackets were bonded to extracted premolars. The adhesion of a minimum of ten bracket/enamel interfaces per material was tested after both 7 and 30 days storage in artificial saliva at 37°C. In addition to results of certain resin systems being better than others, the study showed that 17 per cent of the shear test failures were attributed to defective brackets. Furthermore, scanning electron microscope analysis of the fractured bond sites revealed that most bonds which appeared to be of an adhesive failure when viewed under low magnification actually turned out to have a cohesive failure when viewed under higher magnification.

Buzzitta, Hallgren, and Powers (1982) examined the tensile bond strength of two types of plastic brackets. They reported that the mean tensile bond strength were 0.83 kg./mm.² and 1.10 kg./mm.² for an unfilled cement, 0.58 kg./mm.² and 0.52 kg./mm.² for a low-filled cement, and 0.80 kg./mm.² and 1.08 kg./mm.² for a highly filled cement. Of the two values, the later were of plastic bracket with reinforced metal. The plastic brackets failed more often at the base-cement interface but also within the bracket.

The effectiveness of commercial primers for bonding diacrylate cements to plastic brackets with respect to tensile bond strength and failure location were evaluated by Pulido and Powers (1983). Bond strength of three diacrylate cement to three plastic brackets ranged from 0.03 to 0.34

kg./mm.² without bracket primer and from 0.51 to 0.85 kg./mm.² with bracket primer. Most failures (83 per cent) occurred within the bracket when primers were used. For the seven cements tested, bond strengths were highly dependent on the bracket.

BOND STRENGTH OF CERAMIC BRACKETS

Only a few studies have been published examining the bond strength of ceramic bracket system. Buzzitta, Hallgren, and Powers (1982) first reported tensile bond strengths of ceramic brackets (ZULAUF) with three types of diacrylate adhesives. The mean tensile bond strength was 1.26 kg./mm.² for an unfilled cement, 0.47 kg./mm.² for a low-filled cement, and 0.52 kg./mm.² for a highly filled cement. Bond failures with the ceramic brackets occurred most frequently at the bracket-cement interface except with unfilled cement for which within-cement failures also occurred. The use of a silane primer with the ceramic bracket increased within-cement failure and, with unfilled cement, resulted in several within-bracket failures.

Iwamoto, Kawamoto, and Kinoshita (1987) tested new ceramic brackets for tensile and shear bond strength and compared with metal brackets and ZULAUF ceramic brackets using three types of direct bonding cements (unfilled, low filled, and highly filled diacrylate cements). Variable amount of mechanical retention were built into the bases of the new ceramic brackets. One set of new ceramic brackets was silane treated. Following conclusions were drawn from the study:

- 1) Tensile and shear bond strength decreased as the mechanical retention increased.
- 2) Silane coating did enhance the bond strength.
- 3) A highly filled diacrylate cement gave the highest values of tensile bond strength for both the metal and the new ceramic brackets. An unfilled acrylic cement gave the highest values of bond strength for the ZULAUF ceramic brackets.
- 4) Shear bond strength was always greater than tensile bond strength for each bracket-cement combination.

A similar study was conducted by Gwinnett (1988), who compared the shear bond strengths of metal, ceramic, and ceramic-filled plastic brackets bonded to human incisor teeth with a heavily filled composite resin. The mean shear bond strengths of two types of ceramic brackets were 18.3 MPa¹ and 18.8 MPa, while the ceramic-filled plastic brackets were 15.7 MPa. There were no statistically significant differences ($p < 0.05$) among the mean values for groups of different types of brackets. However, if the data for metal brackets (the mean shear bond strength of 12.1 MPa and 12.9 MPa), were compared with the data of the ceramic brackets excluding the plastic type, then the bond strength of the ceramic brackets was approximately 50 per cent greater and the values were statistically significant ($p < 0.001$).

¹ MPa (megapascal): pascal = N/m.² (1 Mpa = 145 psi)

The site of failure was generally at the resin-bracket interface except for the ceramic-filled plastic brackets, which frequently showed failure of the bracket itself. The author concluded that ceramic brackets should offer a viable alternative to their metal counterparts because they combine esthetics with a bond strength that is comparable to and as reliable as their metal counterparts.

Swartz (1988) also investigated the shear bond strength of several ceramic brackets and a foil-mesh metal bracket. The mean load for the ceramic brackets ranged from 1.7 to 3.0 kilograms while that for the metal bracket was 2.9 kilograms. In order to simulate incidence of sudden loading (i.e. biting or trauma) the author subjected the samples to the load at a rate of 1000 mm./min., and he found that two ceramic brackets demonstrated total failure within the enamel in 5 to 6 out of 10 samples tested for each bracket. The author attributed such failures to the low fracture toughness of enamel (Rasmussen et al., 1976) and rigidity of the ceramic brackets which tend to distribute debonding forces over the entire interfaces.

CHAPTER III

METHODS AND MATERIALS

Four types of commercially manufactured ceramic brackets were tested for tensile adhesive bond strength with respective proprietary bonding adhesives and one bis-GMA adhesive. To compare with the ceramic brackets, one type of mesh-backed metal bracket was also tested. The codes, products, catalog numbers, and manufacturers are listed in Table 1. The codes, batch numbers and manufacturers of the bonding adhesives are listed in Table 2.

As a means of comparison, the nominal area of the base of each bracket was measured by planimetry¹ and enlarged photograph of the bracket base (B) obtained by a scanning electron microscope (SEM). The actual area (A) of bonding base of the bracket was then calculated by equation 1.

$$A = B / \text{square of magnification of SEM} \quad (1)$$

Plastic cylinders, which were used as substrates, were

¹ ALVIN Planimeter, Catalog No. PL655, Elk Grove Blue Print, Elk Grove Village, Illinois.

Table 1

PRODUCT, CATALOG NUMBER, AND MANUFACTURER
OF BRACKETS TESTED

CERAMIC BRACKETS

| <u>Product</u> | <u>Catalog Number</u> | <u>Manufacturer</u> |
|----------------|-----------------------|---|
| STARFIRE | 081-800 | "A" company, Inc. 11436 Sorrento Valley Rd. San Diego, CA 92138 |
| ALLURE | 01-511-02 | GAC international, inc. 185 Oval Dr. Central Islip, NY 11722 |
| TRANSCEND | 2001-602 | Unitek Corporation 2724 South Peck Rd. Monrovia, CA 91016 |
| INTRIGUE | 243-101 | Lancer Orthodontics, Inc. P.O. Box 819 Carlsbad, CA 92008 |

METAL BRACKETS

| <u>Product</u> | <u>Catalog Number</u> | <u>Manufacturer</u> |
|-------------------------------|-----------------------|--|
| Standard edgewise brackets | 002-008 | American Orthodontics Sheboygan, WI 53081 |

Table 2

PRODUCT, BATCH NUMBER, AND MANUFACTURER
OF DIRECT BONDING ADHESIVES USED

| <u>Product</u> | <u>Batch Number</u> | | <u>Manufacturer</u> |
|----------------|---------------------|--------|---|
| ACHIEVE | Universal paste | 7J305 | "A" company, Inc. 11436 Sorrento Valley Rd. San Diego, CA 92138 |
| | Catalyst paste | 7J309 | |
| ACCUBOND | Base past | 062587 | GAC international, inc. 185 Oval Dr. Central Islip, NY 11722 |
| | Catalyst resin | 011086 | |
| DYNA-BOND PLUS | Base adhesive | 051887 | Unitek Corporation 2724 South Peck Rd. Monrovia, CA 91016 |
| | Catalyst adhesive | 051887 | |
| CONTROL | Paste | 012588 | Lancer Orthodontics, Inc. P.O. Box 819 Carlsbad, CA 92008 |
| | Primer | 122187 | |
| CONCISE | Paste A | 7AC2 | Dental Products / 3M 270-5N-02 3M Center St. Paul, MN 55144 |
| | Paste B | 7AC3 | |
| | Paste A | 7AC2 | |
| | Paste B | 7AC3 | |

constructed from 1 inch width acrylic rod¹. Using a hand saw, the acrylic rods were first cut into cylinders, approximately 1 inch long. Both ends of these acrylic cylinders were machined to smooth surfaces such that the surfaces were perpendicular to the long axis of the cylinder. During this procedure, a hole was drilled into one surface of the cylinder to a width of 0.28 inch (7.0 mm.) and a depth of 0.16 inch (4.0 mm.). To provide retention for the bonding adhesives, undercuts were formed inside of the hole using an inverted cone laboratory carbide bur².

A special mounting jig was constructed according to the description of Dickinson et al. (1980) to assure that the bonding bases mounted on the plastic cylinders would be perpendicular to the loading forces during testing (Fig. 1). The jig was made from two pieces of 1 inch, .021 inch x .025 inch rectangular wire and one piece of 1 inch, .018 inch x .025 inch wire. The .021 inch side of two pieces of .021 inch x .025 inch wire were welded to one .018 inch side of .018 inch x .025 inch wire, about 0.4 inch apart. To ensure that the wires stay together and to minimize the distortion of the jig, the welded spots were then soldered. The length of .018 inch x .025 inch wire was reduced to where it was

¹ Catalog No. 8531K23, McMaster-Carr, Chicago, Illinois.

² Catalog No. 951-5225, Darby Dental Inc., Rockville Centre, New York.

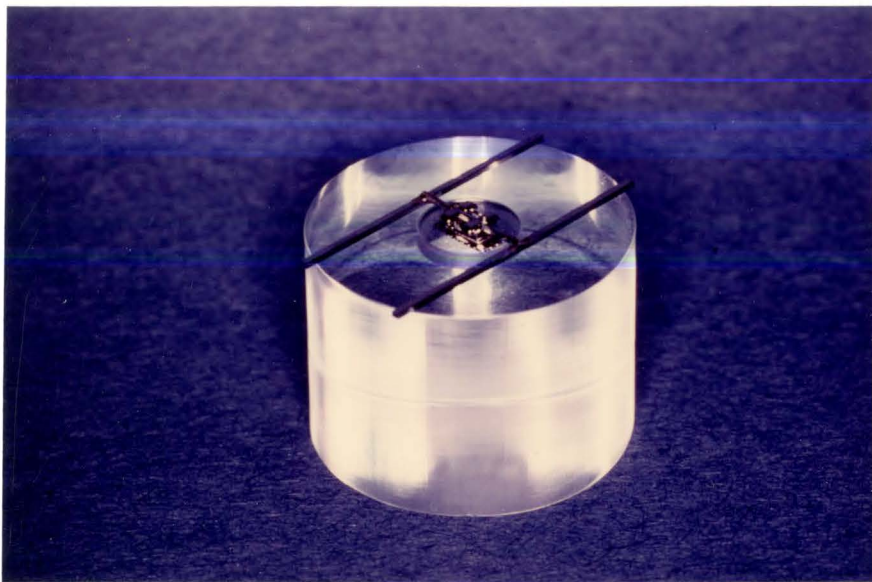


Figure 1. Plastic cylinder with special mounting jig in place.



Figure 2. Special loading jig and the hook, which would be mounted on the upper part of Instron machine.

soldered to the other two wires.

Special loading jig was constructed to engage the bracket wings evenly and maximize the tensile load (Fig. 2). Two .018 inch wires, about 4 inches long, were bent into rectangular loop. The width of the loop was determined by the width of the bracket; it was made just wide enough to engage twin wings of each bracket. Care was taken to ensure that the two wires were of identical dimensions and had 90 degrees corners. The two wires were aligned one on top of the other such that, when they were viewed from the top, only one wire could be seen. They were carefully taped in that state on a piece of glass. Next, .032 inch round wire was cut into a length of approximately 4 inches and bent into a round ended loop. This wire is also taped on the glass, opposite to the rectangular loops, such that the long axis of this wire would meet with the long axis of the rectangular loops in a straight line. Self polymerizing acrylic was sprinkled between these two taped wires.

A hook was needed to engage these loading jig to a testing machine. It was made with .050 inch round wire embedded in an acrylic block (Fig. 2). The acrylic block was trimmed so that the long axis of the hook was parallel to two sides of the block, which were to be used to mount the hook on with the upper part of the testing machine.

The mounting jig was used with each metal bracket-adhesive system and with any ceramic bracket-adhesive system

where the adhesive was too viscous to allow direct placement of the brackets. The rest of the ceramic bracket placements were done directly, without the usage of the jig. When the mounting jig was used, a bracket (.018 inch slot) was tied into the .018 inch side of the rectangular wire with .010 inch steel ligature wire. Care was taken not to contaminate the bracket base and to ensure that the jig wire was fully seated in the slot. The jig was adjusted for a bracket with torqued slot such that the bracket base was parallel to the jig and perpendicular to the loading force.

The bonding adhesives were mixed according to manufacturer's instructions and loaded into the prepared areas in the plastic cylinders with adhesive spatulas. The quantity of adhesive loaded was carefully controlled to make sure it did not overflow onto the bracket. Bonding adhesive was then applied to the bonding base, with special attention to manipulating the adhesive on all surface of the base and into all retention areas of the base, if present. The bonding bracket, tied to the jig, was then pressed into the bonding adhesive in the plastic cylinder. The sample was then immediately examined under a light optical stereo microscope¹ for overlap of bonding adhesive on the bonding base. If any excess was found, it was removed with sharp explorer.

¹ StereoZoom 7, Bausch & Lomb, Rochester, New York.

If the adhesives were firm enough to allow direct placement of the ceramic brackets, then the mounting jig was not used. The bonding adhesive was loaded and slightly overfilled into the prepared areas of the cylinder. A side of a clean adhesive spatula was scraped across the surface of the cylinder making the adhesive flat and even with the surface. Bonding adhesive was also applied to the bonding base of the bracket. Special care was taken to remove any excess adhesive from the base. Brackets were then aligned and dropped onto the adhesive surface of the cylinder. The sample was cautiously examined to make sure that the flat surface of the bracket was parallel to the flat surface of the cylinder. If the bracket started to sink into the adhesive, this procedure was done with the whole cylinder turned upside down. The light optical stereo microscope was also utilized to check for any overlapping of bonding adhesives.

For those ceramic brackets in which wings broke off before separation of bonding bases from the adhesives, special assemblies were built on the brackets after completion of bonding of the brackets to the plastic cylinders (Fig. 3). Strips of cellophane tape were placed around four margins of the bracket base, covering all the remaining adhesive and plastic surface of the cylinder, and exposing only the bracket. A thin coat of a silane coupling

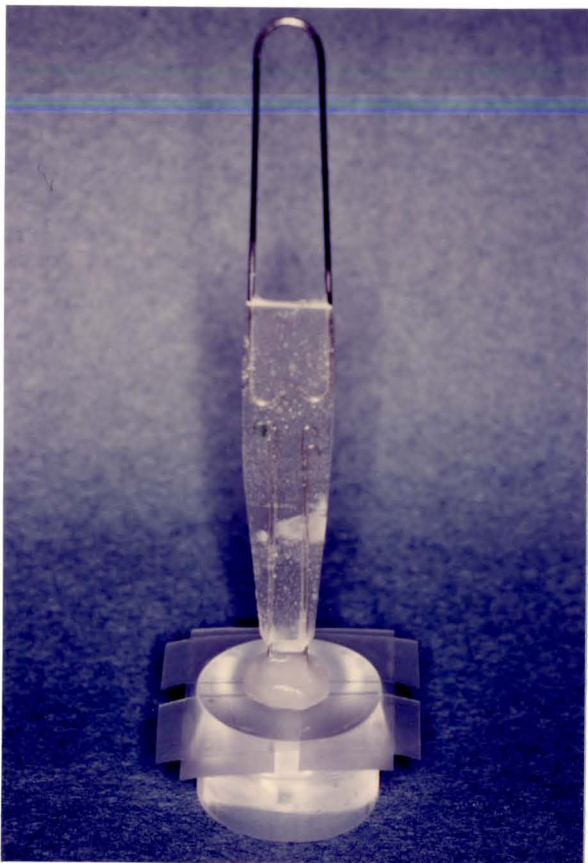


Figure 3. Special assembly built on a ceramic bracket that had fragile wings.

agent¹ was applied on all exposed surfaces of the bracket. The wires of a loading jig were engaged beneath the wings of the bracket. The same bonding adhesive, used to bond the bracket to the cylinders, was mixed and applied all around the bracket and the engaging wires of the loading jig. Special attention was given to insure that the adhesive contacted all the surface of the bracket including the slot and underneath the wings.

All cylinders with the bonded brackets, including the ones with a loading jig attached to them, were stored for 24 hours in 100 per cent humidity in a high humidity chamber at 37.0 C before testing.

After 24 hours, a loading jig was placed on samples that did not had the special assemblies. No silane or adhesive was applied to these samples. Each loading jig was engaged to the hook which was mounted on the upper part of the testing machine². Care was taken to allow centering of the loading jig within the hook in order to minimize shear forces during loading in tension (Fig. 4). The samples were loaded by the testing machine at a crosshead rate of 0.1 inch per minute. The force (L) required to break the bond was recorded and the bond strength (BS) was calculated in units of lbs./in.² by equation 2.

¹ Scotchprime Ceramic Primer, No. 2721, Dental Product Division / 3M, St. Paul, Minnesota.

² Instron Corporation, Canton, Massachusetts.

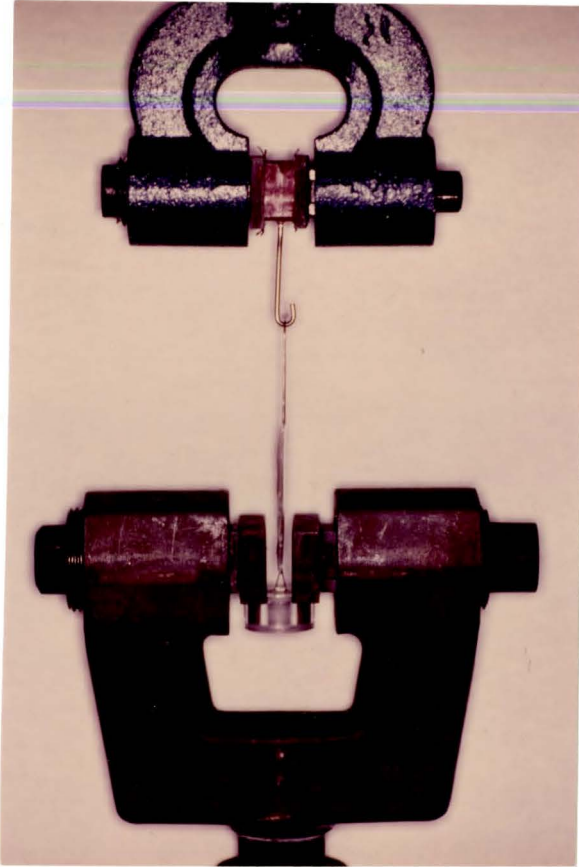


Figure 4. The apparatus for testing tensile bond strength of the bracket-adhesive interface.

$$BS = L / \text{area of bonding base} \quad (2)$$

A minimum of five replications were tested for each ceramic bracket/adhesive system, and ten replications were tested for each metal bracket/adhesive system. After failure of the bond, the fractured surfaces were examined with the light optical stereo microscope and with scanning electron microscope.

Mean values and standard deviations of properties were calculated. The data were analyzed statistically by Student t-test at $p < 0.01$.

CHAPTER IV

RESULTS

The products, types of retention, and nominal areas of the bases tested are listed in Table 3. Mean values and standard deviations of tensile bond force and strength for each of the bracket-adhesive combinations are listed in Table 4 (same data as Table 5 - kg./cm.²). Data used to calculate the mean values of tensile bond force and strength are listed in Table 6. The bracket/adhesive combinations are ranked in Figure 5 for tensile bond force and in Figure 6 for tensile bond strength.

No significant difference was found in bond strength of the metal brackets (METAL) between the different adhesives.

STARFIRE with its proprietary adhesive, ACHIEVE, had the highest bond strength, which was significantly greater than that of METAL with ACHIEVE. STARFIRE with CONCISE had the second highest bond strength that was also significantly greater than that of METAL with CONCISE.

ALLURE III with CONCISE had the third highest bond strength, and it was significantly greater than that of METAL with CONCISE. ALLURE III with ACCUBOND had no

Table 3

PRODUCT, TYPE OF RETENTION, AND NOMINAL AREA
OF THE BRACKET BASES TESTED

| <u>Product</u> | <u>Type of Retention</u> | <u>Nominal Area</u> | |
|----------------|--------------------------|------------------------|------------------------|
| | | <u>in.²</u> | <u>cm.²</u> |
| STARFIRE | Chemical / mechanical | 0.017 | 0.108 |
| ALLURE | Chemical / mechanical | 0.015 | 0.098 |
| TRANSCEND | Chemical / | 0.015 | 0.096 |
| INTRIGUE | Chemical / | 0.017 | 0.108 |
| Metal bracket | / Mechanical | 0.020 | 0.129 |

Table 4

MEAN TENSILE BOND FORCE AND BOND STRENGTH (psi)
FOR EACH BRACKET / ADHESIVE COMBINATIONS USED

| Code | Combination Bracket/Adhesive | No of specimens | Force (lbs.) | | Bond strength (psi) | |
|------|---------------------------------|--------------------|--------------|----------|---------------------|-----------|
| | | | Mean | [+ S.D.] | Mean | [+ S.D.] |
| A | STARFIRE/CONCISE | 5 | 51.2 | [15.2] | 3011.8 | [893.8] |
| B | STARFIRE/ACHIEVE | 3 | 71.0 | [6.3] | 4174.5 | [370.4] |
| C | ALLURE III/CONCISE | 5 | 30.6 | [2.2] | 2041.3 | [148.1] |
| D | ALLURE III/ACCUBOND | 5 | 26.0 | [3.2] | 1730.7 | [213.9] |
| E | TRANSCEND/CONCISE | 8 | 22.0 | [5.6] | 1465.0 | [374.3] |
| F | TRANSCEND/DYNA-BOND PLUS | 6 | 14.3 | [4.2] | 955.6 | [280.1] |
| G | INTRIGUE/CONCISE | 7 | 17.6 | [6.9] | 1035.3 | [406.5] |
| H | INTRIGUE/CONTROL | 5 | 21.1 | [12.3] | 1242.4 | [721.5] |
| I | METAL/CONCISE | 10 | 32.3 | [5.1] | 1612.5 | [255.3] |
| J | METAL/ACHIEVE | 10 | 31.2 | [4.5] | 1557.0 | [223.5] |
| K | METAL/ACCUBOND | 10 | 35.2 | [7.2] | 1760.0 | [368.5] |
| L | METAL/DYNA-BOND PLUS | 9 | 32.5 | [5.4] | 1626.6 | [270.4] |
| M | METAL/CONTROL | 10 | 32.5 | [5.4] | 1623.0 | [268.5] |

Table 5

MEAN TENSILE BOND FORCE AND BOND STRENGTH (kg./cm.²)
FOR EACH BRACKET / ADHESIVE COMBINATIONS TESTED

| Code | Combination Bracket/Adhesive | No of specimens | Force (kgs.) | | Bond strength (kgs./cm. ²) | |
|------|---------------------------------|--------------------|--------------|----------|--|----------|
| | | | Mean | [+ S.D.] | Mean | [+ S.D.] |
| A | STARFIRE/CONCISE | 5 | 23.0 | [6.8] | 213.3 | [63.0] |
| B | STARFIRE/ACHIEVE | 3 | 31.9 | [2.8] | 295.7 | [25.9] |
| C | ALLURE III/CONCISE | 5 | 13.8 | [1.0] | 140.6 | [10.2] |
| D | ALLURE III/ACCUBOND | 5 | 11.7 | [1.4] | 119.2 | [14.3] |
| E | TRANSCEND/CONCISE | 8 | 9.9 | [2.5] | 103.0 | [26.0] |
| F | TRANSCEND/DYNA-BOND PLUS | 6 | 6.4 | [1.9] | 67.2 | [19.8] |
| G | INTRIGUE/CONCISE | 7 | 7.9 | [3.1] | 73.3 | [28.7] |
| H | INTRIGUE/CONTROL | 5 | 9.5 | [5.5] | 88.0 | [50.9] |
| I | METAL/CONCISE | 10 | 14.5 | [2.3] | 112.5 | [17.8] |
| J | METAL/ACHIEVE | 10 | 14.0 | [2.0] | 108.7 | [15.5] |
| K | METAL/ACCUBOND | 10 | 15.8 | [3.2] | 122.8 | [24.8] |
| L | METAL/DYNA-BOND PLUS | 9 | 14.6 | [2.4] | 113.4 | [18.6] |
| M | METAL/CONTROL | 10 | 32.5 | [5.4] | 113.2 | [41.9] |

Table 6

DATA FOR TENSILE BOND FORCE
FOR EACH BRACKET / ADHESIVE COMBINATIONS USED

| Code | Force (lbs.) | | | | | | | | | |
|------|--------------|---------------------|------|--------|------|---------------------|------|------|------|------|
| | #1 | #2 | #3 | #4 | #5 | #6 | #7 | #8 | #9 | #10 |
| A | 26.9 | [15.7] ¹ | 56.5 | [36.7] | 50.6 | 53.5 | 68.5 | | | |
| B | [47.6] | 74.4 | 63.7 | [31.3] | 74.8 | | | | | |
| C | 28.5 | 30.0 | 34.4 | 30.2 | 30.0 | | | | | |
| D | 26.9 | 25.0 | 30.9 | 22.3 | 24.7 | | | | | |
| E | 16.0 | 23.6 | 25.3 | 26.2 | 19.0 | 17.5 | 31.7 | 16.5 | | |
| F | 9.4 | 11.5 | 11.0 | 18.5 | 16.5 | 19.1 | | | | |
| G | 12.4 | 13.7 | 20.2 | 29.1 | 23.9 | 12.9 | 11.0 | | | |
| H | 35.0 | 23.8 | 29.8 | 6.5 | 10.5 | | | | | |
| I | 38.3 | 33.7 | 32.0 | 28.7 | 32.8 | 26.2 | 32.0 | 24.7 | 41.7 | 32.4 |
| J | 30.1 | 35.9 | 28.7 | 30.7 | 25.1 | 35.5 | 32.2 | 28.9 | 25.2 | 38.2 |
| K | 25.7 | 24.0 | 42.6 | 44.1 | 42.2 | 36.1 | 39.6 | 37.3 | 33.0 | 27.4 |
| L | 38.3 | 37.5 | 23.8 | 25.7 | 28.3 | < 4.2> ² | 35.9 | 31.5 | 36.0 | 35.8 |
| M | 36.4 | 26.6 | 36.7 | 42.0 | 33.7 | 28.9 | 23.2 | 32.3 | 33.0 | 31.8 |

¹ []: Excluded from the calculation due to cohesive failure of the bracket despite the usage of the special assembly.

² < >: Excluded from the calculation because of its abnormally small figure.

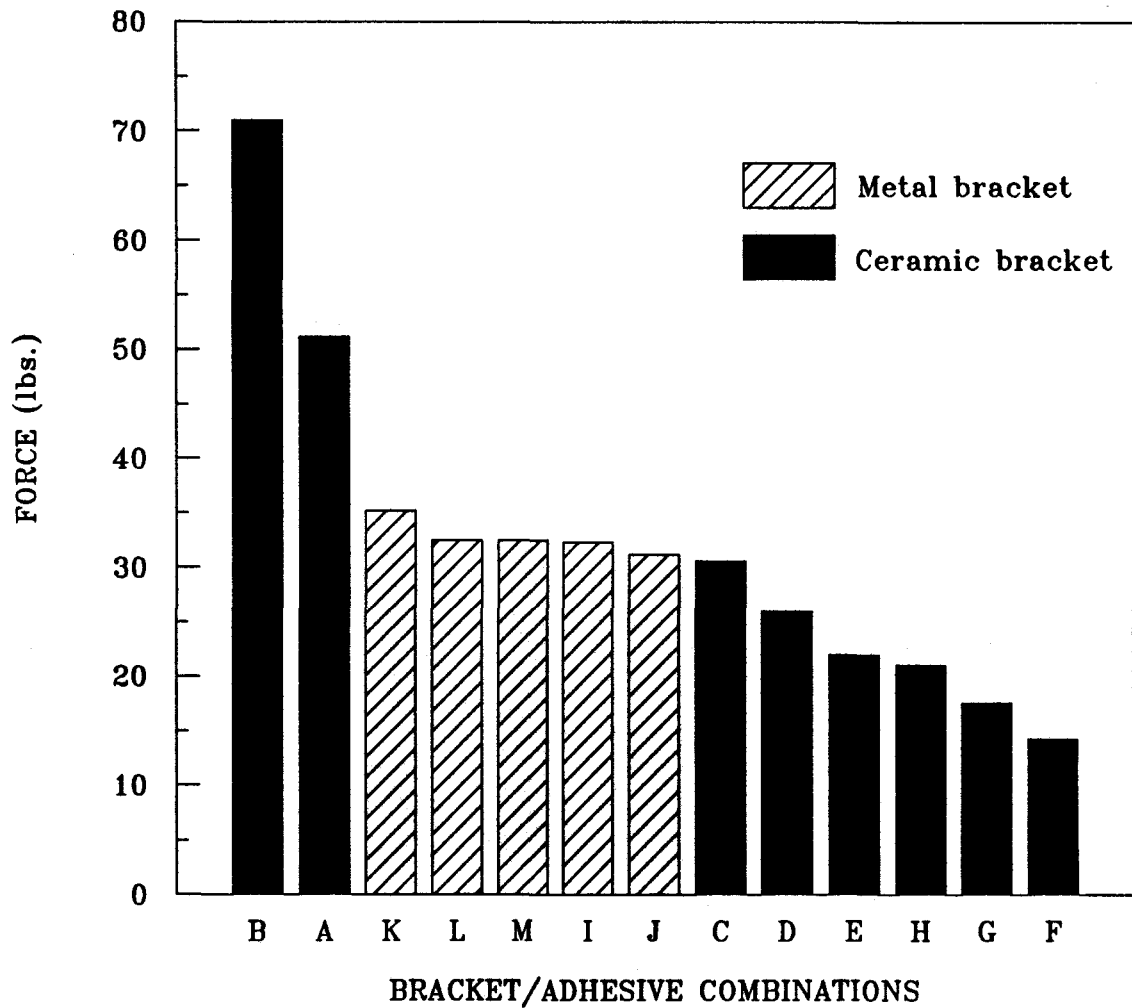


Figure 5. Mean tensile force for bracket/adhesive combinations tested.

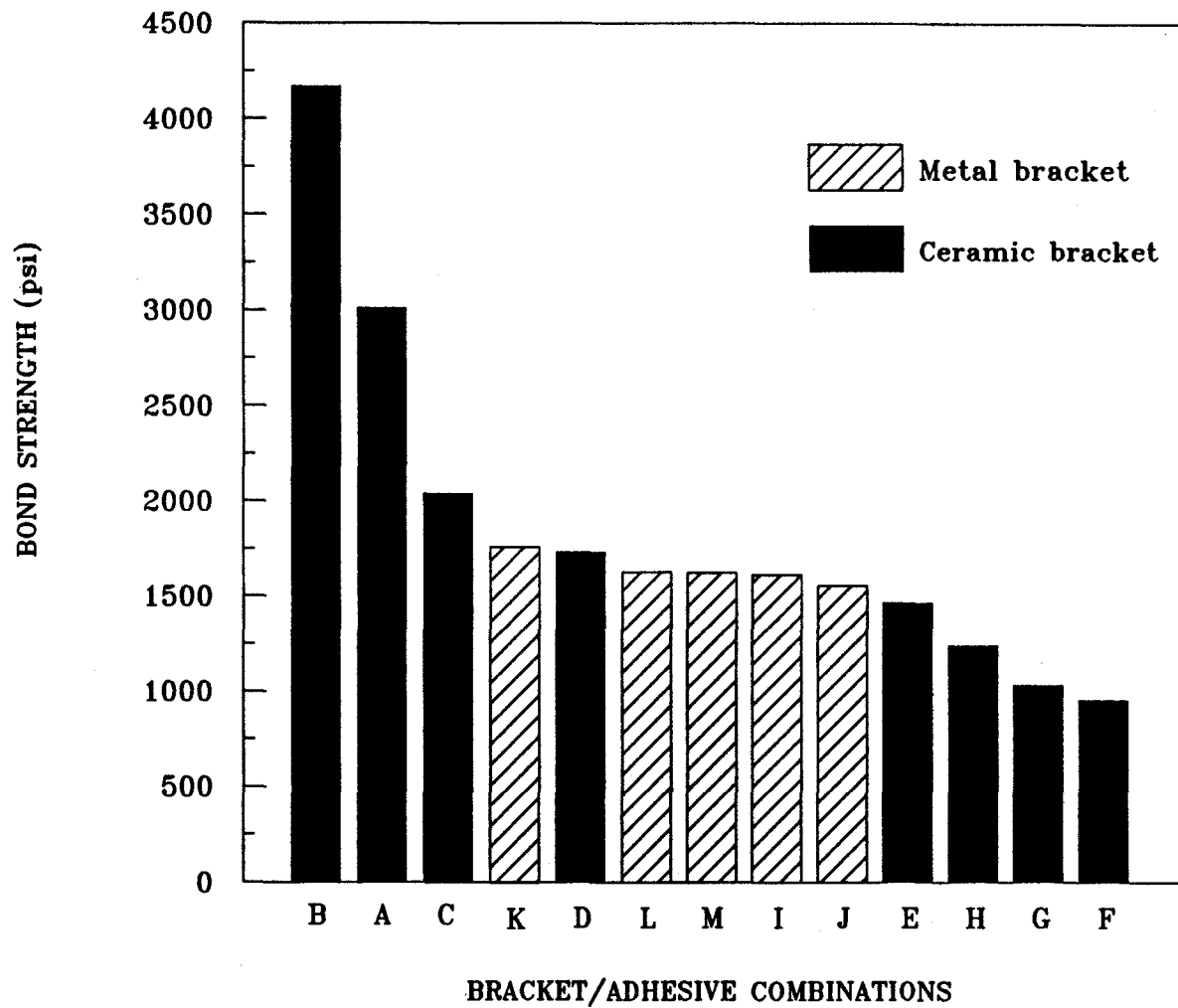


Figure 6. Mean tensile bond strength of bracket/adhesive combinations tested.

significant difference in bond strength from that of METAL with ACCUBOND. There was no significant difference in bond strength between CONCISE and ACCUBOND when used with ALLURE III.

Between TRANSCEND with CONCISE and METAL with CONCISE, there was no significant difference in bond strength. CONCISE and DYNA-BOND PLUS had no significant difference in bond strength when used with TRANSCEND. However, TRANSCEND with DYNA-BOND PLUS had the weakest bond strength, which was significantly less than that of METAL with DYNA-BOND PLUS.

INTRIGUE with CONCISE had significantly less bond strength than that of METAL with CONCISE. However, there was no significant difference in bond strength between INTRIGUE with CONTROL and METAL with CONTROL. CONCISE and CONTROL had no significant difference in bond strength when used with INTRIGUE.

Bond strengths between different ceramic brackets were compared using CONCISE as adhesive. STARFIRE had the highest bond strength, which was significantly greater than that of TRANSCEND or INTRIGUE but not significantly greater than that of ALLURE III. ALLURE III was the second highest in bond strength, which was significantly higher than either that of TRANSCEND or INTRIGUE. There was no significant difference in bond strength between TRANSCEND and INTRIGUE. Data used to compute these results are listed in Appendix,

CHAPTER V

DISCUSSION

This study was conducted to evaluate and compare tensile bond strength of the bracket-adhesive interface of four commercially available ceramic brackets and one mesh-backed metal bracket. All the data were analyzed statistically by Student t-test at $p < 0.01$.

Instead of natural teeth, plastic cylinders were used as retaining devices for the brackets tested. The reasons are as follows: 1) The study was to determine the bond strength of bracket-adhesive interface, not of adhesive-substrate interface. The usage of plastic cylinders eliminated variations that might have been introduced at the enamel-adhesive interface if natural teeth were used. 2) Other previous studies (Dickinson and Powers, 1980; Buzzitta, Hallgren, and Powers, 1982; Pulido and Powers, 1983; Wright and Powers, 1985) had shown that, as substrates, there is no significant difference in bond strength and in failure location between natural teeth and plastic cylinders 3) Many of the ceramic brackets were only available for the anterior teeth, and it would have been very difficult to obtain sufficient quantity of extracted

incisors for the study.

Two techniques of bracket placement were utilized in this study. One involved the usage of the mounting jig and the other, direct placement, did not involve the usage of the jig. The jig was effective with the metal brackets, whose mesh-backed design and wide contoured shape of the base made it necessary for the bracket to be pressed into the prepared area of an adhesive.

Except for one ceramic bracket/adhesive combinations, all ceramic brackets were placed directly on the adhesives. On the premise that the consistency of the adhesives mixed were firm enough to withstand the weight of a bracket, the direct procedure was much more manageable and easier for ceramic brackets than with the jig. Nevertheless, the jig had to be used with one ceramic bracket (INTRIGUE) with its proprietary cement (CONTROL) due to softness of the cement when its paste and primer were mixed together.

SEM examination has indicated that STARFIRE was apparently a non-crystalline substance which is consistent with the manufacturer's claim that the bracket is made from a single crystal aluminum oxide (sapphire). Different from other polycrystalline ceramic brackets, the surface of STARFIRE was very smooth (Fig. 7). The base had four grooves (Fig. 8), whose surface showed what appeared to be a layer of coupling agent (Fig. 9).

The examination of the bases that were successfully

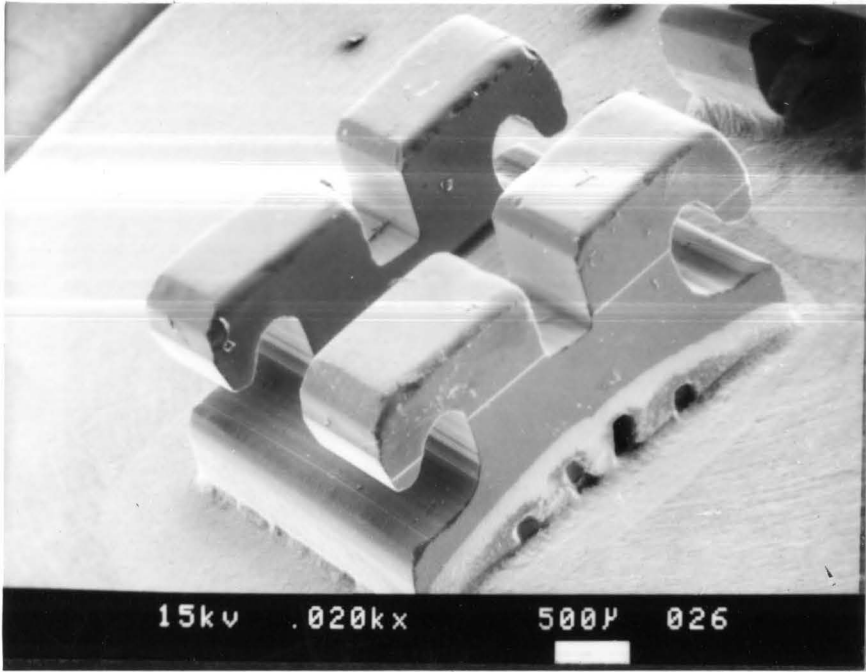


Figure 7. SEM photograph of STARFIRE with its smooth surfaces.

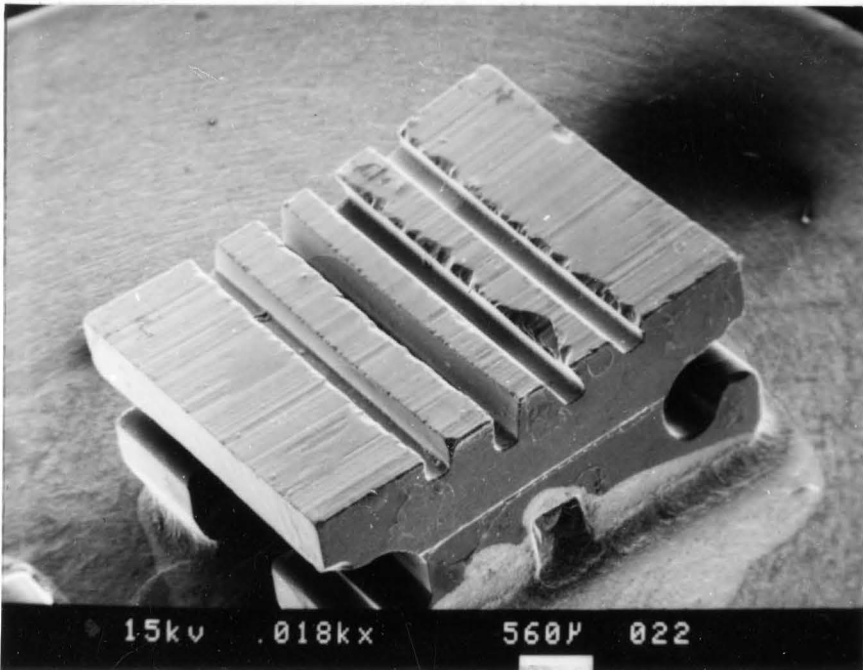


Figure 8. SEM photograph of a base of STARFIRE with its four retention grooves.

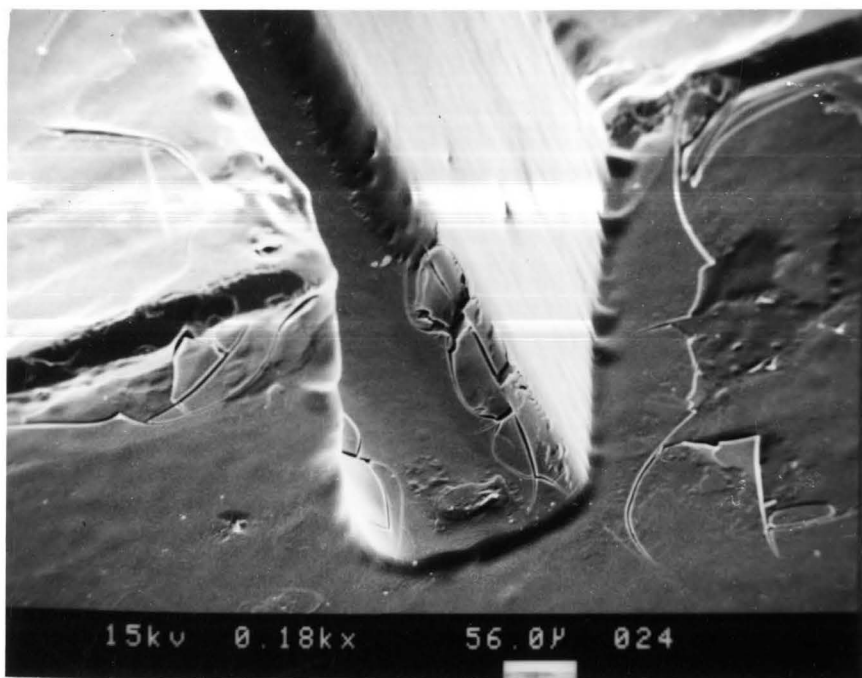


Figure 9. Further enlarged photograph of the base of STARFIRE showing indications of what seems to be a coupling agent.

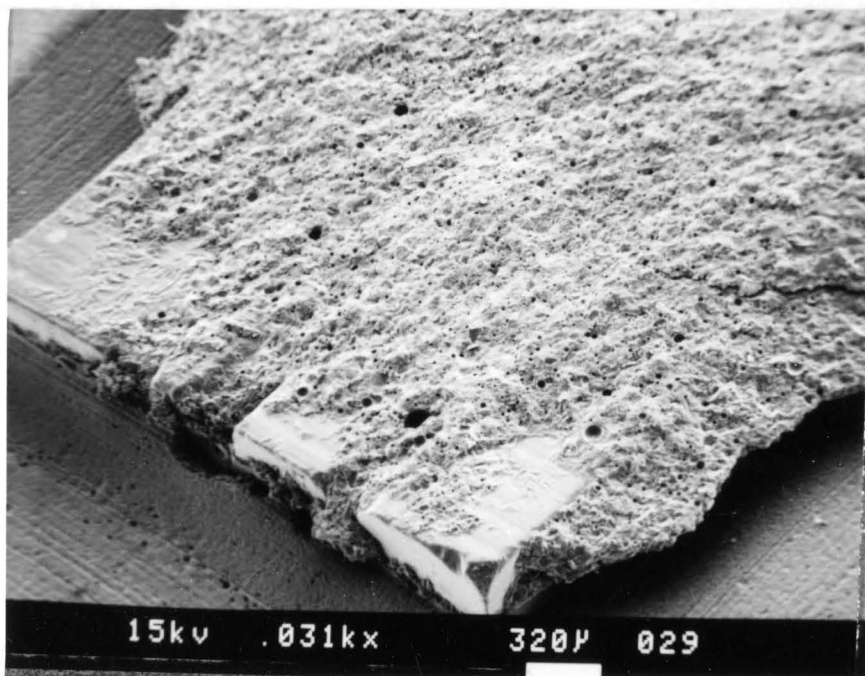


Figure 10. SEM photograph of a base of STARFIRE debonded from ACHIEVE demonstrating mostly cohesive failure of the adhesive.

debonded from the adhesives has indicated that the failures, both with CONCISE and with ACHIEVE, were mostly cohesive failures of the adhesives (Fig. 10). Exposing only small areas, the bases were covered with adhesives, indicating a strong chemical bond of the adhesives to the bases in addition to mechanical retention of the grooves. As a result, STARFIRE demonstrated the two highest bond forces and bond strengths of this study.

The highest and the second highest bond strength were achieved with proprietary cement, ACHIEVE, and with CONCISE, respectively. Although there was no significant difference between these two cements, bond strength with ACHIEVE was greater than with CONCISE at $0.05 > p > 0.01$. In comparing bond strengths of these two adhesives with STARFIRE and with the metal bracket, those with STARFIRE were significantly greater ($p < 0.001$) than those with its respective metal counterpart. Such significant differences were also demonstrated when bond strength of STARFIRE was compared with those of the other ceramic brackets. With CONCISE as a common adhesive, STARFIRE exhibited bond strength that was significantly greater ($p < 0.001$) than those of TRANSCEND or INTRIGUE. It was only greater at $0.05 > p > 0.01$ when compared with bond strength of ALLURE III. more rounded than

However, as a material, the single crystal STARFIRE was the weakest of the ceramic brackets tested. Because brittle nature of these brackets, numerous trials were made

to determine the most favorable configurations for tensile testing. Every single STARFIRE tested without the aid of the special assembly (Fig. 3) fractured at their wings. Even with the help of the special assembly, five out of thirteen brackets fractured either at the wings or at the neck portion of the bracket (Fig. 11). The forces that were found to fracture STARFIRE ranged from 4.6 lbs. to 47.6 lbs., depending on the locations of the fractures (Appendix, Table 8). Because of the geometry of the bracket, it was difficult to establish any significant data from these forces.

On the other hand, a distinct cohesive strength of the bracket material was demonstrated by ALLURE III. Although the special assembly was not utilized, not a single ALLURE III fractured during the investigation. SEM examinations of internal structures of the brackets have shown that ALLURE III, TRANSCEND, and INTRIGUE had similar polycrystalline structures (Fig. 12, 13, and 14), and yet, the wings of TRANSCEND and INTRIGUE fractured frequently while those of ALLURE III did not. Such differences could be the outcome of differences in manufacturing processes, and/or differences in design of the brackets. The wings of ALLURE III appeared to be bulkier and more rounded than those of the rest of the ceramic brackets.

The facial surface of ALLURE III was smooth, as though it had been glazed (Fig. 15). However, the slot,

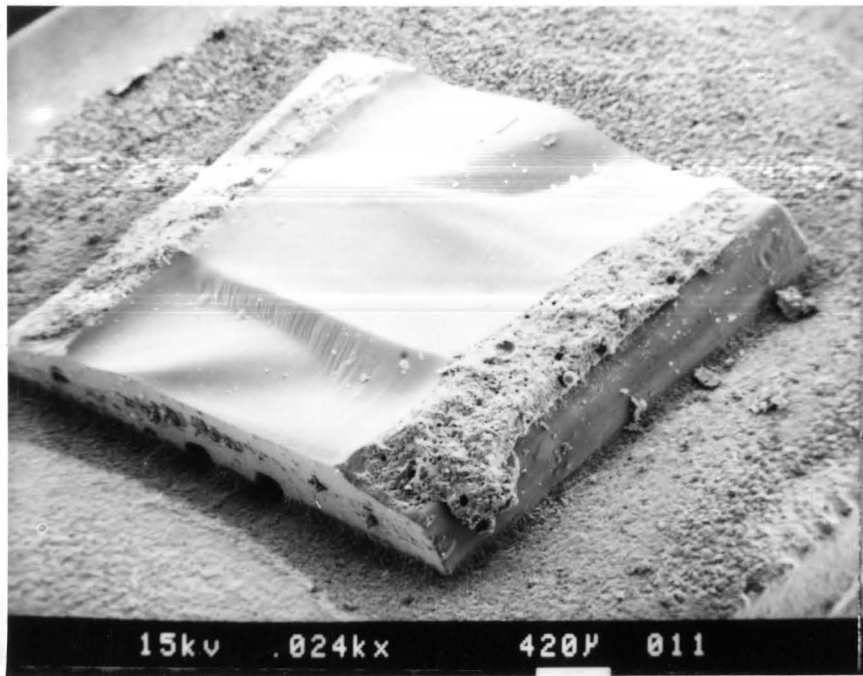


Figure 11. SEM photograph of STARFIRE exhibiting cohesive failure of the bracket. The whole neck portion of the bracket has been fractured.

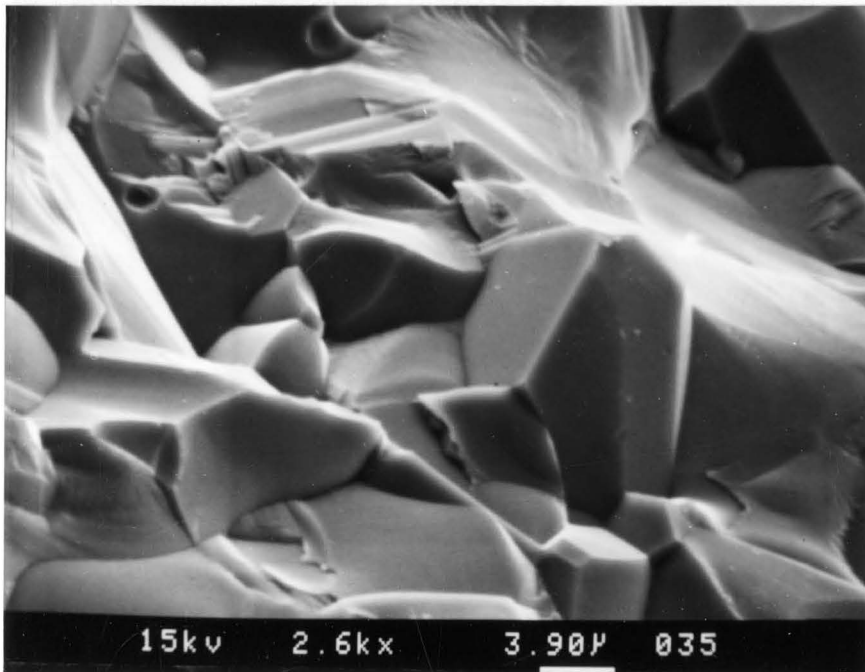


Figure 12. SEM photograph of internal structure of ALLURE III revealing polycrystalline formation.

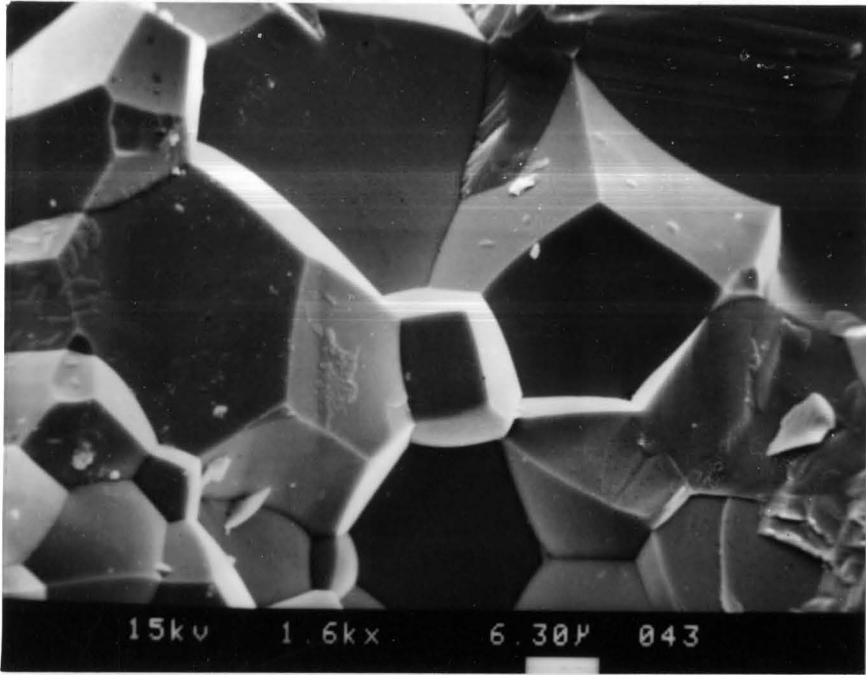


Figure 13. SEM photograph of inner structure of TRANSCEND showing polycrystalline structure.

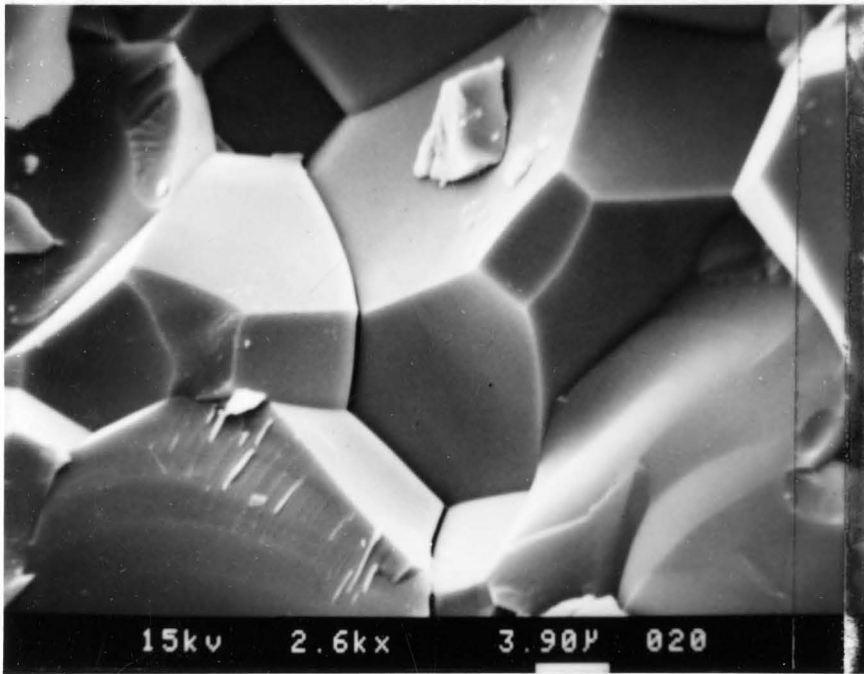


Figure 14. SEM photograph of inner structure of INTRIGUE demonstrating polycrystalline formation.

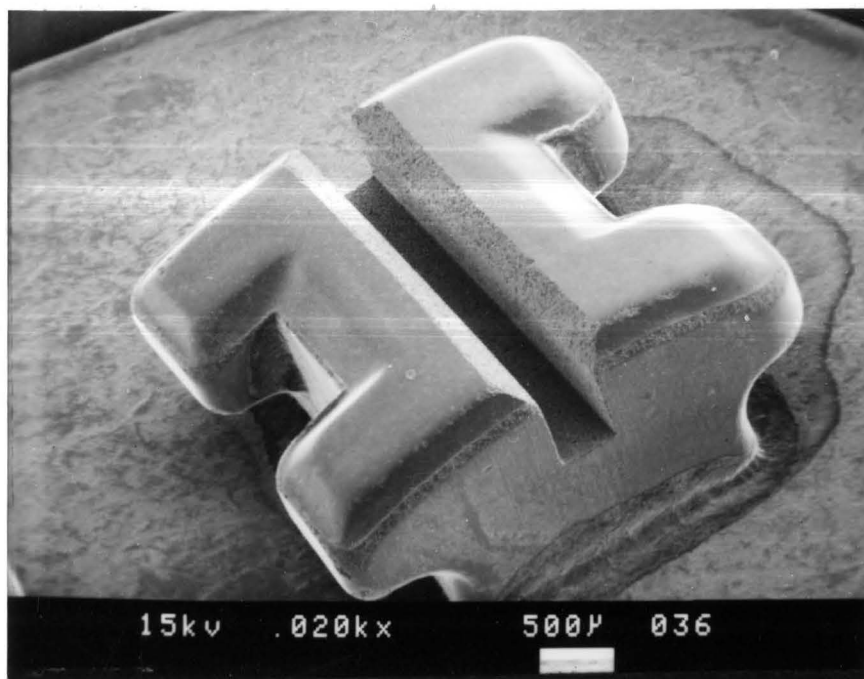


Figure 15. SEM photograph of ALLURE III showing smooth facial surface.

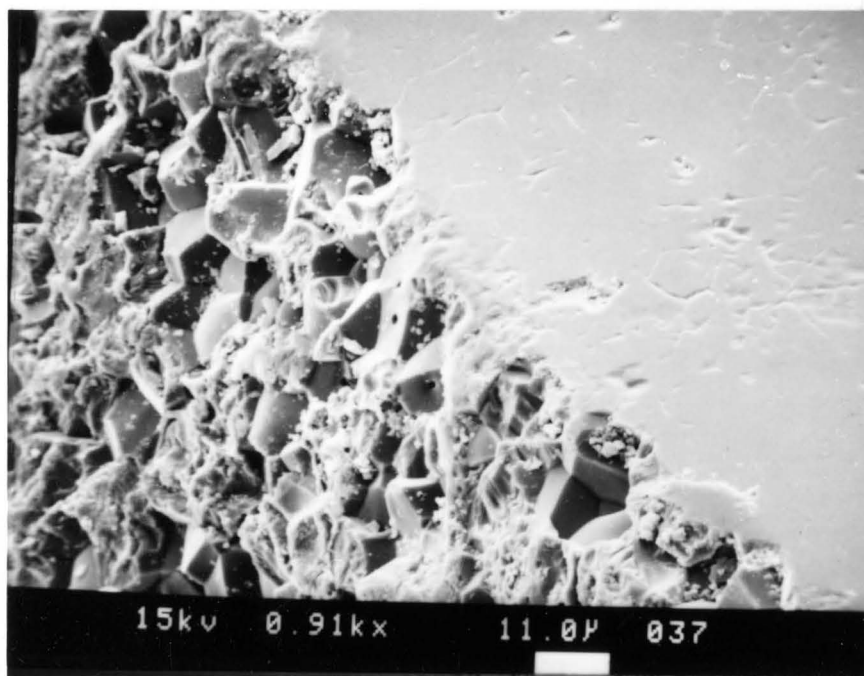


Figure 16. Further enlarged photograph of a corner where the slot was ground into the facial surface of ALLURE III. The contrast between the coarse surface of the slot and the smooth facial surface was evident.

which appear to be ground into the bracket, was very coarse (Fig. 16). This roughness could be a problem due to its potential ability to accumulate plaque. In addition to its detrimental effect on oral hygiene, plaque build up would have adverse effect on sliding mechanics by increasing friction between a wire and the slot of the bracket.

The base of ALLURE III, which had six square-shape indentations for mechanical retention, was a continuation of the neck portion of the bracket; it did not have a typical flare out design of the base that was seen with the other brackets (Fig. 17). It looked as though the thickness needed to make the base was incorporated into the bulk of the wings instead, thus reinforcing the strength of the wings. However, any excess or overflow of adhesives applied to the base could jeopardize the spaces needed for ligature ties.

Using CONCISE as the control adhesive, ALLURE III demonstrated bond strength that was significantly greater than those of TRANSCEND, INTRIGUE or the metal bracket. In comparing CONCISE with ACCUBOND, ALLURE III did not show a significant difference in bond strength. However, the bond strength with CONCISE was greater than that with ACCUBOND at $0.05 > p > 0.01$.

Examination of debonded bases of ALLURE III has indicated that the bond failures were adhesive and cohesive failure of the cements used (Fig. 18). Every ALLURE III had

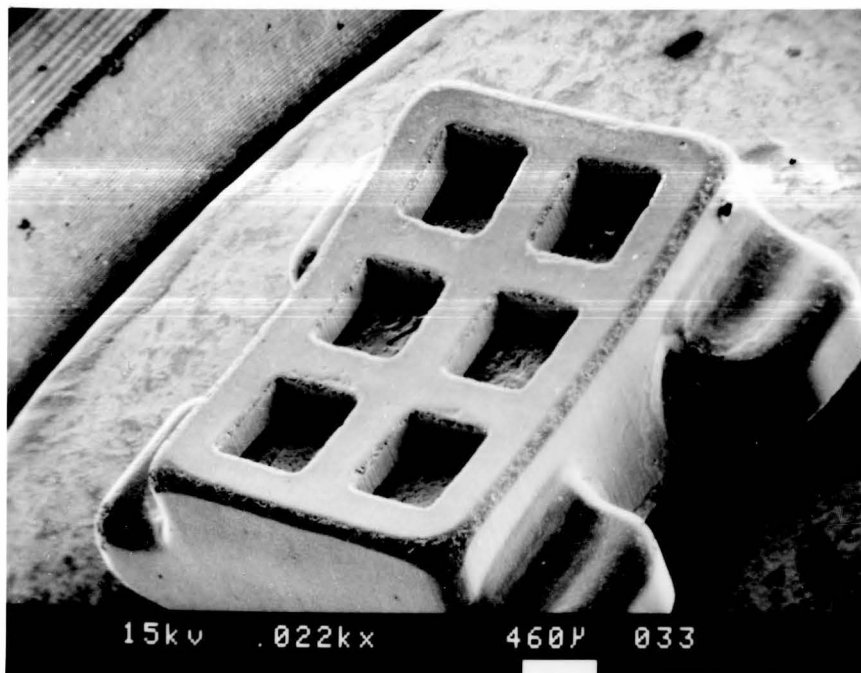


Figure 17. SEM photograph of a base of ALLURE III with its six square-shaped indentations.

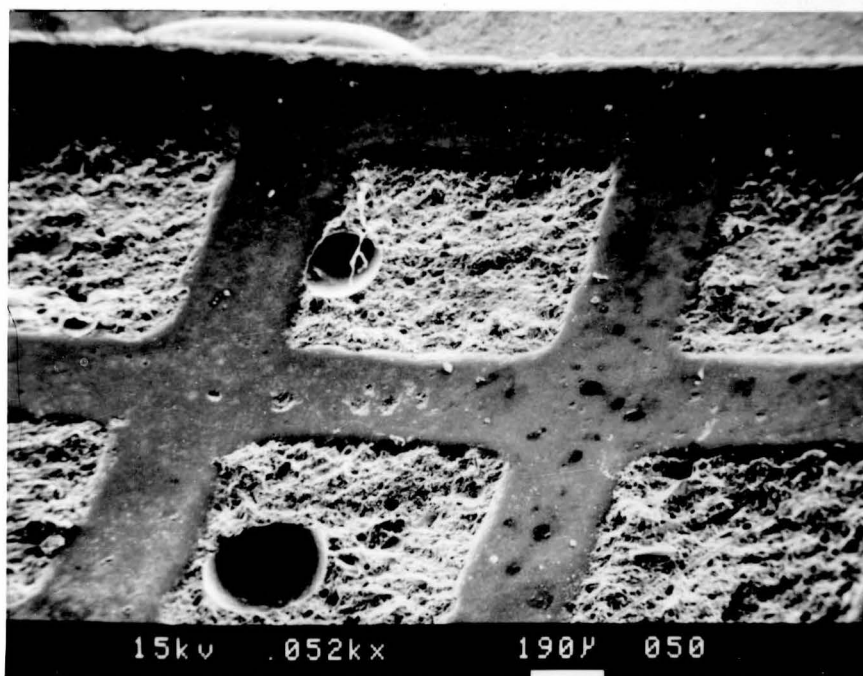


Figure 18. SEM photograph of debonded surface of ALLURE III with indentations filled with ACCUBOND and a few air bubbles.

similar mode of failure. The six retention indentations of the base were filled with cement which was fractured at the level of the base indicating an effective mechanical retention. However, the base portion of the bracket was cleanly detached from the cement revealing poor adhesion to the adhesive. The indentations had their drawback of entrapping air bubbles, which were unavoidable despite a meticulous effort. These voids could reduce bond strength and cause premature bond failures.

Although not as smooth as ALLURE III, the outer surfaces of TRANSCEND also appeared as though the bracket had been glazed. Nevertheless, surfaces of the slot were as rough and coarse as those of ALLURE III (Fig. 19 and 20). As discussed with ALLURE III, such surface irregularities could have significant effects on oral hygiene as well as sliding mechanics due to their potential to gather plaque. It could nullify the advertised benefit of lower coefficient of friction which is theoretically obtainable with ceramic brackets.

The base of unused TRANSCEND was unique because of its glossy appearance (Fig. 21). Whether it was a result of glazing, silica coating, coating with a coupling agent, or any combination of these was not known. However, since it was known that the bond strength was directly proportional to the area of contact in a given surface area, it seems that the design of a smooth surface, which minimizes the

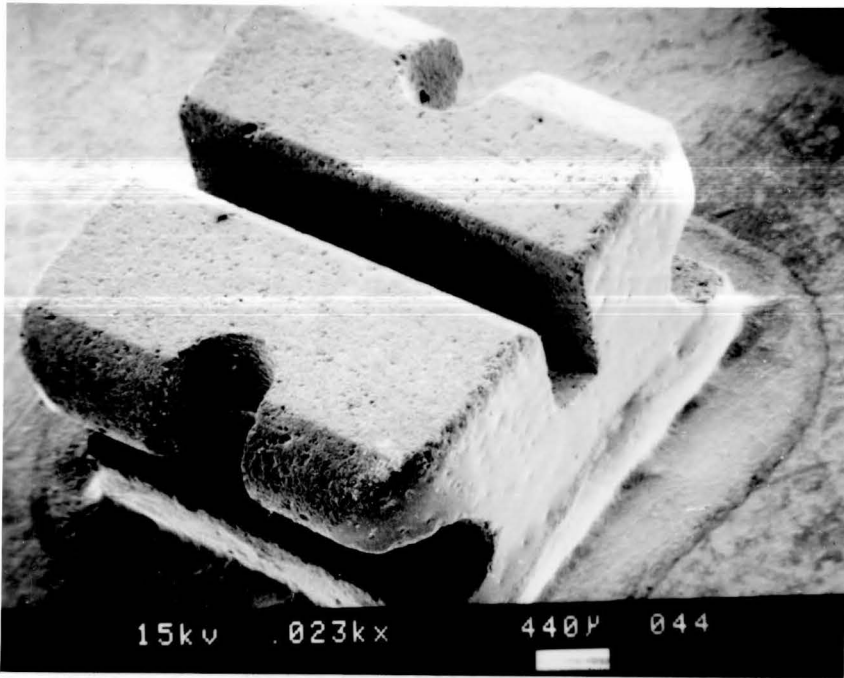


Figure 19. SEM photograph of TRANSCEND.

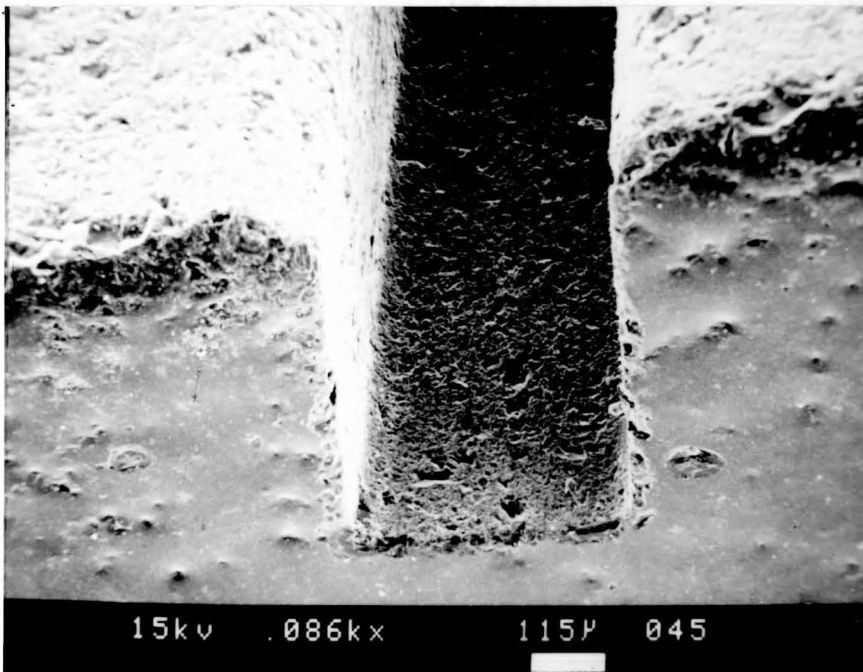


Figure 20. Further enlarged photograph of the slot of TRANSCEND showing the roughness of the slot and much smoother lateral surface of the bracket.

contact area between the base and the cement, might have been intentional to prevent any excessive adhesion of the base to cement and to facilitate the debonding process of the bracket.

The bond strength of TRANSCEND with CONCISE was only greater than those of INTRIGUE with CONCISE, and TRANSCEND with DYNA-BOND PLUS, the proprietary cement of TRANSCEND. However, the differences with both of them were only significant at the $0.05 > p > 0.01$ level. With CONCISE as adhesive, TRANSCEND and the metal bracket did not show a significant difference in bond strength. However, with DYNA-BOND PLUS as adhesive, the metal bracket demonstrated bond strength that was significantly greater ($p < 0.001$) than that with TRANSCEND. This certainly could indicate a weakness in chemical bond between DYNA-BOND PLUS and the base of TRANSCEND, but it was found that problems involving the adhesive itself could have contributed to such weakness.

When CONCISE was used with TRANSCEND, inherent weaknesses of the brackets have caused some cohesive failures of the brackets. Wings of the brackets fractured with or without adhesive failures of the bases from the cement. When the cohesive failures concurred with the adhesive failures, the forces ranged from 18.0 lbs. to 26.2 lbs. with one of the wings broken from each bracket. When the failures were only the cohesive failures of the brackets, the forces were 24.5 lbs. and 27.5 lbs. with all

four wings broken from each bracket (Appendix, Table 8). These values are excluded from the bond strength data.

Careful examinations of SEM photographs (Fig. 22 and 23) of debonded bases of TRANSCEND have revealed the differences in failure modes between two adhesives, CONCISE and DYNA-BOND PLUS. While almost all of the failures with both adhesives were combinations of adhesive and cohesive failures of the cements, the failures with CONCISE were mostly adhesive with detachment of materials which produced glossy appearance of a new base; the debonded base appeared to have rougher texture than the new base (compare Fig. 21 with 22). These observations led to speculation that materials that comprised the smooth surface of the new base were layers of silica and coupling agent, rather than the ceramic base that had been glazed. If the smoothness was due to a process of glazing, then the roughness of the debonded base would have meant a cohesive failure of the bracket, which it did not appear to be.

A base debonded from DYNA-BOND PLUS was very much different from the one debonded from CONCISE. Although it also showed a combination of adhesive and cohesive failure of cement, part of the base that had adhesive failures still appeared to retain the smoothness that was seen with the new base, indicating that adhesion of the cement to the base was not strong enough to detach the base layers from the base (Fig. 23). From a further enlarged micrograph, a slight gap

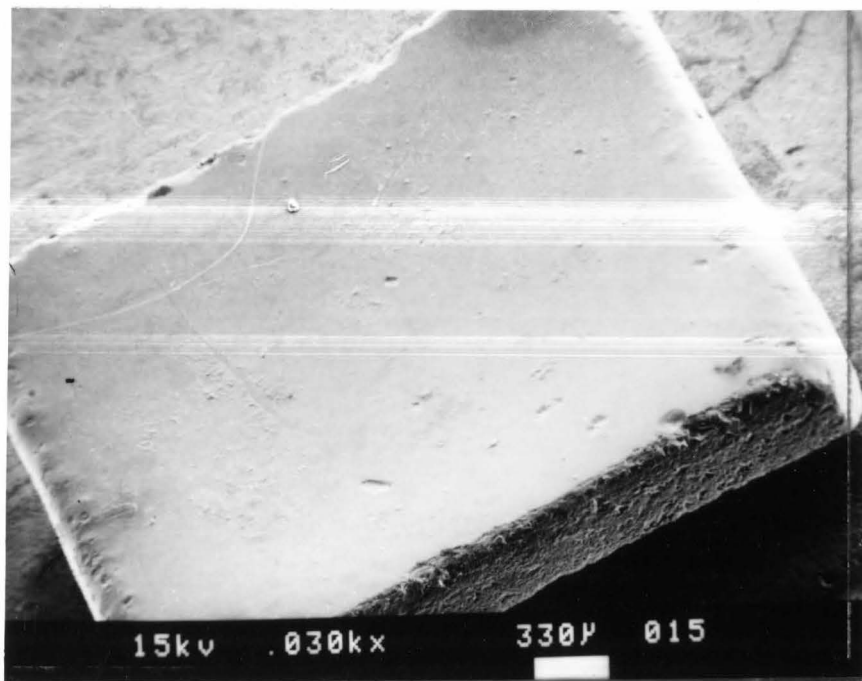


Figure 21. SEM photograph of a base of TRANSCEND. The base was much smoother than the rest of the surfaces.

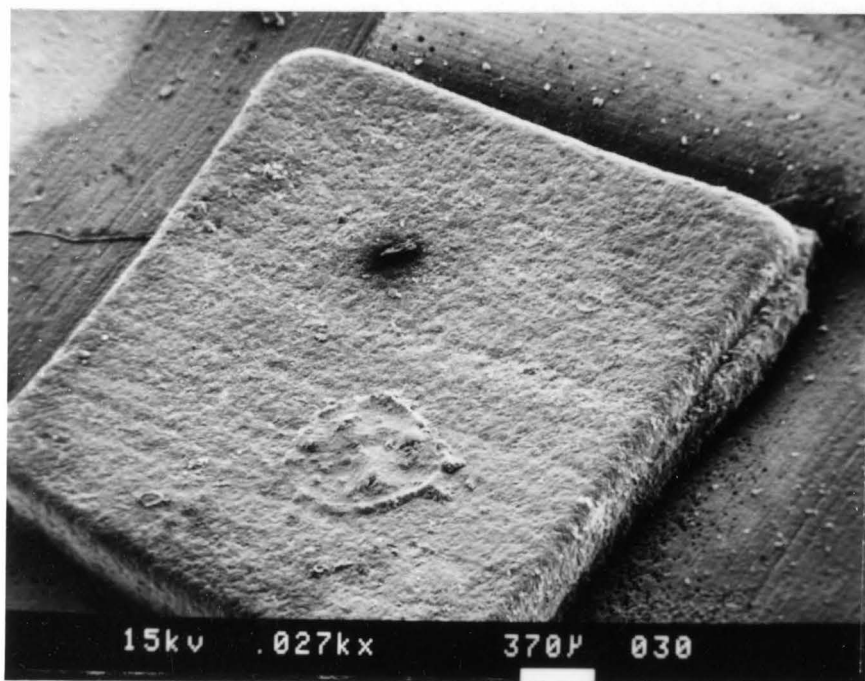


Figure 22. SEM photograph of the base of TRANSCEND debonded from CONCISE. The smoothness of the new base had disappeared.

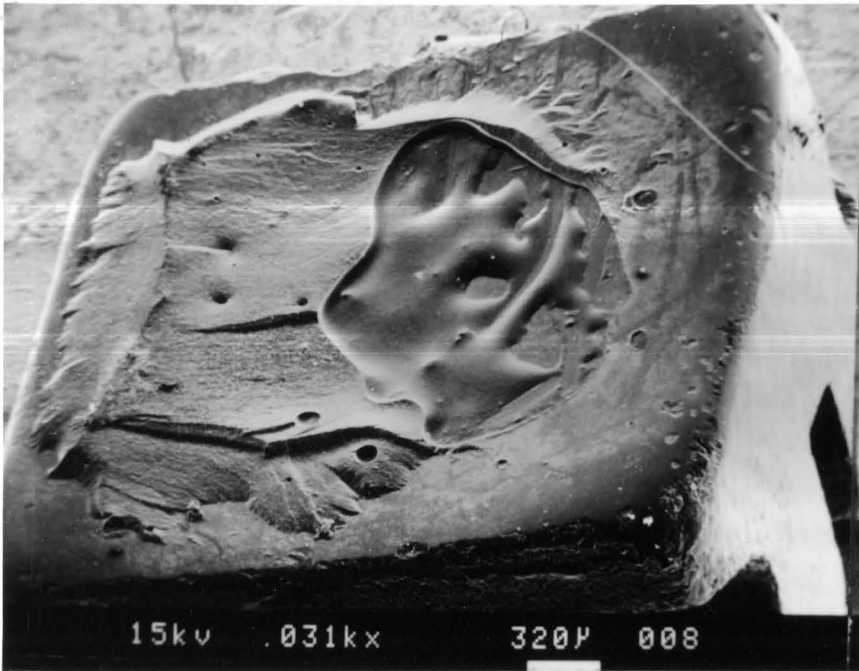


Figure 23. SEM photograph of the base of TRANSCEND debonded from DYNA-BOND PLUS. The part of the base that had adhesive failure was still smooth like the unused base.

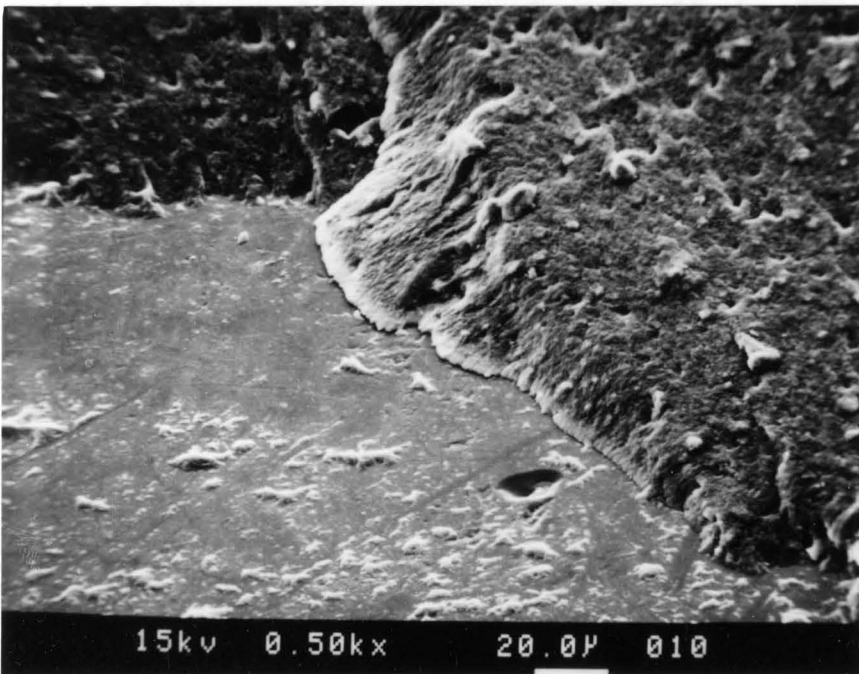


Figure 24. Further enlarged photograph of the base of TRANSCEND debonded from DYNA-BOND PLUS. The slight gap between the cement and the base indicated an incomplete adhesive failure.

could be detected between the cement and the base, indicating partial adhesive failure at the interface between the cement and smooth surface of the base (Fig. 24).

The greater portion of the base debonded from the DYNA-BOND PLUS was a cohesive failure of the cement. Within the cohesive failure, most of the bases contained a void, presumably an outcome of manipulation difficulties encountered with the cement. DYNA-BOND PLUS had very viscous consistency, which made handling difficult. In addition to that, the length of actual setting time¹ of the cement, which was about 45 to 50 seconds, was much less than its claimed time of 120 seconds. If the adhesive was mixed according to manufacturer's instruction, which was 20 seconds, that left working time of only 25 to 30 seconds. Such working time was less than half of what was claimed which was found to be insufficient for proper placement of the bracket. With the consent of the manufacturing company, the mixing time had to be decreased to 10 seconds to slightly extend the working time.

Some difficulties were also encountered with CONTROL, the proprietary cement for the INTRIGUE. In addition to a mushy consistency which necessitated the usage of the mounting jig, CONTROL was a no-mix adhesive system, where the base paste polymerizes upon contact with its primer. This type of adhesive is most effective when an adhesive

¹ From start of mix at 20-22°C (68-72°F).

layer is thin enough for the primer to promote adequate polymerization of the paste. However, for this study large quantity of cement was needed to fill the prepared area of the plastic substrates. After consultation with the manufacturing company, it was decided to mix the paste of the cement with its primer within the prepared area of the substrates to evenly polymerize the adhesive while saving some working time. Different proportions of paste to primer had to be tried preliminarily to find the one that yielded sufficient working time. It has been stated that the degree of conversion from monomer to polymer in no-mix system decreases rapidly as the distance from the site of polymerization initiation increases (Swartz, 1988). If this type of adhesive is used with the ceramic brackets with bases that have relatively deep indentations for mechanical retention, problems could occur with polymerization. ALLURE III is an example of such bracket (Fig. 17).

The surfaces of INTRIGUE were not much different from ALLURE III or TRANSCEND. All surfaces including that of the base, which did not show any sign of glazing process, were rough and coarse with countless micro pores (Fig. 25, 26, and 27). The base was flat and had no mechanical indentations. After it was debonded from either CONCISE or CONTROL, it also displayed adhesive and cohesive failure of the cements used (Fig. 28 and 29). However, to achieve separation of the base from the cements, the special

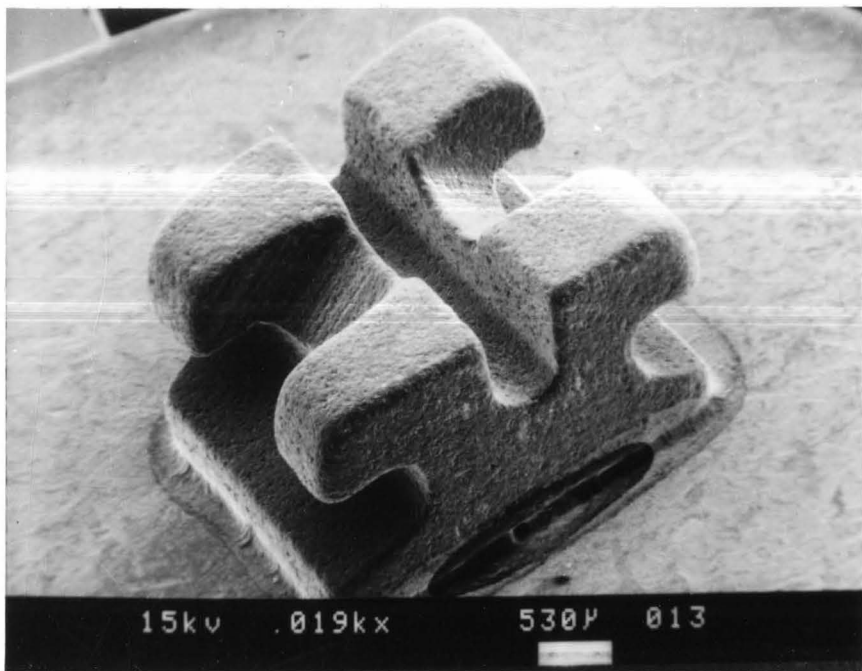


Figure 25. SEM photograph of INTRIGUE.

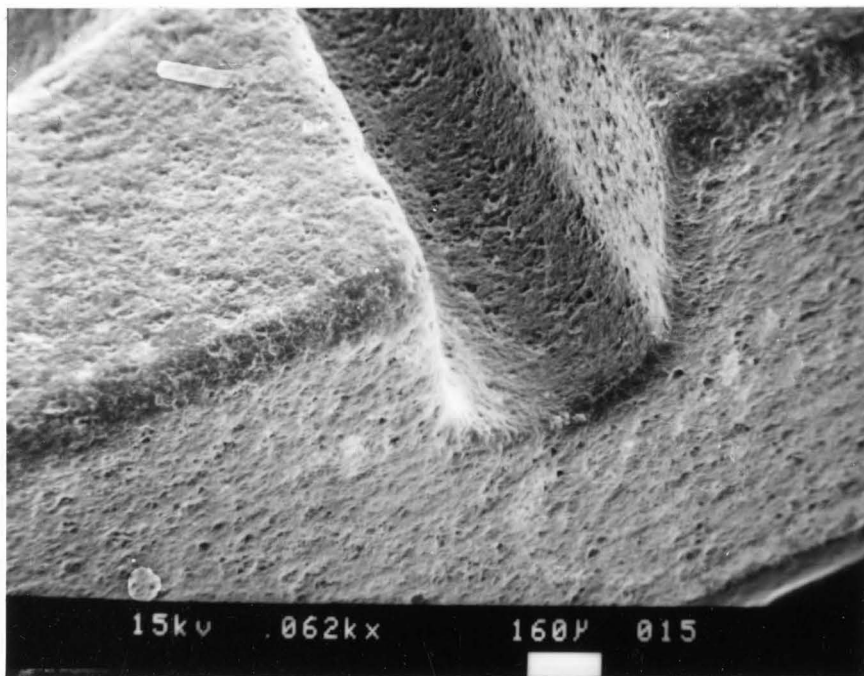


Figure 26. Further enlarged photograph of INTRIGUE showing countless micro porosities.

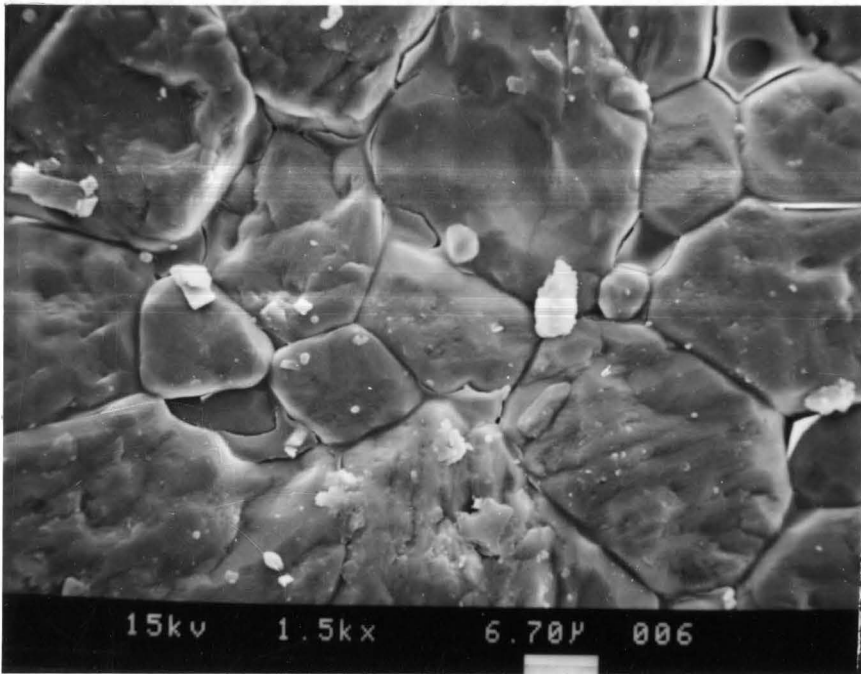


Figure 27. SEM photograph of a base of INTRIGUE revealing polycrystalline structure under high magnification.

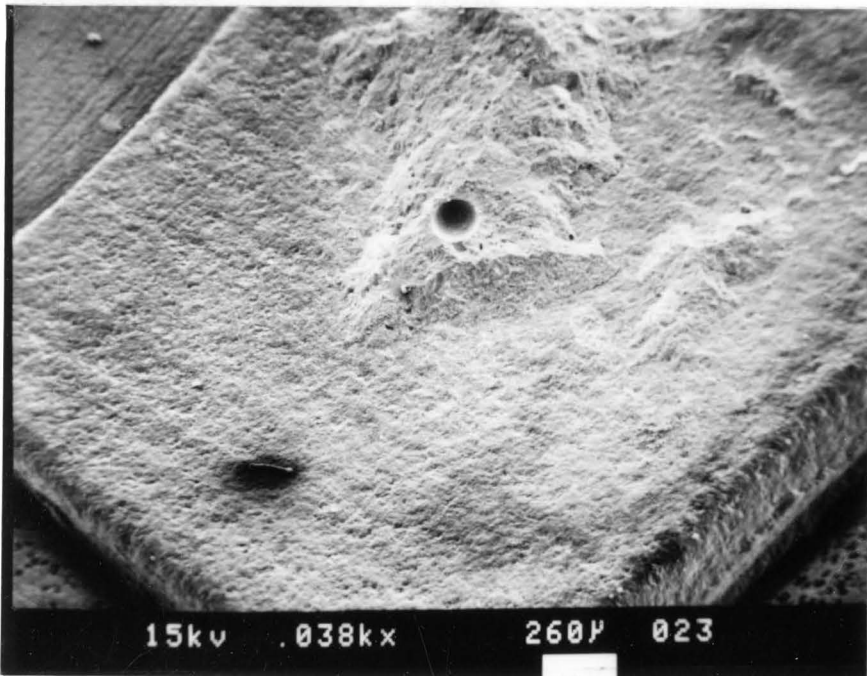


Figure 28. SEM photograph of the base of INTRIGUE debonded from CONTROL displaying both adhesive and cohesive failures.

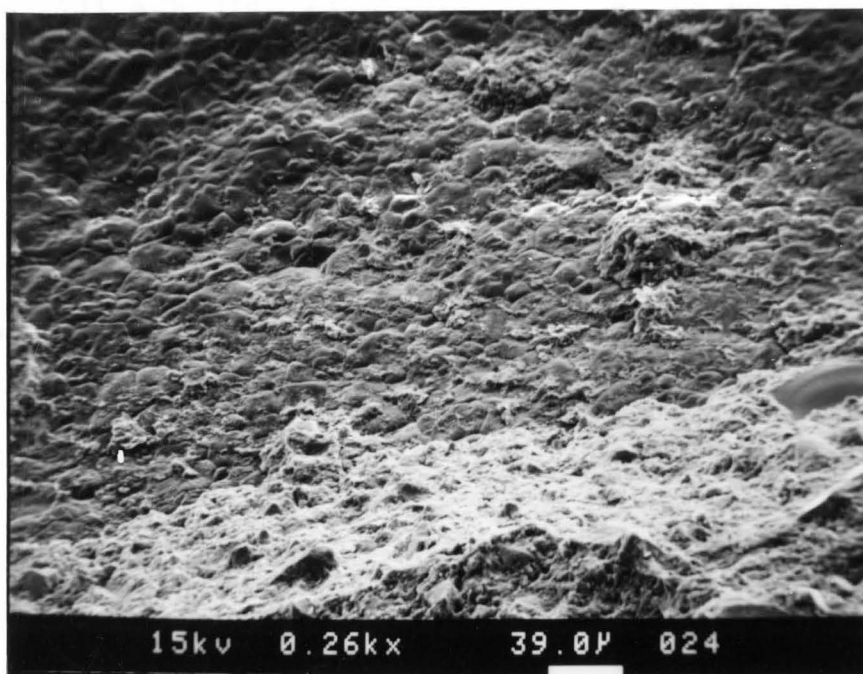


Figure 29. Further enlarged photograph of the base of INTRIGUE at the junction between adhesive and cohesive failures.

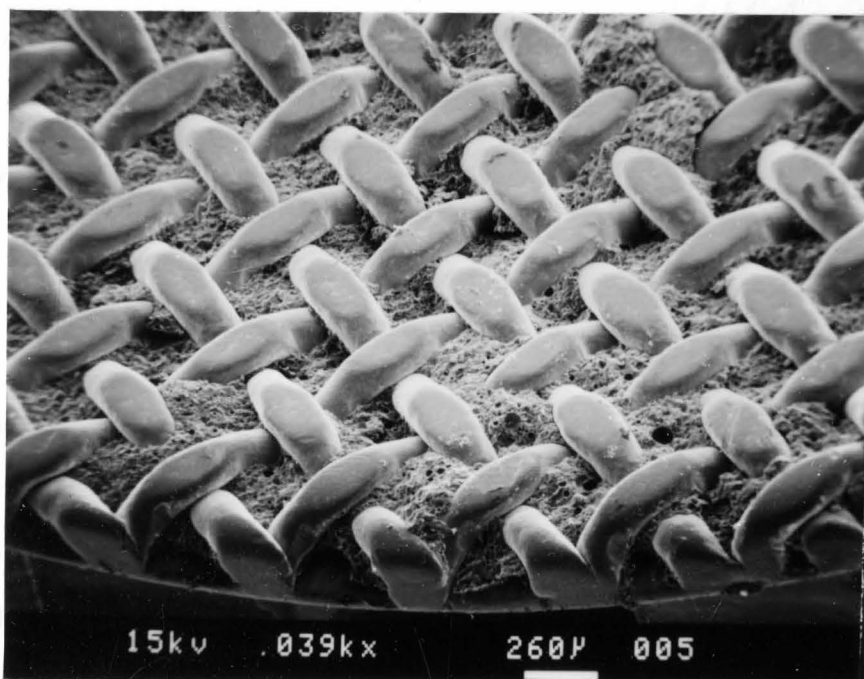


Figure 30. SEM photograph of a base of the metal bracket debonded from CONCISE.

assembly that was used with STARFIRE, had to be built onto the bonded bracket. Without the assembly, inherent weakness of the bracket caused wing fractures in three out of five brackets that were initially tested with CONCISE. The forces recorded for wing fractures ranged from 16.8 lbs. to 20.5 lbs. (Appendix, Table 8). The force values for the two brackets, which were successfully debonded without any wing fracture, were included in bond strength calculations because there was no significant difference between them and the actual data obtained with usage of the special assembly.

The bond strength of INTRIGUE, especially with CONCISE, was one of the weakest tested. Using CONCISE, the bond strength was not only significantly less than that of the metal bracket but also those of STARFIRE and ALLURE III. Although INTRIGUE demonstrated slightly greater bond strength with CONTROL than with CONCISE, the difference was not significant. With CONTROL as the adhesive, the metal bracket showed slightly greater bond strength than INTRIGUE, but the difference was also not significant.

When bond strengths of the metal brackets were compared among different cements, no significant difference was detected. There was also no difference in failure locations among different adhesives. With all cements, the metal brackets failed at the bracket-adhesive interface (Fig. 30).

SUMMARY AND CONCLUSION

The object of this study was to investigate the tensile adhesive bond strength of new ceramic brackets and one mesh-backed metal bracket using the bonding adhesives recommended and distributed by the respective manufacturers and one bis-GMA adhesive (CONCISE).

All metal brackets and one ceramic bracket (INTRIGUE) were mounted using a special jig and the rest of the ceramic brackets (STARFIRE, ALLURE III, and TRANSCEND) were placed directly on the plastic cylinders which were constructed to retain the brackets. To assist debonding of the brackets, special assemblies were built for those brackets which fractured easily. All bonded brackets were kept in 100 per cent humidity at 37.0°C for 24 hours prior to testing with the Instron machine using a special loading jig. The bond failures were examined with an optical stereo microscope and scanning electron microscope. Mean values and standard deviations of bond force and strength were calculated. The data were analyzed statistically by Student t-test at $p < 0.01$.

Although STARFIRE had the greatest bond strength it was the most easily fractured of the brackets studied. INTRIGUE and TRANSCEND had the weakest values. Debonded

bases displayed varying degrees of adhesive and cohesive failure of cement. STARFIRE demonstrated mostly cohesive failure of cements while TRANSCEND exhibited mostly adhesive failure.

Scanning electron micrographs have revealed the texture and the structure of four ceramic brackets tested. The single crystal STARFIRE bracket had smooth surfaces, but the rest of polycrystal ceramic brackets had coarse surfaces, especially inside their slots. It was thought that such roughness could have a adverse effect on oral hygiene and sliding mechanics due to its potentials to accumulate plaque.

Structural weaknesses seen with some ceramic brackets appeared to be the result of both inherent weakness of the material and inadequate design of the bracket. It was shown that with a proper design (e.g. ALLURE III) the inherent weakness could be compensated.

The conclusions drawn from this study were as follows:

- 1) Testing four ceramic brackets for tensile bond strength with CONCISE resulted in the determination of statistically significant differences. STARFIRE and ALLURE III had the highest bond strength while INTRIGUE and TRANSCEND had the lowest values.
- 2) Testing four ceramic brackets for tensile bond strength with CONCISE and with respective proprietary cements

resulted in no statistically significant differences.

- 3) Testing four ceramic brackets and one metal bracket for tensile bond strength with CONCISE resulted in the determination of statistically significant differences. STARFIRE and ALLURE III had bond strengths that were significantly greater than that of metal bracket while INTRIGUE had a value which was significantly less than that of metal bracket.
- 4) Testing four ceramic brackets and one metal bracket for tensile bond strength with respective proprietary cements resulted in the determination of statistically significant differences. STARFIRE had bond strength that was significantly greater than that of the metal bracket while TRANSCEND had the value that was significantly less than that of the metal bracket.
- 5) Excessive bond strength demonstrated by STARFIRE might lead to an enamel fracture if sudden load as in biting or trauma was applied.

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APPENDIX

Table 7

STUDENT T-TEST FOR TENSILE BOND STRENGTH

| <u>Comparison of Combinations</u> | <u>Degrees of Freedom</u> | <u>T-value</u> | <u>Significance at p < 0.01</u> |
|---------------------------------------|-------------------------------|----------------|--|
| A to B | 6 | -2.093 | No |
| A to C | 8 | 2.395 | No |
| A to E | 11 | 4.404 | Yes |
| A to G | 10 | 5.217 | Yes |
| A to I | 13 | 4.737 | Yes |
| B to J | 11 | 15.499 | Yes |
| C to D | 8 | 2.700 | No |
| C to E | 11 | 3.244 | Yes |
| C to G | 10 | 5.230 | Yes |
| C to I | 13 | 3.437 | Yes |
| D to K | 13 | -0.163 | No |
| E to F | 12 | 2.605 | No |
| E to G | 14 | 2.132 | No |
| E to I | 17 | -0.994 | No |
| F to L | 12 | -4.396 | Yes |
| G to H | 10 | -0.638 | No |
| G to I | 15 | -3.611 | Yes |
| H to M | 13 | -1.516 | No |

Table 7 (continued)

| <u>Comparison of Combinations</u> | <u>Degrees of Freedom</u> | <u>T-value</u> | <u>Significance at $p < 0.01$</u> |
|---------------------------------------|-------------------------------|----------------|---|
| I to J | 18 | 0.517 | No |
| I to K | 18 | -1.041 | No |
| I to L | 17 | -0.117 | No |
| I to M | 18 | -0.090 | No |
| J to K | 18 | -1.490 | No |
| J to L | 17 | -0.614 | No |
| J to M | 18 | -0.597 | No |
| K to L | 17 | 0.891 | No |
| K to M | 18 | 0.950 | No |
| L to M | 17 | 0.029 | No |

Table 8

LOAD AT COHESIVE FAILURE OF CERAMIC BRACKETS

| <u>Bracket/Adhesive Combination</u> | <u>Load Range (lbs.)</u> | <u>Fracture Site</u> |
|---|------------------------------|--------------------------|
| A | 22.2 - 32.4 | 4 wings |
| A | 37.4 - 45.5 | 4 wings |
| [A] ¹ | 15.7 - 16.7 | Neck |
| [A] | 17.6 | Neck |
| [A] | 36.7 | Neck |
| B | 10.0 - 12.2 | 4 wings |
| B | 4.6 - 9.4 | 4 wings |
| [B] | 43.1 - 47.6 | Neck |
| [B] | 31.3 | Neck |
| E | <18.0> ² | 1 wing |
| E | <19.2> | 1 wing |
| E | <19.1> | 1 wing |
| E | 27.5 | 4 wings |
| E | 24.5 - 32.7 | 4 wings |
| G | 16.8 | 4 wings |
| G | 18.8 | 4 wings |
| G | 20.5 | 4 wings |

¹ []: the special assembly was used (also listed in Table 5, but excluded in actual computations).

² < >: mixed adhesive/cohesive failure.

APPROVAL SHEET

The thesis submitted by Moon Woo Limb, D.D.S., has been read and approved by the following committee:

James L. Sandrik, Ph.D.
Professor and Chairman
Department of Dental Materials, Loyola

Lewis Klapper, D.M.D., M.Sc.D., D.Sc.
Associate Professor and Chairman
Department of Orthodontics, Loyola

Leslie A. Will, D.M.D., M.S.D.
Assistant Professor
Department of Orthodontics, Loyola

Paul C. Kuo, D.M.D., M.D.
Associate Professor and Chairman
Department of Oral and Maxillofacial Surgery, Loyola

The final copies have been examined by the director of the thesis and the signature which appears below verifies the fact that any necessary changes have been incorporated and that the thesis is now given final approval by the Committee with reference to content and form.

The thesis is therefore accepted in partial fulfillment of the requirements for the degree of Master of Science.

Date

June 25, 1988

Director's Signature

James L. Sandrik