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### THE BOND STRENGTH OF COMPOSITE RESIN TO DENTAL CASTING ALLOYS USING AN ELECTRO-CHEMICAL TIN PLATING SYSTEM

Keith S. Smith, II, M.S.

### The University of Texas Graduate School of Biomedical Sciences at San Antonio

Supervising Professor: Barry K. Norling, Ph.D.

An alternative to the metal ceramic restoration has been the resin veneer crown. The resin veneer offers the advantages of low cost, ability to make clinical repairs, lack of wear to opposing teeth. and ease of fabrication. Clinical use of the resin veneer has been limited due to problems associated with the resin including low resistance to abrasion, high water sorption, a high coefficient of thermal expansion, low proportional limit, and a low modulus of elasticity.

Improvements realized in composite resin technology have prompted the introduction of a number of composite resin veneering materials for prosthodontic applications. An electrochemical tin plating technique has been suggested for bonding composite veneering resins in lieu of conventional mechanical retention. The adhesive bonding of veneering resins to castings

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would prove beneficial by improving esthetics and eliminating the percolation of fluids at the resin-metal interface.

The purpose of this investigation was to examine the shear bond strength of a composite resin processed to gold and base metal specimens that were treated with an electro-chemical tin plating technique. Uniform specimens were cast in a gold alloy and a base metal. Conventional retentive beads on the gold alloy and electroetching of the base metal served as controls in this investigation.

A microfilled composite veneering resin, Isosit, was processed to the four experimental groups which were stored for 7 days at  $37^{\circ}$ C. Following thermal cycling between  $5^{\circ}$ C and  $60^{\circ}$ C, shear bond strengths were determined using an Instron testing machine.

The resulting resin shear bond strengths of the electrochemical tin plating technique with the gold and base metal specimens were significantly lower than those obtained with retentive beads or electroetching. The opaque layer of the electro-chemical tin plating technique was found intact on the metal specimens following shear bond testing. This would suggest that inadequate bonding occurred between the opaque layer and the Isosit resin. Therefore, the actual bond strength of the electro-chemical tin plating technique was not determined. A significant improvement in bond strength was found in a subsequent pilot study when Isosit fluid, a component of the

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Isosit system containing a cross-linking agent, was placed over the opaque layer prior to processing.



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## THE BOND STRENGTH OF COMPOSITE RESIN TO DENTAL CASTING ALLOYS USING AN ELECTRO-CHEMICAL TIN PLATING SYSTEM

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### THESIS

Presented to the Faculty of The University of Texas Graduate School of Biomedical Sciences at San Antonio in Partial Fulfillment . of the Requirements for the Degree of Master of Science

Ву

Keith S. Smith, II, D.D.S.

San Antonio, Texas

June, 1985

### DEDICATION

I wish to dedicate this document, and the effort it represents, to four people who fostered my personal and professional development.

My parents, Keith S. and Vivian M. Smith, have sacrificed, guided, tolerated, taught, financed and inspired me tirelessly for many years. It's time I said thank you.

John D. Adams, D.D.S., Professor and Chairman, Department of Fixed Prosthodontics, (Retired) West Virginia University School of Dentistry, Morgantown, West Virginia taught me patience, professionalism, dedication and a love of Prosthodontics.

Leah Grishkat, my wife, endured the turmoil a residency creates and remained an inspiration and my best friend throughout.

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### I. INTRODUCTION

Esthetic demands have been an important consideration in the evolution of materials used in restorative and prosthetic dentistry. An esthetic restorative material complemented with the required physical properties and recognized as an ideal replacement for tooth structure has yet to be developed. (Phillips, 1982)

The natural "tooth like" appearance of dental porcelain has led to its widespread use in restoring severely compromised teeth. (Craig, 1980; McLean, 1980) In addition to its esthetic qualities, properties such as long term color stability, a resistance to absorption of fluids, excellent gingival response, and a high wear resistance have equally contributed to the popularity of porcelain use. (Phillips, 1982) Porcelain is often reported to have a high compressive strength in relation to its inferior tensile strength. (Craig, 1980, Phillips, 1982) Overcoming this deficiency requires a strict adherence to preparation design to direct resulting forces in a favorable pattern and provide a sufficient bulk of porcelain in the final restoration. (Preston, 1977; McLean, 1980)

An improvement in the clinical performance of porcelain was accomplished with the introduction of a metal substructure. With a metal substructure, preparation design was simplified and a more flexible clinical application was realized. (Dykema et al., 1958; Mumford, 1965) The resulting metal ceramic restoration

has become the standard of care when fabricating esthetic complete crowns. (McLean, 1983)

The desirable property of wear resistance of dental porcelain can become a disadvantage and jeopardize the clinical success. Severe wear of opposing teeth or restorative materials has been reported when porcelain is placed in occlusion. (Mahalick et al., 1971; Monasky and Taylor, 1971) An additional disadvantage of using porcelain as a restorative material is encountered when porcelain fractures. The clinical repair of porcelain surfaces has been extremely difficult to accomplish and has met with little success. (Welsh and Schwab, 1977; Dent, 1979; Highton et al., 1979)

An alternative to the metal ceramic restoration has been the acrylic resin veneer crown. Beginning with its introduction in 1940, it gained widespread popularity due to simplicity of fabrication, ease of clinical repairs, lack of wear to opposing teeth, and relatively low initial cost. (Dykema et al., 1958; Peyton and Craig, 1963) Unfortunatley, a number of deficiencies have been reported with acrylic resin veneer crowns to limit their clinical application.

The low proportional limit and modulus of elasticity of acrylic resins may lead to excessive plastic deformation when placed under the stresses encounted in prosthetic applications. (Peyton and Craig, 1963) The lack of adhesion and considerable differential thermal expansion and contraction between the resin material and the metal substructure results in percolation of oral fluids and may contribute to early discoloration of the

restoration. (Phillips, 1982) A low resistance to toothbrush abrasion, high water sorption, porosity, and compromised esthetic results are additional deficiencies reported with this combination of materials. (Peyton and Craig, 1963; Tylman, 1970; Phillips, 1982) The requirement of developing mechanical retention for the resin veneer has also been criticized. (Tanaka et al., 1978; Tanaka et al., 1979)

The introduction of inorganic filler particles into a newly developed dimethacrylate resin base formulation gave birth to a new generation of direct resin restorative materials in the 1960's. (Bowen, 1962; Phillips, 1982) These so called, "composite resin" filling materials were found to have improved wear characteristics, including lower coefficients of thermal expansion, better color stability, less water scrption, and less polymerization shrinkage. (Peterson et al., 1966; Nuckles and Crosby, 1972) While these new composites have had little application in prosthetic dentistry, other than porcelain repair procedures (Dent, 1979), further modification in the late 1970's gave rise to a new class of potential crown and bridge veneering resin materials. (Raptis et al., 1979; Rupp, 1979)

This new class of resin material incorporates an extremely small filler particle consisting of pyrolytic or precipitated silica. (Phillips, 1982) Clinical research has demonstrated an improvement in surface smoothness with these "microfilled resins" over the larger glass filled composite resins. (Jorgensen and Asmussen, 1978; Mitchem and Gronas, 1982)

These advancements in composite resin technology have prompted the introduction of several new crown and bridge resin veneering materials. Initial laboratory studies have shown an improvement in physical properties with these materials when compared to conventional acrylic veneering resins. (Nathanson ... al., 1985; Osorio et al., 1985) The clinical behavior of these new materials, as assessed by long term clinical study, has yet to be determined.

Bertolotti and Napolitano (1983) reported a technique for bonding a composite veneering material to an electrolytically etched base metal alloy. The stated advantage of this technique would be the achievement of profound retention of the resin without traditional mechanical retentive designs. While electrolytic etching has been useful for bonding composites to base metal alloys, bonding to gold based alloys has not been successful with this technique. (Simonsen et al., 1983).

An alternative technique to the electrolytic etching process has been recently introduced which is reported to be effective in bonding composite resin to both base metal and gold based alloys. (Veen et al., 1984) This system utilizes an electrochemically applied tin plating on the metal substructure which is then oxidized to produce a surface to which resin materials can chemically bond. The ability to produce composite bond strengths comparable to those found with electrolytically etched base metals or produced with mechanical retention in gold based alloys has not been investigated.

The purpose of this investigation is to examine the shear bond strength of a composite crown and bridge veneering resin to a base metal alloy and a gold based alloy utilizing an electrochemical tin plating system.

### **II. LITERATURE REVIEW**

### A. Porcelain Restorations

An ideal restorative material for dental prosthetic applications has been sought for thousands of years. Ivory, human and animal teeth, bone, wood, gold, and porcelain have been employed with varying degrees of success. (Sproull, 1977) However, since the 1780's, porcelain alone or in combination with gold has endured as an acceptable esthetic material for dental use.

The esthetic qualities of porcelain are exemplified by its incorportion into a number of restorative techniques. Established procedures utilizing porcelain include porcelain jacket crowns, partial or complete dentures, porcelain inlays, metal ceramic restorations, and porcelain laminate veneers. (Tylman, 1970; Craig, 1980; Horn, 1983)

The complete porcelain crown has been described as the most esthetically pleasing restoration in dentistry. (Goldstein, 1976; Tylman and Malone, 1978) Utilizing this procedure, the clinician is able to reproduce many of the characteristics and peculiarities of an individual tooth within a given dentition. (Tylman and Malone, 1978)

Gingival tissues are very tolerant to contact with glazed porcelain surfaces. This favorable response has been attributed to the extreme smoothness of glazed porcelain which is resistant to plaque accumulation. (Podshadley, 1968; Karlsen, 1970) In an

in vivo investigation by Clayton and Green (1970), glazed porcelain was found superior to highly polished gold with respect to plaque accumulation.

An additional quality of porcelain is its ability to resist the normal wear that occurs in the oral environment. In a survey by Lehman (1967), porcelain jacket crowns were found to resist clinical wear extremely well.

Several disadvantages have been reported with the all porcelain crown and may account for its limited application in recent times. The tooth preparation for the all porcelain crown is often referred to as an arduous procedure. (Goldstein, 1976; Tylman and Malone, 1978) Because of the low tensile and shear strength of dental porcelain, a 90 degree shoulder preparation is required to support the porcelain and direct forces more towards a compressive mode. (Lehman and Hampson, 1962; Phillips, 1982; Craig, 1985) Preparing teeth sufficiently may result in pulpal damage, particularly in young dentitions with large pulp chambers. (Tylman and Malone, 1978)

Porcelain in combination with various metal substructures has been used as an alternative to the all porcelain crown for a number of years. As early as 1887, Land introduced a technique using a platinum foil matrix under an all porcelain crown. (Sproull, 1977) The incorporation of a foil matrix, which is bonded to the porcelain, has been suggested to improve the strength of the restoration. (McLean and Sced, 1976)

Following the introduction of the lost wax casting technique by Taggert in 1907, several techniques were reported which

modified stock porcelain denture teeth for use as veneers or facings on crowns and pontics. The Hollenback Crown featured a facing ground from a porcelain denture tooth that was cemented into a cast gold crown. (Sproull, 1977) Shooshan (1959) introduced the "reverse pin" modification of porcelain denture teeth for improved retention when used as pontic facings. Woolson in 1955 described a veneering technique that involved firing low fusing porcelain onto 24 karat gold foil that was swaged inside the labial window of an open faced cast gold crown.

These techniques allowed a broader application of the esthetic porcelain materials into areas of greater stress in the oral environment. Yet, due to their being extremely technique sensitive and time consuming, their success in dentistry was limited.

During the mid 1950's, improvements in casting alloy properties and ceramic systems stimulated interest in the concept of baking porcelain to gold alloys. Early attempts, as those described by Woolson (1955), resulted in porcelain crazing due to the different thermal coefficients of the two materials. Further research and refinements in alloys and porcelain composition led to compatible systems that had excellent clinical results. (Lyon et al., 1960; Shell and Nielsen, 1962; Weinstein et al., 1962)

The porcelain-bonded-to-metal restoration, or metal ceramic restoration, is an esthetic restoration providing the strength necessary to function in posterior areas. (Mumford, 1965; Tylman and Malone, 1978) In addition, the qualities of porcelain could now be combined with metal for use in fixed prosthetic

applications. (Tylman and Malone, 1978) Consequently, the metal ceramic restoration has become the restoration of choice in situations where esthetic full coverage is required for a single tooth. (Dresen, 1970; Phillips, 1982)

Despite its wide acclaim and clinical success, disadvantages associated with the metal ceramic crown have been reported. Problems such as accidental fracture of the porcelain or a failure of the porcelain bond have been a common occurrence. (Highton et al., 1979) Faced with such a dilemma, the clinician could remake the restoration totally or attempt to repair the defective crown. Repairs can be performed directly in the operatory, or indirectly using a laboratory procedure. Such a procedure was described by Welsh and Scwab (1977) to provide a more permanent repair. Their technique involved the fabrication of a metal ceramic overcasting that was cemented to the original restoration. Disadvantages of indirect porcelain repairs include additional treatment time and laboratory expenses.

Acrylic resin has been recommended in a direct repair technique for fractured porcelain restorations. (Tylman and Malone, 1978) Simplicity, minimal treatment time, and economics are reported advantages with this technique. However, disadvantages such as poor color stability, a high coefficient of thermal expansion, lack of adhesion, and poor abrasion resistance afforded only interim treatment. (Mumford, 1965; Phillips, 1982)

Organo-silane coupling agents used in combination with acrylic resin or composite resin formulations have been investigated for porcelain repair. (Eames et al., 1977; Eames and

Rogers, 1979; Highton et al., 1979; Dent, 1979; Burgar et al., 1980; Nowlin et al., 1981) Results vary with bond strengths reported as high as 48 per cent (Highton et al., 1979) and as low as 18 per cent (Nowlin et al., 1981) of the strength of unrepaired porcelain. While resin bond strength studies have been performed in the laboratory, their long term clinical performance has not been investigated.

The potential for porcelain restorations to be abrasive to opposing natural teeth or restorative materials when placed in occlusion has been reported. (Mahalick et al., 1971; Monasky and Taylor, 1971; Craig, 1985) In one study, enamel surfaces were found to wear at an accelerated rate when occluding against porcelain as compared to enamel occluding against gold. (Monasky and Taylor, 1971) This behavior has prompted recommendations by authorities to design the metal ceramic restoration such that porcelain is limited to an esthetic veneering material and not used to establish occlusal relationships. (Lau and Yamada, 1980)

### B. Acrylic Resin Veneered Restorations

Synthetic resins were rapidly accepted by the dental profession as esthetic veneering materials for fixed prosthodontic applications in the 1940's due to their ease of manipulation and low cost. The original resin compositions were acrylic, vinyl-acrylic polyester, and epoxy resin formulations. (Peyton and Craig, 1963) The acrylic and vinyl-acrylic materials were supplied as powder-liquid systems and required flasking as typically required for denture base resins. (Craig, 1985)

Further refinement of these materials resulted in replacement of methyl methacrylate monomers with less volatile dimethacrylates which could be rapidly processed to castings in an oven at 135  $^{\circ}$ C. (Craig, 1985)

The acrylic resins were described as translucent which in some instances allowed for excellent shade matching due to the pick up of colors from adjacent teeth. (Skinner and Phillips, 1968; Phillips, 1982) Esthetic masking of the metal substructure was achieved with a minimum veneer thickness of 0.6 mm (Long, 1968) to 1.0 mm (Johnston et al., 1971). This enabled a more conservative tooth preparation than that possible with the porcelain veneered restoration. (Long, 1968; Tylman, 1970) The veneer could be removed and easily replaced prior to restoration cementation if the shade was unsatisfactory. (Johnston et al., 1971) An additional reported advantage was the ability to clinically repair a fractured or defective facing. (Kornfeld, 1974; Rehany and Stern, 1977)

However, disadvantages of the synthetic resin veneering materials outnumbered the advantages and led to a decline in use of the original materials. Low reported values for proportional limit and modulus of elasticity of the resin necessitated a metal substructure for support and protection from occlusal loads to prevent plastic deformation. (Peyton and Craig, 1963; Phillips, 1982) The lack of an adhesive bond between the resin and casting required development of mechanical retention for the veneer. These included various undercut configurations, beads, loops and peripheral reverse bevels on the castings. (Miller, 1962;

Johnston et al., 1971; Kornfeld, 1974; Atsuta et al., 1974; Tanaka et al., 1978) A high differential in thermal expansion between the resin and metal, high water sorption, and high polymerization shrinkage were additional deficiencies reported. (Tylman, 1970; Johnston et al., 1971; Phillips, 1982)

A percolation of oral fluids at the resin-metal interface contributed to a discoloration of resins and a tarnish or corrosion of non-noble casting alloys. (Lamstein and Blechman, 1956; Soremark and Bergman, 1961; Peyton and Craig, 1963; Issa, 1968; Johnston et al., 1971) Plating the substructure with 24 karat gold prior to veneering was recommended to prevent this problem. (Johnston et al., 1971; Kornfeld, 1974)

The lack of abrasion resistance of resin veneers (Peyton and Craig, 1963; Tylman, 1970; Kornfeld, 1974) prompted some authors to recommend soft toothbrushes and mild dentifrices for patients with such restorations. (Johnston et al., 1971; Phillips, 1982)

Processing porosity has been reported to lead to an inherent weakness of the resin, a more opaque milky appearance, the potential for incubating microorganisms, and a potential for tissue irritation due to roughness. (Tylman, 1970) Swartz and Phillips (1957) have described a chronic inflammation of marginal gingiva resulting from seepage at the resin metal interface.

#### C. <u>Composite Resins</u>

The introduction of composite resins in the late 1950's effected an improvement in the clinical performance of resin materials over previous unfilled direct restorative resins.

(Bowen, 1962; Bowen, 1963, Leinfelder et al., 1975; Jordan et al., 1977) The composite resins exhibited higher wear resistance, lower water sorption, better color stability, lower polymerization shrinkage, and a lower coefficient of thermal expansion when compared to unfilled methylmethacrylate resins. (Paffenbarger, 1953; Macchi and Craig, 1969; Roberts et al., 1977)

The composite resin has been defined as a three dimensional system in which filler particles are bound by a coupling agent into a resin matrix formulation. The filler component, or inorganic phase, is typically fused quartz or a glass such as aluminosilicate, strontium, or barium glass. (Craig, 1981; Phillips, 1982) The mean filler particle size has varied from as small as 5 microns to as large as 30 microns. (Phillips, 1982; Lutz et al., 1983) The inorganic filler content of composites has been found to vary from 50 to 84 per cent by weight. (Craig, 1981; Lutz et al., 1983)

The resinous matrix, or organic phase, is reported to be a dimethacrylate such as the BIS-GMA monomer developed by Bowen (1962), modifications of BIS-GMA (Craig, 1985), or a urethane dimethacrylate (Braden, 1978). The coupling agent used to bind the filler particles to the matrix is normally an organosilane such as methacryloxypropyltrimethoxy silane. (Craig, 1981)

The activation of traditional composite resins is a chemical reaction whereby an initiator, such as benzoyl peroxide, comes into contact with an organic amine accelerator to initiate polymerization. (Craig, 1985) The initiator and accelerator are

brought together when the dentist begins mixing the two parters, or powder and liquid components of the composite resin.

A marked improvement was found in the clinical performance of the traditional filled composite resins when compared to the unfilled resins. However, with time the surface roughness of these materials was noticeably poorer and the wear resistance was not greatly improved. (Weitman and Eames, 1975; Deraded and Ehrnford, 1980; Itoh et al., 1981) Abell et al. (1981) has reported that the loss of traditional composite material proceeds at a rate of 1 micron per day when placed in occlusal function.

A new concept in the filler component of composite resins was introduced in the late 1970's. The incorporation of submicron pyrolytic or precipitated silica particles, ranging in size from .04 to .06 microns, gave rise to a "microfilled" or "polishable" class of composite resins. (Phillips, 1982) Without the much larger quartz and glass fillers, surface smoothness was greatly improved and staining and plaque accumulation was decreased. (Fan et al., 1979; Dennison et al., 1981; Christensen et al., 1982; Tyas, 1982)

The microfilled composites are reported to be less filled than the traditional composites. The greater surface area of the small silica particles requires a greater volume of matrix material and has resulted in filler contents of only 30 to 50 per cent for microfilled resins. (Phillips, 1982; Craig, 1985) The increased resin volume is responsible for a higher water sorption and greater coefficient of thermal expansion than for traditional composites. (Swartz et al., 1982; Waknine et al., 1985) The

significance of this higher volume of resin is not totally understood. However, poorer color stability (Swartz et al., 1982) and greater marginal leakage (Hembree, 1983) have been found with microfilled composites in laboratory testing. Robinson et al. (1983) reported on the clinical results of eight composite resins at three years and found traditional filled composites to be superior to microfilled resins with respect to color stability.

As an alternative to the benzoyl peroxide chemical initiator system, either ultraviolet or visible light may be used to initiate the polymerization of composite resins. With ultraviolet light initiation, an organic compound that generates free radicals when exposed to ultraviolet light, such as benzoin alkyl ether, is substituted for the benzoyl peroxide. (Craig, 1981) A diketone such as camphoroquinone is used in combination with an organic amine for the visible light systems. (Craig, 1981; Phillips, 1982)

Advantages reported with the light activated composites have been numerous with respect to the clinical placement of resin restorations. Ease of handling, shorter curing time, decreased porosity, and more rapid finishing are reported examples. (Brown, 1983; Blankenau et al., 1983; Swartz et al., 1983) More recent investigations have suggested an improvement in the physical properties of composite resins with visible light activation. Rice et al. (1984) reported on the improved wear resistance of a resin that was light activated versus chemically activated. A more complete degree of polymerization has also been suggested to

occur with light activated resins. (Lutz et al. 1983; McCabo, 1985)

A three year clinical investigation of light cured versus chemically cured microfilled resins by Murray et al. (1985) resulted in 47.3 per cent of the chemically cured resins in need of replacement. Ninety-two per cent of those were due to bulk discoloration. In contrast, only 4.8 per cent of the light cured restorations were in need of replacement.

A new generation of resin based veneering materials for prosthodontic applications has been introduced as a result of technological advancements realized with composite resins. These materials are reported to have improved properties over previous acrylic resin veneering materials and offer a better alternative to the use of porcelain. (Michl, 1978; Rager, 1983)

Nathanson et al. (1985) observed an increased resistance to toothbrush abrasion with five new resin veneering materials when compared to an acrylic resin material. The tensile strengths however, were not as high as the conventional resin. The invitro color stability as reported by Osorio et al. (1985) was comparable for four new resin materials and an acrylic resin material.

### D. <u>Electrolytic Etching of Alloys</u>

The bonding of composite resins to electrolytically etched alloys was developed as part of a conservative technique for the replacement of missing teeth. This so called "Maryland Bridge" or acid etched resin bonded retainer technique involved the

bonding of a prosthesis with composite resin to abutment teeth which have been acid etched. (McLaughlin, 1981; Thompson and Livaditis, 1982; Simonsen et al., 1983) The electrolytic etching process has been shown to produce greater bond strengths than a previous technique which utilized perforations in the metal. (Rochette, 1973; Livaditis and Thompson, 1982)

While a greater resin metal bond strength is possible with electrolytic etching, a number of variables involved with the process have been shown to influence the results. The type of alloy used, type of electrolyte, proper agitation of electrolyte, porcelain firing, and surface area to be etched, have been shown to influence bond strengths. (Thompson, 1982; Thompson and Del Castillo, 1982; Al-Shamary et al., 1983)

The electrolytic etching process involves the use of a low voltage DC power supply which is connected to a stainless steel cathode while the metal casting serves as the anode. The areas of the casting that are not to be etched are masked with a suitable medium and the anodic casting is immersed in an appropriate electrolyte (e.g., 10% sulphuric acid) while a current of constant density is passed through the electrodes for a specific time. Following etching, the metal framework is usually cleaned with an acid (18% hydrochloric acid) in an ultrasonic unit. (Simonsen et al., 1983)

The greatest success with electrolytic etching has been found with the nickel-chrome-beryllium containing base metal alloys. (Simonsen et al., 1983) With modifications to the etching process, recent reports suggest chrome-cobalt (Thompson

et al., 1984) and silver-palladium (Jensson et al., 1985) alloys are adequate for resin bonded retainers.

The use of electrolytically etched base metal as a foundation for the application of a composite resin veneer was reported by Bertolotti and Napolitano in 1983. The advantage of this technique was the elimination of the need for bulky mechanical retention, as provided by retentive beads. An earlier investigation by Dunn and Reisbick (1976) demonstrated that ceramic coatings could be bonded to chrome-cobalt alloys using pitting corrosion. The bond was reported to be mechanical and related to the increased surface area. A subsequent investigation by Tanaka et al. (1979) demonstrated that acrylic facings could be bonded to a nickel chromium copper alloy using a similar pitting corrosion process. The resulting tensile bond strengths with the pitting corrosion process were greater than that produced using conventional retentive beads.

An alternative technique to the electrolytic etching process has been introduced for use with both base metal and gold based alloys. (Veen et al., 1984) Tin plating is applied by an electro-chemical process and then oxidized to produce a surface to which composite resin materials are able to bond. The ability to produce sufficient bond strengths without the need for mechanical retentive designs would be an attractive clinical feature allowing additional depth for esthetic development in resin faced crowns. The ability to produce composite bond

base metals or produced with mechanical retention in gold based alloys has not been investigated.

### **III. RESEARCH OBJECTIVES**

Because of the potential advantages afforded with the ability to adhesively bond composite resin to casting alloys, this investigation was undertaken to:

1. Evaluate the bond strengths produced between a base metal alloy and a crown and bridge resin using the OVS Bonding System.

2. Determine the bond strength produced between a gold alloy and a crown and bridge resin using the OVS Bonding System.

3. Evaluate the relative bond strengths produced by the OVS Bonding System and conventional mechanical retention in each alloy system.

### IV. MATERIALS AND METHODS

### A. <u>Overview</u>

Twenty specimens each of a gold alloy and a base metal alloy were used in this study. Ten of the gold alloy specimens were cast with traditional retentive beads. The remainder of the gold specimens and all base metal specimens were cast without retentive devices. A composite resin veneering material was processed onto the specimens. Resin retention on the gold specimens was achieved by either mechanically engaging retentive beads or chemically bonding to an oxidized tin plated surface. On the base metal specimens, retention was achieved by either mechanically engaging microretentions produced by electroetching or by chemically bonding to an oxidized tin plated surface.

### B. Metal Specimen Preparation

Forty plastic patterns were prepared by scoring 10 millimeter square grids on 1.5 millimeter thick Ubex plastic sheet (Plastic Supply Co., San Antonio, TX). The patterns broken from the sheet were deburred and flattened on one side by wet grinding on 400 grit silicon carbide paper. Ten patterns were masked by placing an index card with a 5 millimeter diameter hole over the center portion of the pattern. A thin layer of Krylon spray enamel (Borden Inc., Columbus, OH) was applied to the exposed surface. While the enamel was still wet, 20 Microretention beads (Isosit, Ivoclar, USA, Inc., San Marcos, CA) were individually placed to create a 4 by 5 bead square pattern with a diagonal dimension of 5 millimeters (Figure 1). The beads were carried to the pattern surface on the moistened red tip of a red/blue pencil.

Wax sprues 1.5 millimeters in diameter by 5 millimeters long were attached to one corner of each of the 40 patterns with sticky wax. Six patterns were waxed to a 4 millimeter diameter wax runner bar. Two bars were attached at their centers to a crucible former of a 2.5 inch diameter by 1.875 inch high casting ring (Figure 2). All sprued patterns were sprayed with Wax Pattern Cleaner (Jelenko, Armonk, NY) and allowed to dry.

Twenty patterns, cast in Rexillium III (Jeneric Gold Co., Wallingford, CT), were invested in vacuum mixed Hi Temp investment (Whip Mix Corp., Louisville, KY) using the recommended water to powder ratio of 9.5 cc to 60 grams. The rings were lined with one layer of wet asbestos. The investment was allowed to bench set for one hour prior to being placed in a cold oven. The temperature of the oven was brought to 1800  $^{o}$ F and held at that temperature for one hour. The alloy was melted with a natural gas-oxygen torch and cast in a broken arm centrifugal casting machine.

The patterns cast in Baker Inlay Extra Hard Type IV gold (Engelhard Ind., Carteret, NJ) were invested in vacuum mixed Beauty Cast investment (Whip Mix Corp., Louisville, KY) using the recommended water to powder ratio of 14.5 cc to 50 grams. The rings were lined with a single layer of wet asbestos. Immediately after the ring was filled with investment, it was
Figure 1. Plastic Patterns with Micro-Retention Beads





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placed in a 100  $^{\circ}$ F water bath and allowed to set for 45 minutes. The rings were then placed in a cold oven and the temperature raised to 1000  $^{\circ}$ F and held at that temperature for one hour. The alloy was melted with a natural gas-air torch and cast in a broken arm centrifugal casting machine.

After removal of investment, all castings were cleaned by air abrading with 50 micron aluminum oxide and the sprues removed with a separating disk. The castings were visually inspected to ensure surface smoothness (absence of nodules), absence of distortion, and when present, uniformity of retentive beads (Figure 3).

The 10 Rexillium III specimens to be electroetched were spot welded in groups of four, four, and two to 20 centimeter lengths of .030 stainless steel orthodontic wire (Rocky Mountain Dental Products, Denver, CO) (Figure 4). All surfaces except the one centimeter square faces to be etched were masked with fingernail polish (Maybelline Co., North Little Rock, AR) as were the wires except for the last two centimeters. The exposed faces were again aluminum oxide air abraded immediately prior to electroetching. Each group was suspended in a stirred 10 percent sulfuric acid bath (Figure 5). The specimens were made anodic with respect to the stainless steel beaker cathode. A variable DC power supply (Hewlett-Packard Co., Berkeley Heights, NJ) was used to control the current density at  $300 \text{ ma/cm}^2$ . The total etch time was three minutes. After electroetching, the specimens were rinsed in deionized water and ultrasonically cleaned for 10 minutes in 18 percent hydrochloric acid to remove the black film

Figure 3. Completed Castings Removed from Investment



Figure 4. Spot Welded Castings to Stainless Steel Wire for Electroetching

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Figure 5. Electroetching in 10% Sulfuric Acid

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generated by electroetching. (Figure 6) Each etched surface was then examined under a stereo microscope (Olympus, Tokyo, Japan) to ensure uniformity of etch and development of the desired retentive dendritic etch pattern.

The remaining 10 Rexillium III castings and the gold castings without retentive beads were prepared for resin bonding with the OVS tin plating system (Dentsply International, York, PA). The specimens were again air abraded with 50 micron aluminum oxide, steam cleaned, and rinsed with distilled water. While still damp, each specimen was attached to the cathode clip of the OVS applicator (Figure 7). With the OVS power supply set to 9 volts, the plating solution saturated felt point anode was brushed over the specimen surface until a uniform dark gray tin electroplate was developed (Figure 8). Each specimen was then rinsed in distilled water, dried, and oxidized by immersion in the OVS oxidation solution. Oxidation was confirmed by lightening of the plated surface (Figure 9). The specimens were then rinsed with distilled water and air dried.

The 10 gold castings with retentive beads were prepared for resin bonding by air abrading with 50 micron aluminum oxide and steam cleaning.

## C. <u>Resin Veneering</u>

All specimens were veneered with the Isosit N System (Ivoclar Inc., Monrovia, CA) following the instructions in the manufactuer's technique manual. The central portions of the 10 bead retention gold specimens and the 10 electroetched Rexillium

Figure 6. Castings Prior to Electroetching, Following Electroetching, and After Cleaning in 10% Hydrochloric Acid



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Figure 7. Tin Plating of Gold Casting With OVS System



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Figure 8. Appearance of Tin Plated Gold Casting



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Figure 9. Confirmation of Oxidation by Lightened Alloy Surface



III specimens were first painted with a thin layer of opaqueadhesive (Figure 10) followed by two thin layers of opaque made by mixing the opaque powder with the opaque-adhesive liquid. (Figure 11). A 4.85 millimeter internal diameter by 6 millimeter long teflon tube was positioned centrally on each specimen. The Isosit body resin was condensed into the tube using the blunt end of a single ended periodontal scaler. The specimens were cured in the Isosit curing unit at 120°C for 5 minutes under 6 bar pressure.

The 20 tin plated specimens were opaqued with the OVS K&B opaque (Dentsply Corp., York, PA). The opaque was painted in two thin layers on the central portion of the specimens. The Isosit body resin was then applied and cured as described above. (Figure 12)

Prior to bond strength testing, all specimens were stored in distilled water for 7 days at  $37^{\circ}$ C and then thermocycled between 60 and  $5^{\circ}$ C for 560 cycles with a 120 second cycle time.

#### D. Shear Bond Testing

Shear bond strengths were measured using an Instron universal testing machine (Instron Corp., Canton, MA). Each specimen was clamped in a specially constructed vise which oriented the bonding surface vertically (Figure 13). The vice was placed on the Instron load cell platen and the specimen face manually brought into light contact with the shear blade. The blade was made of cobalt cemented tungsten carbide and beveled on the surface facing away from the specimen to produce a 0.5

Figure 10. Application of Opaque-Adhesive to Castings



Figure 11. Application of Two Layers of Opaque-Adhesive Containing Opaque Powder

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Figure 12. Isosit Resin Condensed into Teflon Tubes

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Figure 13. Device Used to Hold Specimens During Shear Bond Tests



## Figure 14. Loading of Specimens in the Instron Testing Machine With Beveled Blade

Machine with Beveled Blade

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millimeter wide surface in contact with the cylindrical resin specimen (Figure 14). The cross-head speed of the Instron was 1 mm/min. A 50 kilonewton load cell was used and the chart speed was set at 100 mm/minute. The force required to fracture the bonded resin from the metal specimen was recorded in Newtons (N). The bond strengths were converted to Mega Pascals by dividing the force in Newtons by the surface area in square millimeters.

## E. Statistical Analysis

The resulting bond strengths for the specimens were analyzed using a one-way analysis of variance (ANOVA). The variable was the retention system employed (gold alloy or base metal and the beads, OVS, or electroetch). A Fisher's Least Significant Difference analysis was performed on all groups to identify significant differences at a probability level of p <.05.

## V. RESULTS

The means and standard deviations of the shear bond strength values are listed in Table 1 and graphically represented in Figure 15. The raw data for all the test specimens are presented in the Appendix. One specimen from the OVS System/Gold Alloy group, and one specimen from the Electroetch/Base Metal group were lost during testing due to improper positioning in the Instron.

The results of the one-way analysis of variance are shown in Table 2. The variable of type of retentive system was found to be highly significant (p <.001). The Fisher's Least Significant Difference post-hoc analysis of the means revealed two significantly different groups (p <.05) within the four test groups. There was no significant difference between the Retentive Bead/Gold Alloy and the Electroetch/Base Metal group. These two groups had significantly greater shear bond strength values than the two OVS groups which were not significantly different from each other.

| RETENTIVE SYSTEM           | N  | MEAN<br>(MPa) | (S.D.) |
|----------------------------|----|---------------|--------|
|                            | 10 | 6 10          | (0.76) |
| Retentive Beads/Gold Alloy | 10 | 6.40          | (0.76) |
| OVS System/Gold Alloy      | 9  | 1.24          | (0.99) |
| OVS System/Base Metal      | 10 | 0.96          | (0.44) |
| Electroetch/Base Metal     | 9  | 5.33          | (1.32) |

# TABLE 1.STATISTICAL MEANS AND STANDARD DEVIATIONS<br/>OF COMPOSITE SHEAR BOND STRENGTHS

Figure 15. Shear Bond Strength Results Graphically Represented


TABLE 2. SUMMARY TABLE FOR ONE-WAY ANALYSIS OF VARIANCE COMPARING RETENTIVE SYSTEM GROUPS

Treatment Variable = Retentive System Between: SS= 7716.027 DF= 3 MS= 25705.343 F= 89.393 \* Within: SS= 9776.809 DF= 34 MS= 287.553 Total: SS= 86892.837 DF= 37

\* P <.001

#### VI DISCUSSION

The composite resin veneered crown has been suggested as an alternative to the metal ceramic restoration. (Meyer and Eichner, 1980) Compatibility with the opposing teeth, ability to make clinical repairs, and lower cost are advantages over the metal ceramic restoration.

Although the tooth preparation for resin veneered crowns has been reported to be more conservative than for metal ceramic crowns, gaining esthetic results requires a certain bulk of resin material. An initial opaquing layer is required to mask the metal substructure followed by an adequate thickness of composite to gain the necessary translucency. Conventional retentive designs, such as beads, limit the available space for esthetic development. In many instances the health of the supporting tissues may be compromised due to overcontouring.

The ability to form a chemical adhesive bond between the resin and the metal substructure, as proposed by the OVS tin plating technique, would eliminate the need for mechanical retention. In addition, adhesive bonding would prevent the percolation of oral fluids between the resin and metal found with convention retentive techniques. (Lamstein and Blechman, 1956; Johnson et al., 1971)

The shear bond strength found in this study with the OVS system was significantly less than that obtained with retentive beads. An examination of the metal specimens following the bond

tests revealed that with both gold and base metal specimens, failure occurred at the resin-opaque interface (Figure 16). Therefore, the actual bond strength of the OVS system was not determined. Rather, the values reported are representative of the bond strength of the Isosit resin to the OVS opaque layer.

The Isosit resin material was selected for this study because the manufacturer of the OVS system identified it as a compatible resin. The Isosit N resin is a recently introduced modification of the original Isosit material which is no longer commercially available. Apparently, the new formulation may not be as compatible as the original Isosit material. A previous study by Veen et al. (1984) demonstrated tensile bond strengths that averaged approximately 9.0 MPa when Silar, a BIS-GMA microfilled resin was bonded using the OVS system. In contrast, the Isosit N resin is a urethane dimethacrylate resin and may not develop the strength possible with BIS-GMA resin materials.

The Isosit N system recommends the use of a special fluid during processing which is used to cover theresin surface, thereby preventing air inhibition during polymerization. The Isosit fluid is activated just prior to use with a cross-linking agent supplied with the system. A pilot investigation was performed using this special fluid between the OVS opaque layer and the Isosit resin. The resulting shear bond strength values are shown in Table 3. In addition to the greatly improved bond strengths, an examination of the metal specimens following the shear tests revealed a failure at the opaque-metal interface. (Figure 17) The Isosit fluid is reported to contain peroxides, a

Figure 16. Example of Specimen Failure at the Resin-Opaque Interface



## TABLE 3.SHEAR BOND STRENGTHS OF ISOSIT TO GOLD<br/>ALLOY WHEN ISOSIT FLUID WAS USED

| Specimen                      | Bond Strength<br>(MPa)  |
|-------------------------------|-------------------------|
| ***************************** | *********************** |
| 1.                            | 10.5                    |
| 2.                            | 12.2                    |
| 3.                            | 11.8                    |
| 4.                            | 7.6                     |
| 5.                            | 7.4                     |
| MEAN STRENGTH                 | 9.9                     |
| STANDARD DEVIATION            | 2.28                    |

Figure 17. Magnification of Bond Area (30x) Demonstrating Specimen Failure at the Opaque-Metal Interface When Isosit Fluid was Used

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trace of monomer, and an oxygen inhibitor. The cross-linking paste contains uncured Isosit monomer and silicon dioxide. The presence of these components would appear to enhance the bonding between the OVS opaque layer and the urethane dimethacrylate resin in the Isosit material.

The base metal alloy was investigated in this study because of the interest in using this alloy for resin bonded retainers and as a possible alternative to gold based alloys in prosthodontics. The bond strengths found with the Isosit resin to the electroetched base metal were considerably lower than previously reported for composite to base metal. (Simonsen et al., 1983) This is probably due to the lower tensile strength of the Isosit resin when compared to conventional composite resins. (Nathanson et al., 1985) Additional evidence for this interpretation arises from examination of the failure mode during shear strength testing. Cohesive failure was observed within the Isosit resin which resulted in a layer of resin firmy attached to the electroetched metal surfaces after shear testing.

The bonding of resin to electroetched base metal has been shown to be highly technique sensitive and results are often variable. (Sloan et al., 1983) The ability to form a chemical adhesive bond to the base metal may offer a more predictable technique. Further research is required to evaluate the OVS tin plating system with base metals using either modifications of the Isosit resin technique or different resin formulations.

The ability of the OVS system to resist the percolation of oral fluids at the metal-resin interface should be investigated.

An in-vitro microleakage study would be a logical step towards answering this question. Finally, a comprehensive clinical investigation would be in order to assess the long term durability of this system prior to any recommendations for routine patient care.

#### VII. SUMMARY

The bonding of a composite veneering resin, Isosit N, to gold and base metal castings with the OVS electro-chemical tin plating system has been investigated and compared to conventional retentive mechanisms. Shear bond strengths were determined on one week old specimens following thermal cycling between  $5^{\circ}$ C and  $60^{\circ}$ C. The following conclusions and recommendations can be made from the results of this study:

1. The bond strength of Isosit N to gold or base metal castings using the OVS system was significantly less than with retentive beads on gold or electroetched base metal.

2. Inadequate bonding was observed between the Isosit N resin and the OVS opaque layer.

3. Applying Isosit Fluid activated with a crosslinking paste to the OVS opaque layer prior to the application of the Isosit resin greatly improved the shear bond strength.

4. Further research is required to evaluate the bond strength of other resin veneering materials to noble and base metals using the OVS system.

APPENDIX

#### TABLE 1

#### RAW DATA OF SHEAR BOND STRENGTHS

#### (Values are in Newtons)

With Gold Alloy:

| Retentive Beads/Isosit                  | <b>OVS/Isosit</b> |
|---|-------------------|
| *************************************** |                   |
| 150.0                                   | 18.5              |
| 116.0                                   | 70.4              |
| 102.0                                   | 20.4              |
| 112.0                                   | 20.0              |
| 120.0                                   | 15.2              |
| 113.5                                   | 6.0               |
| 130.0                                   | 18.0              |
| · 103.0                                 | 18.0              |
| 115.0                                   | 19.8              |
| 125.0                                   |                   |

With Base Metal Alloy:

| Electroetch/Isosit     | OVS/Isosit |  |
|------------------------|------------|--|
| ********************** |            |  |
| 97.0                   | 24.0       |  |
| 113.0                  | 8.0        |  |
| 95.0                   | 30.1       |  |
| 112.5                  | 16.0       |  |
| 119.5                  | 29.0       |  |
| 54.0                   | 15.0       |  |
| 78.0                   | 9.0        |  |
| 133.6                  | 17.0       |  |
| 83.0                   | 9.0        |  |
|                        | 20.0       |  |

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Woolson, A.H. 1955. Restorations made of porcelain baked on gold. J. Prosthet. Dent., 5: 65.

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In the summer of 1970 he entered the West Virginia University School of Dentistry, in Morgantown, West Virginia, and recieved the Doctor of Dental Surgery degree in May, 1974.

Upon graduation from dental school he was appointed to the faculty of the West Virginia University School of Dentistry in the department of Crown and Bridge as an Instructor. He was promoted to Assistant Professor in 1977.

In 1978, he resigned from the West Virginia University School of Dentistry and accepted a commission in the United States Air Force and was assigned to Brooks AFB, San Antonio, Texas. He was married to Leah Ann Grishkat during this year. A subsequent assignment to Lackland AFB in San Antonio followed.

In July, 1982, he entered the University of Texas Health Science Center at San Antonio for graduate studies in Prosthodontics. In 1984 he was admitted as a candidate for the Master of Science Degree at the Graduate School of Biomedical

#### VITA

Sciences. He recieved a certificate in Prosthodontics in June, 1985.

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He has been assigned the position of Assistant Director Area Dental Laboratory, Kadena AFB, Okinawa. He will assume this position in August, 1985.



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