# A MATERIALS EVALUATION OF TEN DIRECT BONDING SYSTEMS UTILIZING POLYCARBONATE BRACKETS

by

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OF TEN DIRECT BONDING SYSTEMS

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Abstract

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An *in vitro* investigation was undertaken to determine significant differences among various dental materials available for possible use in an orthodontic direct bonding system. The materials included three dental restorative resins--two composite resins, Enamelite and Nuvafil, and an unfilled resin, Sevriton; a UV polymerized pit and fissure sealant, Nuva-Seal; and six resin systems currently available for direct bonding--Nuva-Seal-Bracket Bond, Unitek, Directon with and without a Sealant, Orthomite, and O.I.S.

Edgewise polycarbonate brackets were bonded to thirty-six teeth per adhesive material. Tensile, shear and peel stress modes were applied to the brackets; one-half of the sample was tested after one day storage in 37°C distilled water, the remaining samples were tested after one month storage.

Bond strengths were determined,and a scanning electron microscope was used to evaluate the fracture patterns of various bond failures.

The results of this report may be summarized as follows:

(1) There was no statistically significant difference among the systems specifically designed as orthodontic adhesives.

(2) The two composite resin systems and the sealant did not bond satisfactorily to the polycarbonate brackets.

(3) Bond failures included adhesive, cohesive, and a combination of adhesive and cohesive fracture patterns.

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DEDICATED

TO

My Wife Carol, for her love, patience and understanding;

My Children Sharon, Christine, Jeffrey, and Richard,

who have allowed me to retain a sense of humor;

My Parents, for their love and encouragement.

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# INTRODUCTION

Tooth movement during orthodontic therapy can be controlled precisely with the use of a fixed appliance system. Auxiliaries, commonly referred to as brackets, tubes, spurs, etc., are soldered or welded to a piece of band material, which is subsequently adapted circumferentially to a tooth.

Wires can be adapted to the dental arch and attached to the brackets, allowing the tooth to be guided into the desired position. This system is approximately one hundred years old!

Angle (1907) credited a Dr. Magill of Erie, Pennsylvania, as the first practitioner to use a plain band attached to a tooth by a dental cement--oxychloride of zinc. The cement enabled the band to resist displacement under the ordinary strain of tooth movement. Although the materials utilized in the fabrication of the bands and brackets have changed from precious metals (such as gold and platinum) to stainless steel alloys, the most popular form of orthodontic therapy in North America still requires the banding of at least twenty teeth (Moyers, 1958).

## Disadvantages of Banding

Placing a metal band directly on a tooth is not an easy task. First, it usually requires sufficient separation of the teeth to accommodate the thickness of the band material. Although separation

can be easily accomplished by using brass wire ligature, stainless steel springs or small rubber strips, the procedure is usually somewhat painful and may subsequently affect future patient cooperation (Altemus, 1968).

Second, the bands must be closely adapted to the tooth, so as to minimize the cement margin, prevent occlusal interferences, and avoid insult and injury to the gingival tissue. Adapting and cementing bands is a time consuming and expensive procedure (Newman, 1964, 1965, 1969b; Bernstein, 1965; Retief and Dreyer, 1967; Newman et al., 1968).

A third consideration is the thickness of the band material and cement. Newman (1969b) is of the opinion that metal bands and cement impinge on the arch length. The band material may vary in thickness from .003 to .007 inches (Moyers, 1958 and Graber, 1961). If, in a four bicuspid extraction case, bands were placed from first molar to first molar in each dental arch, the arch length would increase by approximately 2.5 millimeters. This "potential space" must be closed during the retention phase of treatment. Some of the precise tooth control, gained during active treatment, may be lost because of band spaces that require closing (Cunat, 1972).

A fourth disadvantage is esthetics. Metal bands placed circumferentially about a tooth, especially an anterior tooth, are not esthetic (Newman, 1965, 1966, 1969a; Grenadier et al., 1969; Schwartz, 1971). This factor may discourage a patient

with a malocclusion who would, were it not for the metal bands, seek orthodontic treatment.

A fifth and final area of concern is the possible gingival irritation and enamel decalcification that may arise due to metal circumferential bands (Newman, 1969b). Schluger (1968) feels that banding is a definite, though possibly unavoidable, shortcoming of the orthodontist's treatment--"one of the prices of therapy." Graber (1961) feels that the appliances may produce "inflammation, redness, swelling and pain" in the gingival tissues. Band irritation, inadequate plaque control, and, in children, possible hormonal disturbances due to puberty, are three important factors leading to gingivitis and periodontal complications in orthodontic patients. Tejedor and Sears (1972) review the clinical effects of orthodontics on the periodontium and confirm the findings of Newman, Schluger and Graber.

Enamel decalcification may be caused by loose or ill-fitting bands which may go unnoticed by the patient and practitioner (Newman, 1965, 1966). As the cement luting agent disintegrates in the oral fluids, plaque may form in exposed areas and cause enamel decalcification. Graber (1961) reports that these "decalcification lines will persist for the lifetime of the individual." He feels that the lingual surfaces of the upper incisors and the buccal and lingual surfaces of the molars are especially susceptible to decalcification and advises that periodic recementation of these banded teeth may be indicated.

In conclusion, placing a band on a tooth has certain disadvantages: (1) it is associated with discomfort and pain, (2) it is time consuming and expensive, (3) it impinges on arch length, (4) esthetics are compromised, and (5) gingival health and enamel integrity are jeopardized.

## Advantages of Banding

In the past, banding offered the only available means of attaching a bracket to a tooth. This is no longer true. Newman (1966) reported a case treated with a fixed appliance utilizing plastic brackets attached directly to the enamel. An "acrylic type adhesive" was the principle bonding agent.

Is there any advantage in using a circumferential band over a directly bonded bracket?

Two advantages are proposed: (1) a circumferential band covers the tooth, possibly decreasing a patient's susceptibility to caries (Graber, 1961) and (2) the efficiency of the bracket may be increased by utilizing a circumferential band which "surrounds" the entire crown, rather than a bonded attachment which contacts only one surface of the crown (Phelps, 1972).

The first proposal waits correlation with future clinical investigation. Bonded brackets are relatively new to orthodontic therapy and, until recently, were used only for limited tooth movement. Caries incidence related to a bonded orthodontic attachment has not been reported.

As to the second proposal, one may consider the enamel as circumferentially covering the dentine core. An attachment bonded to this "enamel band" should have the same efficiency as a metal band around the crown (Johnston, 1972).

## The Bonded Orthodontic Bracket

An orthodontic bracket--especially a plastic bracket--bonded to the enamel surface may offer several advantages to both the orthodontist and patient. Bonded plastic brackets may (1) improve esthetics, (2) reduce chair time for the doctor (auxiliary personnel could play an important role), (3) reduce cost, (4) diminish the degree of gingival irritation, (5) decrease enamel decalcification, (6) eliminate the need for prebanding separation, (7) eliminate an iatrogenic increase in arch length during treatment due to "band spaces", (8) facilitate the application of brackets to teeth which are partially erupted, (9) increase the patient's cooperation and acceptance of the appliance, and (10) increase the demand for orthodontic care (Newman, 1964, 1966, 1969a, 1969b; Newman et al., 1965, 1968; Cohl et al., 1972).

At the present time the main disadvantage of a bonded orthodontic bracket is strength--both of the bracket and the bond. The strength and durability are affected by fluids in the oral cavity, masticatory and mechanical stresses, and rapid pH and temperature changes (Newman et al., 1968).

However, new materials--classified by their manufacturers as "dental adhesives"--are continually being offered to the dental profession. While some of these materials are produced especially for bonding orthodontic brackets to a tooth, still others are promoted as dental "restoratives" or pit and fissure sealants. With continued interest in this area, the orthodontist can hope for acceptable bonding agents. The question that remains, then, is which material, if any, will be best suited for bonding brackets to the teeth?

## Purpose of the Investigation

The present investigation examines a broad spectrum of dental "adhesives" that are currently available to the dental profession. Materials testing has been performed *in vitro* to determine the strength of the material. That is, tensile, shear and peeling forces were applied to polycarbonate plastic brackets bonded to extracted human teeth.

A second examination of the dental "adhesive" includes a scanning electron microscope study of the types of joint failures that are produced.

# REVIEW OF THE LITERATURE

Since this study involves materials which are referred to as "adhesives," basic principles concerning adhesion to tooth structure will be presented.

## Principles of Adhesion

Buonocore (1963) and Retief (1970a) have described adhesion as the "molecular attraction exerted between molecules at an interface." Through molecular attraction via physical or chemical forces, an adhesive substance is capable of adhering (sticking) to a surface and bonding that surface to another material (Brauer, 1961). In this case, the materials involve unlike molecules. The term cohesion is reserved for attraction between molecules of the same kind (Retief, 1970a).

Chemical forces involved in adhesion involve covalent, ionic, or metallic bonds (Buonocore, 1963; Retief, 1970a). These forces are intramolecular and are referred to as primary forces. Electrons are shared or transferred, resulting in a strong bond.

Physical forces include Van der Waal's and hydrogen bonding (Buonocore, 1963; Retief, 1970a). These are intermolecular attractive forces and are termed secondary. Physical forces depend upon the existence of dipoles, induced dipoles and a nonpolar dispersion effect due to the interaction between the random

motions of electrons, ions or atoms. Hydrogen bonding is also a special type of dipole-dipole interaction.

Physical forces are generally rapid, reversible and require no activation energy. Surface molecules remain chemically intact. However, chemical attraction, which is preceded by adsorption, is usually slower due to an activation energy that is required. These strong attractions are not easily reversed (Retief, 1970a).

Although a dentist restoring a carious or fractured tooth may be interested in an adhesive that would chemically bond to tooth structure, an orthodontist requires an adhesive which is reversible. Once the malocclusion has been treated, the adhesive should be able to be removed without injury to the tooth. An adhesive utilizing physical attraction forces would be indicated.

While adhesion can exist only if the adhesive and adherend (or substrate) are in intimate contact, physical forces may be sufficient for good adhesion (Eirich, 1961; Buonocore, 1963; Retief, 1970a; Baier, 1970; Brauer and Huget, 1972; Eick et al., 1972). These forces will often be stronger than the cohesive force of the materials; bond failure would generally involve a cohesive break rather than fracturing at the adhesive-adherend interface (Zisman, 1961; Retief, 1970a; Baier, 1970).

In addition to physical and chemical forces, other factors are involved in adhesion. These include: (1) surface contact phenomena, (2) wetting, (3) contact angle, (4) surface tension,

(5) thickness of the adhesive layer, (6) thermodynamic considerations and (7) setting conditions (Buonocore, 1963; Retief, 1970a).

Surface contact considerations depend upon the smoothness of the materials involved. Surfaces which are smooth at the atomic level will adhere spontaneously. Practically speaking, such smooth surfaces are unattainable, especially in dental applications. Consequently, adhesives are utilized to unite two materials together. Ideally, the adhesive would fill the irregularities and voids of the surfaces. Surface contact also includes surface topography (Eick et al., 1972). Surface topography affects the wetting process and the fracture pattern of an adhesive joint--even though a cohesive failure may be involved.

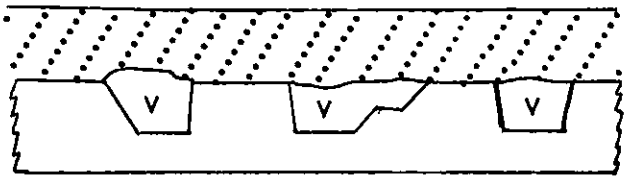
Buonocore (1963) described wetting as a "manifestation of the attractive forces between molecules of adhesive and adherend." Strong attractive forces would produce wetting. Huntsberger (1966) has further stated that complete wetting occurs when the maximum possible number of interfacial contacts is obtained (Figure 1).

Zisman (1966) prefers to define wetting in relation to the contact angle ($\theta$). Retief (1970a) denotes this angle as that "formed between the surface of the adhesive drop and the surface of the adherend upon which it is resting." Figure 2, after Zisman (1961), demonstrates this. A small contact angle indicates greater wetting of the adherend by the adhesive. When $\theta = 0$, the liquid will spread spontaneously over a solid surface. Poor adhesion results with large contact angles.

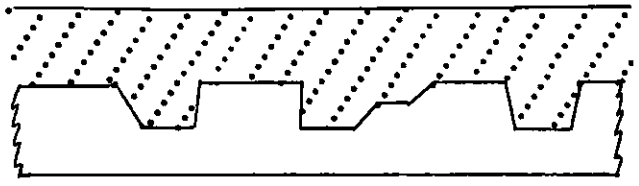
Liquids and solids demonstrate a "tension" in the surface region. Although molecules within the material are attracted equally on all sides by surrounding molecules, those in the surface region are not attracted from above. These molecules are pulled downward due to the unequal force produced. The stronger the force pulling the molecules downward into the bulk of material, the greater the surface tension--expressed in dynes/centimeter (Retief, 1970a).

The thickness of the adhesive layer is another important factor in obtaining and maintaining adhesion (Buonocore, 1963; Retief, 1970a; Patrick, 1961). As the adhesive increases in thickness, the joint strength decreases (Figure 3). Buonocore (1963) and Retief (1970a) propose a number of reasons: (1) a thin layer may produce fewer imperfections in the joint, (2) a thick layer may deform and fracture readily, and (3) a thin layer will produce less shrinkage during polymerization.

Young in 1805 described thermodynamic conditions for adhesion that exist at the phase boundary of a liquid in static equilibrium with a solid (cited by Retief, 1970a). He expressed these conditions in terms of surface tension vectors $\vec{\gamma}$ shown in Figure 2. Zisman (1961) defines a critical surface tension ($\gamma_C$) and utilizes it to generalize the mechanism of wetting. An organic liquid of low specific surface energy will freely wet a clean, smooth, high energy surface provided the critical surface tension ($\gamma_C$) is not surpassed by the liquid. Hence, the surface tension of the liquid




a. Substrate incompletely wetted (v=voids)

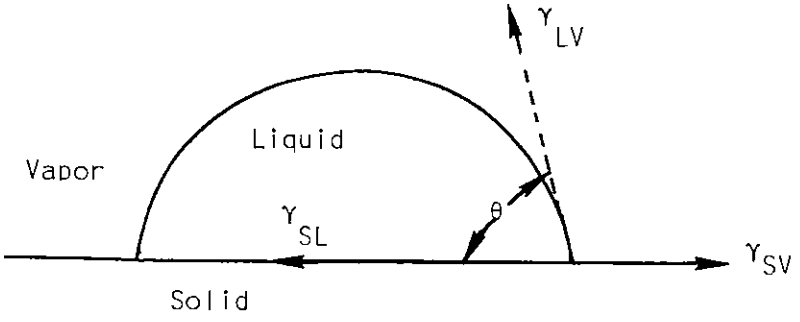


b. Substrate completely wetted

Wetting of a solid substrate by a fluid adhesive

(After Huntsberger, 1966)

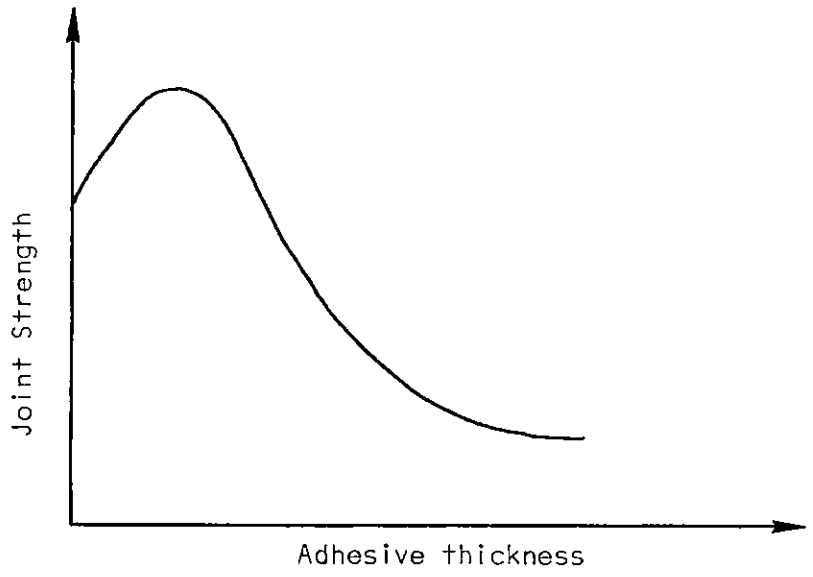
Figure 1




Contact angle

(After Zisman, 1961)

Figure 2




Joint strength as a function of

adhesive thickness

(After Patrick, 1961)

Figure 3

must be lower than $\gamma_C$ in order to wet a surface completely (Retief, 1970a).

Retief (1970b) maintains that adhesion is best obtained and maintained under dry conditions. Industrial adhesives can usually approach this goal; bonding to hard dental structures, however, continually involves an aqueous environment. The effect of the oral environment on adhesion will be discussed in the next section.

In conclusion, the principles involved in adhesion are numerous and complex. Basically, adhesion is most satisfactorily obtained when the liquid adhesive completely wets the solid substrate. The contact angle ($\theta$) and surface tension ($\gamma$) are measures of the wetting process. Wetting is enhanced when the

free surface energy of the liquid adhesive is less than that of the solid substrate ($\gamma_{SV}>\gamma_{LV}$). Complete wetting will produce a joint that is stronger than the bonded materials, i.e., a cohesive failure will occur, as opposed to fracture at the adhesive-substrate interface. Also, a thin joint will be stronger than a thick one.

## The Oral Environment

An adhesive material that is intended for use in the oral cavity must withstand many insults: (1) continual moisture and high humidity, (2) pH fluctuations, (3) temperature extremes, (4) variable stresses and (5) possible bacterial attack.

Moisture within the oral cavity cannot be eliminated. Cannon (1961) and Retief (1970b) observed that even if a vacuum pump were applied to the mouth, the teeth could not be dried thoroughly at room temperature.

Drying a tooth cannot be maintained. Bergman (1963) demonstrated that fluid flows continually from the pulp to the enamel surface. This flow is spontaneous and does not require a rise in intrapulpal pressure.

Although Linden in 1968 showed that fluid flow to the enamel surface does decrease as permanent teeth become older, an adhesive must contend with at least a monolayer of water at the adhesive-enamel interface. The adhesive, therefore, would have to either compete with the water or "bond" to it. Cannon (1961) feels that the presence of a monolayer of water on the enamel surface makes

a true chemical bond to enamel an impossibility. He proposed that prosthetic materials bond to enamel only through hydrogen bonding. Accordingly, the water would aid in establishing adhesion.

Fluctuations in pH are a second factor that an adhesive must be able to withstand. Newman (1964) reports that while the pH of saliva may range from 6.8 - 7.2, that of fluids taken into the mouth may vary greatly. For example, carbonated beverages can have a pH of 1.8, while milk of magnesia may have a pH of 10.

Temperature extremes are a third insult to which adhesives are exposed. While the oral temperature (98.6° F.) may tend to buffer the temperature changes of ingested foods and liquids, ice cream (35° F.) and hot tea (145° F.) may produce sudden and instantaneous temperature extremes. Differences as great as 149° F. have been reported (Skinner and Phillips, 1960; Newman, 1964, 1969b; Retief, 1970b).

Retief (1970b) emphasizes that the instantaneous changes in temperature may be of significance if there is a pronounced difference in the coefficient of thermal expansion of tooth and adhesive. Stress concentration within the adhesive material may develop during temperature cycling.

Stresses applied to an adhesive may include masticatory, soft tissue, and orthodontic. Skinner and Phillips (1960) report that the average static biting force is 170 pounds. Newman (1964) states that the stresses of mastication upon a single cusp on a posterior tooth may vary from 10,000 - 30,000 pounds per square inch (p.s.i.).

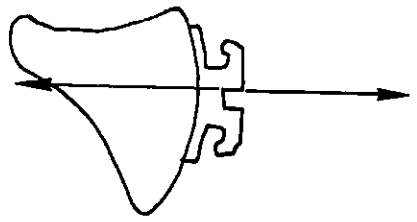
Anterior stresses vary from 1,000 - 4,000 p.s.i. Furthermore, these masticatory stresses involve combinations of tensile, shear, compressive, and torquing modes (Buonocore, 1970b).

In addition to the forces of mastication, Kydd and his associates in 1963 demonstrated that the lips exert from 0.071 to 2.78 p.s.i. of pressure on a maxillary central incisor. Adhesives used to place an orthodontic attachment on an anterior tooth would have to consider these forces.

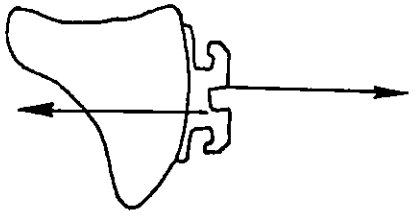
In 1964, Newman illustrated various types of loading in adhesive joints between an attachment and an incisor (Figure 4). He suggests that the forces involved in orthodontic treatment should not exceed one half pound on a tooth, and a load of ten pounds or 200 p.s.i. is the "maximum which probably occurs under clinical conditions." (Newman, 1965). Hence, the shear strength of the joint after 30-45 days in water should be at least 400 - 500 p.s.i. (Newman et al., 1965).

Finally, adhesives are exposed to bacterial and hydrolytic attack (Buonocore, 1970b). Although resins cured at high temperatures demonstrate excellent resistance to bacterial degradation, room temperature cured resin adhesives are probably less resistant to bacterial breakdown.

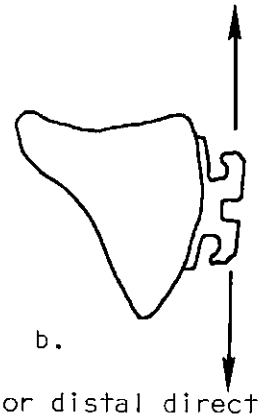
An adhesive must readily adapt to the oral environment in order to maintain the adhesive joint.



Tension: Incisor being moved in a labial direction by the archwire

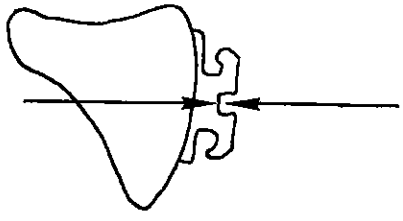


a.

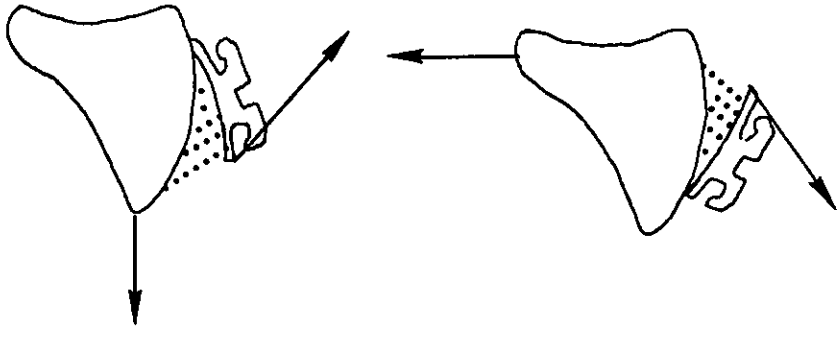


b.

Shear: Incisor being moved in a mesial or distal direction (a.) or incisal or gingival direction (b.)



Compression: Incisor being moved in a lingual direction



Peeling (a.) and cleavage (b.) forces should be avoided

Types of Loading in Adhesive Joints

(After Newman, 1964)

Figure 4

While conditions in the oral cavity constitute a major obstacle in the development of an ideal adhesive, there are three other areas which must be understood: (1) the substrate, or enamel surface, (2) the adhesive material and (3) the adherend, or orthodontic attachment.

## The Substrate: The Enamel Surface

Dental enamel is a thin, protective cap, covering the underlying dentine and pulp. It is composed of both an organic and inorganic phase, the latter comprising 96 percent of the weight in mature enamel. The inorganic portion consists of hydroxyapatite crystals of varying size organized into a basic structural unit--the enamel prism (Scott and O'Neil, 1961; Nikiforuk and Sognnaes, 1966; Buonocore, 1970b; Scott et al., 1971; Brauer and Huget, 1972; Orban, 1972). Covered by salivary products and various organic films, enamel in vivo maintains a hydrophobic, low energy surface (Zisman, 1961; Meckel, 1965; Newman and Sharpe, 1966; Buonocore, 1955, 1970b). Although these films may be removed with abrasives, an organic-rich enamel pellicle still presents a low energy surface inhibiting adhesive potential (Orban, 1972).

Rose and his associates (1955), screening seventeen cements and resins for adhesion to human dentine and enamel, demonstrated that cleaning the tooth surfaces with tissue and 95 percent alcohol was inadequate. None of the materials tested maintained adhesion after prolonged immersion in water.

In 1961 Swanson and Beck, testing Eastman 910[1] adhesive, concluded from their in vivo studies that pumicing the teeth prior to applying the adhesive was not satisfactory--"mechanical retention" was necessary.

If high energy surfaces are required for bonding (Baier, 1970), and if pumicing, abrading and drying the enamel do not fulfill this requirement (Glantz, 1969), what procedure will enhance adhesion?

## Conditioning the Enamel Surface

Buonocore (1955) demonstrated in vivo the improved adhesion obtained when enamel was pre-treated with phosphoric acid prior to placing acrylic filling materials. He concluded that increased adhesion was due to several factors: (1) an increase in the surface area produced by the acid-etch, (2) an exposed organic framework, (3) removal of an inert enamel layer, exposing a fresh reactive surface, and (4) precipitation of polar phosphate groups (from the phosphoric acid) onto the enamel surface. These factors all contribute to the wettability of the enamel surface.

In 1961, Swanson and Beck demonstrated that etching enamel for receiving cyanoacrylate provides better results than untreated enamel, provided the surface is clean and dry.

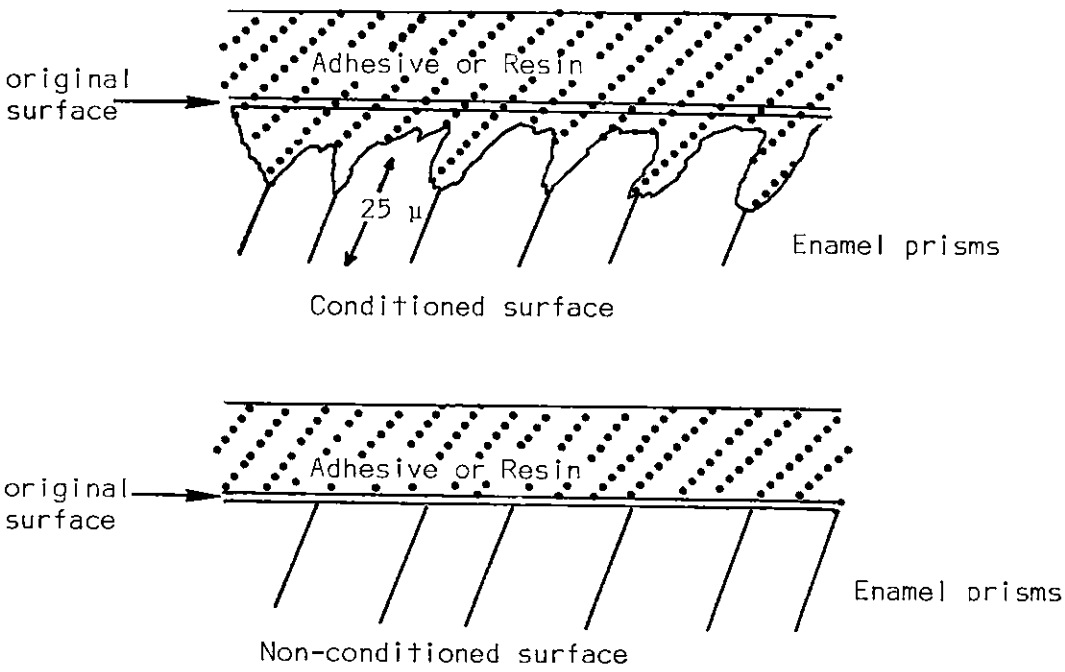
In 1966, Newman and Sharpe evaluated the contact angles ($\theta$) of various liquids on tooth surfaces. Distilled water and an epoxy resin spread spontaneously ($\theta \tilde{=} 0°$) over enamel surfaces treated with

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[1]Eastman Chemical Products, Kingsport, Tennessee

85 percent phosphoric acid. Teeth untreated or pumiced yielded contact angles of 36° - 70°, inadequate for satisfactory wetting. The authors concluded that the acid pre-treatment converts the low energy, hydrophobic enamel surface into a high energy, hydrophilic surface.

Gwinnett and Matsui in 1967 elaborated on the etching process and mechanical retention produced. Eight different materials were applied to acid-conditioned enamel. Optical and electron microscopy studies revealed the formation of "tags"--extensions of the materials 10 - 25 microns into the enamel surface.




The penetration of an adhesive into conditioned and non-conditioned enamel surfaces

Figure 5

(After Buonocore et al., 1968)

Buonocore and his associates (1968) investigated the "prism-like" tags previously reported (Gwinnett and Matsui, 1967). Adhesion of various resins to the enamel surface was enhanced by etching. The authors diagrammatically represented the penetration of the resin into the enamel surface by Figure 5.

Gwinnett and Buonocore (1965) evaluated various conditioning agents for enamel, including: (1) phosphoric acid (10 - 15 percent, with and without modifiers), (2) 4 and 10 percent solutions of hydrochloric acid with sodium fluoride, (3) silicate cement liquid, and (4) salts of ethylene diamine tetraacetic acid (EDTA). The enamel was etched to a depth of approximately 10 microns following a two minute application of the liquids. Mechanical retention was produced when the acid-etch created pores or spaces for an adhesive.

Poole and Johnson (1967), using a scanning electron microscope, examined enamel surfaces etched with various agents--formic, lactic and hydrochloric acids, as well as EDTA. The acids appeared to dissolve the core of the prism, while the EDTA dissolved the periphery, leaving the central portion intact. Their findings on the acid-etch process confirmed the work of Kanthak and Benedict, reported 35 years previously (1932), using a simple reflecting microscope.

Hoffman and his associates (1969) confirmed the work of Poole and Johnson (1967): acid caused demineralization of the enamel prism cores while EDTA removed the peripheries.

Kateley (1970) compared the effects of 50 percent citric acid and 50 percent phosphoric acid on ground enamel specimens. Citric

acid produced a " . . . smoother outlining of the prisms. . . ," while the phosphoric acid gave ". . . a very jagged surface. . . ."

In 1968, Mulholland and DeShazer examined the effects of altering the pH and molarity of various acid pre-treatment solutions. The conditioning was used in conjunction with bonding orthodontic attachments to enamel with Addent 35.[2] Monovalent acids (hydrochloric and hydrofluoric) and polyvalent acids (phosphoric and aspartic) with different pH values were studied. There was a definite correlation between an increase in bond strength and a decrease in pH of the acidic pre-treatment solution.

While most investigators now advocate an acid pre-treatment prior to bonding an adhesive to enamel, the etchant and procedure may vary. These include: (1) phosphoric acid incorporated in a zinc phosphate cement liquid or silicate cement liquid (Gwinnett and Matsui, 1967; Christensen, 1970a and b; Nagel, 1972), (2) a specified concentration of phosphoric acid, usually 50-85 percent by volume (Phillips, 1970; Lee et al., 1971; Sharp and Grenoble, 1971; Laswell et al., 1971; Gwinnett, 1971, 1973a; Retief, 1973), (3) an attenuated solution of 50 percent phosphoric acid with 7 percent (by weight) zinc oxide (Gwinnett and Buonocore, 1965; Cueto and Buonocore, 1967; Buonocore et al., 1968; Welk, 1970; Buonocore, 1970b and c; Sheykholeslam and Buonocore, 1972), and (4) a 50 percent solution of citric acid (Kateley, 1970; Lee et al., 1971; Lee and Swartz, 1971).

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[2] 3M Company, St. Paul, Minnesota

Brauer and Huget (1972) conclude that pre-treating the enamel surface with an acid is a necessary step in achieving satisfactory adhesion. Surface debris is removed, a more wettable surface is produced, and the effective surface area available for bonding is increased. Retention is improved due to the mechanical interlocking that takes place between the demineralized portion of the enamel prism and the adhesive.

## Remineralization of the Etched Enamel Surface

The demineralized enamel surface produced by acid etching has concerned some investigators in the past. Bernstein (1965), while bonding cyanoacrylate to extracted human teeth, did not etch the enamel surfaces in his investigations because of the ". . . criticisms that some members of the profession have for this procedure."

Buonocore in 1955 felt that acid treatments were clinically safe, Newman (1965) answered the criticisms of etching by noting that dental cements contain 35-55 percent phosphoric acid. Also, the cements utilized in band cementation may have a pH of 1.6; this may last for as long as 15-45 minutes after cementation.

Newman and Sharpe (1966) were able to ". . . retrieve the surface existing before phosphoric acid treatment, by pumicing. . . ." The etched, hydrophilic surface became hydrophobic.

In 1971, Newman and Facq used a scanning electron microscope to study the effects of adhesive systems on tooth surfaces. They

found that an etched enamel surface can be restored to its original appearance by pumicing.

In 1973, Retief showed scanning electron micrographs to demonstrate the in vivo recovery of etched enamel. After an etched tooth was exposed to the oral environment for two weeks, recovery was almost complete.

Lenz and Muhlemann (1963a and b), noted that the pattern of prism endings characteristic of an etched surface, disappeared in samples exposed to the oral environment for at least two days. They concluded that the surface was rendered smooth by deposition of a salivary pellicle, rather than actual remineralization.

Newman (1969b) utilized interferometer measurements to show a significant reduction in the ". . . maximum peak-to-valley heights due to acid pre-treatment." He confirmed the views of Lenz and Muhlemann (1963a and b): etching had been eliminated by a pellicle of salivary origin.

Retief and his associates in 1970 reported that the etched enamel regained a normal appearance within a few days. They felt that abrasion or remineralization may have caused the normal appearance.

Wei (1970) studied enamel remineralization with an electron microprobe. He concluded that acid etching was confined to the first 9 - 10 microns from the enamel surface, and calcium and phosphorous were lost during the etching process. When a calcifying solution was applied to the treated enamel, the mineral

content was restored; remineralization had taken place.

Lee and his co-workers in 1970 reported on the in vivo remineralization of enamel etched for one minute with 50 percent citric acid. A replica technic in conjunction with scanning electron microscope studies demonstrated ". . . essentially complete remineralization within 72 hours."

Albert and Grenoble (1971) also found the scanning electron microscope a useful tool in examining etched enamel. They found that as early as one hour after etching, enamel exposed to the oral environment was able to redeposit calcium phosphate from the saliva. After a few days, the etched enamel was normal.

In conclusion, the recovery process of an etched enamel surface appears to include: (1) abrasion or erosion due to artificial means (pumicing), mastication, or the oral environment; (2) deposition of a salivary pellicle which obliterates the prism end pattern, and (3) remineralization due to a deposition of calcium phosphate salts from the saliva.

## The Adhesive: A Materials Analysis

A variety of materials with adhesive potential have been studied in the past. Investigators have tried to produce an ideal dental adhesive, useful in restorative dentistry, preventive dentistry (sealants) and orthodontics. Buonocore (1963) felt that an ideal adhesive should meet the following requirements:

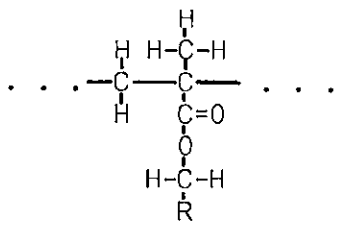
> . . . produce a lasting bond with enamel and dentin; polymerize rapidly near body temperature, with little or no shrinkage; be sufficiently cross-linked to minimize expansion or water absorption; have sufficient strength to resist masticatory forces; have the same coefficient of thermal expansion as tooth structure; be innocuous to the pulp and oral tissues, and be resistant to degradation in the oral environment.

The ideal adhesive material has not been developed; however, various organic polymers are available that fulfill many of Buonocore's requirements. These include acrylics, epoxides, composites of acrylics and epoxides, polyacrylates (polycarboxylate cements), cyanoacrylates, and polyurethanes.

## Acrylics

Acrylic resins have been utilized in dentistry for over 35 years (Bowen, 1956). These resins are derivatives of ethylene ($CH_2=CH_2$) and contain a vinyl group in the structure. The monomer units polymerize via a series of chemical reactions (Phillips, 1973).

The most popular acrylic resin in restorative dentistry is based on the polymethyl methacrylate system, whose monomer is depicted in Figure 6.



Methyl methacrylate (monomer)

Figure 6

The acrylic resins usually contain a catalyst, such as benzoyl peroxide and an accelerator. The accelerator may be an amine system or a sulfinic acid derivative (Stanford, 1971).

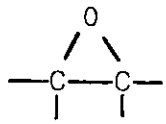
Unfortunately, these resins have serious shortcomings: (1) shrinkage on polymerization, (2) a high coefficient of thermal expansion, (3) color instability, and, in restorative applications, (4) recurrent decay around margins (Stanford, 1971). In addition, these materials lack adhesion to tooth structure.

These disadvantages prompted Buonocore in 1955 to explore a new approach to promoting the adhesion of acrylic materials to teeth. Phosphoric acid (85 percent) was used to pre-treat enamel surfaces prior to placing acrylic beads on human teeth. While acrylic beads on untreated surfaces were retained for an average of 10 hours, those on treated surfaces remained for approximately 1070 hours. The adhesiveness of the resin to enamel had improved, although it is inherently weak.

## Epoxides

A second adhesive system includes the epoxy resins. First synthesized in 1937, these resins have high adhesive strengths, low curing shrinkages, and moderate hardening temperatures. From a dental standpoint, however, the advantages are outweighed by long curing times, poor color stability and high water sorption (Lee, 1969; Brauer and Huget, 1972).

Epoxy resins are characterized by an epoxy group:



The ring is unstable and tends to open and combine with other compounds (Phillips, 1973).

As with polymethyl methacrylate systems, expoxides do not spontaneously adhere to enamel. The enamel surface must be pretreated with a conditioning or priming agent to obtain satisfactory adhesion.

## Composite Resins

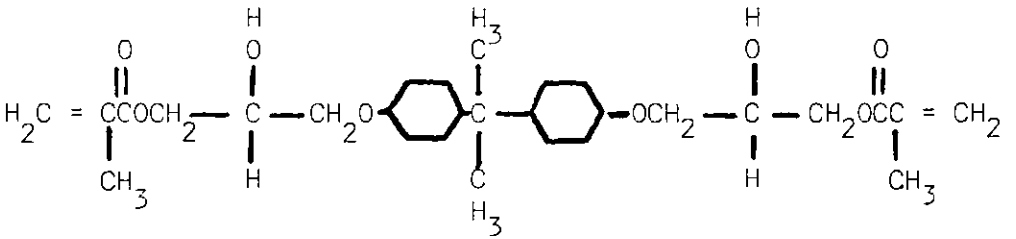
Stanford (1971) reports that Bowen synthesized and patented the first composite resin for dental application in 1962. The matrix resin is a reaction product of an epoxy resin (bisphenol-A) and an acrylic (glycidyl methacrylate). Two possible reactions to form the adhesive monomer are demonstrated in Figure 7. Silane coupling agents are used to bind various inorganic fillers (glass beads or rods, natural silica or borosilicate powders) to the resin matrix (Greener et al., 1972).

Composite resins are in general more rigid, less abrasive, and more stable than unfilled resins. Water sorption is clinically insignificant, polymerization shrinkage is low and the working and setting times are shorter.

Although the benzoyl peroxide-amine (BPA) system initiates the polymerization in most composites (Greener et al., 1972),

1. Diglycidal ether of Bisphenol-A (DGEBA) + Methacrylic acid → BIS-GMA monomer

2. Bisphenol-A + Glycidal Methacrylate → BIS-GMA monomer



BIS-GMA

Figure 7

(After Phillips, 1973)

Buonocore (1970a) substituted benzoin methyl ether, an ultraviolet light-sensitive composition, as catalyst. An important prerequisite is that a source of ultraviolet light must be provided.

Once again, adhesion is promoted in the composite resins primarily through the use of a conditioning agent,

## Polyacrylate (polycarboxylate) Cements

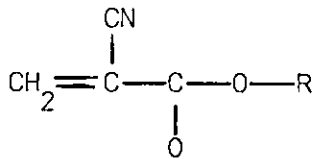
In 1968 Smith introduced a new cement to dentistry; zinc oxide was synthesized with polyacrylic acid to form a polycarboxylate cement. He reported that the cement has a high tensile strength (higher than that of most zinc phosphate cements), demonstrates no significant pulpal irritation, and adheres to tooth structure.

In 1972, Smith claimed that scanning electron micrographs indicate a chemical interaction between the cement and underlying calcified tissues. The enamel surface had been prepared by pumicing; no acid conditioning agents were utilized.

Mizrahi and Smith (1969, 1971) and Mizrahi (1972) reported that metal orthodontic brackets could be bonded directly to enamel, without an acid pre-treatment, utilizing polycarboxylate cements. Moreover, this is the only technique that does not incorporate an acid etchant in the procedure.

## Cyanoacrylates

Cyanoacrylates have been used as adhesives by many investigators. They may be chemically represented by the following structure:



Swanson and Beck (1961) investigated the adhesiveness of a methyl-2-cyanoacrylate[3] to enamel. Even with an acid pre-treatment, water immersion caused the bonds to fail.

Cueto and Buonocore (1967) used a cyanoacrylate as a pit and fissure sealant. While their early investigations appeared promising, the sealant was not retained as expected.

Investigations by Bernstein (1965) and Crabb and Wilson (1971) confirmed a lack of satisfactory adhesion for the cyanoacrylates to enamel for orthodontic uses. Furthermore, Greener and his associates (1972) reported that the cyanoacrylates cause pulpal irritation, thus curtailing their use for many dental applications.

## Polyurethanes

The utilization of polyurethanes as a dental material has not been investigated as extensively as have other organic polymers. Lee and Swartz (1971) and Lee and his co-workers (1971) studied various polyurethane resins as potential cavity liners and pit and fissure sealants. They showed satisfactory adhesion to enamel and dentine, and adequate retention in the pits and fissures, for periods up to one year.

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[3]Eastman 910

A more recent study (Rock, 1972) indicated that a polyurethane pit and fissure sealant[4] retained contact with enamel in only 10 percent of the cases after six months. This compared to 91 percent intact for a Bis-GMA sealant[5].

As a group, the use of polyurethanes as orthodontic direct bonding agents has not been reported. However, all of the other organic polymer groups have been investigated.

## The Adherend: The Orthodontic Bracket

Attachments or brackets for orthodontic direct bonding systems are made of metal (stainless steel) or a resin product. Metal brackets have been used and reported by many investigators (Sadler, 1958; Mitchell, 1967; Retief and Dreyer, 1967; Mulholland and DeShazer, 1968; Mizrahi and Smith, 1969, 1971; Retief et al., 1970; Smaha and Voth, 1972; Mizrahi, 1972; Dijkman and Retief, 1972; Rensch, 1973). While these brackets are strong, they must be constructed so as to mechanically lock into the adhesive; no chemical bonding takes place between the metal bracket and adhesive. Also, the metal brackets defeat one of the reasons for placing direct bonded attachments: esthetics.

Newman and his associates in 1965 fabricated cold cure polymethyl methacrylate brackets. Some of the attachments were

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[4]Epoxylite 9070, Lee Pharmaceuticals, South El Monte, California

[5]Nuva-Seal, L.D. Caulk Co., Milford, Delaware

custom-made, fitting individual tooth dies, while others were cut from plastic sheets.

Newman (1965) also tested a plexiglass attachment; his *in vitro* studies indicated that an epoxy adhesive did not bond satisfactorily to the attachment.

A thermoplastic resin, polycarbonate, was introduced as an orthodontic attachment by Newman and his co-workers in 1968. Newman (1969b) reported that he had used many materials as brackets, including acrylics, epoxies, polysulfones, nylons, polyphenylene oxides, and polycarbonates. The polycarbonate resins appeared to give the best results as orthodontic attachments.

Polycarbonates have many desirable characteristics; they are (1) nontoxic, (2) easily fabricated, (3) resistant to abrasion, (4) clear, (5) odorless and relatively tasteless, (6) resistant to creep and deformation under load, and (7) resistant to impact forces.

Polycarbonates do possess a decided disadvantage: they absorb moisture. While the dimensional change that accompanies water absorption is undesirable, the change is small (0.15 - 0.36 percent), and polycarbonates remain the only type of plastic attachments manufactured for direct bonding systems.

The treatment of the polycarbonate attachment varies with techniques and operators. Newman (1969a) recommended "roughening" the bonding surface of the bracket base with an emery board prior to bonding. Cohl and his associates (1972) etched the bracket

base with methacrylic acid for two minutes. Dean (1972), Sciaretta (1972), and Dietz (1972) recommended an alcohol wipe to eliminate possible contaminants. Touching the bracket without the aid of an instrument was contraindicated.

## Testing Procedures

Testing adhesive joints has been an area of concern to dental investigators. To date, however, standard testing procedures or guidelines have not been established; almost every investigation examines adhesion differently (Brauer and Huget, 1972).

Buonocore (1955) used his thumbnail while attempting to remove bonded acrylic beads.

Rose and his associates (1955) prepared enamel and dentine samples to fit into a hollow end testing device. The specimens were secured with pins and acrylic material; two opposing ground enamel surfaces were held together with one of seventeen adhesive materials. A ". . . simple tensile device. . ." measured in pounds the tensile force required to separate the samples.

Swartz and Phillips (1955) used a Tinius Olsen testing machine to examine the adhesiveness of an acrylic resin and zinc phosphate cement to enamel.

Sadler (1958) used tensile, shear, and torquing forces to test his adhesive systems. Force was gradually applied until the bracket was dislodged.

Swanson and Beck (1961) used a simple tensile device for the *in vitro* portion of their investigation.

> It consisted of a heavy laboratory stand with adjustable clamps so that a perpendicular force could be applied to the bonded surfaces. A platform was attached to the free end of the chain, and weights were added in 100 gm. increments. The breaking point was recorded in kilograms.

Bernstein (1965) used both a tension meter and digital pressure to dislodge his samples.

Newman (1965) and Newman and his associates (1968) used a Chatillon Model DTC Universal Tester. A tensile force at the rate of one pound per second was applied until the joint failed.

Mitchell (1967) determined his bond strengths by ". . . chipping and peeling them with a knife." Also, a shear test was performed by (1) placing the tooth with the bonded attachment in a vise, (2) tying a wire (.016 in.) into the bracket, (3) applying finger pressure to the free end of the wire, and (4) dislodging the bracket.

Retief and Dreyer (1967) applied a tensile disruptive force to their specimens.

Mulholland and DeShazer (1968) applied a tensile force to custom-made metal brackets bonded to enamel with a composite resin. A commercial jar lid supported the specimens. The brackets were placed over a hole in the lid. Force was applied to a bracket by adding ". . . number one buckshot and/or gram weights. . ." to a foam-lined bucket until the bond failed.

Retief and his co-workers (1970), in order to test tensile stresses, imbedded wire loops into their bonded epoxy samples. Twenty-four hours later, a container was fixed to the wire loop;

the container was gradually filled with water until the bond broke. The weight of the container and its contents were recorded; bond strength was expressed in pounds per square inch.

Recently, investigators have begun using an Instron Testing machine to study bond strengths (Mizrahi and Smith, 1969; Sheykholeslam, 1970; Laswell et al., 1971; Crabb and Wilson, 1971; Cohl et al., 1972; Rensch, 1973).

Specimens are mounted on a cross-head that moves at a constant rate preset by the investigator. Tensile, shear, and modified tensile and shear stresses can be applied to a sample. While this procedure is precise, strain rates may vary from study to study.

Lee and Swartz (1970a) summarized the problems involved in the various test methods:

> . . . researchers in the field have not been able to compare data readily, and often have difficulty in assessing the significance of their own data because of the interplay of tensile, peel, and flexural forces in the test devices and jigs.

Since most of these tests are performed in vitro, they can only approximate the conditions that exist within the oral environment (Lee and Swartz, 1970a).

## The Scanning Electron Microscope

Examination of adhesive materials utilizing a scanning electron microscope (SEM) has become extremely helpful within the past few years. Lee and Swartz (1970b) utilized a SEM to examine surface irregularities and cavity margin adaptation of four composite resins.

Sharp and Grenoble (1971) used a SEM to examine the extent of resin penetration into acid-etched enamel surfaces.

Cohl and his associates (1972) examined bond failures after testing a Bis-GMA direct bonding system. Fracture patterns and locations at the interfaces were studied.

It is not the purpose of this discussion to examine in detail the techniques of using a SEM; Boyde and Wood (1969) and Boyde and Knight (1969) reported and reviewed specimen preparation and SEM technique, particularly for dental applications. The Workshop on Biological Specimen Preparation for SEM of the I.I.T.R.I., Fifth Annual SEM Symposium, offers an excellent overall review.

Scanning electron microscopy offers many advantages over light or transmission microscopy: (1) good depth of field, (2) direct examination of a sample without replication, (3) no requirement of thin specimen sections, (4) broad magnification range, (5) three-dimensional imaging, and (6) good resolution (Everhart and Hayes, 1972).

Since a great deal of information is derived from scanning electron micrographs, most of the materials investigations that are reported include a SEM evaluation.

## MATERIALS AND METHODS

### Sample

Human incisors, canines, and bicuspids that had been extracted during the previous 2-8 months were collected and placed immediately in neutral buffered formalin. Following collection, the teeth were washed thoroughly in distilled water and placed in a 0.85 percent NaCl (saline) solution. The teeth were stored at 5°C. until needed.

### Adhesive Materials

For this report, the adhesive materials were divided into two groups. Group A included materials developed primarily for non-orthodontic use, i.e., dental restoratives and a pit and fissure sealant. Group B materials were manufactured or modified specifically for use in an orthodontic adhesive system.

#### Group A Materials

The materials in group A included (1) Enamelite[6], a composite resin manufactured as an "enamel coating" (Plate I), (2) Nuvafil[7], a composite resin polymerized by ultra-violet light (Plate II B), and (3) Sevriton[8]. a polymethyl methacrylate resin restorative, accelerated by a sulfinic acid derivative (Plate III A),

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[6]Lee Pharmaceuticals, South El Monte, California 91733

[7]L.D. Caulk Company, Milford, Delaware 19963

[8]Amalgamated Dental Trade Distributors Ltd., London, England

Group B Materials

Materials in group B included (1) Directon Adhesive and Sealant[9], a relatively new resin bonding system (Plate IV), (2) Directon Adhesive[9] alone (Plate IV B), (3) Nuva-Seal[10] System, described by Cohl and his associates (1972), shown in Plate II A, (4) Nuva-Seal[10]-Bracket Bond System[11], reported by Dean (1972), Sciaretta (1972), Dietz (1972), and Silverman and co-workers (1972), depicted in Plate II A and C, (5) O.I.S. Adhesive System[12], an acrylic adhesive system containing a fluoride agent in the etchant and adhesive, described by Newman (1969a), seen in Plate III B, (6) Orthomite Direct Bonding System[13], a methylmethacrylate resin system utilizing a silane coupling agent (methacryloxyprophyl tri-methoxysilane); Miura and his associates (1971) investigated this system (Plate III C), and (7) Unitek Direct Bonding System[14], a polymethyl methacrylate resin system (Plate III D).

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[9]TP Laboratories, LaPorte, Indiana 46350

[10]L.D. Caulk Company, Milford, Delaware 19963

[11]GAC International, Inc., Farmingdale, New York 11735

[12]Ortho International Services, Inc., Wilmington, Delaware 19899

[13]Rocky Mountain Dental Products Company, Denver, Colorado 80201

[14]Unitek Corporation, Monrovia, California 91016

These materials are presented with detailed instructions for use in the Appendix. These instructions were faithfully followed throughout this investigation.

## Brackets

The orthodontic attachments (brackets) used in this report were of the edgewise design--.018 X .025 posterior width Lexan polycarbonate brackets[13]. There were two reasons for using this bracket: (1) the edgewise bracket is the most widely used in orthodontics today and (2) it was easiest to design testing jigs for this bracket.

## Modifications

Two modifications involving the teeth and brackets were utilized in this investigation.

### Tooth Modification

The roots of the teeth were machined to fit into a small vise in the testing apparatus[15]. The following steps were undertaken (1) the crown of a tooth (Plate V A) was placed into a vise affixed to a milling machine[16] (care was taken not to damage the facial surface of the crown--a thin lead sheet was placed between the

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[15]The testing apparatus (Plate X B) was specially designed and constructed for this study at the Olin Materials Center and the School of Dentistry, Case Western Reserve University

[16]Bridgeport Milling Machine, Bridgeport Machines, Inc., Bridgeport, Conn.

PLATE I

Enamelite Enamel Coating (in kit)

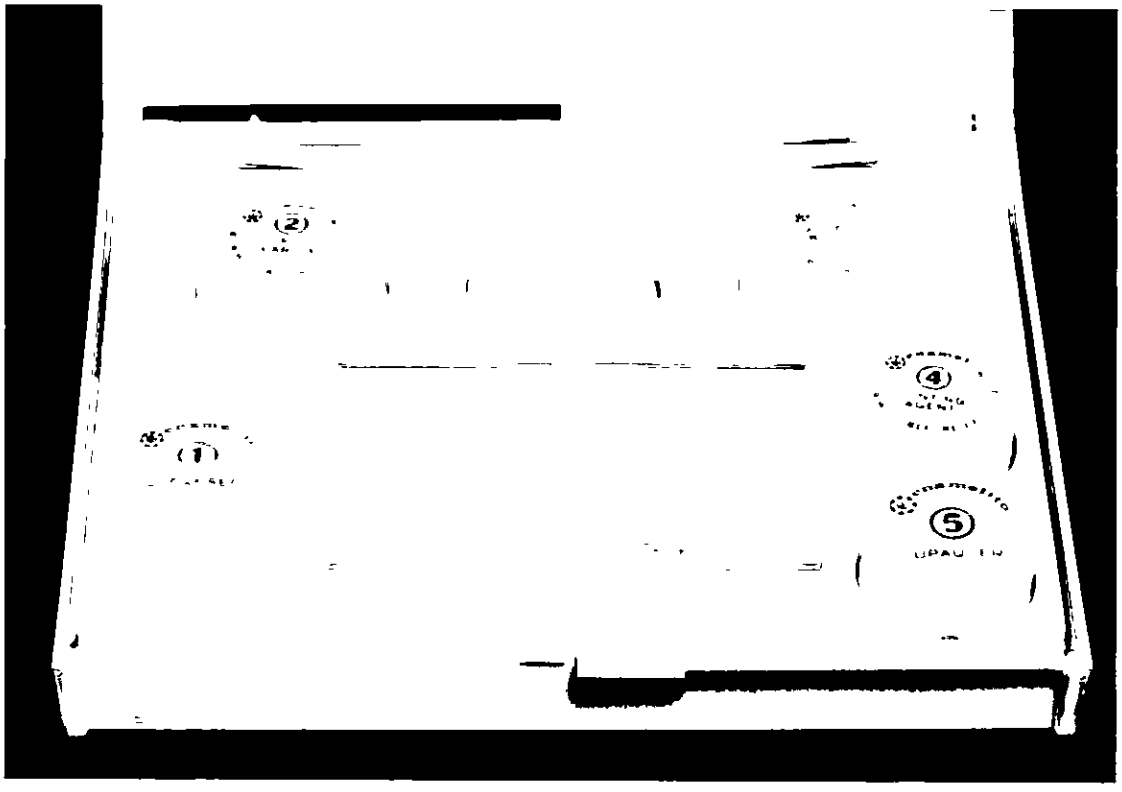
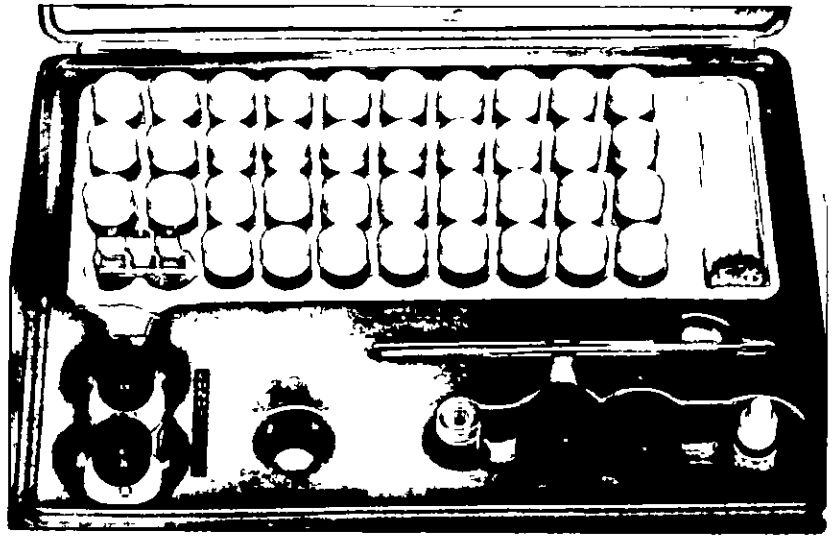

2
1
4
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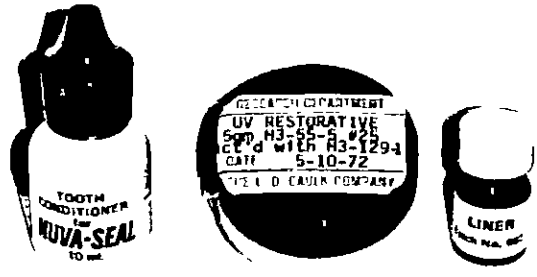
PLATE II

A. Nuva-Seal pit and fissure sealant (in kit)

B. Ultra-violet restorative (now packaged as Nuvafil) with liner and tooth conditioner

C. Bracket Bond Adhesive kit and materials




TOOTH
CONDITIONER
for
NUVA-SEAL
UV RESTORATIVE
5cm H3-55-A #25
Ctd with H3-129-1
DATE 5-10-72
LINER

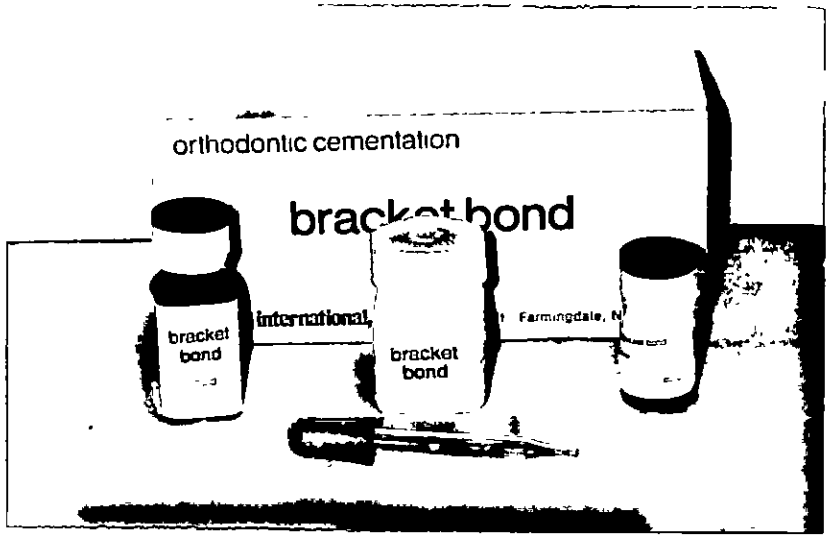

orthodontic cementation
bracket bond
bracket
bond
bracket
bond

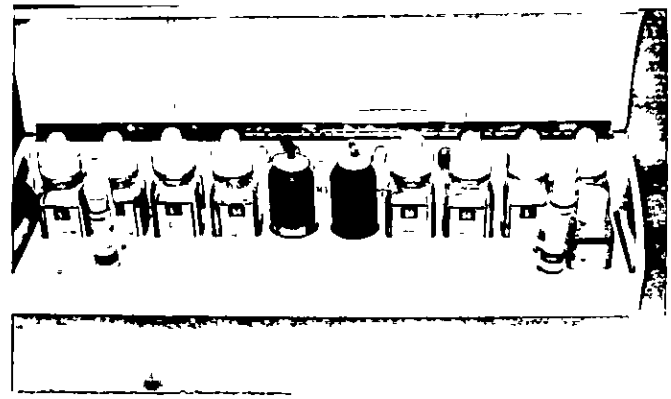
PLATE III

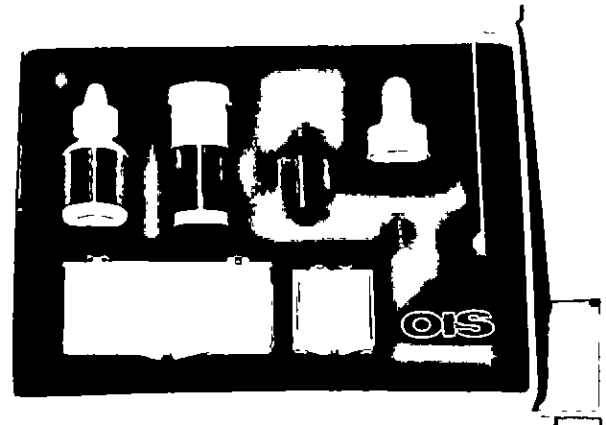
A. Sevriton Resin kit

B. O.I.S. Adhesive kit

C. Orthomite Direct Bonding kit

D. Unitek materials




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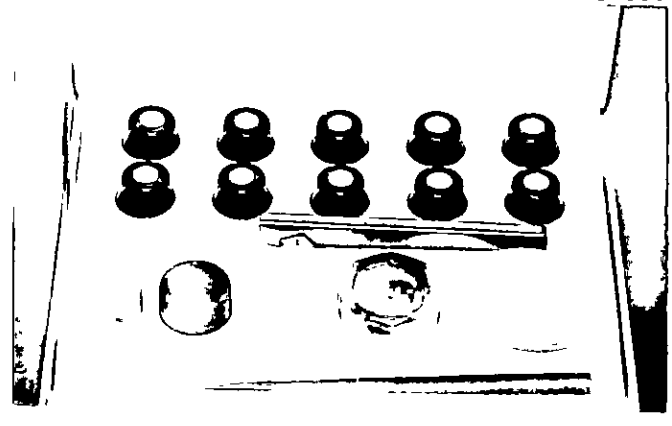
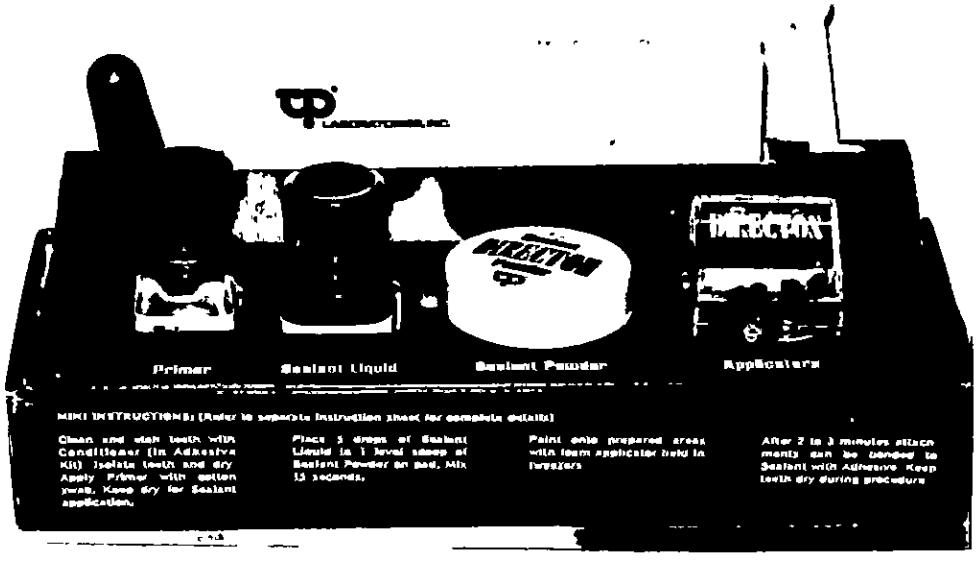




PLATE IV

A. Directon Sealant kit and materials

B. Directon Adhesive kit and materials


Primer
Sealant Liquid
Sealant Powder
Applicators
MINI INSTRUCTIONS: (Refer to separate instruction sheet for complete details)
Clean and wash teeth with Conditioner (In Adhesive Kit) Isolate teeth and dry. Apply Primer with cotton swab. Keep dry for Sealant application.
Place 3 drops of Sealant Liquid in 1 level scoop of Sealant Powder on pad. Mix 15 seconds.
Paint onto prepared areas with foam applicator held in tweezers
After 2 to 3 minutes attachments can be cemented to Sealant with Adhesive. Keep teeth dry during procedure

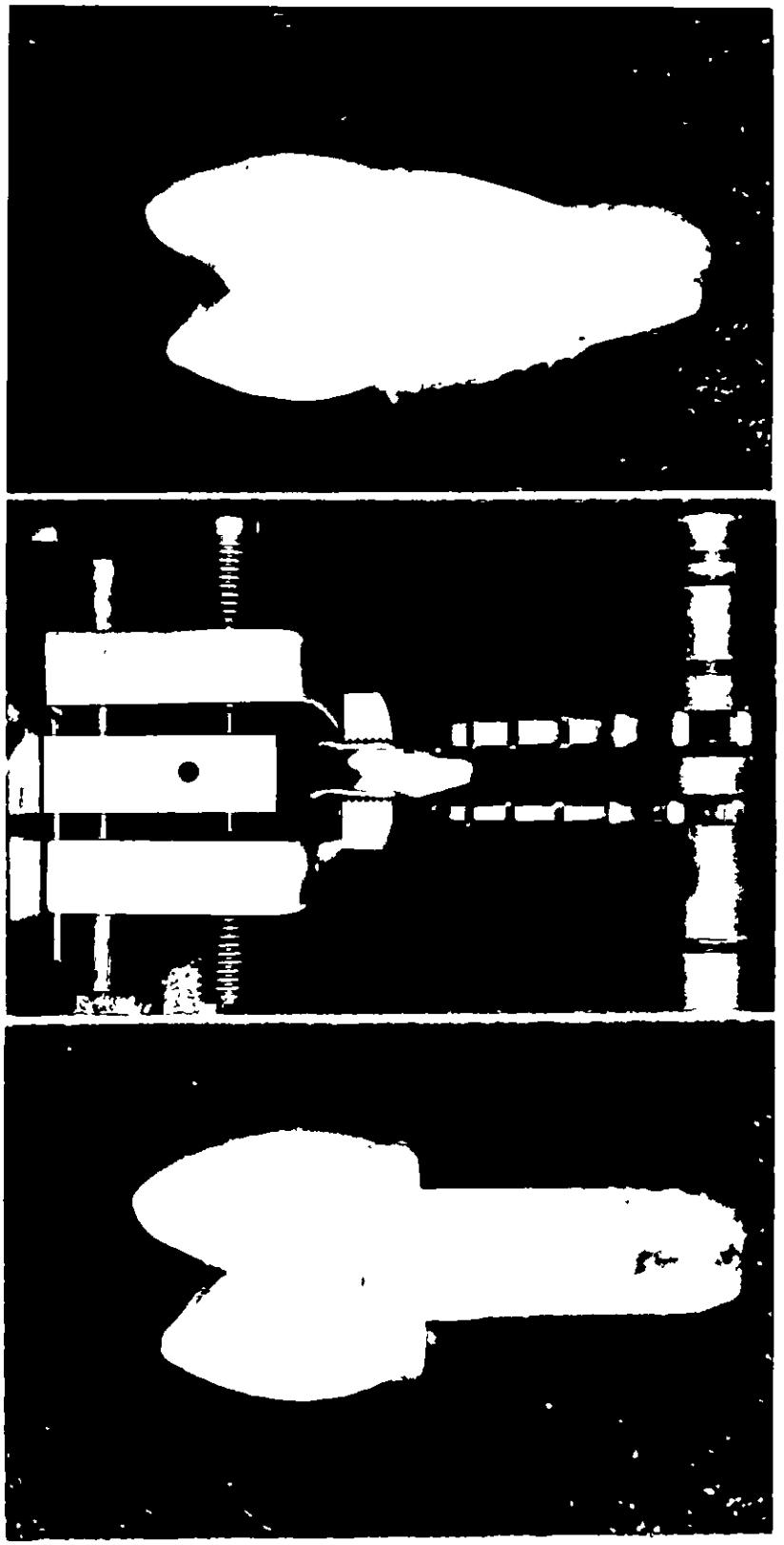

Conditioner
Primer
Adhesive
DIRECTON
Powder
Adhesive
Liquid

PLATE V

A. Bicuspid tooth prior to cutting root

B. Tooth root being cut on Bridgeport Milling Machine

C. Bicuspid tooth after root has been cut



grips of the vise and the enamel surface), (2) the facial and lingual surfaces of the root were cut parallel to each other (Plate V B), yielding (3) a modified tooth (Plate V C).

### Bracket Modification

For the tensile and peel tests, the brackets were modified as follows: (1) the edgewise slots of the brackets were placed in an epoxy resin[17] (care was taken to keep the tooth surface of the bracket out of the resin, Plate VI A), (2) the resin was cured for two days at 57°C., (3) the bracket-epoxy "caps" were separated, and (4) a hole was drilled into the "cap" with a #557 dental bur (Plate VI B). No modifications were needed for the shear test.

### Materials Application

The methods used in the present study were designed to parallel, as closely as possible, the use of the various materials clinically. Consequently, the facial enamel surfaces were prepared as they would be in the oral cavity. While ground enamel specimens produce relatively flat, smooth, and uniform surfaces, this procedure is not feasible in a clinical situation.

The general technique for bonding the polycarbonate bracket included (1) pumicing each tooth for 15 seconds using a stiff dental prophylaxis brush (Plate VII A), (2) rinsing the pumice off

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[17]Maraglas Epoxy, Marblette Epoxy Resins, New Haven, Conn.

PLATE VI

A. Brackets embedded in Maraglas epoxy resin

B. Bracket with epoxy "cap"

C. Tooth with bonded bracket-epoxy "cap"

D. Tooth with bonded bracket

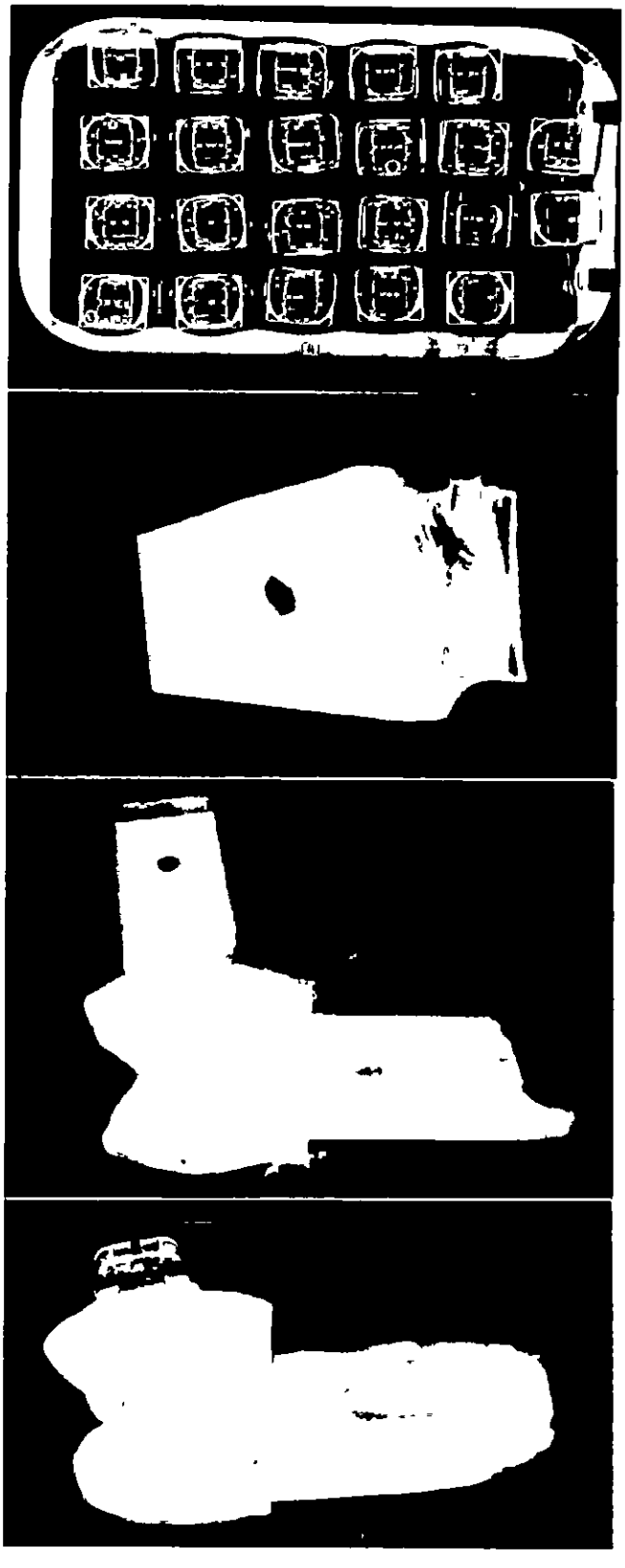
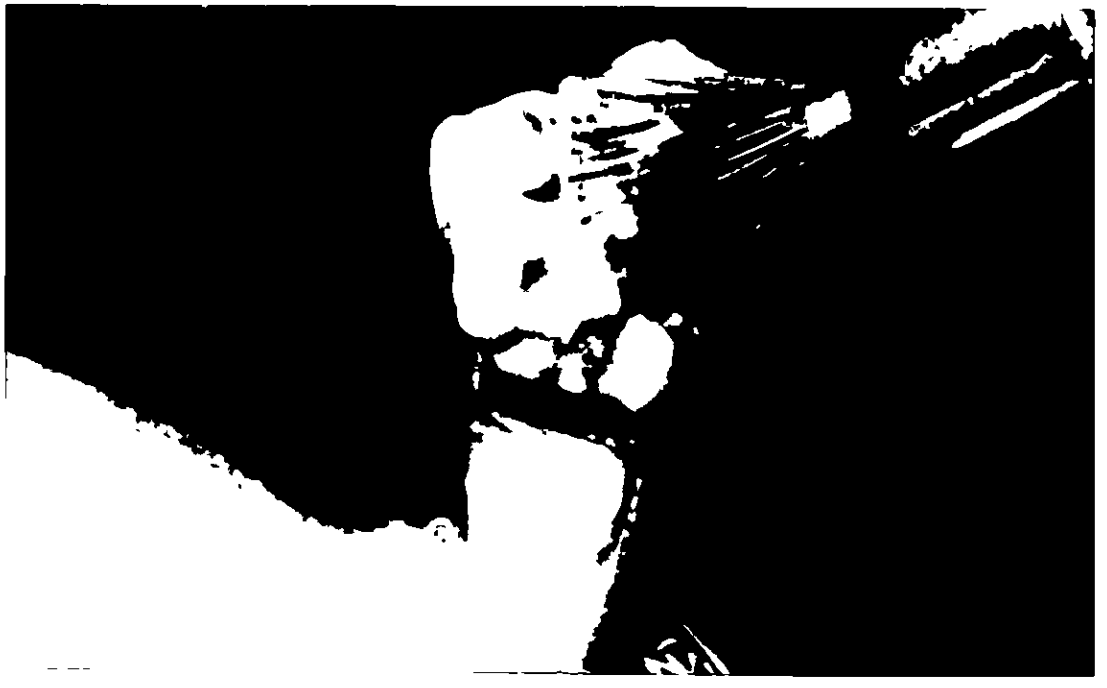
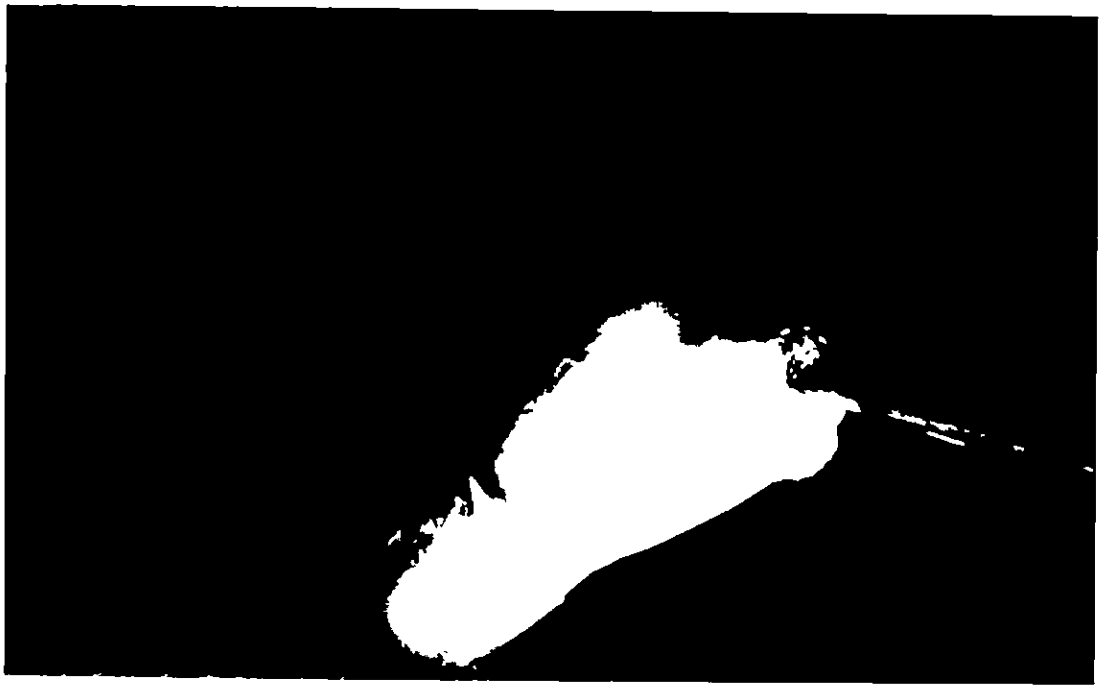


PLATE VII

A. Pumicing tooth with dental prophylaxis brush

B. Conditioning the facial enamel surface with etchant





the tooth, (3) drying the enamel surface with compressed air, (4) etching the facial enamel surfaces with an acid conditioner for a prescribed period of time (Plate VII B), (5) rinsing with water, (6) drying with compressed air, (7) applying sealant (if indicated), (8) mixing adhesive material (Plate VIII A), (9) applying adhesive to bracket base (Plate VIII B), (10) placing the bracket in the middle 1/3 of the facial surface of the crown (Plate VIII C, D; IX), and (11) storing the specimens in distilled water after allowing 15 minutes for setting. Detailed instructions for each material are presented in the Appendix.

## Testing Procedures

Thirty-six samples (teeth) were tested for each adhesive material. Eighteen samples were tested after one day (24 hours ± 6 hours); eighteen samples were tested following one month (30 days ± 2 days) in storage. During the storage periods, the tooth-bracket assemblies were kept in a 37° C. environment.

Each group of eighteen teeth included six incisors, six canines, and six bicuspids, distributed according to Table I.

Tensile, shear, and peeling stresses, indicated by Newman (1964, Figure 4) to be involved in tooth movement, were applied to the adhesive joints.

### Tensile Test

For the tensile test, the following steps were followed: (1) the tooth with the bonded bracket-epoxy "cap"was removed from water storage (Plate VI C), (2) four strands of orthodontic

PLATE VIII

A. An adhesive (Unitek) being mixed in dappan dish

B. Placing an adhesive (Unitek) on the bracket base

C. Placing a bracket-epoxy "cap" on a tooth with cotton pliers

D. Placing a bracket on a tooth with cotton pliers

PLATE IX

Polymerizing adhesive (Nuva-Seal-Nuvafil) with an ultra-violet light (Nuva-Lite)

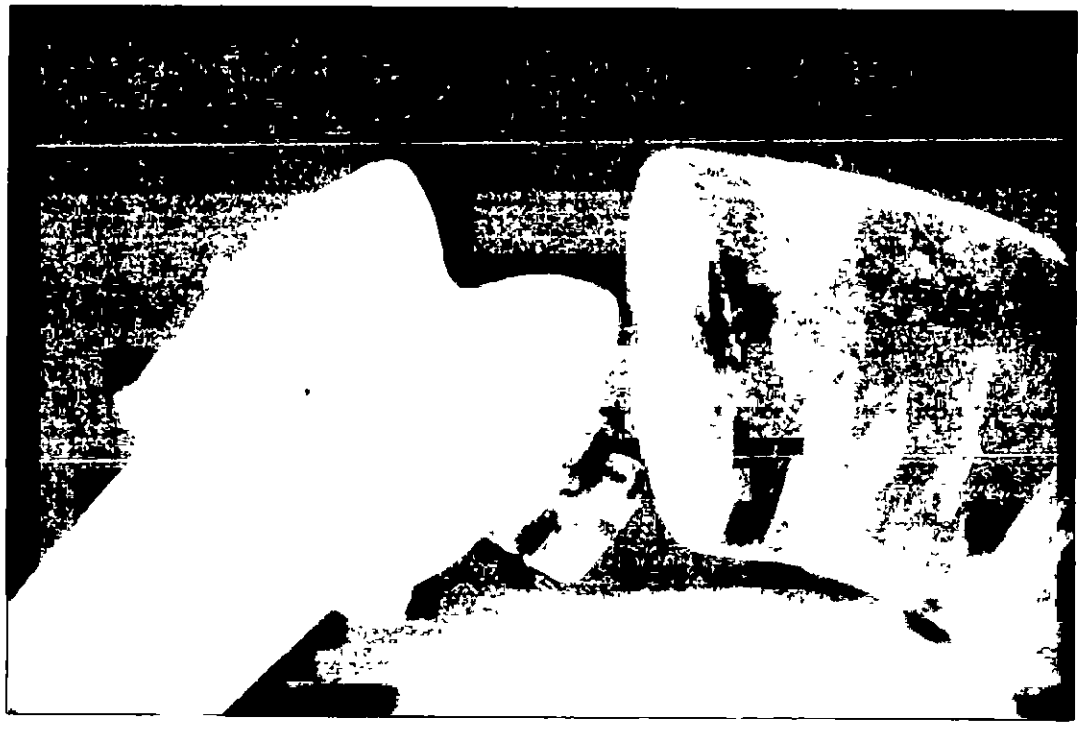


TABLE I

DISTRIBUTION OF TEETH FOR EACH ADHESIVE MATERIAL

| Time | One Day | | | One Month | | |
|---|---|---|---|---|---|---|
| Test | Tensile | Shear | Peel | Tensile | Shear | Peel |
| Teeth | Incisor | Incisor | Incisor | Incisor | Incisor | Incisor |
| | Incisor | Incisor | Incisor | Incisor | Incisor | Incisor |
| | Canine | Canine | Canine | Canine | Canine | Canine |
| | Canine | Canine | Canine | Canine | Canine | Canine |
| | Bicuspid | Bicuspid | Bicuspid | Bicuspid | Bicuspid | Bicuspid |
| | Bicuspid | Bicuspid | Bicuspid | Bicuspid | Bicuspid | Bicuspid |

stainless steel ligature wire (.010 in.), utilized as a jig, were placed through the hole in the epoxy cap, (3) the tooth was secured in the testing apparatus vise (Plate X B 5), (4) the testing apparatus was placed and secured on the cross head of an Instron Universal Testing Machine[18] (Plate X A), (5) the bracket-epoxy "cap" was positioned under the upper member (Plate X B 1) of the testing apparatus so that the bracket base was perpendicular to the wire jig (Plate XI A), (6) the test set-up was checked with a magnifying lens or micrometer microscope to insure accuracy of bracket placement, (7) the set-up was secured and a lucite cap (Plate X B 4) was placed over the tooth, (8) the cap was filled with room temperature distilled water, (9) the cross head rate was set at .05 cm. per min. (a 50 pound tensile load cell was used) and the cross head started, finally (10) a tensile stress was applied until a failure[19] occurred (Plate XI B).

## Shear and Peel Tests

The steps involved in the shear and peel tests were basically the same, with the following important exceptions noted: (1) shear test--the bracket was placed under a jig [.022 stainless steel orthodontic wire or a machined steel alloy (410) jig] so that the

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[18] Model no. TTCML, serial no. 1130
Instron Engineering Corp., Canton, Massachusetts

[19] Failures were recorded as (1) bond failures, (2) epoxy-"cap" failures, (3) tooth fractures, or, in certain shear tests, (4) bracket distortions.

PLATE X

A. Instron Universal Testing Machine (Model TTCML)

B. Testing apparatus, demonstrating:
   1. Upper member connected to 50 pound load cell
   2. Wire jig
   3. Octagonal back
   4. Lucite cap
   5. Vise on insert capable of 360° movement
   6. Base a., for diagonal movement of apparatus
   7. Base b., for horizontal movement of apparatus (base b. secured to cross head)

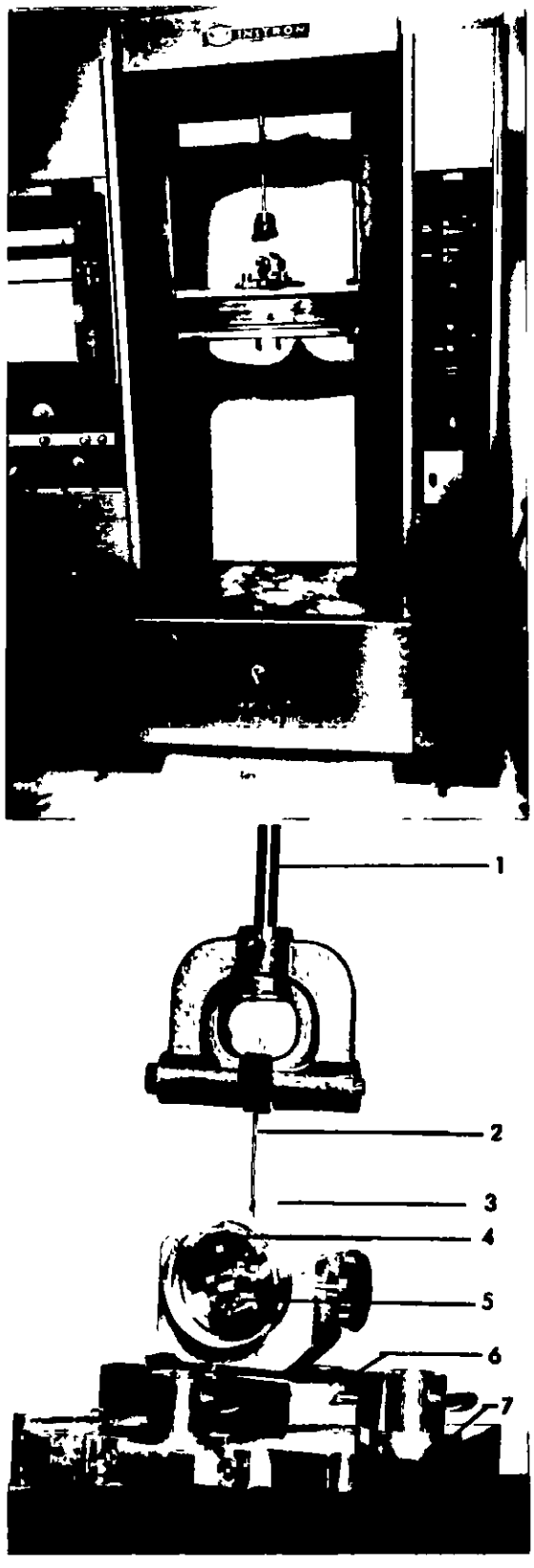

1
2
3
4
5
6
7

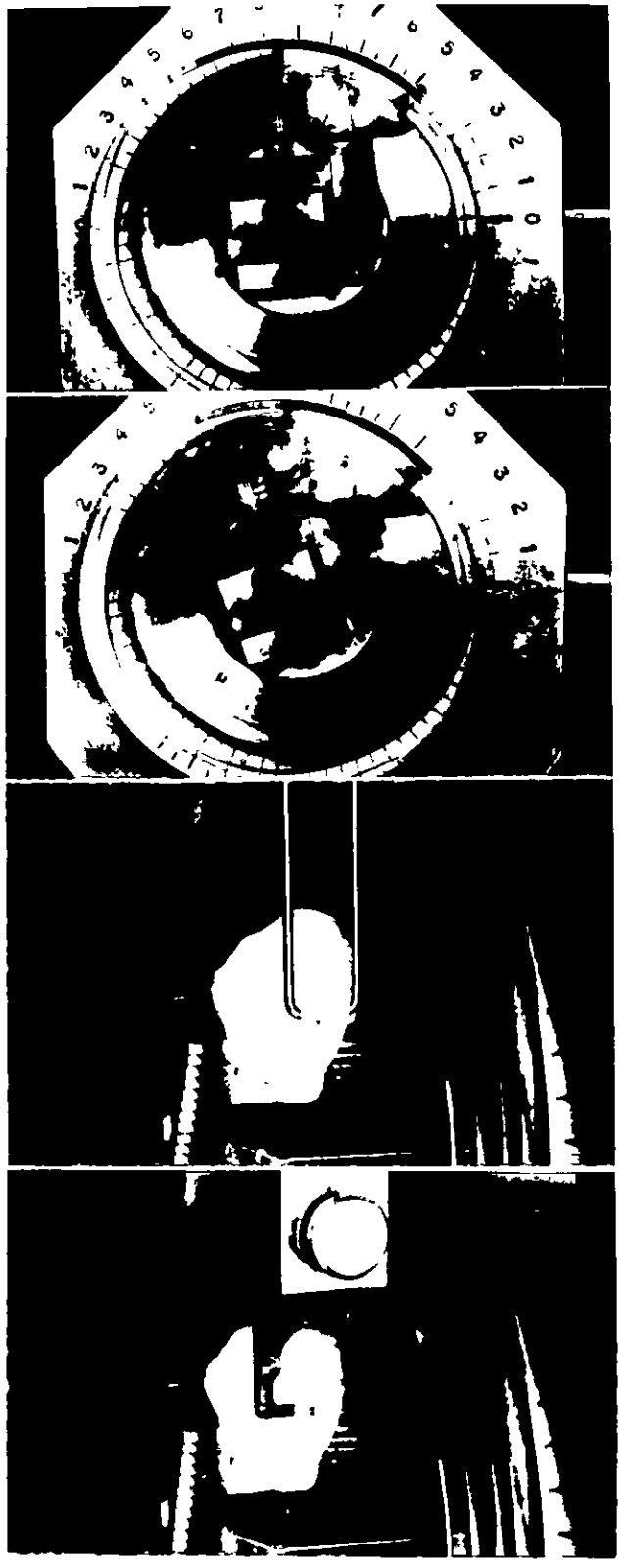
PLATE XI

A. Tooth positioned for tensile test

B. Tooth in testing apparatus following a bond failure (tensile test)

C. Tooth in position for shear test (.022 wire jig)

D. Tooth in position for shear test (machined stainless steel jig)



bracket base was parallel to the jig (Plate XI C and D), (2) peel test--the bracket-epoxy "cap" was placed under the upper member of the testing apparatus so that the bracket base was at a 55° angle to the vertical wire jig (four strands of .010 ligature wire).

## The Scanning Electron Microscope

Following bond failures (adhesive or cohesive), selected specimens were examined with scanning electron microscopes located in Cleveland, Ohio[20] (Plate XII), and Burlington, Massachusetts[21].

The specimens were mounted on aluminum discs (1/2 in. diameter) and conductive coated under vacuum with gold-palladium alloy (approximately 200 Å layer). The specimens were examined according to standard techniques with the SEM operating at 10-20 Kv accelerating potential. Representative micrographs are included in this report.

## Data Reduction

Calculations were performed on a portable electronic calculator[22]. The statistical design employed was a 2 X 10

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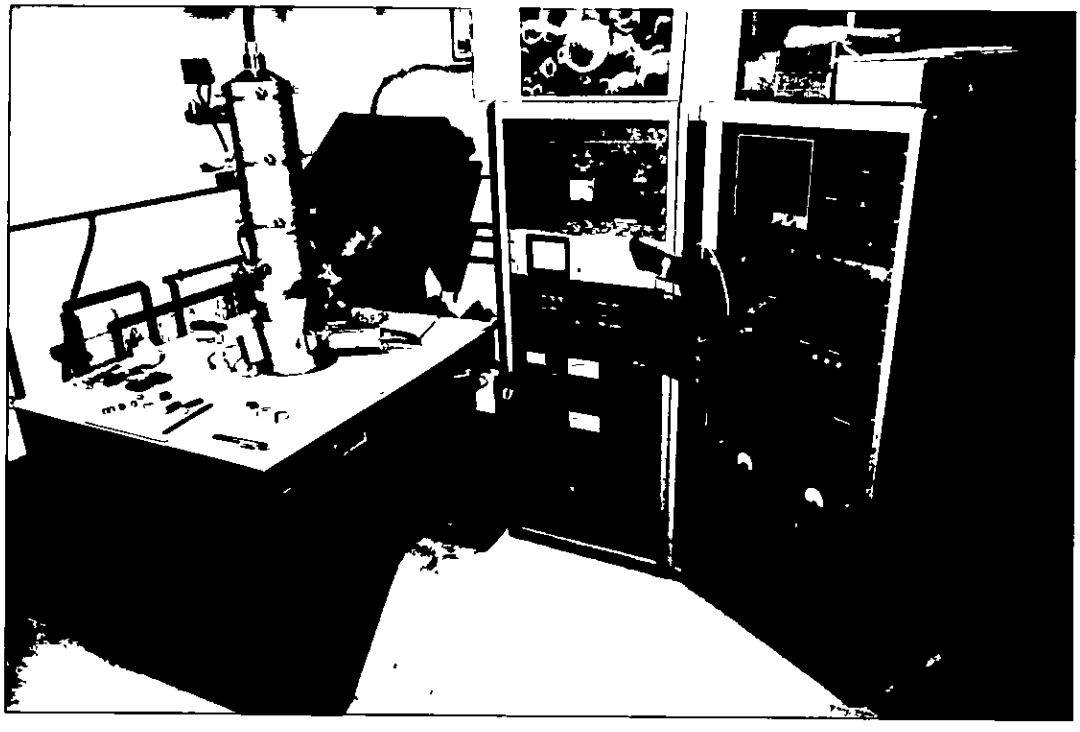
[20] M.A.C. SEM, model no. 700, manufactured by Materials Analysis Company, Palo Alto, California; located at the Olin Materials Center, Case Western Reserve University

[21] A.M.R. SEM, model no. 1000, located at and manufactured by the Advanced Materials Research Company, Burlington, Mass.

[22] Commodore Calculator, model no. US-3, Commodore Business Machines, Inc., Santa Clara, California

PLATE XII

M.A.C. Scanning Electron Microscope



factorial analysis of variance, fixed constants model (Steel and Torrie, 1960). There were two times and ten treatments (materials).

The linear model for this design may be expressed as:

$$X_{ijk} = \mu + \alpha_i + \beta_j + (\alpha\beta)_{ij} + \varepsilon_{ijk}$$

where,

$X_{ijk}$ = any measurement at the ith time, jth treatment (material) for the kth individual (tooth)

$\mu$ = the population mean

$\alpha_i$ = the effect of the ith time (i = 1,2--one day or one month)

$\beta_j$ = the effect of the jth treatment (j = 1,2....10 adhesive materials)

$(\alpha\beta)_{ij}$ = the effect of the interaction between the ith time and the jth treatment

$\varepsilon_{ijk}$ = the experimental error associated with the ith time and the jth treatment for the kth tooth

Details of the experimental design may be found in Steel and Torrie (1960) and Snedecor (1956).

Tukey's test for comparisons among the means was also performed to determine significant differences among main effect factors for each test, i.e., tensile,shear, and peel tests (Snedecor, 1956).

$$D = Qs_{\bar{x}} \quad \text{where,}$$

D = difference among the means
Q = tabulated value at the .05 level
$s_{\bar{x}}$ = sample standard deviation of the mean

$$s_{\bar{x}} = \sqrt{\frac{\text{Error Mean Square}}{n_o}} \quad \text{and,}$$

$n_o$ = effective number of replications for unequal samples.

# RESULTS

The results of the present study will be presented as (1) materials testing and (2) SEM observations.

## Materials Testing

The individual data for each stress modality are presented in Tables II, III, and IV. While all teeth with bonded attachments were placed in the testing apparatus, several samples could not be tested due to bond failures while the jig was being attached. The results were pooled for each time and a 2 X 10 factorial analysis of variance was performed for each test, i.e., tensile, shear, and peel. The results of variance calculations are shown in Table V.

All three tests indicated a significant ($p < .001$) difference among the means of the adhesive materials (treatments). Tukey's test for comparison among means was performed to determine significant differences. The results are presented in Figures 8, 9, and 10. Groups of materials, rather than individual adhesives, differed significantly from other groups. In general, the orthodontic adhesives, as a group, were significantly stronger than the composite resin restoratives and the pit and fissure sealant when used alone.

TABLE II

TENSILE TEST (P.S.I.)

| Treatment (Material) | Directon with sealant | Directon without sealant | Enamelite | Nuva-Seal | Nuva-Seal Bracket Bond | Nuva-Seal Nuvafil | O.I.S. | Orthomite | Sevriton | Unitek |
|---|---|---|---|---|---|---|---|---|---|---|
| Time | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day |
| Teeth | 1269[b] | 924[b] | 255 | 126 | 1314[b] | 351 | 162 | 1140[b] | 1002 | 1215[b] |
| | 900[b] | 909[b] | 360 | 30 | 795[b] | 732 | 609 | 915[b] | 951 | 1005 |
| | 810[b] | 1047 | 96 | . .[a] | 1086[b] | 291 | 126 | 900[b] | 1056[b] | 915[b] |
| | 900[b] | 744 | 117 | 117 | 1290[b] | 375 | 195 | 870[b] | 1230[b] | 1110[b] |
| | . .[a] | 894 | 141 | . .[a] | 882[b] | 390 | 450 | 1125[b] | 939 | 1026[b] |
| | 675[b] | 750 | 207 | . .[a] | 1185[b] | 447 | 633 | 900 | 1467 | 870[b] |
| Time | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. |
| Teeth | 768[b] | 660[b] | 180 | 21 | 615 | 69 | 720 | 615[b] | 645 | 675[b] |
| | 843[b] | 660[b] | 324 | 192 | 1206 | 90[a] | 795 | 567[b] | 1020 | 765[b] |
| | 606 | 510[b] | 120 | 102 | 387 | . .[a] | 234[b] | 510[b] | 873 | 765[b] |
| | 672 | 660[b] | 135 | 72 | 1122[b] | 87 | 432 | 315[b] | 855 | 648[b] |
| | 774[b] | 657 | 51 | . .[a] | 1005 | 102 | 612 | 570[b] | 174 | 924[b] |
| | 399[b] | 675[b] | 156 | 123 | 1215[b] | . .[a] | 372 | 714[b] | 573 | 600[b] |

[a]Denotes a sample which broke during the set-up of the test or a loose set-up

[b]The strength of the adhesive attachment exceeded the breaking stress of the tooth or epoxy cap

TABLE III

SHEAR TEST (P.S.I.)

| Treatment (Material) | Directon with sealant | Directon without sealant | Enamelite | Nuva-Seal | Nuva-Seal Bracket Bond | Nuva-Seal Nuvafil | O.I.S. | Orthomite | Sevriton | Unitek |
|---|---|---|---|---|---|---|---|---|---|---|
| Time | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day |
| Teeth | 855[b] | 1263 | 132 | 132 | 1221[b] | 843 | 276 | 201[b] | 849 | 1098 |
| | 1110[b] | 1218[b] | 141 | 96 | 1101[b] | 855 | 276 | 1161[b] | 1068[b] | 819 |
| | 1143[b] | 1080 | 138 | 75 | 1221[b] | 831 | 255 | 729 | 972 | 888 |
| | 1089[b] | 1107[b] | 195 | 69 | 1206[b] | 906 | 375 | 1056[b] | 1059 | 621 |
| | 954 | 849 | 78 | ..[a] | 1005[b] | 531 | 105 | 1095 | 786 | 654 |
| | 1104[b] | 759 | 171 | 159 | 1194[b] | ..[a] | 471 | 798 | 945[b] | 999[b] |
| Time | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. |
| Teeth | 1092[b] | 1083[b] | 201 | 102 | 735 | 666 | 90 | 1128[b] | 984 | 1071 |
| | 1071[b] | 702 | 129 | 84 | 1164[b] | 711 | 246 | 1074[b] | 1155[b] | 1029[b] |
| | 630[b] | 963 | 78 | 33 | 960 | 504 | 492 | 993[b] | 681 | 1194 |
| | 768 | 705[b] | 69 | 66 | 933[b] | 225 | 690 | 1101[b] | 1020 | 756 |
| | 1089[b] | 834[b] | 99 | 30 | 975[b] | 891 | 918 | 1134[b] | 606 | 786 |
| | 1236[b] | 912 | 63 | 33 | 864[b] | 915 | 447 | 834[b] | 1068 | 627 |

[a]Denotes a sample which broke during the set-up of the test or a loose set-up

[b]The strength of the adhesive attachment exceeded the breaking stress of the tooth or bracket.

TABLE IV

PEEL TEST (P.S.I.)

| Treatment (Material) | Directon with sealant | Directon without sealant | Enamelite | Nuva-Seal | Nuva-Seal Bracket Bond | Nuva-Seal Nuvafil | O.I.S. | Orthomite | Sevriton | Unitek |
|---|---|---|---|---|---|---|---|---|---|---|
| Time | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day | 1 day |
| Teeth | 723[b] | 909 | 255 | . .[a] | 741[b] | 57 | 390 | 561 | 186 | 585[b] |
| | 606 | 831[b] | 252 | 69 | 609 | 129 | 18 | 354 | 444 | 630 |
| | 660[b] | 663[b] | 183 | 30 | 291 | 48 | 144 | 411 | 465 | 1119[b] |
| | 531 | 726[b] | 210 | 30 | 885[b] | 51 | 162 | 615 | 351 | 726[b] |
| | 957[b] | 546 | 165 | . .[a] | 819 | 69 | 78 | 447 | 312 | 651[b] |
| | 378 | 630 | 114 | . .[a] | 813 | 69 | 84 | 519 | 447 | 666 |
| Time | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. | 1 mo. |
| Teeth | 270 | 399[b] | . .[a] | 51 | 399 | 51 | 75 | 345 | 564 | 291 |
| | 771[b] | 618 | 126 | 171 | 855 | 45 | 126 | 414 | 486 | 918 |
| | 450 | 426 | 180 | 39 | 570 | 36 | 459 | 450 | 435 | 819 |
| | 579 | 351 | 45 | 174 | 609 | . .[a] | 72 | 366 | 462 | 528 |
| | 771 | 345 | 24 | 87 | 516 | 33 | 96 | 588 | 654 | 441 |
| | 831[b] | 801 | . .[a] | 48 | 432 | 60 | 381 | 339 | 480 | 855 |

[a]Denotes a sample which broke during the set-up of the test or a loose set-up

[b]The strength of the adhesive attachment exceeded the breaking stress of the tooth or epoxy cap

TABLE V

FACTORIAL ANALYSIS OF VARIANCE

Tensile Test

| Source | df | SSQ | MSQ | F | Significance Level |
|---|---|---|---|---|---|
| Treatment | 9 | 10,231,377 | 1,136,820 | 12.38 | p <.001 [ 3.57] |
| Time | 1 | 1,195,269 | 1,195,269 | 13.02 | p <.001 [11.85] |
| Treat. X Time | 9 | 909,242 | 101,027 | 1.10 | p >.05 [ 2.00] |
| Error | 93 | 8,538,488 | 91,812 | . . | . . |
| Total | 112 | 20,874,376 | . . | . . | . . |

Shear Test

| | | | | | |
|---|---|---|---|---|---|
| Treatment | 9 | 14,509,439 | 1,612,160 | 32.85 | p <.001 [ 3.56] |
| Time | 1 | 36,456 | 36,456 | 0.74 | p >.05 [ 3.95] |
| Treat. X Time | 9 | 525,573 | 58,397 | 1.19 | p >.05 [ 1.99] |
| Error | 98 | 4,809,662 | 49,078 | . . | . . |
| Total | 117 | 19,881,130 | . . | . . | . . |

Peel Test

| | | | | | |
|---|---|---|---|---|---|
| Treatment | 9 | 6,197,525 | 688,614 | 25.14 | p <.001 [ 3.57] |
| Time | 1 | 59,618 | 59,618 | 2.18 | p >.05 [ 3.96] |
| Treat. X Time | 9 | 896,267 | 99,585 | 3.63 | p <.001 [ 3.57] |
| Error | 94 | 2,574,731 | 27,391 | . . | . . |
| Total | 113 | 9,678,141 | . . | . . | . . |

Figure 8

Tensile Bond Strengths (p.s.i.) of Adhesive Materials

for Orthodontic Attachment

[Groups with similar shadings indicate significant differences at p = .05 level]

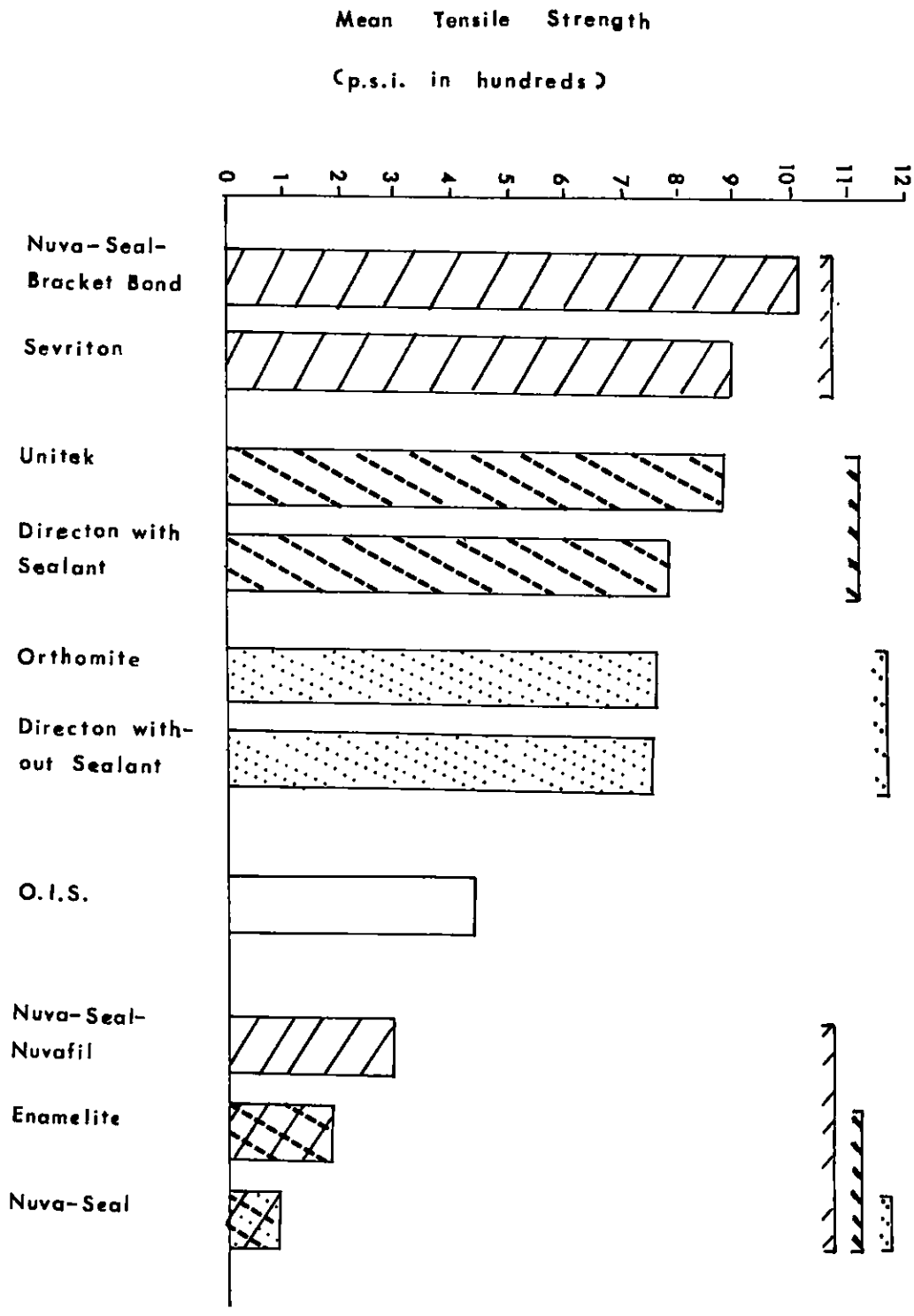

Mean Tensile Strength
(p.s.i. in hundreds)
0
1
2
3
4
5
6
7
8
9
10
11
12
Nuva-Seal-Bracket Bond
Sevriton
Unitek
Directon with Sealant
Orthomite
Directon without Sealant
O.I.S.
Nuva-Seal-Nuvafil
Enamelite
Nuva-Seal

Figure 9

Shear Bond Strengths (p.s.i.) of Adheisve Materials

for Orthodontic Attachment

[Groups with similar shadings indicate significant differences at p = .05 level]

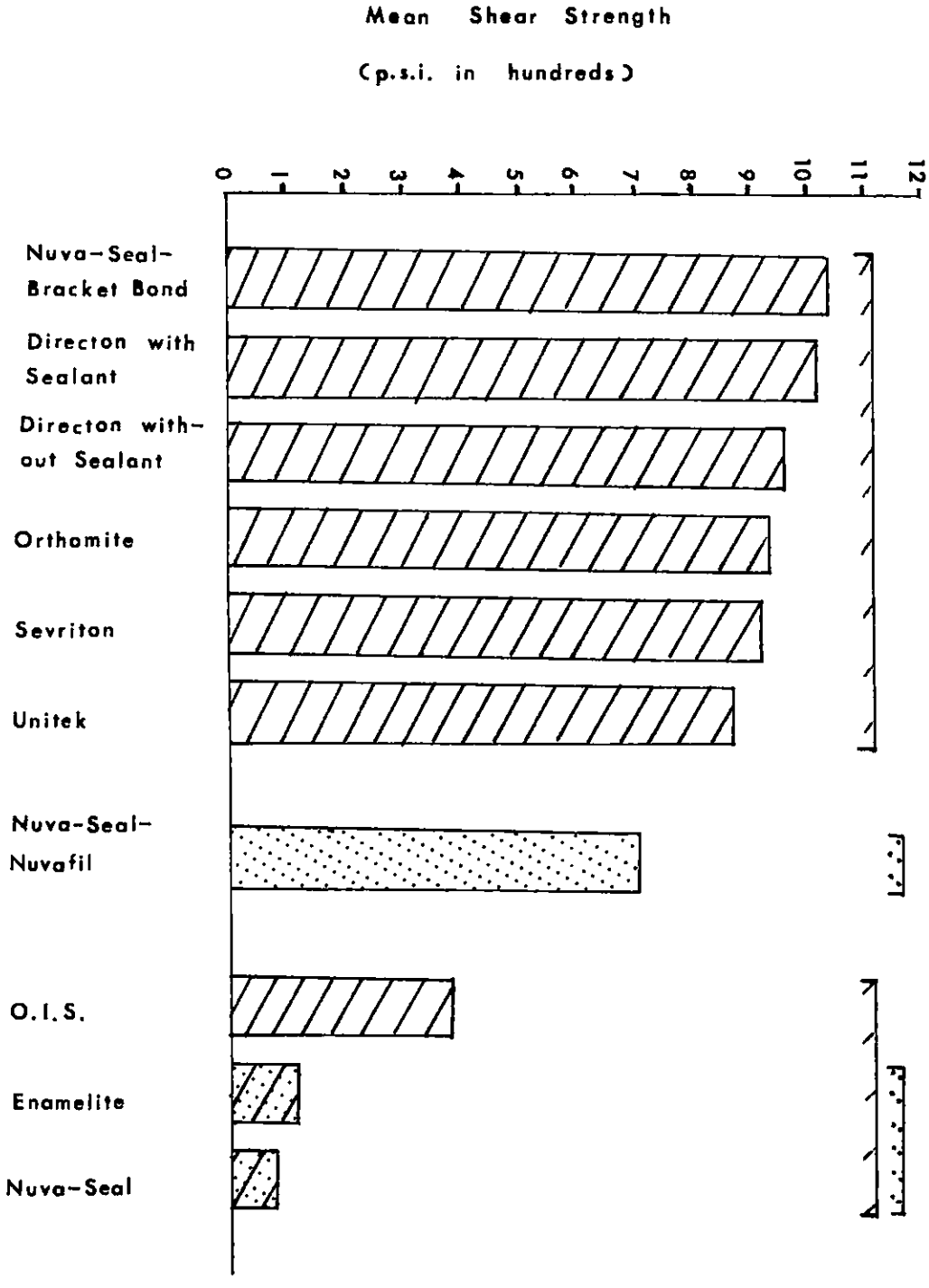
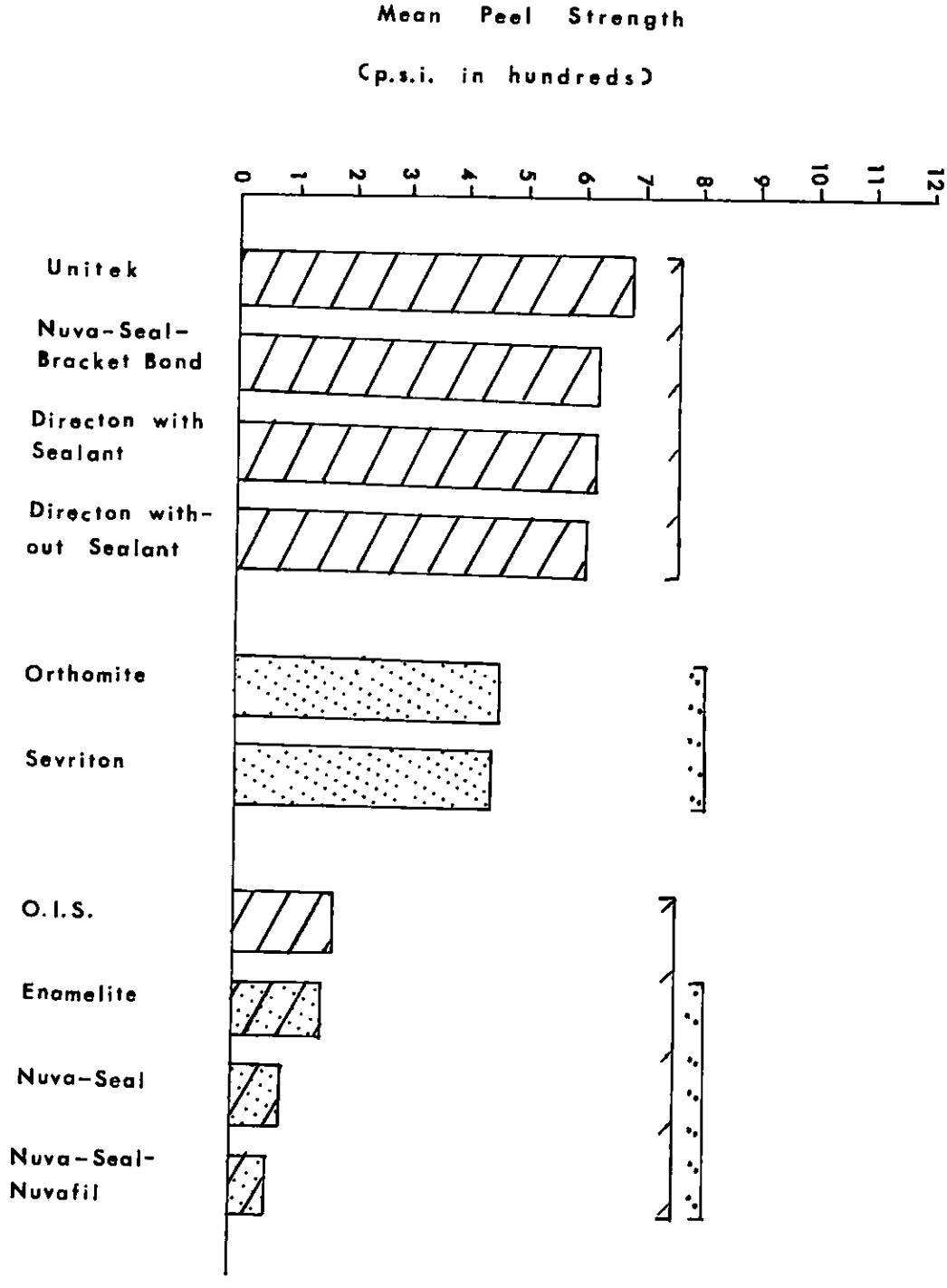

Mean Shear Strength
(p.s.i. in hundreds)
0
1
2
3
4
5
6
7
8
9
10
11
12
Nuva-Seal-Bracket Bond
Directon with Sealant
Directon without Sealant
Orthomite
Sevriton
Unitek
Nuva-Seal-Nuvafil
O.I.S.
Enamelite
Nuva-Seal

Figure 10

Peel Bond Strengths (p.s.i.) of Adhesive Materials

for Orthodontic Attachment

[Groups with similar shadings indicate significant differences at p = .05 level]


Mean Peel Strength
(p.s.i. in hundreds)
0
1
2
3
4
5
6
7
8
9
10
11
12
Unitek
Nuva-Seal-
Bracket Bond
Directon with
Sealant
Directon with-
out Sealant
Orthomite
Sevriton
O.I.S.
Enamelite
Nuva-Seal
Nuva-Seal-
Nuvafil

The time effect (one day compared to one month) was significant only in the tensile test. The data were further analyzed using a student's t test. The data of significance were directly accompanied by a high level of epoxy "cap" or tooth failures.

While no interactions were expected, interaction of effects (treatment X time) did occur in the peel test. Once again, a high incidence of epoxy "cap" or tooth failures was present in this examination.

## SEM Observations

The materials, mixed and unmixed, were observed under the SEM for orientation. Plate XIII shows the difference between a polymethyl methacrylate (unfilled) resin--Unitek (A), and a composite resin (fillers included) adhesive material--Enamelite (B). The polymer beads are apparent in (A); the inorganic filler particles in (B) are referred to as "whiskers" by the manufacturer.

Examination of an untreated bracket base (Plate XIV A) and a bracket base conditioned with methacrylic acid for two minutes (Plate XIV B) indicates a possible "leeching-out" of the high molecular weight molecules by the acid, thus creating an increased surface area available for the adhesive.

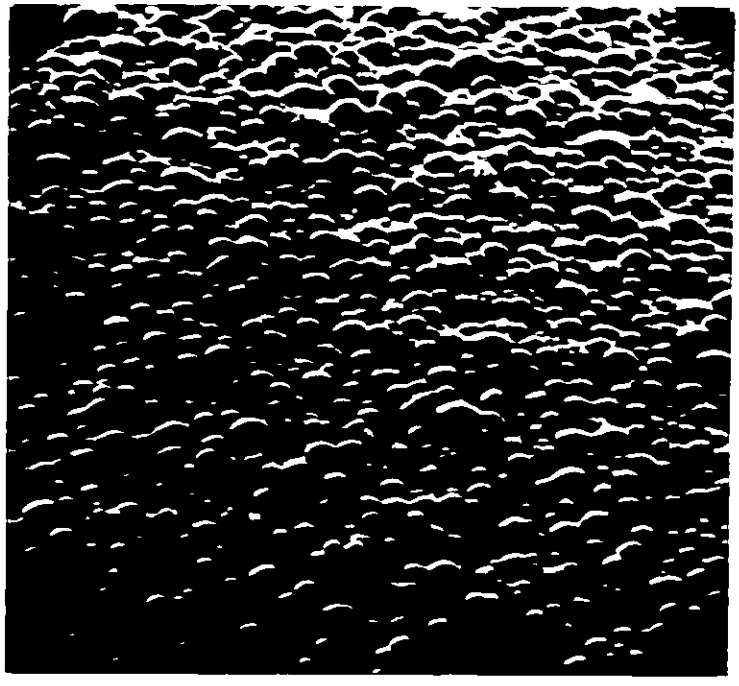
Stereoscopic microscope and SEM observations of the bond failures revealed various types of possibilities: (1) an adhesive failure at the adhesive-enamel interface (Plates XV, XVI, XVII),

PLATE XIII

Scanning electron micrographs

A. Polymer beads of an unfilled resin (Unitek) mixture
200 X

B. Composite resin (Enamelite) mixture containing inorganic "whiskers"
200 X



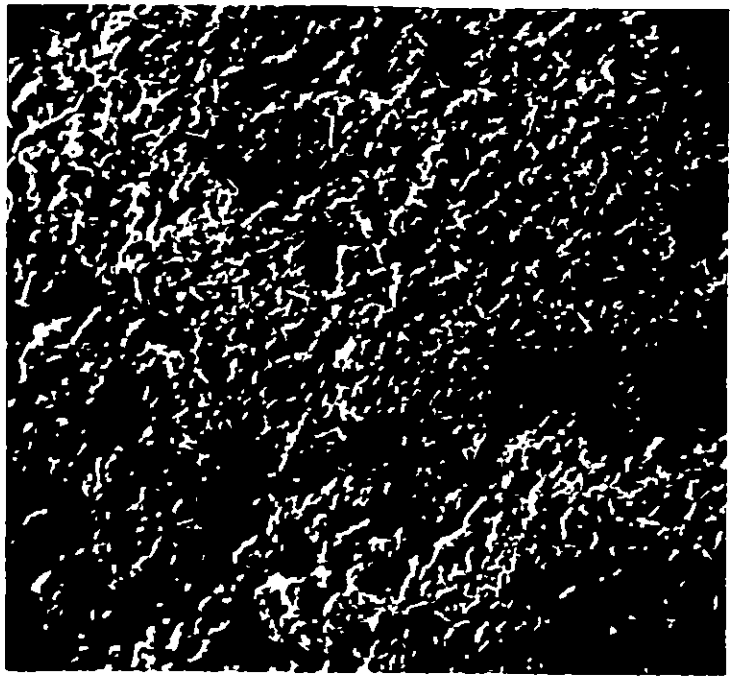


PLATE XIV

Scanning electron micrographs

A. Bracket base of .018 X .025 posterior width Lexan polycarbonate (Rocky Mountain Products) after the base was wiped with an alcohol guaze sponge
500 X

B. Bracket base (as above) conditioned with methacrylic acid for 2 minutes
500 X



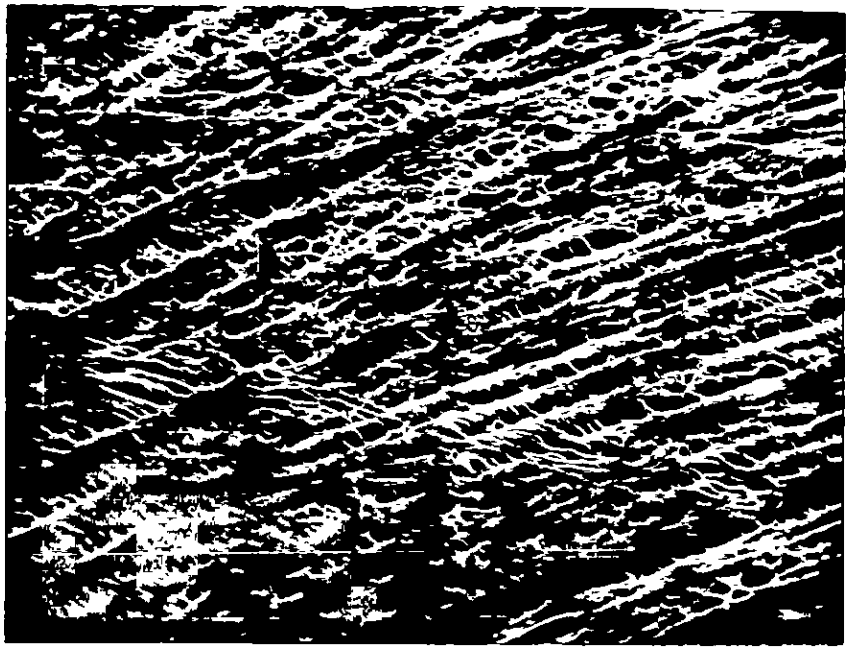


PLATE XV

Scanning electron micrographs

A. Tooth surface following a tensile test joint failure with O.I.S. adhesive. Adhesive failure occurred in the bracket contact area, with excess resin retained.
20 X

B. Enamel prism endings in the central portion of the tooth surface shown above. Preferential etching of the prism cores or peripheries is evident in some areas, while other areas show little effects of the acid attack.
500 X

C. Higher magnification of B., showing enamel prism arcade structure with the peripheries etched. Enamel was conditioned with phosphoric acid.
4000 X

PLATE XVI

Scanning electron micrographs

A. Interface between enamel (E) and O.I.S. resin adhesive (A) following tensile failure
240 X

B. Higher magnification of interface shown in A. indicating remnants of resin tags into etched enamel pores
1600 X


A

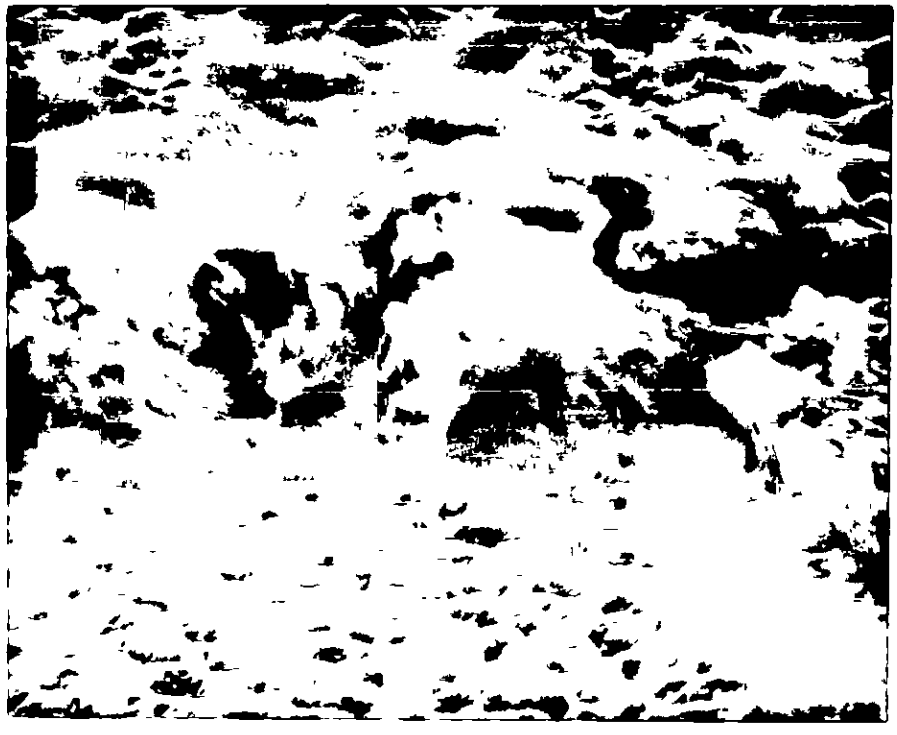


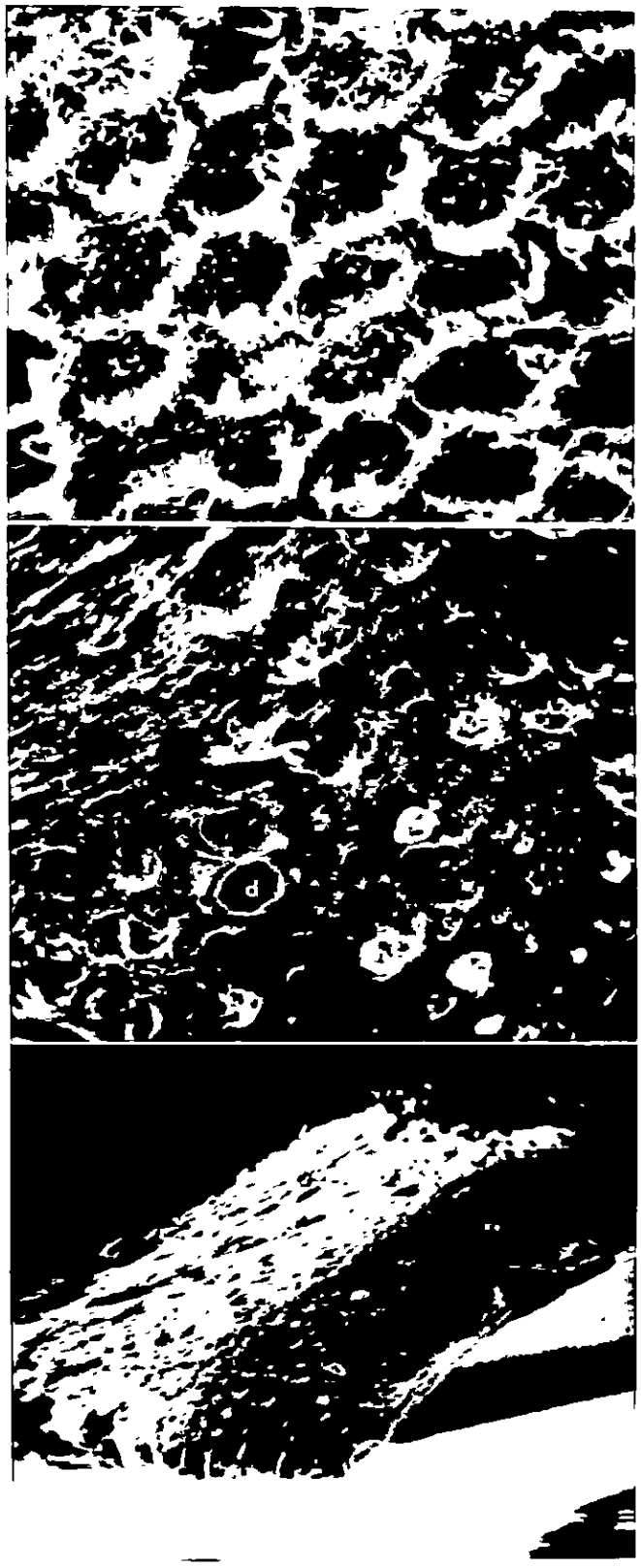
PLATE XVII

Scanning electron micrographs

A. Bracket surface (polycarbonate bracket) following a tensile test failure with O.I.S. adhesive. Joint failure occurred at the enamel-adhesive interface with nearly all resin retained on the bracket.
20 X

B. Central portion of A. showing resin formerly in contact with enamel, with numerous porosities (P) probably occurred during preparation.
100 X

C. Higher magnification of the resin contact area seen in B., indicating adhesive tags. The pattern faithfully replicates the etched enamel prism peripheries as seen in Plate XV C.
4000 X



(2) a cohesive failure within the adhesive, (3) a combination failure--that is, areas indicating cohesive and adhesive failure in the same sample (Plates XVIII, XIX, XX), and (4) an adhesive failure between the bracket base and adhesive material (Plates XXI, XXII). The examples are represented in Figure 11.

In general, a bond failure of an orthodontic adhesive was usually of the cohesive type. The bond between enamel and the adhesive was thus stronger than the adhesive material itself. However, two exceptions were noted: (1) the O.I.S. system (Plates XV, XVI, XVII) consistently yielded an adhesive failure at the adhesive-enamel interface, and (2) the Nuva-Seal system consistently presented an adhesive failure at the bracket-Nuva-Seal interface.

An unfilled resin restorative (Sevriton) consistently produced a cohesive failure whenever a bond failure occurred. The two composite resin restoratives in Group A (Enamelite and Nuvafil) yielded adhesive failures at the bracket-adhesive interface.

PLATE XVIII

Scanning electron micrographs

A. Tooth surface following a tensile test joint failure with Nuva-Seal-Bracket Bond Adhesive System. While the initial failure occurred near the enamel surface (E), failure propagated through the adhesive (A) and bracket (B).
15 X

B. Higher magnification of A. indicating a fracture through the polycarbonate bracket. A portion of the top (facial) surface of the bracket base remained in contact with adhesive on the tooth.
100 X


B
A

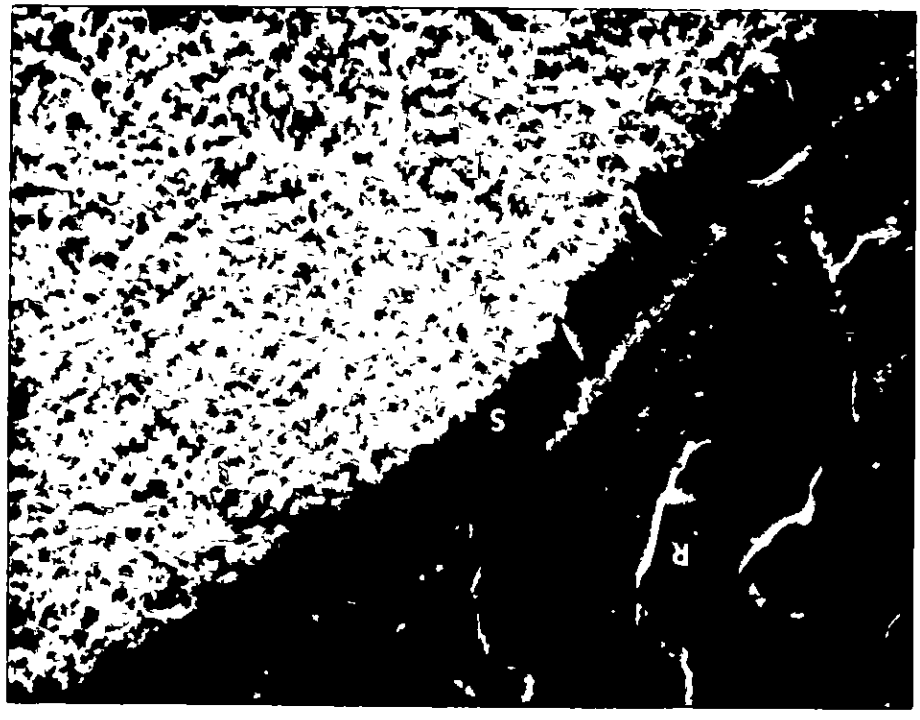


PLATE XIX

Scanning electron micrographs

A. Higher magnification of the enamel surface viewed in Plate XVIII A. A cohesive failure occurred through a portion of the Nuva-Seal, as indicated by a region of sealant (S) that is poorly differentiated from the enamel (E).
200 X

B. Adhesive-enamel interface with the Bracket Bond resin (R), Nuva-Seal sealant (S) and the enamel surface (E). The enamel surface retained portions of sealant which had established tags into the conditioned enamel.
500 X


S
R

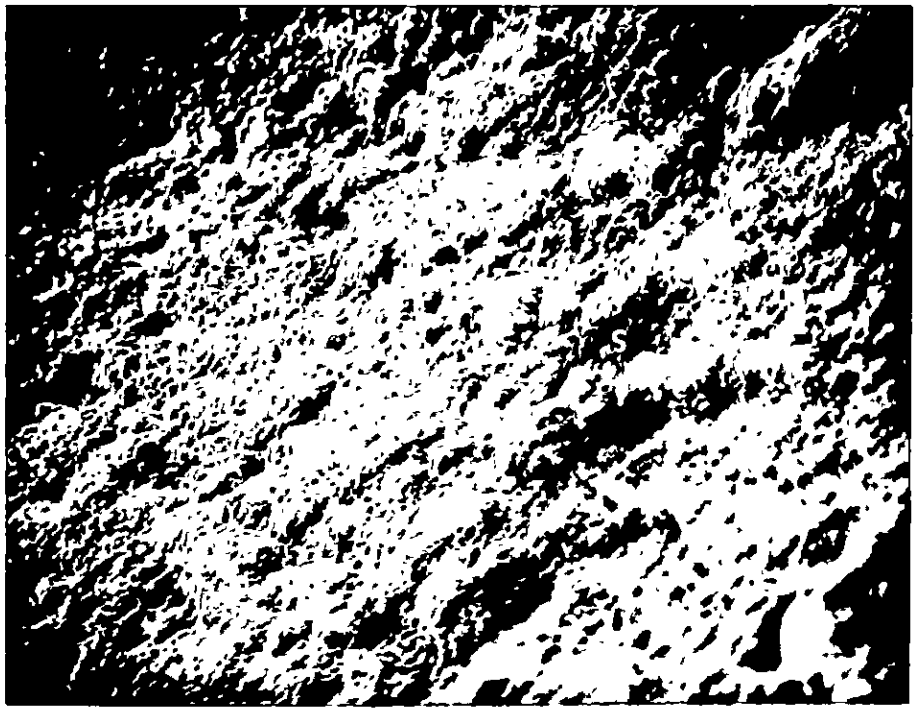

S

PLATE XX

Scanning electron micrographs

A. Bracket surface following a tensile test joint failure with the Nuva-Seal-Bracket Bond Adhesive System. The surfaces correspond to the fracture pattern indicated in Plate XVIII A. Regions indicated include sealant (S), Bracket Bond resin (R), and the region of the "missing" bracket (B).
20 X

B. Higher magnification of the sealant (S)-resin (R) interface. The sealant surface was in direct contact with enamel.
200 X

C. Higher magnification of the sealant surface in B, showing tags (T), formerly in enamel. Incomplete replication of the enamel surface provides further evidence of firm retention of the sealant in enamel.
1000 X

R
B

PLATE XXI

Scanning electron micrographs

A. Tooth surface following a tensile test joint failure with Enamelite adhesive. Almost all of the adhesive was retained on the tooth, with failure occurring at the bracket-adhesive interface. Again, the failure was limited to the area of the bracket base.
20 X

B. Higher magnification of the central portion of the adhesive surface showing a replication of the bracket base surface (BR is replicated base) by the adhesive. Porosity (P) is evident in the Enamelite, exposing the filler component.
150 X

C. Contact surface of a bracket base following a tensile test joint failure with Enamelite adhesive. The smooth bracket surface indicates a pure adhesive failure between the adhesive and bracket.
20 X

BR

PLATE XXII

Scanning electron micrographs

A. Tooth surface following a shear test joint failure with Nuva-Seal-Nuvafil. Most of the tooth surface retained the Nuva-Seal sealant (S) and the Nuvafil composite resin (R).
30 X

B. Bracket surface following the shear test in A. above indicating an adhesive failure between the bracket (B) and adhesive resin. A "chunk" of resin (R) remained on one corner of the bracket.
20 X

C. Higher magnification of the interface of adhesive resin chunk (R) with the bracket surface (B). A film (F) prominent across much of the bracket base may be a layer of unpolymerized, "air-inactivated" resin.
200 X


R
S
R
B
B
F
F

Figure 11

Bond Fracture Patterns Observed Under

Stereoscopic and Scanning Electron Microscopy

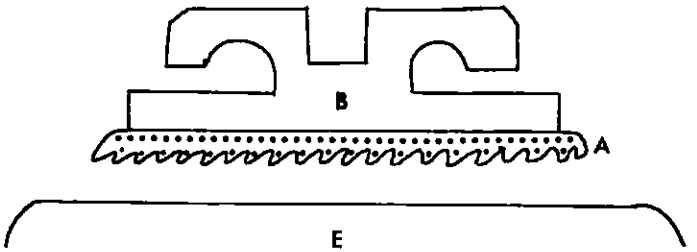
(E = enamel, A = adhesive, B = bracket)

1. Adhesive failure at enamel-adhesive material interface

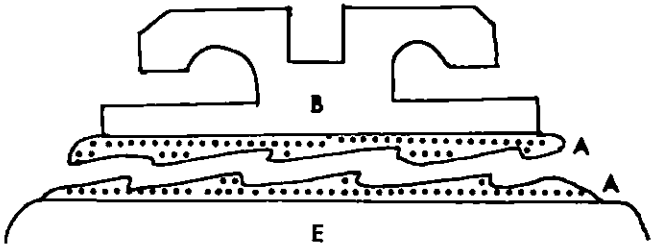
2. Cohesive failure within adhesive material

3. Combination cohesive and adhesive failure

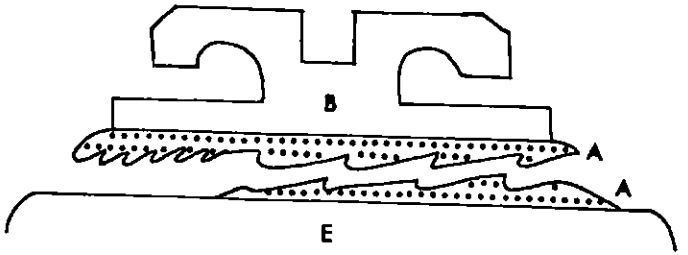
4. Adhesive failure at bracket-adhesive material interface



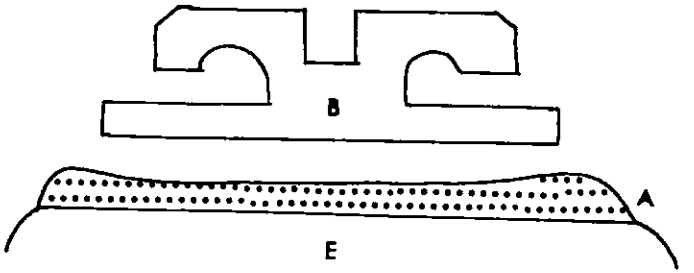

1




2




3




4

# DISCUSSION

The testing modes employed in this study were intended to duplicate the kinds of stresses applied clinically to polycarbonate attachments. Plate XXIII demonstrates clinical examples of the stresses placed upon orthodontic attachments. Pilot tensile and peel tests attempted to examine the tooth-adhesive-bracket system; however, 20 of 36 trail tensile tests yielded bent or distorted brackets. The testing jigs distorted the wings of the edgewise brackets. Orthodontic wire, placed under the bracket wings, yielded similar results (Plate XXIV).

The brackets for the tensile and peel stress modes were thus reinforced with epoxy in an attempt to circumvent this problem. Although reinforcement appeared to be a logical approach, it was not completely successful. Failure within the epoxy or at the epoxy-bracket interface did occur at times.

Testing in shear did not incorporate reinforced brackets; however, in 16 of 118 samples tested, the bracket wings were detached from the bracket base, leaving the latter bonded to the tooth. These results would seem to indicate that the polycarbonate material itself is a weak link in a bonding system.

Adhesive bond failures occurred at and were limited to the bracket base contact area (0.030 sq. in.). Although a flash of

PLATE XXIII

Clinical Examples

A. Case C.T.: An example of a modified tensile load upon the attachments on the maxillary lateral incisors. Treatment with a Begg appliance (Stage I), Orthomite adhesive and brackets.

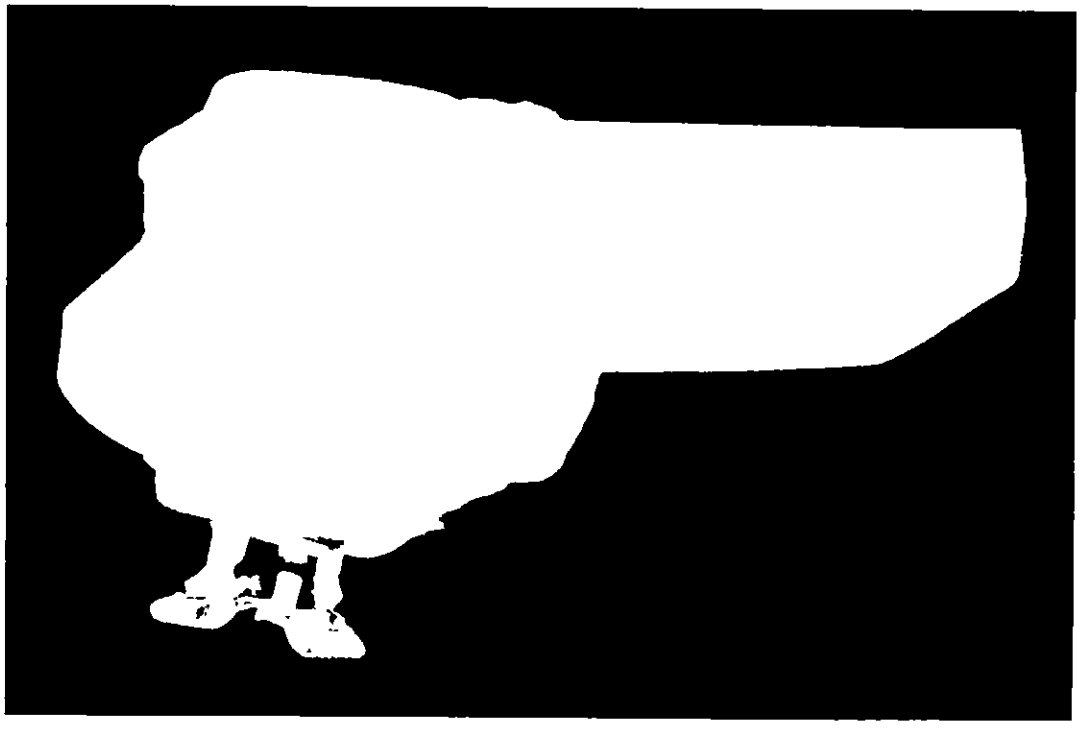
B. Case W.B.: An example of a modified tensile load upon the bracket on the maxillary left lateral incisor. Treatment with a Begg appliance (Stage I), TP Directon adhesive (without sealant) and brackets.

C. Case D.V.: An example of a modified shear load upon the edgewise bracket on the maxillary right canine. Treatment with an edgewise appliance (leveling), Unitek adhesive, and Rocky Mountain bracket--the remaining anteriors are bonded with Nuva-Seal-Bracket Bond and Rocky Mountain brackets.

D. Case K.K.: An example of a peeling stress applied to the maxillary left central incisor. Torquing key checks amount of torque before and after placing edgewise wire in bracket. Treatment with an edgewise appliance, Nuva-Seal-Bracket Bond Adhesive and Rocky Mountain brackets.

PLATE XXIV

Edgewise bracket with bent wings following trial tensile test



adhesive invariably spread beyond the boundary of the bracket base, SEM observations of the excess indicated that it did not extend the fracture surface area when an adhesive failure occurred. Plate XV, an adhesive failure at the tooth-adhesive interface, and Plate XXI, an adhesive failure at the bracket-adhesive interface, demonstrate this observation.

With the exception of the O.I.S. system and Nuva-Seal, the orthodontic adhesives (Group B) yielded cohesive or combination bond failures. When such failures occurred, the flash did become part of the fracture pattern. A strain developing within the adhesive propagated through the excess. For calculations, the bracket surface was still used to approximate the fracture area for strength determination.

As bond strength increased, more cohesive and combination failures occurred; the actual strength of the bond between the tooth and adhesive, or bracket and adhesive, would be greater than the values reported. This finding confirms that of Sheykholeslam (1970) investigating the bonding of adhesive resins (notably Sevriton).

The two composite resins from Group A--Enamelite and Nuvafil--as well as Nuva-Seal from Group B, did not adhere well to the brackets. All bond failures were adhesive, occurring at the bracket-resin interface. Treating the brackets with methacrylic acid, as suggested by Cohl et al. (1972), did not seem to improve the adhesion of Nuva-Seal to the brackets. SEM observations indicated that

etching of the bracket surface by the acid was not sufficiently extensive to lock the sealant to the bracket.

Waller (1972) tested the bond strengths of Nuva-Seal to polycarbonate brackets. His results (0-401 p.s.i.), using untreated bracket surfaces, are still greater than those in this report (0-192 p.s.i.). Waller also observed that all failures were at the bracket-adhesive interface. Cohl and his associates (1972) reported results of 448 ± 93 p.s.i. after 1 day, tested in tension, to 821 ± 116 p.s.i., after 30 days, tested in shear. They further noted a high incidence of failures at the bracket-adhesive interface.

Newman (1965) stated that a load of 10 pounds or 200 p.s.i. is probably the maximum clinical orthodontic load. However, as noted previously, soft tissue and masticatory forces must be taken into consideration. Enamelite, Nuva-Seal, and Nuvafil, when used as orthodontic direct bonding agents utilizing polycarbonate brackets, generally exhibit bond strengths below 200 p.s.i. When the Nuvafil extends beyond and over the bracket base, it appears to lock the bracket to the composite resin. Water, however, eventually decreases adhesion at the bracket-adhesive interface (one day vs. one month, $p < .01$ in tension).

O.I.S. is the only adhesive that incorporates a fluoride agent--sodium fluoride. Newman (1969b) reported that the fluoride acts as an attenuating agent. However, Sheykholeslam (1970)

reported that sodium fluoride applied prior to resin application resulted in reduced bond strength. The O.I.S. material incorporates a fluoride in both the etchant and adhesive powder. It was the only adhesive, in this report, manufactured specifically as an orthodontic bonding system, that consistently produced adhesive failures at the tooth-resin interface.

Two sealant-resin systems (Nuva-Seal-Bracket Bond and Directon Adhesive with Sealant) did not statistically differ from each other. Both incorporate an acrylic resin and a sealant. While no other results have yet been reported concerning Directon, results with the Nuva-Seal-Bracket Bond system compare favorably with those of Waller (1972). He reported bond strengths of 666-1314 p.s.i., using polycarbonate brackets. Most of his failures were bracket failures. The average in this report was 895 p.s.i., with strengths to 1314 p.s.i. recorded. Bond failures were cohesive or combination.

However, is a sealant necessary? Unitek, Orthomite, Directon Adhesive without Sealant and Sevriton, did not differ significantly in strength from each other or from the sealant-resin systems. A possible explanation may arise from the product constituents. All systems contain an acrylic resin: methyl methacrylate or polymethyl methylacrylate. A drying agent may be included (2-Butanone in the Unitek system), a coupling agent (methacryloxyprophyl trimethoxysilane in Orthomite) or a primer (vinyl silane in Directon or glycerophosphoric acid dimethacrylate in Sevriton). However,

drying agents can do no more than improve dryness, silane coupling agents have questionable value (Cass, 1966; Eirich, 1966), and the use of primers to increase the adhesion of a resin to enamel does not appear significant (Lee et al., 1971).

Initial bond strengths do not differ significantly from those obtained after water storage for one month. This confirms the study of Rensch (1973), who noted a significant deterioration in the in vivo bond strength of Sevriton and Durelon (polycarboxylate cement)[23], but not in his in vitro results.

## Clinical Observations

While it was not intended to include a clinical study as part of this investigation, it is important to provide a clinical perspective for these in vitro tests. Over the past seventeen months, adhesive materials have been used by various clinicians in the Department of Orthodontics, School of Dentistry, Case Western Reserve University. Both metal and plastic (polycarbonate) attachments were bonded with various adhesive materials. The results are presented in Tables VI and VII.

While the overall success rate for metal and plastic attachments is 65 percent, the success rate for this investigator approximates 90 percent. This includes all systems, observed over a seventeen

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[23]Premier Dental Product Company, Philadelphia, Penn. 19107

TABLE VI

CLINICAL EXPERIENCE OF THE DEPT. OF ORTHODONTICS, C.W.R.U.

NOV. 1971-APRIL 1973 WITH DIRECT BONDED

PLASTIC ATTACHMENTS

| Adhesive | Number Placed | % Lost[a] | % Removed[b] | Observation Period (mo.)[c] |
|---|---|---|---|---|
| Nuva-Seal-Bracket Bond | 123 | 13 | 19,5 | 9 |
| Directon without Sealant | 43 | 16.3[d] | 11.6[d] | 3 |
| Orthomite | 28 | 17.8 | 0 | 6 |
| Unitek | 119 | 24.4 | 3.4 | 5 |
| O.I.S. | 8 | 25 | 75 | 12 |
| Directon with Sealant | 46 | 52.2[d] | 13[d] | 3 |

[a]Bracket loss indicates a joint failure, either adhesive, cohesive or combination.

[b]Brackets removed indicates an adequate bond; however, the bracket was distorted, bent, broken, or interfering with occlusion.

[c]Observation period from the time the adhesive was first applied in the clinic, not necessarily the duration of bonding to teeth.

[d]The first shipment of TP Directon products included defective brackets (H.D. Kesling, 1973).

TABLE VII

CLINICAL EXPERIENCE OF THE DEPT. OF ORTHODONTICS, C.W.R.U.

NOV. 1971-APRIL 1973 WITH DIRECT BONDED

METAL ATTACHMENTS

| Adhesive | Number Placed | % Lost[a] | % Removed[b] | Observation Period (mo.)[c] |
|---|---|---|---|---|
| Nuva-Seal-Nuvafil | 6 | 0 | 0 | 12 |
| Durelon | 6 | 16.7 | 0 | 17 |
| Enamelite | 7 | 28.6 | 0 | 12 |
| Sevriton | 20 | 35 | 0 | 17 |

[a]Bracket loss indicates a joint failure, either adhesive, cohesive or combination.

[b]Brackets removed indicates an adequate bond; however, the bracket was distorted, bent, broken, or interfering with occlusion.

[c]Observation period from the time the adhesive was first applied in the clinic, not necessarily the duration of bonding to teeth.

month period. Some attachments have been bonded for as long as 14 months, others for only 4 months.

A success rate of 90 percent compares favorably with that reported by the following investigators: Miura and his associates (1971), using the Orthomite system, 93.9 percent; Dean (1972) and Sciaretta (1972), using the Nuva-Seal-Bracket Bond system, 92.2 percent; and Mizrahi and Smith (1971), using Durelon and metal attachments, 88 percent. Dijkman and Retief (1972) reported a 64.3 percent success rate using a modified epoxy system. All of these studies conclude that the success rate decreases significantly when posterior teeth are bonded.

Many factors can affect the clinical results: (1) adequate etching, (2) clinically dry surfaces, (3) adhesive viscosity, (4) case selection, and (5) operator experience and judgment.

## SEM Observations

The observations of etching agents on the enamel surface, and the pattern of bond failure, confirmed several views: (1) acid attack of natural enamel surfaces may include demineralization of the central portion of the prism, periphery dissolution, or a combination of the two (Sheykholeslam, 1970; Nygaard and Simmelink, 1972; Retief, 1973; Gwinnett, 1973c; Gwinnett and Ripa, 1973); (2) resin tags play a significant role in increasing the mechanical interlocking of the resin into enamel (Buonocore, 1955;

Gwinnett and Matsui, 1967; Sheykholeslam, 1970); (3) porosities are often present within an adhesive and may influence the bonding strength of the material (Sheykholeslam, 1970); and (4) the appearance of a smooth border at the Nuva-Seal-Bracket Bond interface can not rule out the possibility of a chemical bond between sealant and resin (Dean, 1972; Sciaretta, 1972; Glanelly, 1973).

Smith (1972) claimed SEM observations confirmed chemical interaction between polycarboxylate cement and underlying calcified tissues; Beech (1972) suggested that ionic bonds form between the calcium ions in the enamel and the polyacrylic acid in the cement. While the present report did not examine the polycarboxylate cements, the adhesives investigated appeared to mechanically lock into the pores created by the acid conditioning.

## Adhesive Systems Review

Since there is no significant statistical difference among the bond strengths of the various orthodontic adhesives, what criteria could be used in selecting a product? Ease of manipulation and unit cost (economic factors) are two important factors.

The Nuva-Seal-Bracket Bond system requires an ultra-violet light, an initial expensive undertaking. However, the mean bond strength was high, and auxiliary help could substantially reduce the patient-doctor chair time.

The Unitek system is moderately priced and has an adequate working time, if the mixing vessel is cool. A mix produced according to instructions will yield more adhesive than needed.

The Orthomite system advocates a bead (brush) technique, allowing for better control of material flow. However, the kit is relatively expensive, and a bottle of monomer, once activated, is stable for only 15 minutes.

The Directon Adhesive system is one of the lowest priced kits, and the adhesive alone appears adequate for direct bonding. Although the resin sets rapidly, small portions can be mixed.

While the O.I.S. system is relatively easy to use, its effectiveness, as presented in this report, is questionable. The clinician must, in the end, make the final decision.

## SUMMARY AND CONCLUSIONS

The purpose of this investigation was to examine the bond strengths of ten dental "adhesive" materials. The materials included three dental restoratives, a pit and fissure sealant, and six orthodontic direct bonding systems. The protocol attempted to duplicate, *in vitro*, the steps necessary to bond polycarbonate edgewise brackets to teeth in clinical cases.

Thirty-six teeth were bonded with each material; the teeth were examined in tensile, shear and peel stress modes, one-half of the sample after a one day storage in water (37°C), the remaining after one month in storage.

Analysis of the data following the tests was accomplished with a 2 X 10 factorial analysis of variance. While there was a significant difference among the means of all the materials ($p < .001$), a Tukey's test to compare individual means yielded no difference among the orthodontic direct bonding systems ($p > .05$). The two composite resins and the sealant demonstrated significantly weaker bond strengths to the polycarbonate brackets.

Testing the samples after one day or one month following storage in 37°C. distilled water was not significant ($p > .05$). The one instance when a significant difference was recorded

(tensile test), it was attributed to the weakness of the reinforcing epoxy cap, which was also used in the peel test.

Examination of the bond failures was undertaken with stereoscopic microscope and scanning electron microscopy. Four types of bond failures were apparent: (1) an adhesive failure at the enamel-adhesive material interface, (2) a cohesive failure within the adhesive material, (3) a combination of both adhesive and cohesive failure, and (4) an adhesive failure at the bracket-adhesive interface.

The orthodontic direct bonding systems, with one exception, demonstrated cohesive failures when bond failures occurred. The composite restoratives and the sealant presented adhesive failures at the bracket-adhesive interface.

With no statistically significant difference noted among the orthodontic bonding systems, the clinician may be free to choose the system that he feels best fulfills his requirements.

The results may be summarized as follows:

(1) There was no statistically significant difference among the systems specifically designed as orthodontic adhesives.

(2) The two composite resin restoratives and the pit and fissure sealant did not bond satisfactorily to the polycarbonate brackets.

(3) Bond failures included several fracture patterns--adhesive, cohesive, or a combination of the two.

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## APPENDIX

## AN OUTLINE OF THE PROCEDURES FOLLOWED IN THE APPLICATION OF THE MATERIALS EXAMINED IN THIS REPORT

### Enamelite Enamel Coating

I. Source: Enamelite Technical Bulletin, February 7, 1972; Lee Research Report 72-111

II. Tooth surface preparation
  A. Cleansing
    1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
    2. Pumice facial surfaces of teeth for 15 seconds.
    3. Rinse with water
    4. Dry with compressed air.
  B. Etching
    1. Apply cleanser (jar #1: 50 percent phosphoric acid) to enamel surface for 120 seconds with a cotton pellet.
    2. Rinse with water,
    3. Dry with compressed air.

III. Adhesive preparation
  A. On first use, activate enamelite kit by adding contents of jar #2' to jar #2; mix thoroughly.
  B. Shelf-life of jar #2 prolonged by refrigerating.

IV. Bracket preparation
  A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
  B. Wipe contact surface of bracket base.
  C. Dry with compressed air.

V. Application procedure
  A. Mix 4 drops of activated jar #2 with 4 drops of jar #3; spatulate for 15 seconds.
  B. Apply to bracket base with spatula.
  C. Place on tooth.
  D. Allow 15 minutes for setting.

VI. Notes
  A. Working time is 90 seconds.
  B. One mix was used for six teeth.
  C. Lot numbers
    1. jar #1: AE 0040
    2. jar #2: AE 0034
    3. jar #3: AE 0030

## Directon Adhesive with Sealant

I. Source: Instructions supplied with kits

II. Tooth surface preparation
  A. Cleansing
    1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
    2. Pumice facial surface of teeth for 15 seconds.
    3. Rinse with water.
    4. Dry with compressed air.
  B. Etching
    1. Apply conditioner (phosphoric acid) to enamel surface for 60 seconds with a cotton pellet.
    2. Rinse with water.
    3. Dry with compressed air.

III. Adhesive preparation

IV. Bracket preparation
  A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
  B. Wipe contact surface of bracket base.
  C. Dry with compressed air.

V. Application procedure
  A. Carefully paint one coat of primer (vinyl silane) over enamel surface.
  B. Sealant application
    1. Combine 1 measure of sealant powder with 5 drops of sealant liquid on mixing pad.
    2. Mix for 15 seconds.
    3. Apply to enamel surface with foam pellet.
    4. Allow 2-3 minutes for setting.
  C. Adhesive application
    1. Combine 1 level scoop of adhesive powder with 4 drops of adhesive liquid on mixing pad.
    2. Mix 10-15 seconds.
    3. Dip base of attachment into mix.
    4. Apply to tooth.
    5. Allow 15 minutes for setting.

VI. Notes
- A. One mix of sealant was used for six teeth.
- B. One mix of adhesive was used for two teeth.
- C. Lot numbers were not provided.

Directon Adhesive without Sealant

I. Source: Instructions supplied with kit

II. Tooth surface preparation
- A. Cleansing
  1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
  2. Pumice facial surfaces of teeth for 15 seconds.
  3. Rinse with water.
  4. Dry with compressed air.
- B. Etching
  1. Apply conditioner (phosphoric acid) to enamel surface for 60 seconds with a cotton pellet.
  2. Rinse with water.
  3. Dry with compressed air.

III. Adhesive preparation

IV. Bracket preparation
- A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
- B. Wipe contact surface of bracket base.
- C. Dry with compressed air.

V. Application procedure
- A. Carefully paint one coat of primer (vinyl silane) over enamel surface.
- B. Combine 1 level scoop of adhesive powder with 4 drops of adhesive liquid on mixing pad.
- C. Mix 10-15 seconds.
- D. Dip base of attachment into mix.
- E. Apply to tooth.
- F. Allow 15 minutes for setting.

VI. Notes
- A. One mix of adhesive was used for two teeth.
- B. Lot numbers were not provided.

Nuva-Seal Adhesive System

I. Source: Cohl et al., 1972

II. Tooth surface preparation
- A. Cleansing
  1. Apply flour of pumice paste to a stiff dental prophylaxis brush.

2. Pumice facial surfaces of teeth for 15 seconds.
3. Rinse with water.
4. Dry with compressed air.

B. Etching
1. Apply Nuva-Seal conditioner (50 percent phosphoric acid with 7 percent zinc oxide by weight) to a cotton pellet.
2. Continually wipe etchant over enamel for 60 seconds.
3. Rinse with water.
4. Dry with compressed air.

III. Adhesive preparation
A. Prepare Nuva-Seal sealant by dispensing 1 drop of initiator into bottle of sealant base.
B. Stir and mix for a few seconds
C. Mixed sealant has a shelf-life of one working day.

IV. Bracket preparation
A. Precondition contact surface of bracket base by moistening with methacrylic acid for 2 minutes.
B. Rinse with water.
C. Dry with compressed air.

V. Application procedure
A. Brush mixed sealant over enamel surface using overlapping strokes.
B. Brush mixed sealant over contact surface of bracket base.
C. Place bracket on tooth.
D. Place tip of Nuva-Lite quartz rod 2 mm. from bracket-tooth interface.
E. Activate light for 20 seconds (20 seconds used for each side of bracket; total of 60 seconds per tooth)
F. Allow 15 minutes before placing in water.

VI. Notes
A. Nuva-Seal base lot no. 9657

Nuva-Seal-Bracket Bond Direct Adhesive System

I. Source: Instructions supplied with Bracket Bond kit; Dean, 1972; Sciaretta, 1972; Dietz, 1972; Silverman et al., 1972

II. Tooth surface preparation
  A. Cleansing
    1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
    2. Pumice facial surfaces of teeth for 15 seconds.
    3. Rinse with water.
    4. Dry with compressed air.
  B. Etching
    1. Apply Nuva-Seal conditioner (50 percent phosphoric acid with 7 percent zinc oxide by weight) to a cotton pellet.
    2. Continually wipe etchant over enamel for 60 seconds.
    3. Rinse with water.
    4. Dry with compressed air.

III. Adhesive preparation
  A. Prepare Nuva-Seal sealant by dispensing 1 drop of initiator into bottle of sealant base.
  B. Stir and mix for a few seconds.
  C. Mixed sealant has a shelf-life of one working day.

IV. Bracket preparation
  A. Moisten contact surface of bracket base with the Bracket Bond liquid (after sealant is on tooth).

V. Application procedure
  A. Nuva-Seal sealant application
    1. Brush mixed sealant over enamel surface using overlapping strokes.
    2. Place tip of Nuva-Lite quartz rod 2 mm. from Nuva-Seal surface
    3. Polymerize Nuva-Seal by activating light for 30 seconds.
    4. Gently wipe unpolymerized sealant from surface with a moistened cotton pellet.
    5. Dry with compressed air.
    6. Wipe sealant surface with cotton pellet moistened with Bracket Bond liquid.
  B. Bracket Bond adhesive application
    1. Combine 1/2 scoop of powder with 3 drops of liquid.
    2. Mix for 7 seconds.
    3. Place mixture on contact surface of bracket.
    4. Place on tooth.
    5. Allow 15 minutes for setting.

VI. Notes
- A. One mix of Bracket Bond adhesive was used for each tooth.
- B. The Nuva-Lite emits an ultra-violet light beam of 3600 Å.
- C. Nuva-Seal lot no. 9657.

Nuva-Seal-Nuvafil System

I. Source: Instructions for Nuva-Seal; Instructions for Nuvafil (ultraviolet restorative)

II. Tooth surface preparation

A. Cleansing

1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
2. Pumice facial surface of teeth for 15 seconds.
3. Rinse with water.
4. Dry with compressed air.

B. Etching
1. Apply Nuva-Seal conditioner (50 percent phosphoric acid with 7 percent zinc oxide by weight) to a cotton pellet.
2. Continually wipe etchant over enamel for 60 seconds.
3. Rinse with water.
4. Dry with compressed air.

IV. Adhesive preparation
- A. Prepare Nuva-Seal by dispensing 1 drop of initiator into bottle of sealant base.
- B. Stir and mix for a few seconds
- C. Mixed sealant has a shelf-life of one working day,
- D. Prepare new jar of Nuvafil by adding one drop of initiator.
- E. Mix thoroughly, and before each use
- F. Mixed restorative has a shelf-life of 3 months.

V. Application procedure
- A. Nuva-Seal application
  1. Brush mixed sealant over enamel surface using overlapping strokes.
  2. Place tip of Nuva-Lite quartz rod 2 mm. from Nuva-Seal surface.
  3. Polymerize Nuva-Seal by activating light for 30 seconds.
  4. Gently wipe unpolymerized sealant from surface with a moistened cotton pellet.
  5. Dry with compressed air.

B. Nuvafil application
   1. Apply Nuvafil to back of bracket.
   2. Press bracket onto sealant-coated enamel.
   3. Activate Nuva-Lite for 45 seconds per bracket side (total of 3 minutes per tooth)
   4. Allow 15 minutes before placing in water.

VI. Notes
   A. Nuva-Seal base lot no. 9657.
   B. Nuvafil (ultraviolet restorative) lot no. H3-148-1, shade #66, dated October 26, 1972, activated February 19, 1973.

## O.I.S. Adhesive System

I. Source: Instruction manual supplied with kit; Newman, 1969a.

II. Tooth surface preparation
   A. Cleansing
      1. Apply fluoride pumice paste (Nupro gold, manufactured by Janar Company, Grand Rapids, Michigan, 49501) to a stiff dental prophylaxis brush.
      2. Pumice facial surfaces of teeth for 15 seconds.
      3. Rinse with water.
      4. Dry with compressed air.
   B. Etching
      1. Place 2 drops of surface treating liquid (low pH phosphoric acid with 1 percent sodium fluoride and zinc oxide) on a cotton pellet.
      2. Apply liquid etchant to tooth.
      3. Wait 60 seconds.
      4. Rinse with water.
      5. Dry with compressed air.

III. Adhesive preparation

IV. Bracket preparation
   A. Roughen the contact surface of the bracket base with an emery board or sand paper disc.
   B. Dry with compressed air.

V. Application procedure

   A. Combine 7 drops of the liquid monomer with 2 scoops of the powder (polymer containing 3 percent monofluorophosphate).
   B. Mix for 15-30 seconds.

C. Apply to bracket base with a spatula.
D. Press on tooth for 2 seconds.
E. Allow 15 minutes for setting.

VI. Notes
A. Setting time is rapid; one mix of adhesive was used for two teeth.
B. Lot number not provided

Orthomite Direct Bonding System

I. Source: Instruction manual supplied with kit; Miura et al., 1971; DBS/Notes, issue no. 2, 1972.

II. Tooth surface preparation
A. Cleansing
1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
2. Pumice facial surfaces of teeth for 15 seconds.
3. Rinse with water.
4. Dry with compressed air.
B. Etching
1. Apply solution A (65 percent phosphoric acid) for 30-60 seconds to the enamel surface; 60 seconds was used.
2. Rinse with water.
3. Dry with compressed air.

III. Adhesive preparation
A. Use press provided to press-down on top of container C.
B. Mix catalyst (tri-n-butylborane derivative) with monomer by shaking bottle for 4-5 seconds.
C. Solution C is stable for 15 minutes.

IV. Bracket preparation
A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
B. Wipe contact surface of bracket base.
C. Dry with compressed air.

V. Application procedure
A. Apply solution B (methacryloxyprophyl tri-methoxy-silane) to a cotton pellet and place on tooth.
B. Spread liquid over enamel surface with a forceful stream of compressed air.
C. Use a brush-on technique to apply beads of monomer-polymer (powder D) mixture to enamel surface.

D. Clean brush with dry 2 X 2 gauze pad after each application.
E. Place bracket on tooth.
F. Fill voids with beads of adhesive.
G. Allow 15 minutes for setting.

VI. Notes
A. One bottle of monomer was used for six teeth.
B. Lot numbers
1. Monomer #B044
2. Powder #013
3. Solution A #013
C. Kit expiration date: June 1973.

Sevriton Simplified Acrylic Filling Material

I. Source: Instructions for use, Amalgamated Dental Trade Distributors, Ltd., London, England; Instruction: Clinical Application of Direct Bonding of Brackets, Unitek Corporation, 1971.

II. Tooth surface preparation
A. Cleansing
1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
2. Pumice facial surfaces of teeth for 15 seconds.
3. Rinse with water.
4. Dry with compressed air.
B. Etching
1. Apply etchant (50-60 percent phosphoric acid--Ames zinc oxyphosphate cement liquid) to a cotton pellet.
2. Apply to tooth for 4 minutes.
3. Rinse with water.
4. Dry with compressed air.

III. Adhesive preparation

IV. Bracket preparation
A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
B. Wipe contact surface of bracket base.
C. Dry with compressed air.

V. Application procedure
A. Using a cotton pellet, place cavity seal (glycero-phosphoric acid dimethacrylate, in methyl methacrylate monomer) over enamel surface.
B. Place 8 drops of liquid monomer (methyl methacrylate) into a mixing vessel.

C. Add the powder (polymethyl methacrylate containing sulfinic acid accelerator and sodium fluoride) until a dry excess is obtained.
D. Tap mixing vessel on a firm surface several times.
E. Tap-out excess powder.
F. Add 2 drops of liquid monomer; stir for 15 seconds.
G. Place resin on bracket base with spatula.
H. Apply to tooth.
I. Hold with light pressure for a few seconds.
J. Allow 15 minutes for setting.

VI. Notes
A. Working time is approximately 100 seconds.
B. One mix was used for three teeth.
C. Lot numbers not available.

Unitek Direct Bonding System

I. Source: Instruction manual supplied with kit

II. Tooth surface preparation
A. Cleansing
1. Apply flour of pumice paste to a stiff dental prophylaxis brush.
2. Pumice facial surfaces of teeth for 15 seconds.
3. Rinse with water.
4. Dry with compressed air.
B. Etching
1. Apply etching gel (60 percent phosphoric acid) to facial surfaces of teeth.
2. Allow to remain for one minute.
3. Rinse with water.
4. Dry with compressed air.
5. Dry with drying agent (2-Butanone) applied with a cotton pellet.

III. Adhesive preparation

IV. Bracket preparation
A. Apply ethanol (70 percent) to a 2 X 2 gauze pad.
B. Wipe contact surface of bracket base.
C. Dry with compressed air.

V. Application procedure
A. Place one level scoop of cement powder (polymethyl methacrylate) into a dappen dish.
B. Empty measured portion of cement liquid from pipette into dappen dish containing powder.

ACCOUNT NO. 541-2501

TITLE NO. 5(59)

ISSN NUMBER

VOL/YR | FREQUENCY | PICKUP

NEW TITLE ☐ RUB SENT ☐ SAMPLE SENT ☐

| | F | R | NP | | IN | OUT | EX. |
|---|---|---|---|---|---|---|---|
| TITLE PAGE | | | | FRONT COVER | | | |
| TABLE CONT. | | | | REAR COVER | | | |
| INDEX | | | | ADVERTISING | | | |

COLLATE INST.

SEE PATTERN S81

| | | | |
|---|---|---|---|
| POCKET | YES NO | SEW-THRU-FOLD | YES NO |
| SINGLES S.T.F. | YES NO | DON'T RESEW | YES NO |
| ADHESIVE BIND | YES NO | EXTRA TIME | |

STAMP INST.

P 11

F 86

Double Slug

PICA | TITLE | STYLE

VERTICAL LETTERING

NAGEL, N. J.

A MATERIALS EVALUATION OF TEN DIRECT BONDING SYSTEMS UTILIZING POLYCARBONATE BRACKETS

1973

CALL NUMBER

PRINT COLOR WHITE

TRIM HEIGHT

BUCKRAM COLOR 2075

BUCKRAM SIZE 16

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