AN 'in vitro' STUDY OF THE EFFECT OF COUPLING AGENTS ON ADHESION IN FIXED BANDELESS ORTHODONTICS

by

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A thesis embodying original work submitted in fulfilment of the requirements for the degree of Master of Dental Surgery.

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ACKNOWLEDGMENTS

For their interest, assistance and support in the preparation of this thesis, I would like to thank Associate Professor K. Godfrey and Dr. R. Earnshaw. I would also like to thank Associate Professor P. Barnard for his assistance with the evaluation of my results for significance, and Miss Bischoff from the photography section of the Dental Hospital of Sydney.

I would like to thank Mr. John Jennings of Rocky Mountain Dental Products for the provision of the Orthomix Direct Bonding System supplies.

For their encouragement and their patience, I would like to thank my partner, Mr. Archer Broughton and my wife, Judith. I am, in addition, especially indebted to my wife for her toil at the typewriter.
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CHAPTER 1

INTRODUCTION

With the advent of improved adhesion of resins to tooth enamel by acid pretreatment* and improvements in the physical character of resins themselves, principally by the use of bonded particle fillers,** also more diverse handling properties by the introduction of U.V.L. initiated polymerization,*** the profession now seems poised for a further advance in adhesive dentistry. This advancement would seem well served by further investigation of the possibility of achieving improved bonding to tooth structure through the medium of a coupling agent.

Baier(6) says that "there would be many advantages in knowing how to exert better control over surface interactions in vivo* either to promote or inhibit adhesion. We would like to improve adhesion of filling materials in teeth and of the opposed faces of cut tissues; we would like to impede adhesion of bondages to some wound surfaces and of marine biota to structural materials. The concept of tailor-making surface-active modifiers holds considerable promise for eventually

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meeting those needs.

One of the coupling agents to be discussed in this thesis is a silane coupling agent. It will be introduced in two different contexts: one is in a mechanism that has been postulated as bonding tooth enamel to a plastic resin; the other is in the bonding of a plastic resin to glass.

Problems facing adhesives chemists in the industrial field seem to have been largely in the realm of pure chemistry. The materials involved have been such that their chemical and physical nature has been entirely understood. Success has been more predictable, not apparent only from hindsight.

It would be false to make the same claim for tooth surfaces. When a dental research worker outlines a mechanism on the chemistry of a chelating reaction, or a coupling phase, or an ionic bond, in relation to a tooth surface, he is hypothesising. When the adhesives chemist is describing the reason why silane will bond glass to plastic he is outlining a chemical law. The law of ionic bonding is being invoked in both cases, but the evidence indicates that in the
case of silica and glass it is correct, whereas with silica and enamel it is hypothetical and may be incorrect.

The two different contexts in which silica features in this thesis seem to illustrate the problem that faces research in the field of adhesives in dentistry. If tooth enamel were a chemical complex as well understood as, say, the surface of glass, mechanisms by which a silica coupling agent could enhance adhesion would be readily explainable on the basis of current knowledge. But although tooth enamel is not considered to be a vital tissue it cannot be regarded only as a chemical complex. It must be viewed as a biological entity.

Dental cements in general use, with one possible exception, are not adhesives; they are lutes. Most general adhesives for everyday use are either not effective or not suitable as dental adhesives. Dentistry does not appear to have benefited from the enormous advances in adhesives in the industrial field.

The purpose of this investigation is to test the bond strengths of two direct bonding systems involving the cementation of orthodontic brackets to tooth enamel after immersion in water and storage at 37°C in vitro. These two systems are to be tested with and without the coupling agents recommended, in an endeavour to evaluate their effectiveness.

Due to the difference in tooth samples, namely that one half consists of teeth which are being bonded for the first time, whilst the other consists of teeth which have been previously bonded (and the brackets removed during testing and the surfaces re-prepared), a comparison is to be made between the adhesive bonds formed under the different conditions.

In addition to the tests of bond strength, the site of the bond failures is to be examined with a stereoscopic microscope to determine the nature of the failures and to note anything appropriate to the experiment.

The requirement is that the adhesive form a bond between the adherend and the substrate that is capable of resisting degradation from the oral environment and the various displacing forces encountered during orthodontic therapy. The adhesive, in addition
to its primary function, must be non-toxic and must be capable of removal from the tooth surface without impairment to the tooth and its supporting structures. The properties of the adhesive must be such that it can be manipulated satisfactorily under mouth conditions and without undue discomfort to the patient.

Extensive research has been undertaken in an endeavour to find a material that will bond adequately to tooth structure.* The requirement for adhesion of orthodontic brackets to enamel is for bonding for a limited period, whereas that for a tooth restoration is much more demanding.

In a review of the literature it is proposed to discuss various modes of adhesion and the particular problems relating to adhesion in the mouth. Specific adhesives and coupling agents, and conditioning of tooth surfaces for bonding are also discussed.

In an endeavour to place a relatively new procedure such as acid etching of enamel in better perspective, a comparison has been made between its effect and that of routine instrumentation in dental prophylaxis on tooth surface.

The original work in the thesis embraces the development

of a laboratory testing technique and an evaluation of the effect of two coupling agents on adhesion of plastic brackets to enamel.

The author has had four years of clinical experience of the direct bonding techniques described in this thesis, involving the placement of two hundred brackets, mainly on upper anterior teeth. Although this work cannot be included in an "in vitro" study such as this one, it would not seem inappropriate to mention some of the factors that will be evident to any clinician using such a technique.

Firstly, it becomes manifest that existing direct bonding techniques, (all of which involve the same principle of resin cements and acid-etched enamel) do not constitute a replacement for conventional bonding. They represent another tool in the orthodontist's armamentarium.

Secondly, the shortcomings of polycarbonate brackets with regard to strength, when it comes to rotations and uprighting procedures in particular, emphasise the need for improved bracket materials. Giannelly(62) says Lexan brackets are superior to other polycarbonates. Metal brackets can be used by mechanically interlocking resin and a special base.(86)(149)(150) Although this overcomes failure in the bracket itself, it offsets some of the aesthetic appeal of a plastic bracket.
Thirdly, one of the greatest disadvantages of directly bonded brackets in a clinical situation is in their replacement in the event of dislodgment or damage. It is very time-consuming, as it often involves complete removal of an arch wire in addition to re-preparation of enamel for re-bonding. The replacement of a dislodged conventional metal band is much more simple.

Lastly, the advantages claimed for bonded brackets with regard to decreased gingival irritation and plaque retention may be partly valid, but there are considerable problems. These problems still seem to arise in those cases in which gingival involvement can be foreseen even before commencement of treatment and even where the patient is co-operating to a high degree to overcome his predisposition to periodontal disease.

Despite those drawbacks, the achievements in adhesive dentistry are encouraging. In the removal of bonds at the conclusion of active therapy, considerable difficulty can be encountered in the displacement of all the resin from the enamel surface. Such a phenomenon suggests very high retentive forces at work. Further investigation of the potential for improved adhesion through the medium of coupling agents would seem well worthwhile.
CHAPTER 2

REVIEW OF LITERATURE

ADHESION

Introduction

Adhesion to a tooth surface, whether it is attempted directly by an adhesive or through the medium of a surface-active modifier, needs to be considered firstly in the light of what is known of the nature of adhesion.

The Concise Oxford Dictionary defines adhesion as:

"the action of sticking to, by attraction viscosity of surface or grasping". This term is narrowed down to a more technical level, definitions such as these are given:

Adhesion is the joining together of two dissimilar materials. (138)

Adhesion is sticking to a surface. (63)

Adhesion is the bonding of dissimilar materials by the attraction of atoms or molecules. (141)

Adhesion is the state in which two surfaces are held together by interfacial forces which may consist of valence forces or interlocking action, or both. (138)
When the term is applied to the adherence of resin to tooth structure there seems to be some disagreement on just when adhesion has been achieved.\(^{(2)(103)}\)

Further to this, Parker and Taylor\(^{(136)}\) say that an adhesive can be defined as any material that causes one body to stick to another and Brauer\(^{(24)}\) that "an adhesive substance is a material capable of adhoring or sticking to a surface, primarily to promote a bond between that surface and another material."

It may seem pedantic to labour over such a fundamental term as adhesive or adhesion, but it is becoming increasingly obvious that the nearer that true adhesion to tooth surface is approached clinically, the more difficult it will become to define the term satisfactorily.\(^{(2)(103)}\) There is considerable agreement that adhesion of resin to acid-etched tooth enamel is mainly a process of mechanical interlocking.\(^{(36)(54)(72)(98)}\) However, there are areas in the literature where it is obvious that other factors are operating: the apparent coupling action associated with acidulated sodium fluoride in a study by Shaykhoulou et al.\(^{(173)}\) supported by Skinner\(^{(176)}\) and Glantz\(^{(63)}\) remarks about fluoride and adhesion; the various
claims made for a silane coupling agent (86)(97)(102)(117)(120)
for Sowriton sealer (30)(38)(186) and for Bouan's surface-active
comonomer (18)(19)(20)(41)(159)

The Theory of Adhesion

The attractive forces involved in adhesion can be either
chemical or physical.

Chemical Forces

Chemical forces link atoms together (e.g., in crystals and
molecules). These involve sharing or transfer of valency electrons
amongst neighbouring atoms. An example of this type of bonding
is a spot weld which consists of molten and resolidified alloy.
The joint between the pieces of metal is as strong as any part
of the whole. When it is appreciated that the distances of
separation are of the order of one ten millionth of a millimetre
it can be seen that in adhesives technology true chemical
bonding is a rare thing.

The chemical forces that link atoms together to form
molecules are:
1. Ionic (heteropolar) bonds

These bonds involve the transfer of electrons from one valency orbit to another. Solid crystalline salts are typical electrovalent compounds. (136) The ionic bond in such a salt as a sodium chloride crystal is very strong and extremely stable to heat, but readily dissociated by a polar solvent such as water, forming positively charged sodium ions and negatively charged chloride ions. Ionic bonds are not significant in adhesive forces. (136)

2. Covalent (homopolar) bonds

These bonds involve the sharing of electrons between valency orbits as opposed to the transfer that occurs with ionic bonds. They may be polar or non polar, depending on whether the atoms are dissimilar (polar) or whether they are similar and involve equal sharing of electrons (non polar).

Covalent bonds are found in the whole field of organic chemistry, embracing gaseous, liquid and solid states, and are important in adhesion. (136)

3. Metallic bonds

Chemically clean metal surfaces have sites available for bonding due to electron deficiency on the surface. Electron mobility gives rise to some properties of metals.
Physical Forces

Physical forces are those which hold separate molecules together. They are weaker than chemical bonds and they are weak forces due to stray electrons. They are sometimes called "forces of adhesion" as they are usually the ones responsible for adhesive bonds. If attraction by physical forces takes place, the distance of separation of molecules across the interface cannot be more than two or three Angstrom units. If two surfaces could be prepared to a degree of flatness within the limits demanded by either physical or chemical bonding, then no adhesive would be necessary to create a bond between these two surfaces. The surfaces would merely need to be chemically clean and by pressing them together a bond would be formed.

An example of this type of adhesion is the bond between mica sheets. The strength of the adhesion is in the order of 14,000 lbs/sq. inch, which is as strong as the mica itself.

Under the most ideal circumstances, the finest surface that can be produced has a degree of roughness that is about one hundred Angstrom units. That sort of finish is quite impossible with dental instruments, in fact the best that can be produced in cavity preparation is a surface about one million times as rough as the ideal. It is for this reason that we need
adhesives. They fill the spaces caused by surface irregularities thus increasing the forces of attraction.¹⁵³

Lee¹⁰⁰ in examining the mechanism of adhesion, lists the following forces which can appear in a bonded joint:

1. Mechanical locking (present in some rough surfaces).
2. Gravitational forces (negligible).
3. Electrostatic forces (usually negligible).
4. Physical-chemical absorption forces which include van der Waals, London dispersion forces and hydrogen bonds (principal adhesive forces).
5. Covalent or polar bonds (present in some type of adhesives.)

The physical forces that hold separate molecules together are referred to as:

1. Van der Waals forces

Van der Waals forces are generally classified into Keesom, Debye and London forces.

Keesom Forces

Keesom forces result from the interaction of two
permanent dipoles, the result of a polar covalent bond. They are important in adhesive action and examples are those bonds formed from carbon and oxygen, carbon and nitrogen, carbon and a halogen etc. (136) They are considered the principal van der Waals forces. (102)

Debye Forces

Debye forces result from the interaction of a permanent dipole with a bond system capable of being polarised. Several systems are capable of being polarised, especially carbon—carbon bonds. These bonds are not significant in adhesion.

London Forces

London Forces (dispersion forces) do not depend on permanent dipoles. They result from the polarisation of one molecule by another due to the oscillation of electron clouds. (122) The term dispersion refers to the fact that these attractive forces arise from the same oscillations that are also responsible for the dispersion of light by the molecules. (102)

2. Hydrogen Bonds

Hydrogen bonds are regarded as a special type of dipole —

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* Glantz (63) defines a dipole as an atom or molecule in which the effective centres of the positive and negative charges are separated.
dipole interaction. \((41)(151)\) It is not considered of importance in adhesion. The bonds are found in water, not as \(H_2O\) but as \((H_2O)_3^*\).

3. Pi-Adducts

These are special types of bonds mentioned by Glantz, \((63)\) occurring in unsaturated hydrocarbon chains. They involve the so-called pi and sigma-electrons. Pi-electrons are the comparatively free electrons in peripheral bonds, whilst sigma-electrons are the more stable ones in long hydrocarbon chains. Under certain conditions the pi-electrons may interact with elements of other molecules, giving rise to attractive forces.

Smith \((179)\) points out that the diverse origins of the terms used to describe substances which bond materials together are an indication of their various compositions and functions. The broad term to describe materials whose attachment to a surface principally involves molecular attraction is "glue" and "adhesive", whereas those whose function is principally mechanical interlocking or gap-filling are called "lutes" and "cements".

The principle behind the use of an adhesive is that it is applied to a surface in a liquid form so that it wets the surface. It will then be close enough for adhesive bonds to be formed. \((138)\)
By inducing this liquid to solidify without appreciable dimensional changes that will disrupt the bond, adhesion between two solids is achieved.\(^{(97)(193)}\)

In looking at the forces operating in a joint, firstly there may be ionic bonds, although that would be rare. Boeck\(^{(10)}\) used spectroscopic evidence to show that adhesion of polycarboxylato cement to enamel results from ionic bonds formed at the interface. Most of the bonds would be physical with Keesom forces predominating. With organic adhesives the common polar groups are carboxyl, carbonyl, nitro, hydroxyl, nitrile, sulfonic, amide etc. For strong adhesive forces an abundance of these polar groups is needed, also there are other criteria, otherwise polar liquids such as nitrobenzene, ethyl alcohol and acetic acid would be good adhesives. They are not, and two of the factors that predispose to this are their high mobility and low molecular weight.\(^{(136)}\) On the other hand, a high molecular weight polymer with very strong cohesive forces (such as polytetrafluoroethylene) which might be expected to display good adhesive properties, is in fact, adhesive, resisting adhesion.\(^{(136)}\)

It can be seen that a good adhesive needs, in addition to strong polar bonds, also high cohesive forces, also that the
factors which will determine likely adhesives are complex. High molecular weight has been cited as desirable in an adhesive.\(^{(136)}\) Generally an ideal molecular weight represents a compromise between ease of application and freedom from stress and loss of cohesive strength. For this reason an adhesive formed at the surface by the polymerization of polar monomers should yield good adhesion.\(^{(136)}\)

Theories of adhesion based on molecular forces have been discussed. Other theories have been put forward:

**Diffusion**

This theory maintains that adhesion occurs by the diffusion of micromolecules from the adhesive layer into the adherend layer and vice versa. It requires that the adhesive-adherend systems are mutually soluble.\(^{(136)}\)

**Adhesion based on free energies**

It has been proposed that any two materials can be made to adhere strongly. It has to be known which of the two materials must be fluid when brought into contact. This theory is based on critical surface tension of wetting.\(^{(136)}\)

**Surface wetting and surface energy**

The wetting of the surface of the solid substrate by the
liquid is important. If it fails to wet the surface, bonding cannot take place. (153) Wetting in fact is a manifestation of attractive forces between the liquid and solid. (63) Whether or not a liquid will wet a solid surface is decided by the relative surface energies. (63) (194) If the surface energy of the liquid is lower than that of the solid, it will flow over the surface of the solid and wet it. The greater the difference in surface energies, the more effective will be the wetting. (63) The surface energy of a liquid is a familiar phenomenon called surface tension. Liquids which readily wet surfaces have low surface tensions. (194)

Solids also possess surface energy and although it is more difficult to measure in the case of a solid, it is known that the surface energy of most solids is high and at its highest when they are chemically clean. (63) This is why a clean surface is wet more easily than one that is oxidised or greasy, or in some way contaminated. The degree to which a particular liquid will wet a particular solid can be measured by the contact angle formed between the two surfaces. (153) By measuring this angle with an instrument such as a goniometer, a reading can be taken which is a direct expression of the ability of that liquid to wet that solid. (194) A high angle is an indication of poor wettability in which either the liquid has a very high surface energy or the solid a very low one. (Wax, Teflon, graphite or a surface contaminated with grease or oxide film are examples of low surface energy.
surfaces). If the contact angle is small then this is an indication of improved wettability and the liquid flows over a relatively larger area. The ideal contact angle, of course, would be zero, in which case the liquid would flow over and entirely wet the surface to the point of forming a molecular layer.

Parker and Taylor (136) say that 'just as we are careful to rub down woodwork before applying undercoat and topcoat when painting, so must we consider surface preparation before application of adhesives'. The analogy may seem a rather hazy one, but it serves to emphasise the point, even when tooth surfaces are concerned. Mention is made of the avoidance of highly polished surfaces for efficient bonding and emphasis placed on the need for a degree of friction involving roughened surfaces. Mechanical interlocking is discussed with regard to its desirable influence in providing an enhanced contact area for adhesion.

Ratlof (153) confirmed the observation of other authors (36)(54)(72)(98)(150) that mechanical retention plays a major role in the bonding of many adhesives to acid-conditioned enamel surfaces. He noted from his photomicrographs the improved interfacial contact between resin and enamel surface and commented
that the universal London dispersion forces may operate as a result of this. He also concluded that additional chemical bonding occurs at the interface. In this case the distances of separation would not have to exceed four Angstrom units. (153)(189) Rottef added that van der Waals dispersion forces, polar forces and hydrogen bonding may play a part in the bonding.

In the case of intact tooth enamel or of a prepared cavity wall, irregular surfaces are being considered and under these conditions a low contact angle is desirable so that a liquid will flow into grooves and pits and rough surfaces easily, such as for sealing purposes.

It is also desirable that the viscosity of the liquid be low so that it flows and wets the surface relatively quickly. Low viscosity also assists in the avoidance of trapping air bubbles. Bond strength will be considerably weakened by the inclusion of trapped air bubbles which, when the joint has hardened, will produce unequal stresses at the interface and there will be stress concentration around these regions.

Even when a liquid adhesive appears to have flowed evenly over a surface viewed by a microscope at low magnification
closer investigation at high magnification may reveal voids. Eick et al.\textsuperscript{(51)} demonstrated bubble formations, particularly along scratches on dentine surfaces in their scanning electron microscope study. They concluded that the topography of the adherend plays an important role in the formation of an adhesive bond and in the fracture pattern of an adhesive joint, even when cohesive failure is involved.

Stress concentrations can also be formed in the hardened adhesive during setting and are due to dimensional changes that occur at this time. Almost all adhesives contract upon setting so that surface roughnesses cause interlocking of adhesive and substrate resulting in restricted strength. Residual stresses are set up in the hardened adhesive and the effect is one of weakening the final bond.\textsuperscript{(51)(105)(153)}

However, another factor is involved in the hardened adhesive joint, and this is the degree to which the potential for severe stress concentration can be alleviated by diffusion of stress patterns through the bulk of the adhesive due to a degree of deformation in the material.\textsuperscript{(138)} Britteness is mentioned by Newman\textsuperscript{(128)} as an undesirable characteristic in a bond. Patrick\textsuperscript{(138)} cites one of the deficiencies of high molecular weight polymers as their being frequently brittle in the cured state.
Saier(6) mentions polymers as being the most likely surface-active adhesion modifiers for promoting strong bonds to hard tissues. Gowan(134) and Eirich(52) refer to multipoint adsorption of polymer molecules at an interface and, due to the unique nature of polymer molecular chains, a continuous interweaving and intermeshing over several hundred Angstrom units distance, resulting in a substantial interphase.

Patrick(138) describes five definite areas in any adhesive bond:

1. the substrate (adherend), which may be virtually any solid material;
2. an interphase formed by the interaction of the substrate material and the surface of the adhesive;
3. the bulk of the adhesive material;
4. another interphase; and
5. the substrate material.

He describes adhesive failure as occurring only where there is poor wetting. Generally, the failure of an adhesive joint is in the bulk of either the substrate or the adhesive; cohesive failures in fact. He outlines some of the reasons
for adhesive failure:
1. Improper wetting of the substrate.
2. Inherent flaws in the bulk adhesive.
3. Crystallization of the adhesive causing volume diminution.
4. Joint starvation.
5. Differential modulus.
6. Residual stresses after curing.
7. Chemical interaction at the interfacial bonds, such as hydrolysis by water.

Adhesion in the Mouth

Some of the factors in dealing with adhesion to tooth surfaces need consideration. Firstly, the surface is microscopically rough and pitted. (152) Secondly, there is a superficial layer of organic material (34) and the surface layer of enamel is fully reacted and unsuitable for bonding. (193) This makes wetting very difficult. Adhesives have to be moderately viscous in order to be manipulated clinically. They have to set quickly compared to adhesives in, say, industrial use. Under industrial conditions, hours, or perhaps days, can be allowed for setting and the environment can be more easily controlled. (136) Further considerations in the mouth
are the presence of moisture, of bacteria and debris (34) the likelihood of sudden and relatively severe temperature fluctuations (140) and also shock in service.

From what is known of dentine and enamel it seems unlikely that a given adhesive will bond equally well to both. (152) In an evaluation of a resin formulation designed for protection of gingivally eroded areas, Friedman and Retief (60) noted poor adhesion to etched dentine and cementum. However, for adhesion to etched enamel with this material, they recorded the highest ever strength for tensile bonds (1100 N/cm²) for a wide range of restorative materials tested in their laboratory under similar experimental conditions. Buenocore (28) has said that if a choice had to be made between the two tissues, then bonding to enamel would be preferred to dentine, but that it would be preferable to modify existing cavity designs to increase the contact area between enamel and adhesive.

The composition of enamel is roughly 95% inorganic, being calcium phosphate in the form of a hydroxyapatite with a calcium/phosphorus ratio of a little more than 2/1 by weight. (34) The remaining 5% consists of an organic material, mainly a keratin-like protein 0.5%, and water 4%. Retief (153) states
that this relatively small organic component plays a very important part in determining adhesion to tooth structure. Eastoe (50) has said that it is present either as a continuous gel or a viscous sol. This non-uniform composition of enamel presents a problem in that it is unlikely that a given adhesive will bond equally well to both fractions. (152) This introduces stress concentrations due to these conditions occurring at the enamel surface. Posner (146) Schwartz and Galligan (168) and Glantz (63) believe that hydrogen bonds can contribute to adhesion to enamel.

When the enamel surface is considered as a substrate for bonding, one problem which arises concerns the presence of a layer of organic material on the surface of the tooth. (34) This layer has low surface energy and under normal conditions it is markedly hydrophobic, possessing a large contact angle with water or a liquid adhesive. With distilled water the contact angle is about 70°; if the enamel surface is cleaned with pumice paste on a prophylactic brush, the contact angle is not greatly reduced, since it remains at about 50°. (131) The wettability of enamel can be greatly improved by microscopic etching with a 50% phosphoric acid solution. (29) (36) (73) (98) This renders the enamel surface hydrophilic and water or liquid adhesives can wet it easily with essentially zero contact angles. A tooth surface can be restored to its original hydrophobic condition by brushing it again with
pumice. (120) As it acquires its layer of organic material so will its hydrophobic character increase.

In looking at the question of moisture, it is necessary to consider its presence both in the enamel itself (11)(12)(110) and in the oral environment. (149) It is not possible to completely dry the enamel because of the strong affinity that hydroxyapatite has for water, and also because of the fact that moisture will always be present on the enamel surface, if only as a molecular layer. (194) There is the further question of fluid flow through the enamel. Rotiñ et al. (160) reported that laboratory experiments they conducted substantiated the findings of Bergman (11) and Linden (110) with regard to the percolation of fluids in a peripheral direction.

There seem to be two courses open — one is to use an adhesive which is itself compatible with water, (177) the other to use an adhesive which will replace water on the tooth surface. (144)(194) An example of the latter is a system developed by Bowen (15) in which a coupling agent displaces water from hydroxyapatite and combines with it chemically. The former is exemplified in the so-called polycarboxylate composites. In these the liquid is an aqueous solution which diffuses into the water on the tooth surface and bonds with the underlying hydroxyapatite. (179)
With respect to temperature changes, if the adhesive material has a coefficient of thermal expansion different from that of tooth enamel, then the adhesive bond may be subjected to severe stresses. The range of temperature fluctuations registered on the tooth surface appears to be in the vicinity of $30^\circ C(140)(102)$ and the changes during ingestion of hot or cold food can be very rapid. Synthetic polymers are likely dental adhesives but have very high thermal expansion rates. Their use as a restorative material has been improved considerably by incorporating inert fillers with low coefficients of thermal expansion which reduce the overall coefficient of thermal expansion of the material to a degree where they are much more acceptable as restoratives.

**ADHESIVES**

In a survey of materials that have been investigated or are under investigation as adhesives for dental use, the first group to be considered is that which hardens by polymerization of the adhesive.

**Acrylic Resins**

In the late 1940's plastics came into general structural use as adhesives in the industrial field when the epoxy resins
were introduced as miracle materials.\(^{(86)}\) It is interesting to note that epoxy resins were first synthesized in the laboratories of a dental materials company (Dr. Troy Freres, Zurich) by Dr. Pierre Costan in 1937.\(^{(97)}\)

By the mid 1950's, aspects of the growing adhesive technology were being viewed from the dental standpoint. Possible adhesion to enamel and dentino was being researched.\(^{*}\) Although the primary use of the acrylic plastics, introduced to dentistry in 1937,\(^{(127)}\) had been as a denture base, these materials have had a wide variety of other applications in the field of dentistry. One of these has been as a cement.\(^{**}\)

From the original heat-cured resins came developments which led to chemically cured resins.\(^{(127)}(128)\) These have been used as tooth restoratives since their introduction in Germany in World War II.\(^{(127)}\)

Very little success was experienced with adhesion of acrylic resin to tooth enamel\(^{(184)}(185)\) until 1955, when Buonocore reported that adhesion of acrylic resin to enamel was enhanced by acid pretreatment.\(^{(29)}\) It is now realized that this

\(\ast (29)(38)(57)(113)(182)(184)\)

represented one of the major advances in the use of adhesives in dentistry. However, it seemed to pass relatively unnoticed at the time, as it was not until the late 1960's that successful techniques for direct attachment of acrylic and diacrylate resins to enamel were employed.*

Whether the systems were of the chemically cured or ultra-violet light (UVL) activated type, the principle of bonding employing acid pretreatment of the enamel was essentially the same. One disadvantage of chemically cured resin is its slow set compared to UVL activated resins. This has been diminished considerably by the use of a hot air blower.* (190)

Neuman*(128) and others, including Miura et al. (128) gave early reports in the literature on clinical treatment with direct bonding of attachments to tooth surfaces with acrylic adhesives and pretreatment,

Acrylic adhesives are discussed more fully in a separate section of the literature review.

**Epoxy Resins**

Another of these materials to be tried was the epoxy resins. They are very effective adhesives for general use, (e.g. Araldite) and they adhere by chemical bonding with very little dimensional change in the adhesive. The materials that can be bonded by epoxy resins include: metals and alloys. In fact, alloys in aircraft construction are being bonded with epoxy resins instead of riveting them. Lee (97) says that the adhesively bonded structure is often much stronger than a riveted one and will more often fail cohesively rather than adhesively.

Retief et al. (160) reported an early study they made of the direct bonding of orthodontic attachments to teeth using an epoxy resin formulation. For the laboratory tests, separate attachments were not used. The resin itself was forced into blocks on the surface of the tooth and a wire loop embedded in order to carry out load tests. Results were encouraging with optimal tension bond strengths in the region of 3.45 to 4.14 MPa. However, clinical trials conducted with this adhesive system (Epikote 828 / Epikure U) and specially constructed stainless steel brackets gave variable results.

Disadvantages cited for epoxy resins as dental adhesives
are that they cure slowly and bonding is affected by water; following hardening they have a high water absorption rate and poor colour stability.\(^{(97)}\)

**Bowen's Resin**

A third material that hardens by polymerization is Bowen's resin.\(^{(15)}\) Bowen started with an epoxy resin and succeeded in removing a number of its deficiencies by reacting it with an acrylic monomer, producing an epoxide in the form of a rather thick liquid. The liquid is too viscous for clinical usage\(^{(21)}\) and various diluents are added in amounts up to 50 per cent to improve fluidity. These diluents may be monofunctional, such as methacrylic acid or methyl methacrylate, or difunctional, such as ethylene glycol dimethacrylate.\(^{(86)}\) This epoxide can be polymerized in the same way as a cold curing acrylic resin, by benzoyl peroxide and a tertiary amine. It can also be modified so that polymerization can be initiated by U.V. radiation, as in its use as a pit and fissure sealant with the Nuva Seal/Nuva light technique.\(^{(33)}\) This system is also employed for the restoration of fractured anterior teeth.\(^{(35)}\)

To make an experimental filling material Bowen combined his liquid component with powdered vitreous silica, which has a
very low (almost zero) coefficient of thermal expansion. The inert filler (vitreous silica) was bonded to the resin matrix by use of silane coupling agent.\(^{(15)(16)}\) Unfortunately, this resin did not show lasting adhesion to a tooth cavity under mouth conditions. However, Bowen's material has become the basis from which have been developed various composite resins now available as tooth fillings.

In an historical note\(^{(86)}\) on adhesive restorative materials this comment is made: "Although other plastics have been used in adhesive restorative procedures, the materials technology had its true genesis in the work of R. L. Bowen, American Dental Association Fellow at the National Bureau of Standards." It is well deserved.

Bowen\(^{(14)(17)(18)(19)(20)}\) then went on to study the use of coupling agents which would provide a chemical link between his filling material and tooth structure. Coupling agents have been discussed in a separate section of this thesis, particularly with reference to their development on the industrial side.

**Cyanoacrylates**

A fourth group of polymerizing adhesives comprises the cyanoacrylates. These consist of liquid monomers which
polymerize very rapidly when exposed to even small traces of moisture. \textsuperscript{(26)} The simplest one is methyl cyanoacrylate, available commercially as Eastman 910 Adhesive. \textsuperscript{(3)} This has been widely used as a tissue adhesive in various surgical procedures. It can replace suturing. \textsuperscript{(108)} When used as a thin film, it polymerizes to form a tough polymer in thirty to sixty seconds. Unfortunately it breaks down in the tissue fairly rapidly and one of the products is formaldehyde \textsuperscript{(3)} which causes local irritation. Butyl cyanoacrylate breaks down much more slowly than methyl cyanoacrylate and this has been tried as an adhesive in oral surgical procedures, including periodontal surgery. \textsuperscript{(13)} It is hemostatic in addition to being adhesive.

In a laboratory study of the bond strengths of three cyanoacrylates (ethyl, methyland butyl), Crabb and Wilson \textsuperscript{(45)} found that initial high values were rapidly reduced to relatively ineffective levels by twenty four hours immersion in normal saline at 37\degree C. Newman \textsuperscript{(128)} found problems with manipulation and hydrolysis with cyanoacrylates and also thought the bonds too brittle.

Custo and Buonocore \textsuperscript{(47)} used methyl cyanoacrylate in the sealing of pits and fissures. In a series of experiments
they produced an 86% reduction in dental caries after one year, but experienced some shortcomings in the adhesive system which necessitated a six-month recall examination and replacement of the adhesive in about 20% of the cases, due to loss of adhesive.

Porteous and Winter (137) found alkyl cyanacrylates unpromising as fissure sealants.

Beach (9) in an "in vitro" study of bonding to both enamel and dentine, using a group of cyanacrylates, claimed good bond strengths with dentine, but generally unsatisfactory ones with enamel. Acid pretreatment of the enamel enhanced adhesion but with dentine did not significantly improve bond strengths.

The American Dental Association Council on dental materials and devices published a report in December, 1974, (2) recommending that cyanacrylates were not suitable for routine use in dentistry on the basis of current knowledge.

**Ultraviolet-Light-Sensitive Resin**

Buonocore (33) undertook additional study and reported on a method of sealing pits and fissures with a new adhesive material that hardens when exposed to UVI and maintained 100%
cohesive protection after one year. The adhesive used in this work was a relatively stable thick liquid containing as the major ingredients three parts by weight of the reaction product of Bisphenol A and glycidyl methacrylate (BIS-GMA) and one part by weight of methyl methacrylate monomer. Just before use, 2% benzoin methyl ether was dissolved in the adhesive liquid to form an ultraviolet light-sensitive composition that was painted on the tooth surface with a fine camel hair brush. The technique also involved pretreatment of the pits and fissures with a 50% phosphoric acid solution containing 7% dissolved zinc oxide by weight. The polymerization of the material was accomplished by use of ultraviolet light. There have been numerous reports in the literature of studies involving this technique. \(67(73)(172)(192)\)

In 1974 the Council on Dental Materials and Devices classified two pit and fissure sealants as provisionally acceptable. One was Nuva-Seal (L. D. Caulk Co.) which is a UVL initiated BIS-GMA resin.\(^1\)

The application of this technique to direct bonding of brackets to enamel may be seen as a possible advance on the technique of bonding by a self-polymerizing resin. Being able to have virtually unlimited time in which to position brackets on the tooth surface and then to harden the adhesive in a relatively short time (twenty seconds) can be seen to have obvious
advantages over a self-polymerizing system. Bonding of clear plastic orthodontic brackets using an ultraviolet sensitive adhesive has been reported on by Cohl, Green and Eick.\(^{42}\) These workers used Fujiwara's system which in this review of polymerizing adhesives constitutes a fifth system. A comparison of bracket failure rates of their system with Fujiwara's showed better adherence in Fujiwara's methacrylate system than in Cohl, Green and Eick's UV initiated adhesive.

**Polyurethanes**

A sixth system of adhesives that has also been investigated is known as polyurethanes. They are used commercially as adhesives and in the dental field they have been tried as cavity liners.\(^{61}(128)\) The technique is to use a liquid prepolymer which is an isocyanate that is applied to the cavity walls. This reacts with the tooth surface to form a polyurethane liner and theoretically the liner should bond chemically to the tooth. However, there is a problem with moisture involving the liberation of carbon dioxide gas.\(^{61}\)

It will readily be appreciated that gas bubbles are a severe threat to any adhesive system so that it is probable that the long term adhesion of a polyurethane liner made from an isocyanate under mouth conditions would be poor.
Polycarboxylate Cements

The second type of adhesive material is one that hardens other than by the action of polymerization. Polycarboxylate cements such as Durcon, Poly C and PCA\(^{(177)}\) are examples of the type of adhesive to be discussed. The hardening of these cements involves a chelation reaction and so they incorporate a chelating agent as the reacting substance. Chelates can combine with another ion by forming bonds with it from two different directions. This can form a strong bond and chelates can be biologically very stable. Materials which adhere naturally to teeth, e.g. mucin proteins, dextrans and tobacco tars are chelating agents.

In the polycarboxylate cements the chelating agent is a polymer, polyacrylic acid, which is soluble in water. The advantage of using a large molecule like a polymer is that each molecule will have a great many different sites for chelation and simultaneous rupture of these multiple sites is required for dislodgement.\(^{(102)}\) These cements consist basically of zinc oxide powder and a 50% aqueous solution of polyacrylic acid. When mixed and applied to the tooth, the polyacrylic acid chelates with calcium in the hydroxy apatite to form a chemical bond.\(^{(178)}\) Zinc polyacrylate is formed in the set adhesive.
Early studies on the adhesion of zinc polycarboxylate cements to tooth structure were reported by Mizrahi and Smith, Phillips et al., Mizrahi and Richter et al.

In a laboratory investigation of the bond strengths of some dental cements including three zinc polycarboxylate cements, Manning states that in his tensile and shear bond strength tests, the site of bond failure was generally within the cement itself, i.e., a cohesive failure. He goes on to say that when failure was not entirely cohesive, the partial adhesive failure was at the cement-metal interface.

Polycarboxylate cements have not been the subject of recent investigation in the dental adhesives field.

Bioadhesivos

A third type of adhesive being investigated at present is a biological one. Reference has been made earlier to the secretions of marine creatures, such as barnacles, that have solved very successfully the problem of adhesive bonding to a solid surface under aqueous conditions. Docking makes reference to the fact that adhesion under wet conditions has already been solved in nature and mentions the investigations of Harrison and Philpott on bio-adhesivos.
The secretory mechanism of barnacles has been identified by Hillman and Nace(77) and Cook,(43) but the exact chemical composition of the bioadhesive has not been traced. Hillman and Nace(77) thought it to be principally a protein or polypeptide. They commented on the adverse conditions under which barnacles show ability to attach themselves to a diversity of substrates: metal, wood, concrete and plastics, and under such conditions as pounding surf or exposure from falling tides. Fossil barnacles have been found firmly adhering to other shells after thousands of years.

Other marine biota, such as oysters and mussels, also come into this group and various researchers(7)(43)(44)(109) have been making a close study of barnacle cement. Only a preliminary analysis has been reported and this suggests that the cement is a protein with a minor lipid content.(28) This liquid cement has been collected painstakingly from the marine life and has been shown to harden in contact with moisture by what appears to be a hydrolysis reaction. Once it has set it is extremely insoluble and even in hot aqua regia it will not dissolve. It is very resistant to almost all organic solvents; so far these workers have found only one which has any effect and this is hot glacial acetic acid.(34) The adherence of this cement to
human enamel has been demonstrated by simply growing barnacles on extracted teeth. The results have shown a very strong bond.

The available literature on the adhesion of various forms of marine life to solid surfaces makes fascinating reading. The biological approach to adhesion is an intriguing one and the mechanisms are not always as slow moving as might be imagined. (75)

COUPLING AGENTS

Introduction

The function of a coupling agent is to improve bonding between an adhesive and a substrate where the molecular configurations of the two materials are sufficiently divergent as to produce weak chemical links. (102)(136)(144) Ideally the molecular structure of the coupling agent will be such that one end of the molecule will bond chemically with one material and the other end bond chemically with another. (34)(102) Coupling agents known broadly as vinyl silanes are employed in bonding the glass filler used in composite resins to the resin matrix. The filler particles are coated with vinyl silane and a chemical bond is formed between them and the resin matrix, and also between the silane and the glass filler. (15)
Pluetailedmann\(^{144}\) has said that attempts to understand the nature of adhesion in the bonding procedures involved in reinforced plastics are unsuccessful because the results bear little relationship to basic concepts. Contact angles, wettability, surface tensions, potential weak boundary layers, surface morphology, chemistry of mineral surfaces or any of the other fundamental concepts show little correlation to adhesion of paints, rubbers and sealants to mineral surfaces and strengths of glass-reinforced plastics, especially after exposure to water. Because of consistent and marked improvements observed in adhesion through the introduction of appropriate silane coupling agents in mineral/plastic composites, the key to adhesion may lie in the understanding of this mechanism. Pluetailedmann\(^{144}\) states that:

Any meaningful theory of adhesion of plastics to minerals must be consistent with a broad range of observations on adhesion. It should be consistent with the known effects of:

1. Fundamental surface chemistry and physics of minerals and plastics.

2. The action of water and other potential weak boundary layers at the interface.

3. Morphology of the plastic at the interface.

4. Mechanical requirements of a plastic-mineral interface to allow distribution of shrinkage stresses especially at edges of voids or other areas of stress concentration.
5. Benefits and limitations of adhesion through silane coupling agents.

6. Adhesion of ico and bio-adhesives to surfaces.

A sound working theory of adhesion of plastics to minerals should be beneficial in developing improvements in present plastic mineral composites.

The Chemical Structure of Silane Coupling Agents

All commercial silane coupling agents are of the general structure \( X_3 Si(CH_2)_n Y \) where \( n \) equals 0 to 3. \( X \) is a hydrolyzable group on silicon and \( Y \) is an organofunctional group selected for compatibility with a given resin. The total amount of silane coupling agent applied is generally 0.1 to 0.5 per cent of the weight of glass. \(^{144}\) Since silane coupling agents are generally applied to glass from water solutions, the hydrolyzable groups are only essential for generating intermediate silanols. For example, essentially equivalent performance was obtained from aqueous vinylsilane coupling agents derived from all common hydrolyzable vinylsilanes.

Aqueous solutions of silane coupling agents have only limited stability. The composition depends on the nature of the organofunctional group on silicon and the pH of the solution.
Neutral solutions hydrolyze rapidly to silane triols and then condense slowly to oligomeric siloxanes. (144)

The coupling agents, as usually applied from solution, are largely monomers or dimers with smaller proportions of higher oligomers. Films of coupling agents deposited from solution are oils that are soluble in common organic solvents, but insoluble in water. Then hydrolyzed coupling agents are completely condensed to siloxane structure, the products are no longer effective as coupling agents. (144)

Amount of Silane required for coupling

From reports by Zisman, (195) Lee, (99) Tutas et al., (187) Bascom, (8) Schrader et al., (167) and Stark et al., (180) it has been agreed that silane coupling agents are not generally deposited on mineral surfaces as simple oriented monomolecular films, but as multi-layers with variable orientation, depending upon conditions of deposition. However, the retained portion of the silane can often be less than a monomolecular film. A major part of the film deposited on a mineral surface is removed readily by water or organic solvents, and, in practice, the plastic used to bond to the surface dissolves much of the silane. However, only a small trace of coupling agent is necessary for improved bonding and this film is retained tenaciously by the surface.
FIGURE 1

Step I. Reaction of Silane with Surface Water at Tooth Surface

3H₂O + \( \text{CH}_3\text{CH}_2\text{O} \)\( \overset{\text{SiCH}_2\text{CH}_2\text{N}}{\text{SiCH}_2\text{CH}_2\text{N}} \text{H} \) → HO\( \text{SiCH}_2\text{CH}_2\text{N} \)\( \text{H} \)

Surface water Silane Coupling Agent Trihydroxysilane and Ethanol (by product)

Step II. Reaction Between Tooth Surface and Silane

Tooth Surface Silane

Silane Primed Tooth Surface

Step III. Reaction of Primed Tooth Surface with Epoxy Resins

Silane Primed Tooth Surface Epoxy Resin

Epoxy Resin Bonded to Tooth Structure Through Silane

FIGURE 19. Reaction between silane coupling agent, tooth structure, and epoxy resins.

(Fig. 19, Lee, 1977)
The deposition of silane on a substrate can be achieved by adding it as an integral blend to the resin system prior to adding the inorganic filler. The silane then migrates to the hydrophilic surface where it reacts with absorbed moisture to form silanol groups which bond to the surface. Sufficient time must be allowed for migration of the silane in the resin matrix.

Silane coupling agents do not function by depositing a tough rubbery water resistant boundary layer. The amount of silane coupling agent required on a glass surface is too small to measure by ordinary analytical means. By use of radioactive tracers\(^{(167)}\) it has been determined that less than a monomolecular layer of the proper silane on a glass surface will function as a coupling agent. Silane coupling agents are equally as effective when added as integral blends in resins at concentrations calculated to be equivalent to a monomolecular layer of silane on the mineral surface. Thicker films deposited by hydrolysis and condensation of silane coupling agents are oily, cheesy gels, or friable powders, but not tough, water-resistant polymers. They are not effective unless they are deposited in a silanol form. When applied as alkoxy silanes, it is essential that water be present at the interface, and, if condensed completely
to siloxanes and deposited from organic solvents, they are not effective as coupling agents.

Silane coupling agents were first introduced to improve the water resistance of reinforced plastics. It was soon observed that they improved significantly the initial properties of laminates. The degree of improvement varied with the resin used, the glass content and the nature of the test.

Moisture at the Interface

In a discussion by Plasdemann (144) on the problems in bonding to hydrophilic mineral surfaces he examines shrinkage stresses and also water at the interface. The discussion on water at the interface seems to be a relevant one to the use of bonding agents and attendant coupling agents in the mouth. (194) It is known that a clean glass surface exposed to ordinary atmospheric conditions immediately picks up a molecular layer of water. Water can penetrate to the glass-resin interface through even the most water resistant resins by diffusion through the resin, by filtering through cracks or by capillary migration along the fibres.

From the above observations it must be concluded that
water cannot be excluded from the interface between resin and a hydrophilic mineral reinforcement and that the effect of water will vary with the nature of the mineral surface. Silane coupling agents do not exclude water from the interface but somehow function to retain adhesion in the presence of water. Flueddemann (144) has proposed a new working theory of adhesion at the polymer mineral interface in which silane coupling agents provide a bond at the interface that is capable of using the hydrolytic intrusion of water, with self healing, as a means of stress relaxation without disrupting the overall bond between plastic and mineral surfaces.

All resins bond to solid hydrophilic surface through a reversible hydrolyzable bond mechanism. Since the only requirement for bonding to a hydrophilic mineral surface is that a material approach the surface with functional groups capable of competing with potential weak boundary layers, usually water, and solidify to a rigid structure, this mechanism is not limited to silanols but applies generally for adhesion.

A rigid interface is necessary so that reactive positions are held in such proximity that free silanols resulting from
hydrolysis of a bond have no alternative but to eventually reform the original bond or make a new bond with an adjacent group. As long as the interface is rigid, the making and breaking of bonds in the presence of water is reversible. Even subjection of the bond to boiling water will not disrupt sufficient bonds to cause complete loss of adhesion as long as the resin retains its integrity.\(^{(144)}\)

A study by Faulkner and Harcourt\(^{(53)}\) on the adhesion of acrylic resin to stainless steel, employing silane coupling agents, showed increased impact strength of silane-treated specimens over controls at room temperature in air. However, storage in water caused a gradual decrease in all samples, although the silane-treated samples were still significantly stronger than the controls in all cases. Silane A 189 gave best bond strengths after curing, when tested in air, but A P 131 (mercaptosilane) produced superior bond strengths after storage of samples in water at 37°C.

The adhesion of plastics and ice to most surfaces, barnacles to underwater structures, plaque to living teeth, etc., are all examples of the same mechanism. Bonding of epoxy resins through hydroxyl groups (hydrogen bonding or alkoxide formation)
to a hydrophilic surface is hydrolytically reversible and, although the equilibrium is less favourable for bond retention than that of a silanol, water resistant bonds are obtained if the hydroxyl concentration at the interface is high enough.\(^{(144)}\) Bonding through reactive silanols or organic hydroxyl groups differs only in degree, and not in kind.

In a study by Saroyan et al.\(^{(166)}\) the adhesion of barnacles to underwater structures was observed and barnacle larvae were seen first to attach themselves mechanically by means of suction cups in their antennae and then to secrete an adhesive cement that builds up and envelops the antennae. The cement appears to be a liquid protein that hardens as it is secreted. Adhesion must of necessity result when a polar material like a mucopolysaccharide or a protein is hardened at the interface by crosslinking or some other chemical reaction. Reproduction of a similar mechanism of hardening of synthetic polymers at a moist interface would be valuable in problems such as wound closure and healing and in adhesive bonding of materials to tooth structure.

Water is necessary for bonding to mineral surfaces. A coupling mechanism will not operate between a rigid resin and a non-hydrophylic surface like graphite. Water does not attack
the interface and there is therefore no mechanism for relaxation of stresses set up during resin cure and cooling. (144)

The foregoing discussion on the use of silane coupling agents has had an application in prosthetic dentistry with the bonding of porcelain teeth to acrylic denture bases by the use of silane as an intermediate coupling agent. (141)

When we come to apply the use of silane to the direct bonding of acrylic resin to enamel, the process is somewhat puzzling. Most bonding systems involving tooth enamel have been shown to improve from an ineffective to an effective level by pretreatment of the enamel surface, mostly involving acid etching. (29) Rizzoli and Smith (123) have suggested that the bonding with Masuhara’s system is mechanical, enhanced by the etching of the surface of the dentine with the phosphoric acid pretreatment.

What then is the effect of the silane in Niura’s bonding system? If the silane coupling agent improved the bond between enamel and an acrylic resin, one would think that cleansing of the enamel surface and the precautions which are normally taken to exclude moisture etc. in bonding procedures, would be sufficient to produce an effective bond. Yet acid pretreatment
is advised as being a necessary prerequisite to effective bonding.

Miura\(^{(120)}\) states: "The silane is blown away with air completely to create a thin film which reduces the surface tension so that the viscous adhesive can flow freely into the crevices of the enamel". It has recently been shown\(^{(58)}\) that even more viscous adhesives do not require an intermediate film of lower viscosity to achieve intimate penetration into the enamel pores.

Miura\(^{(120)}\) also says that silane creates a chemical couple between the adhesive resin and the calcium of the enamel and that he has evidence of this from an electron microscope study. He makes further assurance that exclusion of the silane from his bonding system will result in reduced adhesion. It has been suggested\(^{(139)}\) that silane may improve resin bonding to enamel by increasing its duration rather than by increasing the bond strength.

Newman et al.\(^{(152)}\) reporting on a study of acrylic adhesive systems which involved the use of a silane coupling agent, found that the inclusion of 2.4 per cent of coupling agent "substantially decreased joint strength". This
coupling agent, Dow Corning Z6030, is the same silane that is used with Fujiwara's bonding system.\(^{(120)}\)

The following statement of Lee\(^{(100)}\) refutes recent conclusions that adhesion of composites to enamel is mechanical: "That adhesion to enamel and dentin is not 'mechanical' can readily be demonstrated by coating the freshly etched surface with a dilute solution of silicone oil. The adhesive will promptly 'pop off' upon setting. If retention were mechanical, it would not do so."

This somehow seems a rather trite comment and one that the reader would be inclined to dismiss lightly. It serves to confuse, even more, the whole question of what is adhesion—a question that Lee has certainly involved himself in with some asperity.\(^{(2)(101)(103)}\) He adds\(^{(103)}\) that if adhesion of resins to tooth surfaces were exclusively mechanical with no physical-chemical or chemical forces involved, higher values for adhesion to dentin would be expected, because of its better developed surface. He says that experimental data indicate that adhesion forces to enamel are usually four to seven times greater than to dentin. He further says that etching provides a greater surface area for wetting with the adhesive.
and thus more numerous sites for adhesive attachment forces, while at the same time increasing roughness to improve mechanical adhesion. The two effects are intermingled. However, the overall bonding, no matter what the mechanism, is referred to as an "adhesive bond" and the material as an "adhesive".

**Sevriton sealer**

This surface priming agent represents the earliest application of a coupling agent in dentistry. It has been considered the most commonly used and perhaps the best one for acrylic restoratives. The material is disclosed in British Patent 687,299 (1953).

Sevriton sealer is a polymerizable formulation containing glycerophosphoric acid dimethacrylate. A more detailed description of its composition is given in Chapter 4. It bonds more strongly to dentine than to enamel. (39)(39) Peterson et al. (140) found that Sevriton, when used in conjunction with the recommended sealer, separated from prepared dentine surfaces at appreciably greater stress than did three other resins being tested. This value was reduced approximately by half when the liner was omitted. However, a study involving the bonding of Sevriton (with and without
sealer and with and without acid etching) to bovine enamel revealed that the sealer increased bond strengths significantly (from 4.01±1.58 MPa to 6.16±1.34 MPa) where enamel was acid etched.

The function of such a formulation as Sevriton sealer is based on the assumption that a molecule containing an acid phosphate group may form strong bonds with the crystal lattice of the tooth, while at the same time providing adhesion with the resin. (186)

The study of Lee et al. (98) demonstrated that initial adhesion of Sevriton to unconditioned enamel is enhanced by the use of sealer. However, these bonds suffered complete degradation during thermal cycling whereas those to etched enamel were resistant to the cycling stresses. The histograms from this study are reproduced on pages 55, 56 and 57.
Adhesion after 24 hours storage in water of a poly (methyl methacrylate) direct filling resin to bovine enamel as influenced by treatment of the tooth surface. 

(Fig. 1, Loo et al., 98)
Adhesion after 30 days storage in water of a poly (methyl methacrylate) direct filling resin to bovine enamel as influenced by treatment of the tooth surface.

(Fig. 2 Lee et al. (98))
Adhesion after six months storage in water of a poly (methyl methacrylate) direct filling resin to bovine enamel.

(Fig. 5, Loo et al. (98))
Bowen's surface-active comonomor

Bowen (21) stated that because of polymerization shrinkage (minimised by inert fillers), thermal expanosity and lack of adhesion of resins to tooth surfaces, a coupling agent interposed between resin and tooth might fulfill the requirement for improved restoratives.

This coupling agent, Bowen states, should have chemical groups capable of favourably competing with water and other agents in the oral environment for the chemical constituents of the dentine or enamel to which it is applied. It must also be capable of forming stable bonds to the resin.

The surface-active comonomor is formed by the addition-reaction product of N-phenylglycine and glycidyl methacrylate (NPG-GMA). One end of the molecule has the potential to displace water from the hydroxyapatite surface and bond chemically with the calcium ions in it. The other end contains a methacrylate group and this co-polymerizes with the hardening organic matrix as the composite material sets. Using this coupling agent, Bowen (17)(18)(19)(20) has demonstrated in laboratory tests that he can obtain significantly improved adhesion between composite filling materials and tooth surfaces. The theory
of the coupling of a multiphase structure is represented by Bowen (21) in Fig. 5.

There have been two recent studies (41)(159) involving the use of Bowen's surface-active comonomer. One study (41) involved the clinical assessment of the coupling agent in the placement of composite resin restorations and showed significantly better marginal seal over a period of three and a half years. The other (159) evaluated bond strength to both enamel and dentine of De Troy Cosmic (a composite resin) used both with and without Cosmic Bond (Bowen's surface-active comonomer). Retief (159) was unable to confirm the manufacturer's claim that Cosmic adhered to untreated enamel and dentine surfaces, nor with treated enamel surfaces. However, he recorded substantially higher values for adhesion to treated dentine than those claimed by the manufacturer. Bowen (21) has said that the coupling agent should be applied to the "prepared surfaces" of the tooth. He used hydrochloric acid pretreatment to increase adhesion to dentine (18).

Antonucci and Bowen (5) have recently synthesized compounds having several chelating moieties per molecule and, in addition, having groups that can function as oxime polymerization
Figure 1. Hypothetical schematic diagram of manner in which the various phases of a composite material might be connected by chemical bonds.
accelerators. These surface-active agents have been designed to improve adhesion between tooth surfaces and restorative composites or preventive ("cusalant") resins. They are the reaction product of an epoxidized ortho-cresol-formaldehyde novolak and the sodium salt of L-phenylglycine and N-methyl-p-toluidine.

**EVOLUTION OF MIURA’S BONDING SYSTEM (ORTHOFITE)**

Miura’s technique of direct bonding of orthodontic attachments to tooth enamel, described in an early report of clinical use of plastic attachments, seems to stem from the work of the Japanese chemist and dentist, Masuhara. In 1956 Masuhara was in Germany in connection with a study trip and worked with C. H. Fischer in Gottingen. He was interested in the chemical coupling of protein-containing natural products with synthetic materials. Fischer and Masuhara were working at this time on the possibility of developing a chemical bond between synthetic filling materials and protein components of dental hard tissues.

There was a need to find a reagent which would react with dentinal collagen at mouth temperature to produce a stable
chemical bond of covalent character. In 1960 it was maintained by Masuhara and his co-workers\(^{(113)}\) that certain organo-boron complexes, when used in a synthetic dental filling material, can affect a bonding with the organic matrix of dentine. The boron complexes have been known for a long time in synthetic chemistry as polymerization catalysts. It was found that when these catalysts (tri-n-butyl borane being one) were used, polymerization of monomeric methacrylate begins in the presence of water at the phase surface, although polymerization with other presently available catalysts always occurs from within to the outer surface and therefore the systems react differently. The reaction which begins at the phase border points to the special significance of this synthetic material for its usefulness at the moist dentinal surface.

In further studies Masuhara's group postulated that in this system adhesion to dental hard tissues involves a true chemical bond. A special kind of polymerization reaction involved includes polymerization of new side chains as well as the previously present polymeric chain. Further work of Masuhara's group involved projects connected with the use of inorganic filler materials and the use of coupling agents for bonding the fillers to the organic synthetic matrix.\(^{(58)}\)
This work is of direct interest with regard to development of Miura's bonding system, particularly when we consider the use of acrylic resin as an adhesive used in conjunction with a coupling agent.

Fischer (57) states that adhesion to the dentine can only exist if the liquid synthetic material forms a film on the cavity walls. To obtain the optimal adhesive qualities, therefore, the use of cavity liners was waived. Fischer explains that the wide spectrum of results from his study was due in part to the types of cavity preparations that were used, some of these relying entirely on adhesion alone. Fischer agrees with Masuhara in saying that promising adhesiveness depends on a whole series of factors (56). Thus the surface condition and cleanliness of the cavity has an effect. The presence of a film of saliva between the dentine and the filling material certainly has a deleterious effect on adhesiveness.

In contrast to this, he points out that the normal moistness of newly exposed dentine surfaces is not undesirable. Miura (120) in explaining the effect of his adhesive, mentions the effect of moisture at the enamel interface being a desirable feature of the setting of the cement. This relates to Fischer's
statement that polymerization of monomeric methyl methacrylate begins in the presence of water at the phase surface, although polymerization with other presently available catalysts always occurs from within to the outer surface. Niura maintains that part of his adhesive effect is due to this factor, so that there is minimal shrinkage of the acrylic at the enamel/cement interface.

Niura and Nakagawa [119] reported on the development of an appropriate method for the direct bonding of orthodontic brackets to the enamel surface using 3-methacryloxypropyl trimethoxysilane. This silane was stated as having been deemed the most suitable coupling agent tested.

Pre-treatment of enamel was investigated by electron microscopy in this same study and the use of a sixty-five per cent solution of phosphoric acid for thirty seconds recommended as most suitable for acid etching. It was stated that "a slight decalcification of the enamel surface was observed electron microscopically", but that this faded after regular brushing two thousand times.

Coupling agents, by their nature, can increase the range and effectiveness of an adhesive to a substrate. However, it does not follow that because a specific coupling agent such as a silane facilitates the bonding of glass to resin that such an
agent will act similarly for bonding resin to tooth enamel.
In fact, in considering what is known of silane and of tooth
enamel (and its heterogeneous nature), it seems unlikely.

In early studies of the bonding of resins to etched tooth
enamel, Guinnott and Buonocore (69) and Buonocore et al. (36)
demonstrated microscopic tag-like projections of adhesive that
had penetrated up to twenty five microns into the enamel. As
a result, it was concluded that mechanical retention plays an
important role in such bonds. In the light of this, the
introduction of a coupling agent of doubtful efficacy in forming
bonds with enamel could only be viewed as a likely impediment
to the most effective bonding.

ADVANTAGES OF BONDED PLASTIC BRACKETS

Rotier et al. (160) list the following advantages
of bandless orthodontics:

1. Contact points. In a high percentage of
all cases to be treated orthodontically there is
some degree of lack of space. This is frequently
associated with crowding. Under these circumstances,
it becomes imperative to separate the teeth to
permit the fitting and cementing of bonds. When
the attachments are bonded directly to the tooth,
this procedure is unnecessary. (Fig. 14)
2. Soft-tissue irritation. Preformed bands have considerably reduced gingival trauma during the fitting of bands, but a certain degree of gingival trauma is still unavoidable when the bands are fitted for a full-banded technique. In addition, the edges of the band material and the subsequent stagnation in this area tend to predispose to, if not cause, the gingivitis so frequently associated with a full-banded technique. The minimal gingival irritation associated with full epoxy bonding is shown in Fig. 15.

3. Oral hygiene. Despite all the modern appliances for cleaning the mouth during orthodontic treatment, a number of our patients still present with soft debris on and around the bands. It has been our experience that the patients with bonded brackets have far cleaner mouths. The reduction of overhanging edges, especially in the interdental area, seems to reduce the cleansing problem. (Fig. 16).

4. Space occupied by bands. The total space occupied by the bands and cement could amount to as much as 0.8 centimetres. There are times when this additional space is most welcome during the final stages of treatment. In most instances, however, the last stage of treatment has to be devoted to space closure after removal of the bands. Fig. 17 shows the final stage of treatment with bonded brackets and shows that no space closure will be required after removal of the brackets.

5. Attachments to partially erupted teeth. It is generally accepted that tooth repositioning is accomplished much more speedily during the eruptive phase. It would often be advantageous to be able to fix an attachment to a partially erupted tooth, particularly in cleft palate patients. The conventional banding is often extremely difficult, or even impossible, in such cases. The epoxy resin adhesive can readily be used for these patients. (Fig. 18).
6. Danger of decalcification under detached bands. Preformed bands have reduced the percentage of bands which become loosened during treatment, allowing stagnation and decalcification under them. Some of these, to our subsequent embarrassment, remain undetected and often lead to considerable decalcification. This problem is not encountered with epoxy-bonded attachments, for loosening of these attachments results in complete displacement. In addition, Buonocore and associates have shown that the resistance of enamel to acid decalcification is increased after it has been coated with a dental resin which has then been dislodged. They suggest that the presence of a thin covering layer of adhesive and/or penetration of the adhesive could produce enamel protection.

Where the attachments are of clear or tooth-coloured plastic, there is the added advantage of improved aesthetics. Lee and Swartz (105) state that the orthodontist requires a temporary adhesive to replace the bulky, unhygienic and unsightly metal bands now employed to attach brackets to the enamel. There is universal agreement from all authors using directly bonded plastic attachments on the vastly improved aesthetics.

Swartz (183) has found that not only has there been a much readier acceptance of fixed appliance therapy with bonded plastic brackets by patients, but, more importantly, that it has opened an avenue to orthodontic correction for many who previously would not have availed themselves of the
service.

Many advocates of the systems of direct bonding have emphasized seemingly obvious time-saving advantages. Schwartz (103) refutes this idea and states that to the contrary, three years of experimentation with plastic brackets has convinced him that they are more time consuming than conventional banding. The present author's clinical experience of four years would certainly confirm this latter view, although there has been some narrowing of the gap with increased facility in handling the technique.

A further advantage in the present author's clinical experience of the system, is in the case of interdental stripping in the case of minor disharmonies of tooth size/jaw relationships. Adjustments of this nature are often more satisfactorily carried out during fixed appliance therapy than after band removal. The latter is a necessity with conventional banding.

**TOOTH RESTORATIVE PLASTICS AS CEMENTS**

**Introduction**

Acrylic resins were first used as tooth restoratives in Germany in World War II (141). In the intervening period various modifications have been made in an endeavour to overcome some
of the undesirable characteristics of the material. More recently, tooth restorative plastics based on the free radical polymerization of such adducts as that formed from N-phenyiglycine, Bis-phenol A and glycidyl methacrylate with a bonded inorganic filler incorporated, have shown more promise than previous ones. The adduct referred to is usually known as BIS-GMA. It is not an epoxy resin, but an aromatic dimethacrylate. These restorative materials have been given the general name of composite resins. A recent study using these composite resins for direct bonding of brackets demonstrated good adhesion to enamel.

The inorganic filler represents about seventy per cent by weight of the composite resin. It may be a lithium aluminium silicate, quartzite, or other such solids which have been bonded to the organic resin filler with an intermediate coupling agent. Microscopic spherical glass beads are favoured as a filler as their shape presents the smallest surface area consistent with maximum bulk. The coupling agent is silane.

**Composition**

The acrylic resin which is to be of major interest in this study is of the unfilled type. Examples of commercial products are Bonfil and Sovrilet.
For practical purposes the composition of chemically accelerated powder-liquid denture base acrylic resins is the same as that for tooth restorative plastics.\(^{(141)}\) There is a difference in the size of the powder particles. Those of the tooth restorative are generally smaller as this has the effect of shortening the setting time,\(^{(141)}\) which is a more desirable property for a tooth restorative.

The liquid is made up of methyl methacrylate monomer, an inhibitor such as hydroquinone and originally an amine accelerator, such as \(p\)-toluene sulphinic acid or a derivative, and may contain a cross linking agent such as glycol dimethacrylate. The use of tertiary amine accelerators has been found to affect colour stability of the set plastic.\(^{(141)(172)}\)

The powder consists of poly(methyl methacrylate), benzoyl peroxide initiator, pigment and specifiers.

Originally these tooth restoratives were supplied in three components: liquid, powder and initiator. The latter was the \(p\)-toluene sulphinic acid initiator. The benzoyl peroxide (when present) is included in the powder.\(^{(142)}\) Resins of the latter cyton are marked by their rapid polymerization and excellent colour stability.
Setting Time

Peyton and Craig (141) list the following methods of decreasing the setting time of acrylic tooth restoratives:

1. Increasing the concentration of accelerator or initiator,
2. Increasing the relative surface area of the powder,
3. The presence of moisture,
4. Increasing the mass of materials, and
5. Increasing the temperature of the reactants.

The decrease in the reaction and setting time caused by increasing the concentration of accelerator or initiator results in a decrease in the molecular weight and strength of the set plastic.

Increasing the relative surface area of the powder can be achieved by decreasing the particle size. Iwaku et al. (89) reported, in a study of acrylic resin restoratives, on the effect of milling the originally pearl-shaped powder particles of Sevriton Simplified. They were crushed into irregular and smaller particles with attendant increase in relative surface area. This made the restorations more homogeneous and with significantly decreased separation and porosity. It also
slightly increased the hardness, maximum bonding and bonding strength.

The coarser spherical particles with less surface area are dissolved much less readily by the liquid monomer, so that Sovriton Simplified powder, as supplied, produced a sandy mix with undissolved sphere particles and resulted in a granular structure. Self-polymerizing matrices tend to be softer and less well cured than heat cured resins, resulting in a softer, working resin in which the curing shrinkage is concentrated in the matrices and porosity is produced when curing is prevented by residual particles. Iwata et al. also commented that the brush-on technique of resin mix application was made difficult by the pear-shaped particles but was facilitated by the small and irregular particles.

The short initiation period of polymerization will be further shortened by the presence of moisture. Care should be exercised to see that all equipment which comes in contact with the materials at mixing time should be moisture-free and at a temperature above the dew point.
Water Absorption

Neuman (128) states that a "hydrophobic adhesive, one which repels water or prevents water absorption, would be ideal". Acrylic resins absorb water and expand. Although Neuman did not undertake specific investigations into the effect of moisture absorption by the adhesive he was using, it was his opinion that the measurable dimensional change in a thin layer of adhesive would be small. He believed that water absorption by the adhesive was of less practical importance than is usually presumed. This opinion has been supported by more recent investigations of acrylic adhesive systems (129)(175) for there is no mention of adverse effects attributed to water absorption of the adhesive.

Ross et al. (163) showed that the inclusion of inorganic fillers decreased the water absorption of poly(methyl methacrylate). In another paper, Ross, Lal, Green and Corneli (164) showed that the addition of cross-linking material has no significant effect on water absorption, nor does a change of pH. However, Peyton and Craig (141) show a range of values for a composite plastic from a little over half that of unfilled acrylic resin to nearly double that value. The most likely explanation of this is that unfilled acrylic resins tend to absorb water rapidly over the
first twenty-five days of immersion and approach equilibrium at approximately fifty days. While some filled resins follow a similar pattern but with a lower total absorption, others absorb more slowly but continue to gain water over a much longer period. 

Buonocore demonstrated the different rates in the uptake of water between two filled composite restoratives. Peterson et al. found that after two hundred days one filled resin had absorbed approximately the same amount of water as unfilled specimens and there was still no indication that equilibrium was being approached.

Results of tests conducted on dry specimens were not shown. However, mention was made of a significant increase in hardness over the one-week test period. It was assumed this was due to continued polymerization and that with wet specimens (immersed 15 minutes after placing restorative) increase from polymerization was counterbalanced by water absorption.

The present author has found that periods of up to 15 minutes are not impractical to allow for setting of acrylic adhesives in a clinical situation. Patient comfort can be maintained for this period, even with quite young patients, and the field kept free of gross moisture. However, use of a hot air blower with self-polymerizing resins can reduce setting time to 20 or 30 seconds.
Swartz and Phillips (184) found that resin materials reach maximum adhesion in one hour.

**Leakage and Temperature Fluctuations**

Petersen et al. (140) in their laboratory study of four restorative resins (two filled and two unfilled), examined the influence of temperature change and storage in water on marginal leakage of cariously restored extracted human teeth. The method used was an evaluation of the ability of an isotope tracer (calcium $^{45}$) to penetrate between the restoration and the walls of the cavity. Penetration was detected by the use of autoradiographs prepared from longitudinal sections of the teeth cut through the restorations.

One of the restorative materials was Sevriton and it was felt that the tests could have a direct bearing on some aspects of the use of Sevriton as an adhesive, particularly with regard to temperature fluctuations.

To investigate the effects of temperature cycling on the marginal seal of the restorations, the restored teeth were transferred from a water bath held at $15^\circ C$ to one at $45^\circ C$. The immersion time in each bath was thirty seconds, and
three series of tests were conducted using 10, 50 and 100 temperature cycles.

The respective temperatures of the water baths had been determined by in vivo use of a thermocouple on two subjects drinking, alternately, hot coffee at 60°C and ice water at 0°C and registering the temperature range on the tooth surface; it was 15°C to 45°C. It was interesting to note that the reading could be lowered to 10°C by retention of the ice water in the mouth, but that this was uncomfortable and both patients agreed not their usual drinking habit.

To determine the time for the temperature change to penetrate the restorations, thermocouples were once again used, but in vitro, beneath the restorations. By alternate immersions in water baths of 0°C and 60°C it was found that 15 seconds was needed for the restoration floor to register the temperature of the surrounding environment.

Results showed that Sauritan restorations demonstrated little or no isotopic penetration, even after three months storage. Specimens cycled between temperatures of 15°C and 45°C showed leakage patterns similar to those held at a constant
temperature. However, as the temperature cycles increased from 10 to 100, a trend to greater penetration was observed. A substantial increase in isotope penetration was observed when a 60°C temperature differential was employed. This trend increased as the cycling was stopped up.

The authors have noted that their data indicates that in temperature cycling tests, the ranges used are as important as the number of temperature changes employed, and also that the more moderate temperature range is more indicative of the actual conditions to which the resin is exposed in the oral cavity.

An interesting note is that the reported reduction in coefficient of thermal expansion for the composite resin (Addent) was not reflected in the leakage pattern, which was mostly similar to the unfilled resins.\(^{(140)}\)

In a similar investigation Rose et al.\(^{(163)}\) reached the same conclusion using bacterial penetration as a measure of microleakage. In a recent study involving the bonding of three composite resins to enamel, using a standard acid etching technique, Rayonids and von Fraunhofer\(^{(149)}\) felt that moisture contamination, either from within the enamel or from external sources, was a
major contributing factor to adhesion failure in clinical practice.

Renesch (150) made some observations on marginal leakage of bonds at the enamel interface using a polycarboxylate cement and an acrylic (Sevriton). Cement margins were exposed to and protected from the moist environment by coating the exposed cement margins of half the sample with EpoxyLite pit and fissure sealant, with which it was assumed, moisture absorption would be eliminated. No significant difference in bond strengths was noted between the two samples.

**Powder/Liquid Ratio**

Phillips et al. (142) state that polymer/nomomer ratios are not critical, but that if the mix is excessively thin there is a tendency for bubbles to form in the set resin. Johnson et al. (93) considered adhesion not to be greatly affected by powder/liquid ratio in acrylic cements. However, Peyton and Craig (141) mention that use of too much monomer results in excessive shrinkage. This would increase stress concentration (21) in the case of mechanical interlocking of resin and etched enamel.

The relationship between powder/liquid ratio and the resultant viscosity of the adhesive mix would be thought to be
significant in surface wetting and flow into the etched porosities. Lee,\textsuperscript{(104)} discussing diacrylate resins as fissure sealants considered viscosity and critical surface tension to be the more important of physical properties. However, Jorgenson and Shimakada\textsuperscript{(95)} have shown by scanning electron microscopy study that more viscous resins (composites) flow into enamel micropores as efficiently as thinner ones (Niva-Seal). The authors themselves remarked that this fact was surprising.

**MIURA'S BONDING SYSTEM (ORTHOMITE)**

Miura's bonding system (Orthomite) is in three components. The powder is poly(methyl methacrylate), the liquid is methyl methacrylate monomer. The catalyst is tri-n-butyl borane (T.B.B.) derivative in place of the previously mentioned initiator system of the conventional self-curing resins. Breuer\textsuperscript{(25)} has also outlined the mechanism of T.B.B. derivative.

Miura et al.\textsuperscript{(120)} state:

When compared with the conventional self-curing resin (benzoyl peroxide - amino catalyst system), the polymerization mechanism by the T.B.B. catalyst system varies considerably. When the latter is polymerized on contact with water, the reaction is seen first at the interface. Thus, the resin mixture, when applied to the wet tooth surface, can harden even under the slightly wet conditions.
this creates more stable and durable bonding, because no shrinkage of the resin will occur on the side of the enamel surface. Conversely, the polymerization process of the conventional self-curing resin is susceptible to the existence of the water; as the polymerization progresses and the subsequent shrinkage occurs, it shows a tendency to peel off from the tooth surface. These facts explain the superiority of the new bonding system in its bonding strength.

Firstly, it must be understood that the "wet tooth surface" referred to, is one that has been isolated by cotton rolls and dried with alcohol and a stream of dry air. Moisture will still be present at the enamel surface but it will be at a microscopic level. There is also the internal enamel fluid to consider. (12)(110)

It is difficult to see where a distinction can be drawn between the mode of polymerization of the resin system of Miura et al. and that of a conventional catalyst system, although Miura's contention about T.8.8. is supported by Sanjo. (165) From the theory advanced in the foregoing quote it is hard to see how such a conclusion is reached. Present day techniques of handling unfilled acrylic resins as tooth restoratives have been directed at minimizing any tendency for the resin to pull away from the cavity walls. (141)
Further to this, the resin does not come in contact with tooth surface. It is applied to the silane coupling agent which has previously coated the prepared enamel surface. The chemical structure of a silane is such that it possesses an organofunctional group selected for compatibility with a given resin at one end and at the other has a hydrolyzable group on silicon.\(^{116}\) As the particular silane is designed to couple to an unsaturated polymer, good bonding can be expected to the resin. But what of the hydrolyzable group on silicon? Miura et al.\(^{120}\) report that the silane "was selected as the best of many similar agents that react chemically with calcium on the enamel surface". Lee\(^{101}\) agreed that the mechanism of silane coupling works only partly understood but that they often worked well. An additional comment by Miura et al. mentions that the silane has an effect of increasing the wettability of the etched enamel surface and suggests that the resin flows into more intimate contact with the substrate.\(^{119}\) This would certainly be a factor in improved bonding.

However, Lee\(^{100}\) states that "the silane cannot be said to induce better flow into cracks, per se, since this same enhancement of adhesion is observed when the silanes are used on polished glass".

The properties of both unfilled acrylic resin and a filled composite plastic are shown in Table 1.
## Table 1

Comparison of properties of unfilled acrylic plastics and composite plastics

<table>
<thead>
<tr>
<th>Property</th>
<th>Unfilled Acrylic</th>
<th>Composite Plastics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inorganic phase (wt. in %)</td>
<td>0.4</td>
<td>70-78</td>
</tr>
<tr>
<td>Setting time (min.)</td>
<td>4.0</td>
<td>3.5-4.5</td>
</tr>
<tr>
<td>Free volumetric contraction during setting (%)</td>
<td>5.2</td>
<td>1.0-2.1</td>
</tr>
<tr>
<td>Thermal coefficient of expansion (°C)</td>
<td>9.2×10⁻⁶</td>
<td>2.3-4.0×10⁻⁶</td>
</tr>
<tr>
<td>Thermal conductivity (cal./sec./cm²)</td>
<td>5.7×10⁻⁴</td>
<td>1.1-1.7×10⁻³</td>
</tr>
<tr>
<td>Water sorption (mg./cm²) 24 hrs.</td>
<td>0.80</td>
<td>0.4-0.9</td>
</tr>
<tr>
<td>14 days</td>
<td>0.84</td>
<td>0.5-1.6</td>
</tr>
<tr>
<td>Solubility in water (mg./cm²) 14 days</td>
<td>0.38</td>
<td>0.25-0.44</td>
</tr>
<tr>
<td>Modulus of elasticity in compression (lbf./inch²)</td>
<td>0.34×10⁶</td>
<td>1.6-2.4×10⁶</td>
</tr>
<tr>
<td>Compressive strength (lbf./inch²)</td>
<td>10,000</td>
<td>29,000-34,000</td>
</tr>
<tr>
<td>Diametral tensile strength (lbf./inch²)</td>
<td>3000</td>
<td>5500-6500</td>
</tr>
<tr>
<td>Yield strength, 0.1 % offset (lbf./inch²)</td>
<td>8000</td>
<td>17,000-23,000</td>
</tr>
<tr>
<td>Shear strength (lbf./inch²)</td>
<td>6000</td>
<td>7000-10,000</td>
</tr>
<tr>
<td>Modulus of resilience (inch lbf./inch³)</td>
<td>90</td>
<td>90-110</td>
</tr>
<tr>
<td>Indentation — 3/8 inch diameter ball — 30 kg. load (mm.)</td>
<td>0.113</td>
<td>0.060-0.080</td>
</tr>
<tr>
<td>Recovery from indentation — 10 min. (%)</td>
<td>74</td>
<td>68-87</td>
</tr>
<tr>
<td>Average surface roughness after</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mylar matrix (microinch)</td>
<td>0.3</td>
<td>0.3-0.5</td>
</tr>
<tr>
<td>Sandpaper strip</td>
<td>27</td>
<td>37-46</td>
</tr>
<tr>
<td>Silex</td>
<td>9</td>
<td>60-72</td>
</tr>
<tr>
<td>Tin oxide</td>
<td>6</td>
<td>36-74</td>
</tr>
<tr>
<td>Adhesion to enamel etched with 0.2 N citric acid 24 hr. storage in 37°C water (lbf./inch²)</td>
<td>1200</td>
<td>1200</td>
</tr>
</tbody>
</table>
ENAMEL PRETREATMENT

Introduction

Gwinnett and Buchanan (69) have stated:

The increased adhesion following acid treatment
of the enamel surfaces may have been due to a
number of factors:

a. a tremendous increase in surface area
due to etching;
b. exposure of the organic materials of
enamel to which acrylic can adhere;
c. removal of old, fully reacted and
inert enamel surface to expose a
more reactive surface;
d. adsorption of highly polar phosphate
groups from the acid used;
e. increase in the wettability of the
surface.
ACID ETCHING

With existing techniques of securing an effective bond between dental enamel and acrylic adhesives, acid etching of the enamel is an essential part of pretreatment.*

Gwinnett and Matsui(72) reported that "presently there is no known material capable of forming a permanent bond with untreated enamel surfaces under oral conditions". This hypothesis is substantiated by the work of Buonocore, (29) Neuman et al., (132) and Cutts and Buonocore, (47)

It would obviously be preferable to be able to dispense with acid pretreatment, but it should be borne in mind that any objection to such pretreatment of enamel is more than offset by comparison with conventional bonding techniques with regard to undetected enamel decalcification beneath cemented stainless steel bands, as against the complete displacement of a dislodged directly bonded attachment.

The degree of acid etching necessary to produce an ideal bonding surface has been outlined by Gwinnett(65) from a histological standpoint. The clinical assessment of a suitably

(154)(155)(156)(157)(158)(159)
etched surfaces is nearly done visually as described in the
directions for bonding with the Orthomite system. Watson (188)
considers it an adequate means and points out the present lack of
an alternative. However, his conclusions with regard to the
inadequate etching times recommended by the manufacturers and his
suggested new exposure times in the light of his research,
emphasize the need for an improved method of clinical control
of etching patterns.

Optimum exposure times have been assessed accurately
by Watson (188) by means of the scanning electron microscope
and the exceeding of these exposures by a margin of 20% has
been seen to produce an undesirably rough surface. This is
assuming that the treated enamel has displayed a uniformly
good bonding surface. In the light of various reports, the
heterogeneity of enamel surfaces and the varied
effect of acid etching is very clear. Such variations
may occur on different surfaces of each tooth, in
different areas of the same surface, in different teeth
from the same mouth and in different mouths.

It can be seen from these observations just how arbitrary is the visual assessment of a suitably etched surface. However, it will also be appreciated that due to the complete lack of uniformity it is difficult to see how a more suitable method may be approached. It would account for such wide variations in acid etching times suggested, using the same agent, as from thirty seconds (120) to six minutes. (147)

Yet, etched enamel surfaces and yet, pumiced enamel surfaces appear similar. (188) It is necessary to examine the surfaces dry to evaluate etching. Etched surfaces will appear whiter and duller (less reflective).

Orthophosphoric acid, at various strengths, has been the solution most employed for acid etching in enamel pretreatment. Some authors have researched large numbers of alternative etching solutions. (28)(145) Williams et al. (192) in a recent investigation claim higher retention with some resins using 30% orthophosphoric acid. Buonocore (17) first used 56% orthophosphoric acid, attenuated by the addition of seven parts by weight of ZnO. In later work (33) he used a 50% attenuated solution. Most bonding systems use strengths ranging from 50-65%. (40)(120)(130)(175)
Natauda,\(^{(114)}\) in a recent study on etching patterns of phosphoric acid and eight different organic acids, claimed that the organic acids (2% pyruvic and 1% malonic, in particular) were preferable in that less enamel dissolution produced surface roughness comparable to the deeper etching of phosphoric acid. No details of fluoride level are discussed.

Numbers of scanning electron microscope (S.E.M.) studies have been reported in the literature.\(^*\) The photomicrographs reproduced on the following pages are from a recent study by Stetler\(^{(158)}\) and demonstrate preferential sites of etching. These, together with surface profiles, readily demonstrate the reason for assuming that resin bonding to etched enamel is mainly mechanical.

The penetration of resinous tags of adhesive into the micropores of the etched enamel surface indicate the depth of the etched surface. Reports vary from five microns\(^{(36)}\) to 50 microns\(^{(154)}\).

\(^*\) (70)(71)(83)(92)(145)(158)
FIGURE 6

Featureless surface topography of normal enamel surfaces (SEM X 1000)

(Fig. 1, Rothf., 157)
FIGURE 7

Characteristic 'prism-end' structure produced by etching with 50% H₃PO₄ for 1 min (SEMx2000). Note variable etching pattern of prism peripheries and corne.

(Fig. 2, Rotsiep (157))
Honeycomb appearance of enamel surface etched with 50% H₃PO₄ for 1 min (SEM x5000).

(Fig. 3, Roti et al. (1977))
*Prism-and* structure produced by etching enamel surface with 50% H₃PO₄ containing 7% zinc oxide (SEM×2000).

(Fig. 4, Relief(157))
Preferential etching of prism cores by 50% citric acid applied for 2 min (SEM x 2000).

(Fig. 5, Ratio of 157)
Etching of enamel surface by 50% citric acid applied for 2 min (500x5000)

(Figs 6c, & d, Fig. 157)
FIGURE 12

Preferential etching of ridges of porphyra (SEM x200). Note relative absence of etching pattern in grooves.

(Fig. 7, Ratief 157)
Etching confined to ridge of perikymata (SEM x1000)

(Fig. 8, Relief \(157\))
Marked interfacial separation between adhesive (AD) and unconditioned enamel surface (EN) (SEM x1000).

(Fig. 15, Ratier (157))
Intimate interfacial contact between adhesive (AD) and enamel surface (EN) etched with 50% H$_3$PO$_4$ for 1 min (SEM x1000).

(Fig. 16, Ratier (187))
Reduced interfacial contact between adhesive (AD) and enamel surface (EN) etched with 50% H₃PO₄ for 1 min (SEM×1000).

(Fig. 17, Retief, 157)
Slightly increased interfacial separation between adhesive (AD) and enamel surface (EN) etched with 56% citric acid for 2 min (SEM x1000).

(Fig. 18, Ratiof 157)
Exposure of adhesive aspect of interface that had contacted unconditioned enamel (SERx200).

(Fig. 19, Ref. 157)
Fig. 20.

Featureless surface topography of adhesive surface that contacted unetched enamel (SEMx2000). (Fig. 20, Natarajan (157).)
Dissolution of enamel revealed resin tags extending from adhesive surface opposed to enamel surface etched with 50% H₃PO₄ for 1 min (SEMx2000).

(Fig. 21, Retief(357))
Fig 11.—Photomicrograph shows typical tags projecting from resin after dissolution of etched enamel. Tags represent resin penetration into etched enamel (orig mag $\times 450$, reproduced at 73%).
Fig. 6. Graphic presentation of mean bond strengths and standard deviations of adhesive to enamel surfaces etched with different concentrations of phosphoric acid.

(Fig. 6, Ratios (158) Table IV)
Fig. 8. Surface profile of enamel surface polished with 600 grit silicon carbide paper.
Fig. 9. Surface profiles of polished enamel surfaces etched with: A, 10 per cent $\text{H}_3\text{PO}_4$; B, 20 per cent $\text{H}_3\text{PO}_4$; C, 30 per cent $\text{H}_3\text{PO}_4$; D, 40 per cent $\text{H}_3\text{PO}_4$; E, 45 per cent $\text{H}_3\text{PO}_4$; F, 50 per cent $\text{H}_3\text{PO}_4$; G, 60 per cent $\text{H}_3\text{PO}_4$; H, 65 per cent $\text{H}_3\text{PO}_4$.
Table 3. Rate of etch of enamel surfaces exposed to three etching solutions

<table>
<thead>
<tr>
<th>Etching procedure</th>
<th>No. of teeth etched</th>
<th>15 s</th>
<th>30 s</th>
<th>45 s</th>
<th>60 s</th>
<th>120 s</th>
</tr>
</thead>
<tbody>
<tr>
<td>50% H₃PO₄</td>
<td>10</td>
<td>4.3±1.2</td>
<td>10.0±2.3</td>
<td>16.4±3.2</td>
<td>23.1±3.8</td>
<td>—</td>
</tr>
<tr>
<td>50% H₃PO₄ + 7% ZnO</td>
<td>10</td>
<td>2.5±0.4</td>
<td>5.7±0.7</td>
<td>9.4±1.1</td>
<td>13.4±1.5</td>
<td>—</td>
</tr>
<tr>
<td>50% citric acid</td>
<td>10</td>
<td>0.8±0.02</td>
<td>1.9±0.4</td>
<td>3.1±0.2</td>
<td>4.6±1.1</td>
<td>10.1±1.8</td>
</tr>
</tbody>
</table>

(Table 3, Retief (156)).
THE ROLE OF FLUORIDE

Hoffman et al. (84) found that topical fluorides prevented formation of typical enamel changes when etched with acetate buffer. Guinnett et al. (71) have reported similar findings and concluded that the presence of a reaction product of the enamel surface as a result of topical fluoride treatment was doubtless the reason for reduced bond strengths observed with both Sevriton and Buonocore's pit and fissure sealant.

In a scanning electron microscope study, these authors demonstrated the lack of penetration of acrylic resin into surfaces which had received topical fluoride by the absence of resinous "tags" of the type described by Guinnett and Matsui (72), Buonocore et al. (36), and Guinnett (65).

Sheykholsalam et al. (173) made similar observations with regard to absence of resin penetration into enamel pores in relation to uptake of various fluorides. They thought the strength of attachment of reaction products to the enamel surface could be of considerable importance if the adhesive resin were in turn capable of forming strong bonds with the reaction product; in fact a coupling action. They thought this could be the case with acidulated sodium fluoride,
since in spite of the fact that few or no tags were obtained, the bonding strength was not significantly different from controls.

The reaction product described by Guinnott et al\(^{(71)}\) was shown to appear as minute globules that occluded the porous structure created by the acid etching. Little et al.,\(^{(111)}\) in a study of the site of fluoride accumulation in intact enamel, showed the major incorporation of fluoride to be limited to areas of discolouration, either slight or extensive, depending on whether the tooth was newly erupted or had been in the mouth for a decade or more. As much as ten per cent of an unerupted tooth shows some discolouration. This study also confirmed earlier findings that surface enamel is considerably richer in fluoride than subsurface enamel.\(^{(88)}\)

Miura et al.\(^{(120)}\) used thirty-second pretreatment with sixty-five per cent phosphoric acid with no suggestions for varying the procedure. The instructions for the Orthozone (Miura's) system suggest allowing forty-five seconds acid pretreatment for adults or patients in fluoridated areas and repeating the sequence if the bonding surface does not appear dull. It is known that Miura\(^{(120)}\) approved of this procedure
as he strayed adhering strictly to the instructions issued
with the Orthomite system.

Watson (186) reported that:

The unexpectedly light etching produced by
the tested etching solutions, at the
recommended exposure times, was considered
to be due to increased surface resistance
to etching as a result of a post-developmental,
pre-eruptive (and to a minor degree, post-eruptive)
uptake of fluoride in the enamel surface.

A COMPARATIVE EVALUATION

In order to make some comparative assessment of the
effect of pretreatment of enamel in direct bonding of
orthodontic attachments, the effect of routine instrumentation
of tooth surfaces in dental prophylaxis has been examined.
It was felt that a scanning electron microscope study of
treated tooth surfaces could be compared with similar studies
of tooth surfaces following acid pretreatment for direct bonding
of attachments.

There is quite a wide field of scanning electron
microscope studies of normal enamel. (22)(82)(121) Studies of
treated enamel examined by the scanning electron microscope
include: Hoffman et al.,(63) Guinnott et al.,(71) Guinnott and Buonocore,(70) Johnson et al.,(92) Watson(188) and Rafter(157).

However, the literature on scanning electron microscope studies of tooth surfaces following routine prophylaxis is scarce.

Some studies have been made of the effects of instrumentation on cementum but only two were available that included an examination of other tooth surfaces. One report by Jones, Lozden and Boyd,(94) examined the morphology of tooth surfaces treated with tungsten-carbide curettes, steel scalers and the Cavitron ultrasonic scaler, by the use of scanning electron microscopy. The authors found that the damage to enamel by curettes and scalers may be considerable and is especially severe near the cement-enamel junction. The damage frequently appeared as an inelastic deformation or smearing of the surface layers, with the smeared layer not always well attached to the underlying normal tissue. Photomicrographs revealed fractures along incremental lines and heavy grooving from hard instruments. The Cavitron caused least damage.

A study by Pamaijar et al.(135) reached similar conclusions with regard to a comparison of hand and ultrasonic instrumentation. However, the report did not
detail the findings in the same way as that of Jones et al. (94) but stated that no changes in the enamel were observed. Most deleterious effects were attributed to faulty technique in handling instruments.

**Enamel Recovery**

No report could be found in the literature of a scanning electron microscope study of the effects of polishing of enamel surfaces with polishing cups and brushes and pastes per se. However, Watson (188) discusses the appearance of the polished surface of enamel under scanning electron microscopy and compares it to the same surfaces after acid etching, adhesive bonding, removal of adhesive and repolishing. He noted that the repolished surface appeared very similar to the original polished surface at lower levels of magnification and that good enamel recovery had occurred after polishing, following bonding procedures, although recovery was not absolute.

The incompleteness of enamel recovery was only observed at magnifications in excess of 500X and by further polishing with a finer prophylactic paste a higher degree of recovery was observed.
Newman and Focq (130) reported that the tooth surface is restored to its original pumiced appearance after surface treatment, bonding, removal of the attachment and adhesive, and pumicing. The reader is referred to a scanning electron microscope photomicrograph at a magnification of 1,000X for comparison with the original pumiced surface.

Lenz and Mühlemann (107) reported that the characteristic prism-end pattern of etched enamel disappeared on exposure to conditions in the oral cavity for two to 48 days. They attributed this to remineralization or abrasion. Miura et al. (120) reported that 2,000 toothbrush strokes restored enamel to its preconditioned appearance.

Rekaw et al. (160) treated the occlusobuccal surfaces of two premolars with fifty per cent phosphoric acid for sixty seconds, while the gingivobuccal halves were left untreated. One tooth was extracted ten minutes later and the other one week later. Microscopic examination of the surfaces of the specimens revealed a marked difference in the two areas on the ten minute specimen, whereas the one week specimen showed a less marked difference.
CHAPTER 3

EVOLUTION OF A SUITABLE TESTING TECHNIQUE

Preliminary tests were done with a group of four cements, namely Poly C (polycarboxylate), Durelon (polycarboxylate), Zinc Cement Improved (zinc phosphate) and Kryptox (silico-phosphate), using strips of 0.1 mm stainless steel band material.

Extracted bovine teeth were used and prepared according to the instructions issued by the makers of Durelon. Pieces of band material, approximately an inch in length, were cemented with each of the four cements mentioned and a tag end of the metal was left to grasp to test the bond. It was appreciated that these simple tests would give some indication of bonding, but only with regard to peel strength, due to the flexible nature of the bond material. Because of the size of the enamel surfaces involved, numbers of these metal strips were cemented to each tooth. All were prepared without acid etching and some were immersed in tap water and stored at 37°C. Not many of the bonds showed any cohesion, some of them even parted
from the enamel surface while immersed in water at 37°C awaiting testing. The failures were observed at the cement/metal interface. It was noted that there was an acceleration of failure rate with water immersion.

Tests were also conducted in a similar manner, using these four cements to attach stainless steel lingual buttons to the enamel of extracted human teeth. The buttons were removed after a suitable time by grasping the button with a pair of pliers and simply pulling it off the enamel. It was noted that more adhesion had been achieved with a rigid type of metal base in the case of Poly C and Surelon.

Acrylic patch brackets were also made to cover the entire labial face of extracted human teeth and these were tested in vitro with the four cements mentioned and proved to be unsatisfactory. A further set of in vitro tests was conducted on the 3M Scotch system.* This adhesive system consisted of clear plastic brackets, an enamel pretreatment solution designated as a phosphate containing 1½ fluoride, and a powder and liquid resin adhesive. Instructions for use were similar to Rocky Mountain Orthomito system. Some

* Ortho International Services Inc., Wilmington, Delaware.
difficulty was experienced with the setting of the adhesive and a request for further information from the manufacturer met with no response. Further tests with this system were abandoned.

A laboratory investigation of another direct bonding system (Orthomite)* involving the attachment of polycarbonate brackets directly to tooth enamel, by the use of an acrylic resin adhesive, was commenced with a series of preliminary tests. These tests were initiated in order to find a suitable method of testing the bonds for both shear and tensile strength.

The teeth used in the tests were extracted human bicuspids teeth, which had been stored in tap water after being immersed for a short period in a bactericide and having the pulps of the teeth removed where possible through their open splices. Lingual buttons were selected as the preferred attachment for two reasons. Firstly, it was proposed to use a system whereby the pull exerted on the bracket during tests could be achieved by coupling a wire to the lingual button. Secondly, the contour of the base of these attachments was seen to conform more closely to that of the bicuspid teeth chosen for the tests.

* Orthomite, Rocky Mountain Dental Products Company.
It was not proposed to re-contour the bases of any of the brackets to be tested, as it was thought that this would constitute a variable in testing, in that it was thought that if the bases were contoured with pliers as suggested in the instructions, due to release of strains there was almost certain to be added strain set up in the bond in the ensuing period before actual testing. The brackets were bonded to the buccal surfaces of the teeth, according to the instructions from Rocky Fountain Dental Products Company. These instructions are reproduced "in toto", as it cannot be emphasised too strongly that successful bonding of resins to enamel "in vivo" is an exacting technique.* Daft and Lugassy(48) thought it "too exacting". They also admitted to less than careful bonding techniques and a very limited experience of clinical usage of direct bonding. Also they had no experience of torquing auxiliaries with plastic brackets. Mura(116) in a personal communication, emphasised the importance of following meticulously the instructions given for the Orthomite Direct Bonding System.

They are as follows : 

1. ADAPTING PLASTIC BRACKET TO TOOTH SURFACE

a. Brackets are adapted to the anatomical form of the labial surface of each tooth on the study model.

b. The bracket must be shaped so that its base fits the contour of the tooth surface. Note: if contouring is done several hours before bonding, the brackets may tend to return to their original shape. Make final adjustments at time of bonding. Do not attempt to bond an overcontoured bracket to the tooth. It will spring away from the adhesive when placed.

c. Clip the corners of the base, if needed for better fit or to avoid gingival interference.

d. Store the brackets in a Bend and Bracket tray for easy identification.

2. PRETREATING OF ENAMEL TOOTH SURFACE

a. Clean the teeth with prophy paste and rinse. (Do not use fluoridated prophylactic paste).

b. Isolate the area with cotton rolls and dry with air.
Note: Be sure that air spray is dry and free of compressor oil.
If combination air and water syringe is used, allow any remaining
water to be expelled before applying air stream to teeth.

g. Wipe the tooth surface with ethyl alcohol (70-80%) and dry it well with air. (Do not use isopropyl alcohol).

d. Apply Solution A (phosphoric acid) liberally, but carefully, to the full tooth surface to be bonded using a cotton pellet.
Avoid contact with soft tissue. Phosphoric acid will cause mucosal irritation if left in contact for an extended period.
Note: Neutralize the cotton pellet after use in a diluted baking soda solution to prevent damage to clothing or fixtures.

3. Allow Solution A to remain for 30 seconds. Remove cotton rolls and spray water on treated tooth to dilute and remove solution. Have the patient rinse. Note: For adults or patients in fluoridated areas, allow solution to remain for 45 seconds.

f. Insert fresh cotton rolls and dry teeth with air.

g. Wipe the surface again with alcohol and air dry. The bonding surface should appear dull. If it does not, repeat sequence from application of Solution A (step d).
h. Apply Solution B (silane coupling agent) to the tooth surface with a cotton pellet, leaving a light coating. Silane is non-toxic and does not cause gingival irritation; however, it is unpleasant tasting. Keep container tightly closed when not in use.

i. Immediately apply a forceful stream of air to teeth to create a thin film of silane. Insufficient thinning permits silane to fill tooth crevices and prevents adhesive from achieving a secure bond. Enamel surface appears lustrous again. Do not dry with evacuator; a thin film of silane cannot be created. Be sure air is dry and free of oil. Keep teeth dry for remainder of application.

3. MIXING OF BONDING MATERIALS

a. The press is placed on top of Container C and pushed down firmly. This punctures the metal capsule in the neck of the vial and frees the catalyst to be mixed with the monomer.

b. Visually inspect through the glass container to be sure the inner capsule has opened.

c. Tip Container C upcide down and shake for 4-5 seconds to mix catalyst with monomer.
d. Use the press as a holder for Container C. Remove the
cap with the opener.

   * Use care when opening
   the vial so as not to
   break the container.

   * It is very important to use
   Solution C within 15 minutes
   after mixing. Caution: The
   liquid appears the same after
   15 minutes but it may not form
   a secure bond.

g. Place some resin powder D into the dappen dish.
Keep container tightly closed when not in use.

4. BONDING BRACKETS

a. Brush-on technique: Dip the brush into Container C
and then dip the brush into powder D until a bead of adhesive
is formed.

   * The ideal ratio of liquid
   and powder will create a bead
   approximately this size.
b. Apply the adhesive to the tooth surface.

c. Repeat if necessary, and spread the adhesive over a slightly greater area than the bracket base. Apply enough adhesive so that a slight excess of adhesive will form around the base when the bracket is placed.

d. Wipe the brush clean on a piece of gauze each time before reinserting brush in monomer-catalyst mixture, (C). Contamination with resin shortens the active life of Solution C.

e. Use DBS Tweezers (Mesanaga) to carry the bracket to the tooth and to determine its position and angle. Once the bracket is properly positioned, press it into the adhesive with moderate pressure. Do not move the bracket after pressure is applied.

   Place the bracket immediately after adequate adhesive is on the tooth. Do not allow adhesive to dry before placing bracket.

f. Smooth the adhesive margin around the base with the wet brush. Add more adhesive if necessary to fill any voids. Use care not to flow material into the ligating area of the bracket.
Do not wax the bracket or use elastics to keep the wings free of adhesive.

9. The bonding of one bracket is now completed.

h. The same procedure is used to attach the brackets to each tooth.

i. If all brackets are not placed within 15 minutes after mixing Solution C, discard and mix another Container C.

1. Keep the entire area dry after all teeth are bonded and wait for 10-15 minutes for hardening of the adhesive.

In the meantime, clean the brush thoroughly before storing by washing it in any remaining Solution C, or use chloroform.

Discard any excess Solution C into the sink and rinse with water.

Note: Solution is flammable.
5. **FINISHING DETAILS**

   a. Make sure that the adhesive is fully hardened by pressing with an explorer.

   b. If any part of the adhesive interferes with the insertion of a wire, grind that area with a polishing stone.

   c. Here is a case in which both maxillary and mandibular arches have been bonded.

   d. Levelling arches or other gentle forces may be tied in approximately 30 minutes after bonding. However, the adhesive is completely cured and reaches maximum strength in 24 hours.

**KEYS TO GOOD BONDING**

For best results, the following steps are essential:

   a. Adapt the bracket base carefully to the tooth surface on the study model.

   b. Avoid contamination of the bracket base from hand lotion or
excessive finger oils. Clean with alcohol if necessary.

c. Clean the tooth thoroughly with rubber cup and prophylactic paste before pretreatment.

d. Wash away Solution A completely from the tooth.

e. Disperse Solution B thoroughly over the tooth surface using air. It is important to create a very thin film.

f. Dry the tooth thoroughly before each bonding.

g. Wipe the adhesive from the brush each time before dipping into Container C.

h. Place the bracket on the adhesive immediately after adequate adhesive is applied to the tooth.

i. Do not jiggle the bracket after positioning.
j. Keep the tooth dry until the adhesive is hardened.
k. Discard any exposed powder D or unused Solution C.

REMOVAL OF BRACKET

a. Grasp the bracket base with a pin and ligature cutter and twist slightly to remove.

b. Soften the remaining adhesive with chloroform, and remove it with a scaler.

c. Polish the surface with prophylactic paste and a rubber cup for 10-15 seconds. Polishing will restore the original lustre of the enamel surface.

REPLACEMENT OF BRACKET

If, for some reason, a bracket loosens during treatment, remove cement, clean the area thoroughly with chloroform, then repeat the entire bonding procedure.
CAUTION

- Store the DBS kit below 68°F (20°C).

- Do not expose Containers B and C to heat or direct sunlight.

- Close the caps of Containers A, B and D tightly after use.

- Discard Solution B if it is viscous or if it has colour.

- Do not use Solution C more than 15 minutes after mixing.
Showing test specimen mounted on tensometer in original wire jig for shear test.
Some difficulty was encountered in the bonding procedure, firstly in using the brush supplied, which fell apart after the first few bonds had been made, and secondly some difficulty with the speed of set of the acrylic cement was encountered. As no substitute brush was available on this occasion, an alternative procedure of mixing the powder and liquid in a dappen glass was used and the mix was applied to the bracket base with a stainless steel instrument and this was then placed in position on the tooth surface and the cement allowed to set. Due to the concave surface of the bracket base, it was felt that it was preferable to place the adhesive inside this base and so lessen the risk of including air voids in the final set.

With regard to the discarding of the 'bead and brush' technique, apart from the reasons stated, certain other inadequacies of such a technique are apparent. One is that contamination of the brush with partly set adhesive must be inevitable, no matter how well it is cleaned after each use. The other is the lack of control of the viscosity of the adhesive mix. It is essential that there is a liquid phase available on the cement surface to promote intimate contact with the micropores of the enamel surface. (96) As this has
to be judged visually it is felt than an observed manual mix would be more suitable.

To mount the tooth with its bonded bracket on the testing equipment, a hole was drilled in the crown of the tooth mesiodistally and directly beneath where it was anticipated the bracket would be cemented. A 1.25 mm stainless steel wire was inserted through the hole and two right angled bends made on either side of the hole. Approximately half way between this hole and the root apex, another bar of wire was soldered across the two ends of wire, so that the tests being made in shear would immobilise the tooth by preventing the root from moving (see Plate 1).

A Hounsfield Tensometer was the instrument used to test the bond strengths and in these first tests it was set at a strain rate of 0.2 mm/min. The two wires that have been described, which were attached to the tooth, were inserted in the grip at one side of the tensometer and firmly clamped and a 0.7 mm stainless steel wire, bent in the form of a loop (as shown in Plate 1), was attached to the button neck of the bracket and these wires clamped into the other grip of the tensometer.
Six bonds were tested and the results are given in Table 2.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>62</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>31</td>
<td>-</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>49</td>
<td>-</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>29</td>
<td>-</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>18</td>
<td>-</td>
</tr>
<tr>
<td>6.</td>
<td>Shear</td>
<td>-</td>
<td>Failed during setting up on tensometer.</td>
</tr>
</tbody>
</table>

The bond failure sites were examined with a Zeiss stereoscopic microscope at a magnification of sixteen with oblique illumination. All bonds were observed to have failed at the enamel interface.

Those teeth were re-used for a further set of preliminary tests. The enamel surfaces were scaled, then polished with
pumice and water paste and rebonded with the button brackets as before, except that the etching time was increased from thirty seconds to two minutes following a discussion with B. I. Watson. His scanning electron microscope study (188) had included an evaluation of acid etching. After two hours, the bonds were again tested on the tensometer by the method previously described, excepting that instead of a speed of 0.2 mm/min, a new setting of 0.8 mm/min was used. Three of the bonds were tested in shear and at loads of 67N, 69N and 64N respectively, the buttons sheared off the base of the brackets (a cohesive failure of the polycarbonate brackets). The remaining three teeth with their bonded brackets were then modified by embedding a metal ring in self-curing acrylic resin and attached to the button on the bracket base and the resin flowed around the button and on to the base so that the ring had a direct connection to the base and did not have to rely on the strength of the neck of the button. These three bonds were then tested for tensile strength by simply suspending the tooth and attached bracket between the grips of the tensometer in the manner shown in Plate 2 and 3. Those plates show the new wire jig described on Page 141.
Showing two views of test specimen mounted on toncometer for shear test in new wire jig with modified attachment to plastic lingual button.
TABLE 3

Preliminary tests at a strain rate of 0.8 mm/min using Orthomite with silane

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>39</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>49</td>
<td>-</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>82</td>
<td>-</td>
</tr>
</tbody>
</table>

Microscopic examination of the first bond revealed large voids in the cement which had apparently been produced by over compression of the cement at the time of attaching the bracket and a "regurgitating" effect during subsequent release of pressure. In the second bond, failure was observed at the enamel interface, while in the third, bond failure was noted in the cement itself and one corner of the bracket base fractured and was left adhering to the enamel. A further set of tests was done on seventeen fresh samples which had the rings bonded to the bases by the method previously described, but instead of a thirty second pretreatment of the enamel, two minutes was given to these samples. They were placed in water at 37°C for 28 days and then tested. The results are given in Tables 4 and 5.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Shear</td>
<td>24</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Shear</td>
<td></td>
<td>Bond failed during setting up*</td>
</tr>
<tr>
<td>3</td>
<td>Shear</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Shear</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Shear</td>
<td></td>
<td>Bond failed during setting up*</td>
</tr>
</tbody>
</table>

The sites of the failures were examined under the microscope and in all five were found to be at the enamel interface. With the two bonds that failed during setting up on the Tensometer, a strong peel effect was noted in both and it was realised that the high placement of the ring was producing this effect in shear tests. It was decided to test the remaining twelve bonds for tensile strength, as it was felt that there was no point in conducting further shear tests under those conditions.
Table 5 shows the results of the tensile tests conducted at the same rate of 0.8 mm/min on the tensometer. Observations made during the tests and afterwards by examination with the stereoscopic microscope are noted after the failure strains in each case. This has been done with the preliminary tests to see that the relationship of some individual readings to the observations is significant. It had been intended only to summarize the comments after each group of tests in the main experiments. However, it was seen that this would obscure some significant relationships.
TABLE 5
Preliminary tests at a strain rate of 0.5 mm/min using Orthomite with oil and agar.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>55</td>
<td>Bond intact, ring pulled out of plastic.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>58</td>
<td>Bond intact, ring pulled out of plastic.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>64</td>
<td>Bond failure at enamel interface, some porosity noted in cement.</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile</td>
<td>46</td>
<td>Bond failure at enamel interface, a few small voids seen.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>60</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>29</td>
<td>Bond failure at enamel interface, one medium sized centrally placed void.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>55</td>
<td>Bond intact, ring pulled out of plastic.</td>
</tr>
<tr>
<td>8.</td>
<td>Tensile</td>
<td>27</td>
<td>Bond failure at enamel interface, small void in cement.</td>
</tr>
<tr>
<td>9.</td>
<td>Tensile</td>
<td>67</td>
<td>Bond intact, ring pulled out of plastic.</td>
</tr>
<tr>
<td>10.</td>
<td>Tensile</td>
<td>67</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>11.</td>
<td>Tensile</td>
<td>46</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>12.</td>
<td>Tensile</td>
<td>40</td>
<td>Bond failure at enamel interface.</td>
</tr>
</tbody>
</table>
In five of the tests the ring pulled out of the plastic. Bond failure of the remaining seven occurred at the enamel interface and in four of these, voids were observed in the cement.

On the five composites in which the ring had pulled out of the plastic, but in which the bond between bracket base and enamel was still intact, a new technique for shear tests was tried. A block of acrylic resin was built up on the original bracket base and covered the button. A hole was drilled through this as close to the bracket base as was possible and centred visually occluso-gingivally. Through this hole a 0.7 mm wire was fitted with a 90° bend either side of the bracket so that the free ends could be clamped in the grip of the Tensometer. These bonds were then tested on the Tensometer, as before, for shear strength.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.</td>
<td>Shear</td>
<td>31</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>75</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>73</td>
<td>Bond failure at enamel interface. Piercing of bond site noted.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>-</td>
<td>Failed during setting up.</td>
</tr>
</tbody>
</table>
Showing test specimen mounted on tensometer in new wire jig with ultimate modifications for shear test.
Showing test specimen mounted on tensometer in new wire jig with ultimate modifications for tensile test.
In four tests, bond failure occurred at the enamel interface. In two of these, the bond site had been pierced by the hole in the acrylic block, but this was not consistent with lower readings. The remaining bond failed during setting up.

A further forty three bonds were made on fresh samples of teeth, using the previous method, with the exception that the acid pretreatment was extended to two and a half minutes. Twenty-three of these teeth were stored in tap water at 37°C and the remaining twenty were stored in tap water at room temperature.

The original method of mounting the tooth on the Tensometer was superseded by a system which was more flexible with regard to removing and replacing each tooth to be tested. Instead of an individual system for each tooth, as shown in Plate 1, a new jig was made up from 1.25 mm stainless steel wire as shown in Plate 4. The wire, which was inserted through the hole in the tooth, was not fixed and could be withdrawn and replaced in each successive tooth. It allowed the tooth to be positioned quite firmly and retained horizontally for shear tests and also to be repositioned for tensile strength tests, see Plate 5, as with the first system.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>44</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>87</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>55</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>18</td>
<td>Bond failure at enamel interface. Bond had a shiny appearance (glassy) compared to preceding sample; no voids observed.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>51</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>6.</td>
<td>Shear</td>
<td>51</td>
<td>Bond failure partly at enamel interface and partly in bracket base; half of which was left adhering to the enamel.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>64</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>8.</td>
<td>Shear</td>
<td>49</td>
<td>Bond intact, failure in acrylic block.</td>
</tr>
<tr>
<td>9.</td>
<td>Shear</td>
<td>—</td>
<td>Bond failed during setting up.</td>
</tr>
<tr>
<td>10.</td>
<td>Shear</td>
<td>149</td>
<td>Bond failure at enamel interface. White patches of enamel decalcification observed on tooth.</td>
</tr>
<tr>
<td>11.</td>
<td>Shear</td>
<td>—</td>
<td>Bond failed during setting up.</td>
</tr>
</tbody>
</table>
TABLE 7 (continued)

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>Sheer</td>
<td>125</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>13</td>
<td>Tensile</td>
<td>91</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>14</td>
<td>Tensile</td>
<td>91</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>15</td>
<td>Tensile</td>
<td>15</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>16</td>
<td>Tensile</td>
<td>187</td>
<td>Bond failure at enamel interface. Again white patches were noted on the enamel.</td>
</tr>
<tr>
<td>17</td>
<td>Tensile</td>
<td>-</td>
<td>Bond failed during setting up.</td>
</tr>
<tr>
<td>18</td>
<td>Tensile</td>
<td>38</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>19</td>
<td>Tensile</td>
<td>118</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>20</td>
<td>Tensile</td>
<td>44</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>21</td>
<td>Tensile</td>
<td>71</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>22</td>
<td>Tensile</td>
<td>144</td>
<td>Failure in acrylic block.</td>
</tr>
<tr>
<td>23</td>
<td>Tensile</td>
<td>24</td>
<td>Bond failure at enamel interface.</td>
</tr>
</tbody>
</table>
TABLE 8

Preliminary tests at a strain rate of 0.8 mm/min in specimens stored for 28 days in tap water at room temperature using orthodontic with silicone.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>49</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>125</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>27</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>87</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>131</td>
<td>Bond failure at enamel interface, No flash left adhering to enamel,</td>
</tr>
<tr>
<td>6.</td>
<td>Shear</td>
<td>76</td>
<td>Bond failure at enamel interface, Due to shape of tooth an inclined angle of shear was noted,</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>59</td>
<td>Bond failure at enamel interface, Some flash left adhering to enamel,</td>
</tr>
<tr>
<td>8.</td>
<td>Shear</td>
<td>93</td>
<td>Bond failure partly at enamel interface, partly in bracket, Approximately half bracket base left adhering to enamel,</td>
</tr>
<tr>
<td>10.</td>
<td>Shear</td>
<td>91</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>11.</td>
<td>Tensile</td>
<td>78</td>
<td>Bond failure at enamel interface,</td>
</tr>
<tr>
<td>12.</td>
<td>Tensile</td>
<td>76</td>
<td>Bond failure at enamel interface, No flash left adhering to enamel,</td>
</tr>
</tbody>
</table>
### TABLE 8 (continued)

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>Tensile</td>
<td>76</td>
<td>Bond failure at enamel interface. Some flash left adhering to enamel.</td>
</tr>
<tr>
<td>14</td>
<td>Tensile</td>
<td>29</td>
<td>Bond failure at enamel interface, dark specks observed and larger amount of flash left adhering to enamel.</td>
</tr>
<tr>
<td>15</td>
<td>Tensile</td>
<td>49</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>16</td>
<td>Tensile</td>
<td>53</td>
<td>Bond failure in acrylic.</td>
</tr>
<tr>
<td>17</td>
<td>Tensile</td>
<td>69</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>18</td>
<td>Tensile</td>
<td>27</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>19</td>
<td>Tensile</td>
<td>64</td>
<td>Bond failure at enamel interface.</td>
</tr>
<tr>
<td>20</td>
<td>Tensile</td>
<td>53</td>
<td>Bond failure at enamel interface, with a small amount of flash adhering to enamel.</td>
</tr>
</tbody>
</table>

This completed the preliminary testing in which seventy seven bonds had been tested by the various means described and a technique had been developed which seemed to be reliable and which seemed a fair test of shear strength and tensile strength of the bonds.
CHAPTER 4

MATERIALS AND METHODS

TOOTH SAMPLES

The teeth used in the investigation were freshly extracted human bicuspids from both upper and lower jaws. Most of them were teeth that had been extracted from young patients in preparation for orthodontic therapy. There were very few teeth that needed to be discarded because of any visible injury to the enamel caused by forcope extraction. The samples used were deemed to be satisfactory for the purposes of the investigation from a visual examination of the enamel surfaces under strong light.

After extraction, the teeth were washed and stored in an aqueous solution of Zephiran. Due to the age of the teeth samples, it was possible to remove most pulps by instrumentation through the wide open apices. Following this, the tissues adhering to the root areas were removed by scaling and
polishing and the samples were then washed thoroughly and stored in tap water, which was changed daily.

By use of a diamond fissure Burr in a water-cooled air-driven high speed handpiece, a hole was drilled mesio-distally through the crown of the tooth to admit a 1.25 mm stainless steel wire. The hole in the tooth was located so that, after bracket placement and when the bond was being tested for tensile strength, the pull would be directed as near to 90° to the enamel surface as possible.

The wire jig, which was evolved in the preliminary experiments to determine a suitable method of testing, could then be fitted to each tooth during testing with a minimum time loss.

**ATTACHMENT OF BRACKETS**

Polycarbonate brackets were prepared, as described in the ultimate testing technique, by adding an acrylic block to the base and preparing the bracket to receive the testing jig. After light stoning of the bracket bases with an Alpina green abrasive stone, pattern number A 122 (Amalgamated Dental (Australia))
Pty. Ltd.), the attachment of the brackets to the teeth was then proceeded with as follows:

The instructions for the Orthomite direct bonding system (given on page 118) were followed for the bonding of each of the four different systems, with the following alterations:

1. The bracket bases were not adapted (as suggested in the Orthomite instructions) in any way, either by re-shaping or clipping, for reasons previously stated.

2. Solution A (65% phosphoric acid) was left on the enamel surface for two and a half minutes. (Due to tendency of the etching solution to evaporate, the enamel surface was kept moist with a pledget of the solution throughout the two and a half minute period.) This was the standard etching time used for all systems in the tests.

3. In the tests conducted with the Orthomite system without using silane, this part of the pretreatment was simply dispensed with.

The same procedure was used for Sevriton, excepting that
silane was not used. Senviron cement was applied instead. As previously described, the application of the cement to the bracket base was not achieved with a brush-on technique, as described in the instructions, but a process of mixing the appropriate powder and liquid (Orthoamic or Senviron) in a deep glass was used and the mixed cement was then applied to the bracket base with a stainless steel plastic instrument.

When the bracket was seated on the tooth during cementing, it was merely positioned and then left to harden without using any seating pressure on the bracket. This was deemed to be an important part of the technique, as it was thought this would eliminate any chances of moving the bracket during setting and also to eliminate varying responses from inconsistent seating pressures. It was also thought that a non-pressure seating technique would avoid any tendency to joint starvation, particularly in cases where the concavity of the bracket base was greater than the convexity of the tooth surface to which it was being applied.

After hardening, the bond was not interfered with in any way by any method of finishing or polishing, but after fifteen minutes the tooth were immersed in tap water and stored at a temperature of 37°C until tested. A comparison between the results of preliminary tests recorded in Tables 7 and 8 showed no significant differences in bond strengths for specimens stored either at room temperature or at 37°C.
COMPOSITION OF CEMENTS, PRETREATMENT SOLUTIONS AND SEALER.

Orthomite*

Liquid : monomer - methyl methacrylate
Powder : polymer - poly(methyl methacrylate)
Catalyst : tri-n-butyl borano derivative
Pretreatment solution A : 69.5% phosphoric acid, containing 7% of zinc oxide by weight
Pretreatment solution B : methacryloxypropyl tri-methoxysilane

Sovriten Simplified**

Liquid : monomer - methyl methacrylate
Powder : polymer - poly(methyl methacrylate)
benzoyl peroxide initiator
pigment
opacifiers
Sealer : an analysis by Buonocore et al.(38) indicated it contained:
5 to 10 parts of the phosphoric acid ester
(glycorophosphoric acid dimethacrylate)
5 to 10 parts of methacrylic acid
80 to 90 parts methyl-methacrylate
with perhaps a trace of methacrylic anhydride.

* Orthomite, Rocky Mountain Dental Products Company.
** Sovriten Simplified, Amalgamated Dental Trade Dist. Ltd., London.
ULTIMATE TESTING TECHNIQUE

The method used for preparing and testing of the samples was as follows:

The lingual button brackets were embedded in a block of Paladur clear self-curing acrylic resin, which conformed in two dimensions to the shape of the bracket base, without in any way interfering with the base itself. This acrylic block was approximately 5 mm in depth and was prepared by placing the bracket base on wax, so that acrylic could not flow onto the base area and a metal strip was used as a former, into which the mixed acrylic liquid and powder was poured. After setting this block was examined to see that it conformed in size and any flash was polished off with a sandpaper disc. The bracket base was then cleaned with ethyl alcohol to make sure no wax film might be left adhering to it. Just prior to the cementation of the brackets, the bases were lightly ground with an Alpine green abrasive stone (pattern number A 122, Amalgamated Dental (Australia) Pty. Ltd.) to ensure a suitable bonding surface for the acrylic resin.
The prepared brackets were then cemented to the teeth, using the enamel preparation and pretreatment already described and one of the following four different methods of bonding:

1. The Orthomito system, used with silane pretreatment.

2. The Orthomito system, used without silane pretreatment.

3. The Suvrilon system, used with Suvrilon sealer.

4. The Suvrilon system, used without Suvrilon sealer.

It was hoped by this method to make a comparison between these four systems, particularly with regard to the use or exclusion of the silane coupling agent.

Due to a shortage of supplies of suitable teeth for testing of bonds, it was decided to re-use some already used in preliminary work. It was thought that this would expand the scope of the results in helping to interpret comparative performances of the systems being tested. It was also seen that tests done on previously bonded enamel might provide an indication of what to expect in the clinical situation where a bracket had been
displaced and required re-positioning.

A sample of fifty six teeth to which no cement had been applied was available for testing and this was matched with another fifty six teeth which had previously been used for the testing of bonds and following bracket removal had been re-prepared by scaling and polishing, as described before. An overall sample of one hundred and twelve teeth was tested.

The two fractions, each of fifty six teeth, comprising the overall sample of one hundred and twelve teeth, are henceforth to be referred to as initial bonds and repeat bonds, where initial bonds represent those fifty six teeth having brackets cemented for the first time and repeat bonds represent those fifty six teeth having brackets bonded to a surface which has been re-prepared, as described, following removal of a previous bond.

Fourteen teeth were used in each of the eight tests and of these fourteen teeth, seven were tested for shear strength and seven for tensile strength.
CHAPTER 5

RESULTS

The results of the experiments which comprise the tests proper are shown in Tables 9 to 24.

All tests were conducted on the Tensometer at a strain rate of 0.8 mm/min.

The ruptured bond sites examined with the Zeiss stereoscopic microscope at a magnification of X16 with oblique illumination have been grouped into three categories according to predominant surface appearance:

Matt — Dull and lustreless with a fine granular appearance. There is no comment to made in the results on ruptured surfaces the appearance is matt.

Granular — Coarse granular somewhat frosted or crystalline appearance.

Glazey — Glossy undulating featureless appearance.
TABLE 9

Test A with initial bond* using Orthomite with silane

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>60</td>
<td>Enamel interface failure, small flash.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>64</td>
<td>Enamel interface failure, moderate flash.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>58</td>
<td>Enamel interface failure, rare flash.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>36</td>
<td>Enamel interface failure - little flash - smooth chew on enamel.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>51</td>
<td>Enamel interface failure - small flash - crystalline enamel surface appearance.</td>
</tr>
</tbody>
</table>

In the above tests, all bond failures occurred wholly at the enamel interface. The appearance of the enamel following bond failure from one of the lesser loads was smooth and shiny and from two of the greater loads more crystalline. The remainder presented a matt appearance. Varying amounts of cement flash left adhering to the enamel did not appear significant.

* Tooth samples being bonded for the first time.
**TABLE 10**

Test B with initial bonds using Orthomite without silane

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Shear</td>
<td>76</td>
<td>Enamel interface failure - similar appearance to silane treated tooth surface.</td>
</tr>
<tr>
<td>2</td>
<td>Shear</td>
<td>91</td>
<td>Enamel interface failure - quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>3</td>
<td>Shear</td>
<td>62</td>
<td>Enamel interface failure - quarter of bracket base adhering to enamel, moderate flash.</td>
</tr>
<tr>
<td>4</td>
<td>Shear</td>
<td>64</td>
<td>Enamel interface failure - moderate flash.</td>
</tr>
<tr>
<td>5</td>
<td>Shear</td>
<td>20</td>
<td>Enamel interface failure - large flash, tooth surface very smooth looking.</td>
</tr>
<tr>
<td>6</td>
<td>Shear</td>
<td>133</td>
<td>Enamel interface failure - quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>7</td>
<td>Shear</td>
<td>127</td>
<td>Enamel interface failure - moderate flash.</td>
</tr>
</tbody>
</table>

In the above tests four bond failures occurred wholly at the enamel interface and three involved some degree of cohesive failure in the bracket base. The appearance of the enamel following bond failure from the lowest load was smooth and shiny and from the remainder matt and similar to the silane treated tooth surfaces. Varying amounts of cement flash left adhering to the enamel did not appear significant.
TABLE II
Test C with initial bonds using Sovriton with Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>95</td>
<td>Bracket base/cement interface failure. All flash left on tooth.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>104</td>
<td>Bracket base/cement interface failure. All flash left on tooth.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>149</td>
<td>Enamel interface failure — quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>89</td>
<td>Failed half in adhesive bond and half at enamel interface.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>95</td>
<td>Failed partly in adhesive bond, partly at enamel interface, partly at bracket base/cement interface.</td>
</tr>
</tbody>
</table>

The diversity of failure sites cannot be summarized so as to demonstrate any consistent tendency.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>69</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>111</td>
<td>Bracket base/cement interface failure.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>113</td>
<td>Enamel interface failure—flash partly on tooth, partly on bracket.</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>133</td>
<td>Enamel interface failure, flash partly on tooth, partly on bracket.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>98</td>
<td>Bracket base/cement interface failure.</td>
</tr>
<tr>
<td>6.</td>
<td>Shear</td>
<td>120</td>
<td>Enamel interface failure with quarter of bracket adhering to enamel.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>109</td>
<td>Enamel interface failure.</td>
</tr>
</tbody>
</table>

In the above tests, five bond failures occurred at the enamel interface, with one of them being partly cohesive failure in the bracket base. The remaining two were adhesive failures at the bracket base/cement interface.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>49</td>
<td>Enamel interface failure – moderate flash, smoother enamel surface appearance.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>62</td>
<td>Enamel interface failure – moderate flash, some left on tooth, smooth enamel surface.</td>
</tr>
</tbody>
</table>

In the above tests all bond failures occurred at the enamel interface, with one involving some cohesive failure of the bracket base. The microscopic appearance of the enamel was not consistent with previous failure trends.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>40</td>
<td>Enamel interface failure, with large glassy area on cement.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>80</td>
<td>Enamel interface failure—moderate flash and matt appearance of enamel.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>15</td>
<td>Enamel interface failure—large flash, enamel matt appearance, but cement on bracket glassy in appearance as against more usual dull appearance.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>100</td>
<td>Failure in acrylic block on bracket.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>40</td>
<td>Enamel interface failure—both cement face and enamel look glassy.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>85</td>
<td>Enamel interface failure—small corner of bracket adhering to enamel.</td>
</tr>
</tbody>
</table>

In the above tests, five bond failures occurred wholly at the enamel interface, one other involved a small degree of cohesive failure of the bracket base as well as adhesive failure at the enamel interface and with the remaining test there was cohesive failure of the acrylic block on the bracket. With the three lowest readings, large areas of the cement face appeared glassy.
### TABLE 15

Test 6 with initial bonds using Sovriton with Sealor

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tensile</td>
<td>120</td>
<td>Failed mainly in adhesive and quarter in bracket base.</td>
</tr>
<tr>
<td>2</td>
<td>Tensile</td>
<td>109</td>
<td>Failed mainly at bracket base/cement interface, quarter bracket base adhering to enamel.</td>
</tr>
<tr>
<td>3</td>
<td>Tensile</td>
<td>267</td>
<td>Tensometer was stopped - bond etc., still intact.</td>
</tr>
<tr>
<td>4</td>
<td>Tensile</td>
<td>127</td>
<td>Failure at bracket base/cement interface.</td>
</tr>
<tr>
<td>5</td>
<td>Tensile</td>
<td>62</td>
<td>Failure in acrylic block.</td>
</tr>
<tr>
<td>6</td>
<td>Tensile</td>
<td>100</td>
<td>Failure at bracket base/cement interface.</td>
</tr>
<tr>
<td>7</td>
<td>Tensile</td>
<td>158</td>
<td>Failure at bracket base/cement interface and half bracket base adhering to enamel.</td>
</tr>
</tbody>
</table>

The diversity of failure sites cannot be summarized so as to demonstrate any consistent tendency. The stopping of the tensometer at a load of 267 N in one test was to avoid damage to the jig holding the teeth and bracket.
TABLE 16

Test H with initial bonds using Sevrotan without Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>93</td>
<td>Enamel interface and half bracket base failure.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>104</td>
<td>Enamel interface failure, quarter bracket base adhering to enamel.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>62</td>
<td>Failure in acrylic block on bracket.</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile</td>
<td>140</td>
<td>Enamel interface failure, quarter bracket base adhering to enamel.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>60</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>165</td>
<td>Failure in acrylic block.</td>
</tr>
</tbody>
</table>

In the above tests, three bond failures involved both the enamel interface and part of the bracket base, two others involved partial failure in the adhesive and the remaining two failed in the acrylic block.
TABLE 17

Test I with repeat bonds* using Orthomite with silane

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Shear</td>
<td>169</td>
<td>Bracket failure and quarter adhesive failure at enamel interface.</td>
</tr>
<tr>
<td>2</td>
<td>Shear</td>
<td>149</td>
<td>Bracket failure only.</td>
</tr>
<tr>
<td>3</td>
<td>Shear</td>
<td>109</td>
<td>Enamel interface failure with some fracture of enamel at enamel interface.</td>
</tr>
<tr>
<td>4</td>
<td>Shear</td>
<td>165</td>
<td>Enamel interface failure, quarter of bracket adhering to enamel.</td>
</tr>
<tr>
<td>5</td>
<td>Shear</td>
<td>73</td>
<td>Enamel interface failure, quarter of bracket adhering to enamel.</td>
</tr>
<tr>
<td>6</td>
<td>Shear</td>
<td>9.75</td>
<td>Enamel interface failure - small void in cement.</td>
</tr>
<tr>
<td>7</td>
<td>Shear</td>
<td>142</td>
<td>Enamel interface failure, quarter of bracket adhering to enamel.</td>
</tr>
</tbody>
</table>

In the above tests, four bond failures occurred at the enamel interface, three of these involved some cohesive failure in the bracket base. One failure was a completely cohesive failure in the bracket, another was partly cohesive and partly adhesive. The remaining one was at the enamel interface but also involved some cohesive failure of the enamel itself.

* Teeth replicas being re-used for a second bond after removal of an initial one.
TABLE 18
Test J with repeat bonds using Orthomize without Silane

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>85</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>129</td>
<td>Enamel interface failure, with some enamel surface fracture.</td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>120</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
<td></td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>92</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>104</td>
<td>Failure of acrylic block on bracket.</td>
<td></td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>147</td>
<td>Enamel interface failure with some enamel surface fracture - bracket base cement dull crystalline appearance.</td>
<td></td>
</tr>
</tbody>
</table>

In the above tests, three bond failures occurred partly at the enamel interface and partly within the bracket base; two others involved some cohesive failure of the enamel along with failure at the enamel interface; one failure occurred within the acrylic block and the remaining one wholly at the enamel interface.
TABLE 19
Test K with repeat bonds using Sovriton with Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>178</td>
<td>Failure at bracket base/cement interface.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>111</td>
<td>Failure at bracket base/cement interface and quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>3.</td>
<td>Shear</td>
<td>113</td>
<td>Failure at bracket base/cement interface and quarter at enamel interface.</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>138</td>
<td>Failure in acrylic block.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>69</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>6.</td>
<td>Shear</td>
<td>95</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>67</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
</tr>
</tbody>
</table>

The diversity of failure sites cannot be summarized so as to demonstrate any consistent tendency.
TABLE 20

Test L with repeat bonds using Sauriton without Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Shear</td>
<td>33</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>2.</td>
<td>Shear</td>
<td>69</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>4.</td>
<td>Shear</td>
<td>151</td>
<td>Enamel interface failure and partly in adhesive.</td>
</tr>
<tr>
<td>5.</td>
<td>Shear</td>
<td>98</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>7.</td>
<td>Shear</td>
<td>100</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
</tr>
</tbody>
</table>

In the above tests four bond failures occurred wholly at the enamel interface, one involved the enamel interface and a small part of the bracket base and two involved the enamel interface and the adhesive.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>96</td>
<td>Enamel interface failure — quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>176</td>
<td>Enamel interface failure — quarter of bracket base adhering to enamel and some enamel fracture.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>98</td>
<td>Failure in acrylic block on bracket.</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile</td>
<td>176</td>
<td>Enamel interface failure with quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>69</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>178</td>
<td>Enamel interface failure with quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>122</td>
<td>Enamel interface failure with quarter of bracket base adhering to enamel.</td>
</tr>
</tbody>
</table>

In the above tests, five bond failures involved both the enamel interface and part of the bracket base; one other failed wholly at the enamel interface and the remaining one failed in the acrylic block on the bracket.
<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tensile</td>
<td>51</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel. Glassy cement appearance.</td>
</tr>
<tr>
<td>2</td>
<td>Tensile</td>
<td>87</td>
<td>Enamel interface failure and half bracket base adhering to enamel.</td>
</tr>
<tr>
<td>3</td>
<td>Tensile</td>
<td>122</td>
<td>Enamel interface failure, quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>4</td>
<td>Tensile</td>
<td>171</td>
<td>Failure in acrylic block on bracket.</td>
</tr>
<tr>
<td>5</td>
<td>Tensile</td>
<td>91</td>
<td>Enamel interface failure – quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>6</td>
<td>Tensile</td>
<td>151</td>
<td>Enamel interface failure and half bracket base adhering to enamel.</td>
</tr>
<tr>
<td>7</td>
<td>Tensile</td>
<td>73</td>
<td>Failure of acrylic block on bracket.</td>
</tr>
</tbody>
</table>

In the above tests, five bond failures involved both the enamel interface and part of the bracket base and two failures occurred in the acrylic block on the bracket.
### Table 23

Test 0 with repeat bonds using Sovriton with Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>122</td>
<td>Bracket base/cement interface and quarter of bracket base adhering to enamel.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>167</td>
<td>Tooth fractured through jig hole.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>93</td>
<td>Failure in cement - rather thick bond.</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile</td>
<td>216</td>
<td>Failure in bracket base.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>93</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>200</td>
<td>Failure in bracket base.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>44</td>
<td>Failure in bracket base/cement interface.</td>
</tr>
</tbody>
</table>

In the above tests, there was a wide diversity of failure sites, but two occurred in the bracket base.
TABLE 24

Test P with repeat bonds using Sovrilon without Sealer

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Type of Loading</th>
<th>Load at Failure (N)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Tensile</td>
<td>98</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>2.</td>
<td>Tensile</td>
<td>53</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile</td>
<td>61</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile</td>
<td>89</td>
<td>Enamel interface failure.</td>
</tr>
<tr>
<td>5.</td>
<td>Tensile</td>
<td>107</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>6.</td>
<td>Tensile</td>
<td>60</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
<tr>
<td>7.</td>
<td>Tensile</td>
<td>116</td>
<td>Enamel interface failure and quarter in adhesive.</td>
</tr>
</tbody>
</table>

In the above tests, five bond failures occurred partly at the enamel interface and partly within the cement (cohesive) and two occurred wholly at the enamel interface.
## COMPARISON OF BONDING SYSTEMS

### TABLE 25

Mean values for tests with initial bonds

<table>
<thead>
<tr>
<th>Test</th>
<th>Bond System</th>
<th>Type of Loading</th>
<th>Mean Load 7 Samples (N)</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Orthomite with silane</td>
<td>Shear</td>
<td>54</td>
<td>13</td>
</tr>
<tr>
<td>B</td>
<td>Orthomite without silane</td>
<td>Shear</td>
<td>82</td>
<td>40</td>
</tr>
<tr>
<td>C</td>
<td>Sevriton with sealer</td>
<td>Shear</td>
<td>112</td>
<td>28</td>
</tr>
<tr>
<td>D</td>
<td>Sevriton without sealer</td>
<td>Shear</td>
<td>103</td>
<td>20</td>
</tr>
<tr>
<td>E</td>
<td>Orthomite with silane</td>
<td>Tensile</td>
<td>53</td>
<td>32</td>
</tr>
<tr>
<td>F</td>
<td>Orthomite without silane</td>
<td>Tensile</td>
<td>61</td>
<td>30</td>
</tr>
<tr>
<td>G</td>
<td>Sevriton with sealer</td>
<td>Tensile</td>
<td>153</td>
<td>65</td>
</tr>
<tr>
<td>H</td>
<td>Sevriton without sealer</td>
<td>Tensile</td>
<td>97</td>
<td>43</td>
</tr>
<tr>
<td>Test</td>
<td>Bond System</td>
<td>Type of Loading</td>
<td>Mean Load 7 Samples</td>
<td>Standard Deviation</td>
</tr>
<tr>
<td>------</td>
<td>----------------------</td>
<td>-----------------</td>
<td>---------------------</td>
<td>--------------------</td>
</tr>
<tr>
<td>I</td>
<td>Orthomite with silane</td>
<td>Shear</td>
<td>126</td>
<td>40</td>
</tr>
<tr>
<td>J</td>
<td>Orthomite without silane</td>
<td>Shear</td>
<td>106</td>
<td>27</td>
</tr>
<tr>
<td>K</td>
<td>Sevriton with sealer</td>
<td>Shear</td>
<td>110</td>
<td>39</td>
</tr>
<tr>
<td>L</td>
<td>Sevriton without sealer</td>
<td>Shear</td>
<td>78</td>
<td>42</td>
</tr>
<tr>
<td>M</td>
<td>Orthomite with silane</td>
<td>Tensile</td>
<td>131</td>
<td>46</td>
</tr>
<tr>
<td>N</td>
<td>Orthomite without silane</td>
<td>Tensile</td>
<td>107</td>
<td>43</td>
</tr>
<tr>
<td>O</td>
<td>Sevriton with sealer</td>
<td>Tensile</td>
<td>136</td>
<td>63</td>
</tr>
<tr>
<td>P</td>
<td>Sevriton without sealer</td>
<td>Tensile</td>
<td>82</td>
<td>27</td>
</tr>
</tbody>
</table>
STATISTICAL ANALYSIS

Analysis based on a "t" test, shows that the results are not statistically significant for the number of samples used. However, by ignoring surface pretreatments with sealer or silane and combining the samples of seven into groups of fourteen, certain differences became significant. The results are shown below in Table 27.

<table>
<thead>
<tr>
<th>Bond System</th>
<th>Type of Loading</th>
<th>Mean Load of 14 Samples ± N</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Initial Bonds</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Orthomite</td>
<td>Tensile</td>
<td>57 ± 44</td>
<td>Significant at 95% level of confidence.</td>
</tr>
<tr>
<td>Seviriton</td>
<td>Tensile</td>
<td>116 ± 77</td>
<td></td>
</tr>
<tr>
<td>Orthomite</td>
<td>Shear</td>
<td>68 ± 42</td>
<td>Significant at 95% level of confidence.</td>
</tr>
<tr>
<td>Seviriton</td>
<td>Shear</td>
<td>110 ± 35</td>
<td></td>
</tr>
<tr>
<td><strong>Repeat Bonds</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Orthomite</td>
<td>Tensile</td>
<td>118 ± 64</td>
<td>Not significant at 95% level of confidence.</td>
</tr>
<tr>
<td>Seviriton</td>
<td>Tensile</td>
<td>109 ± 68</td>
<td></td>
</tr>
<tr>
<td>Orthomite</td>
<td>Shear</td>
<td>116 ± 49</td>
<td>Not significant at 95% level of confidence.</td>
</tr>
<tr>
<td>Seviriton</td>
<td>Shear</td>
<td>94 ± 58</td>
<td></td>
</tr>
</tbody>
</table>
Sites of Bond Failures

The comments on bond failure patterns have been examined and expressed as units (or part thereof) in various categories and tabulated below. From this table a percentage distribution of failure sites and ruptured surface appearances has been made and commented on in Chapter 6.

### Table 28
Bond Failure Patterns

<table>
<thead>
<tr>
<th>Test</th>
<th>Failure Sites</th>
<th>Enamel/Cement Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Enamel Bracket</td>
<td>Bracket Base</td>
</tr>
<tr>
<td>A</td>
<td>7.0 - - -</td>
<td>- - -</td>
</tr>
<tr>
<td>B</td>
<td>6.25 - - -</td>
<td>0.75 - -</td>
</tr>
<tr>
<td>C</td>
<td>2.0 2.75 -</td>
<td>0.25 -</td>
</tr>
<tr>
<td>D</td>
<td>4.75 2.0 -</td>
<td>0.25 -</td>
</tr>
<tr>
<td>E</td>
<td>6.75 - - -</td>
<td>0.25 -</td>
</tr>
<tr>
<td>F</td>
<td>6.0 - - -</td>
<td>- - -</td>
</tr>
<tr>
<td>G</td>
<td>- 3.25 1.0</td>
<td>0.75 -</td>
</tr>
<tr>
<td>H</td>
<td>2.75 0.75 1.0</td>
<td>0.5</td>
</tr>
<tr>
<td>I</td>
<td>4.5 - - -</td>
<td>2.5 -</td>
</tr>
<tr>
<td>J</td>
<td>5.25 - - -</td>
<td>0.75 -</td>
</tr>
<tr>
<td>K</td>
<td>3.25 2.25 -</td>
<td>0.5 -</td>
</tr>
<tr>
<td>L</td>
<td>6.25 - - -</td>
<td>0.25 0.5</td>
</tr>
<tr>
<td>M</td>
<td>4.75 - - -</td>
<td>1.25 -</td>
</tr>
<tr>
<td>N</td>
<td>3.25 1.75 -</td>
<td>1.25 -</td>
</tr>
<tr>
<td>O</td>
<td>1.0 1.75 -</td>
<td>2.25 1.0</td>
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<tr>
<td>P</td>
<td>5.75 - - -</td>
<td>- 1.25</td>
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<td>Total</td>
<td>69.5 12.75 12.75</td>
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| 69\% | 12.5\% | 12.5\% | 6\% | 12\% | 80\% | 8\% |
CHAPTER 6

DISCUSSION OF RESULTS

债券失效模式

分析微观观察结果显示，用Zeiss立体显微镜在16X下观察到以下结果:

69%的失效发生在牙釉质界面。

12.5%的失效发生在支座基底/胶粘剂界面。

12.5%的失效发生在支座基底内。

5%的失效发生在胶粘剂内。

对斜照照明的显微镜的使用似乎很合适。通过改变光源的倾斜度和方向，可以获得不同强度和对比度的视野。这有助于澄清失效的部位。然而，损坏的表面纹理被观察到时，它可能引入了一个变量。
(a) The enamel interface

Adhesive failure at the enamel interface occurred in an estimated 69% of the tests. An inspection of the surfaces involved showed:

80% of surfaces showed a "matt" appearance.
12% of surfaces showed a "glassy" appearance.
8% of surfaces showed a "granular" appearance.

The "glassy" appearance of the enamel and cement surfaces was consistent with bond failures at low loads. All occurred with Orthomite, both with and without silane.

The "granular" appearance of the enamel and cement surfaces showed only with Orthomite in conjunction with silane. However, such a small sample may not justify a suggestion of any pattern.

The "matt" appearance of the enamel and cement surfaces represents the more usual one and showed in fifteen of the sixteen series of tests. In this series of fifteen tests the wide scatter of the results was such that no pattern emerged.
(b) The bracket base interface

Adhesive failure at the bracket base interface occurred in an estimated 12.5% of the tests and all occurred with Sevriton, both with and without sealer.

Cohesive failures in the bracket base (estimated at 12.5%) do not bear on the results of the experiment. However, it has been noted that they are of a higher order than cohesive failures in the adhesive (6%). This seems to suggest the relative strengths of the two materials are fairly closely matched.

(e) The cement

Cohesive failures in the adhesives are of a relatively low order, at an estimated 6%, even allowing for a wide margin of error in assessment. The results suggest a pattern, in that all the cohesive failures in the cements occurred with Sevriton, both with and without sealer.

*Working Time of Adhesives*

A comment on the working time of the adhesives seems appropriate to a discussion of the test results.

The average working time for both Orthomite and Sevriton
was eleven seconds. This included four seconds for mixing and seven seconds for loading and seating of the bracket. The high speed of set would mean that it would be doubtful that all brackets were being seated with the adhesive at precisely the same consistency. In the experimental bonding procedures the consistency was judged visually and was confirmed to some degree by tactile sense at the time of seating. This was deemed a more satisfactory method of maintaining a uniform mix than a "bead and brush" technique.

However, such a short working time would seem most likely to introduce variation in consistency, particularly when it is considered how rapidly the setting is accelerated once the adhesive is in contact with the polycarbonate of the bracket base.

In a recent study of the working time of polymeric restorative materials, Jacobsen (90) made the following remark about an unfilled acrylic resin: "There was a significant deterioration in adaptation during the working times which suggests that small changes in viscosity early in the setting reactions radically affect the materials mobility".

This factor of small changes in viscosity of the mixed resin
would seem likely to have a marked effect on degree of penetration of the adhesive into the micropores of the enamel. It could have contributed to the wide scatter in bond strengths recorded in the present study.

However, it has recently been shown that viscosity in resin mixes is probably not a governing factor in penetration of enamel pores.\(^{(95)}\) A fact which the authors themselves note may appear surprising. However, Huntsberger\(^{(85)}\) says that the rate of wetting is important in adhesion and that it is inversely proportionate to the viscosity of the liquid. He also claims it is essential to distinguish between "spreading" and "wetting". He adds that even though an adhesive may have a zero contact angle, if there is insufficient time for setting during bonding then adhesion will be poor. A short working time would seem very relevant to the foregoing remarks and would serve to emphasise the competent handling required of self-curing resin adhesives in a clinical situation. It also illuminates the advantages of UWL initiated resins.

**THE ROLE OF SILANE**

With initial bond tests, both in tension and shear, the results indicate that the use of silane with the Orthomite system may have improved the bond strength, but not to a significant degree.
From what is known of the chemistry of coupling agents, it would be recognized that in bonding plastic to glass, silane would improve the bond. Lee (105) says that not only is the bond improved but that strength retention time has been shown to increase 50 fold by the use of silane in glass/resin bonding. He says that several silanes have enhanced adhesion to tooth structure and his postulated mechanism for such a reaction has been reproduced elsewhere in this thesis (see Fig. 1). However, the chemical nature of the silane would appear unlikely to confer any advantage on a bond between enamel and plastic if the nature of the enamel surface is considered as a biological entity and not merely as a chemical complex.

If silane could enhance the bonding of Orthomite to tooth enamel, then it would seem reasonable that such an improvement in bond strength could be demonstrated with an enamel surface which had been only mechanically cleaned. The recommended acid etching prior to use of silane can only be viewed as improving adhesion by means of mechanical interlocking in the micropores and by providing a larger bonding surface. If the silane coupling agent had promoted true adhesion then not only should it have been evident in significantly superior bond strengths, but also in the bond failure patterns: cohesive failures in either
the enamel substrate or the adhesive would have predominated.
No significant failures occurred in the enamel substrate and
cohesive failure in the adhesives was of a low order (6%).

With repeat bond tests, both in tensile and shear, the
results indicated that the use of silane with the Orthomite
system improved the bond strength, but not to a significant degree.

It appears that there could be some relationship between
the results and the differing samples represented by the two
groups. However, if a relationship exists it cannot be explained.

THE ROLE OF SEVRITON SEALER

In an evaluation for significance, the effect of Sevrito
sealer in tests 0 and 4 was almost significant. An examination
of the results for tests K and L shows a considerable gain in
mean bond strength for the use of sealer, though not to a
significant degree. No such gains are evident with the use
of sealer with initial bonds.

A NOTE ON AN UNUSUAL COUPLING REACTION

Mention was made in the section on enamel pretreatment of
the role of fluoride and absence of resinous "tags" (36)(65)(72)(173) in acid etched enamel which had previously applied topical fluoride. The apparent coupling action associated with the acidulated sodium fluoride (173) is worthy of another mention. Such a chance observation is of the sort that should assist in building up a fuller picture of the significance of coupling to tooth surfaces. Skinner (176) has also noted that the classical concept of fluoride displacing the hydroxyl group in hydroxapatite to form fluorapatite did not hold for topically applied fluoride; the whereabouts of the fluoride ion in the enamel structure was not known and that further study of this phenomenon might provide knowledge concerning surface adhesion. Blantz (63) says that hydrogen bonds should form only between hydrogen and the most electronegative atoms such as fluorine. He says that empirically it has been found that fluorine forms very strong hydrogen bonds.

**Initial and repeat bonds**

Although analysis of the results based on a "t" test does not show a significant difference at the 95% level of confidence between initial and repeat bonds, comparison of the

* See p. 105.*
results shows that the Orthomite system indicated quite substantial gains in bond strength with repeat bonds over initial bonds. These increases were apparent in both shear and tensile and both with and without silane. The results do not indicate any such improvement in bond strengths for Sevriton. Brauer and Termini(28) mention obtaining increased adhesion of resins with repeat bondings to teeth.

If the significance of this result lies in the fact that it occurred as a result of the teeth having been previously bonded, then a discussion of the re-preparation of the surface would seem appropriate.

After bond fracture, any plastic visible on the enamel surface was removed by a scaler and the surface was then polished with pumice and water paste and a rubber polishing cup. Chloroform was not used. It is conceivable that the microscopically roughened surface produced by the original acid etching, and presumably filled by the original cement bond, could still be present. However, the discussion in the section on enamel recovery would seem to indicate that the enamel should have returned to its original state. The degree of pumicing would obviously be an important factor. It seems unlikely that a
second acid etching in preparation for re-bonding could augment the first acid etching, for either the entire acid-etched surface would have been polished away, or whatever remained would be filled with cement.

If, as it appears from the results, the enamel has improved as a substrate for bonding due to the original bonding and subsequent re-preparation, then the assumption must be that a change has taken place; moreover, that this change in the enamel surface affects improved bonds with Orthomite but not with Sevriton and that the use of silane might improve bond strength slightly under the conditions obtaining for repeat bonds.

Bowen (17) reported a parallel finding with reapplication of NPG-GMA without resurfacing the dentine specimens between tests. He gained successively increasing bond strengths which dropped significantly when the dentine specimens were reprepared with a new bonding surface. Bowen wondered if the increase was due to incremental removal of a fractured surface layer of dentine, or to an increase in the population density of NPG-GMA molecules on the substrate surface or other unspecified reasons.
Watson (188), reporting on the photomicrographs of enamel and cement surfaces after bond failure, mentions some cement surfaces as having the appearance of a complete negative of the enamel surface, which would indicate that no cement would have remained within the etched surface of the enamel. Some enamel surfaces are noted as having the cement sheared off "very close to the level of the original enamel surface and never along the base plane of the etched porosities". Considerable amounts of adhesive were observed remaining in the porosities and patches of adhesive seen on the enamel surface after completion of removal and polishing procedures. (188)

If changes such as these have improved bond strength, no explanation can be offered as to why it should be so.
CHAPTER 7

CONCLUSIONS

EVALUATION FOR SIGNIFICANCE

With initial bonds, there was a significant difference at the 95% level of confidence in both tensile and shear between the Orthomite and Sevriton systems. The bonds with Sevriton were superior to those of Orthomite.

With all systems the use of silane or sealer showed no significance in improving the bond at the 95% level of confidence. The tests on Sevriton bonds in tensile stress on repeat bonds (tests O and P) were almost significant for the use of sealer.

THE ENAMEL SURFACE

The empirical nature of assessment of a suitable bonding surface is most likely to account for the wide range of values found in load tests. The reports examined in the section on Enamel Pretreatment stress the heterogeneity of enamel surfaces.
within the same mouths, on different surfaces of each tooth and
in different areas of the same tooth surface. Even greater
differences would be expected to be met when teeth from different
mouths were being assessed.

Add to this the individual uptake of fluoride, which has
such an inhibiting effect on bonding, and something of the
unpredictable nature of the enamel surface as a substrate may
be grasped.

**BOND THICKNESS**

An examination of the failure strains in which part or whole
cohesive failure of the Sevriton cement occurred indicates no
consistent pattern.

Bond thickness is probably significant in the light of
Rensch's (158) findings. He was using Sevriton with a special
metal bracket base, undercut for retention and mentions that
his adhesive layer was bulky. (The area of bonding was not
indicated). His in vitro tests were conducted in tension and
gave average failure strains of 62.8 N. There were no adhesive
failures at the interface, they were all cohesive failures within the adhesive. He concludes that a more cohesive cement would produce superior bonds.

The present author's results showed a low incidence of cohesive failures in the adhesive (6.0%) and of these, the nine readings that involved such failures in tensile stress gave an average failure strain of 40 %.

**SILANE COUPLER**

It must be concluded that the value of silane in improving the bond strength between Orthomite and enamel is doubtful.

Analysis based on a "t" test showed it not to be significant at the 95% level of confidence.

**QUANTITATIVE RESULTS**

In order to relate directly the quantitative results of the tests to the clinical requirements of such a system, an examination of the literature on studies involving the application of a load to a tooth through the medium of an
orthodontic band and attachment was made.

Such studies involved the use of arch wires (both passive and activated), torquing auxiliaries and elastic band pull. Varying types of loading were employed.

Reitan (148) suggested low initial forces, 0.245 N in adults and 0.392 N in young patients, increasing the force later to obtain further continuous tipping of the teeth. He found that a force of 1.275 N allowed a torquing movement to proceed without any hyalinisation.

Jarabak and Fizzell (91) in an evaluation of the applications of mechanics to orthodontic force systems, discussed the activation of arch wires in which both shear and tensile loads were applied to brackets, and the pull of elastics used intraorally. The initial forces employed did not exceed 4.90 N and were more generally in the range of 1.471 - 2.452 N. 

Fine (55) in a discussion of the Begg light wire technique recommended a force of 0.539 N for elastic band pull, uprighting springs, intrusive force and torque.
Storay and Smith (161) in an in-vivo study of distal movement of a cuspip tooth employing springs with fixed appliances, assumed 4.448 N to be a large force. Heavy and light springs were used and the greatest initial forces employed were 3.923 - 5.884 N.

In this range of force there was no movement of the cuspip tooth. The anchor unit moved until the range 1.951 - 2.942 N was reached and then the cuspip commenced to move rapidly and the anchor unit ceased to move.

In his experimental study of the mechanical properties of Begg spur torquing arches, Scully (108) measured the resultant forces produced 15mm apically from the bracket slot. They rarely exceeded 0.588 N. However, the tensile loading of the bracket as a result of activation of the spur torquing auxiliary would be very difficult to calculate when it is appreciated that much of the resultant force is dissipated via the main archwire and its bend around the distal end of the molar tube.

In any of the preceding studies the greatest initial force employed was 5.884 N by Storay and Smith (161).
In the procot study the lowest load failures for a sample of 14 were produced with a mean load of 57 N ± 44. The lowest single reading in this group was 13 N which is still more than double the greatest force employed in the studies under review.

**DEGREE OF ADHESION REQUIRED**

It often seems to be assumed in the literature that a true adhesive bond is what is needed in fixed banded orthodontics. It may not be known just how much adhesion is necessary or acceptable until that point has been exceeded. However, anyone with clinical experience of the removal of resin cement bonds would agree with the author in stating that the strongest of the bonds encountered would prove embarrassing if they were more retentive.

Craig(18) referred to the fact that after decades of research into dental adhesives, the profession seemed to have reached an impasse. No truly adhesive permanent dental restorative has been forthcoming. The adhesives that have been used successfully are of a temporary nature only. Brauer(26) concludes that evidence exists which suggests that continuing progress will lead to
clinically acceptable adhesive restoratives that will substantially improve the quality of dental services.

It is the author's opinion that Orthodontics is fortunate in that developments in dental adhesives appear likely to satisfy the requirement for direct bonding of attachments to enamel for orthodontic therapy.

Although the 'in vitro' study in this thesis has not shown that the two coupling agents tested have significantly enhanced adhesion of the two resin systems used, at the same time some of the results are encouraging. The gains in bond strength with the Orthomite system with repeat bondings cannot be explained, but if such a phenomenon were further investigated it might show the way to achieving a degree of adhesion more acceptable for a permanent tooth restorative.

**Recommendations Concerning Further Tests**

As a result of this study the following list of suggestions for areas of further investigation is made:

1. Clinical trials of direct bonding system.


5. Corroboration of Ronsch's finding that with Sevriton and Duralon water absorption is not the major cause of bond weakening. He found that average bond strength after six months in a water bath was 52 N, compared to an initial bond strength of 55 N. If Ronsch's findings are correct, then storage in water during an in vitro experiment for the two cements studied is unnecessary and as a result rapid screening of many techniques would be possible.

6. Consistent reports of polycarbonate bracket failures in uprighting procedures in clinical
practice suggest the need for investigation of bracket materials and modifications in design.

7. Investigation of other uses of bonding, such as:
   a. Retention following active therapy by interproximal bonding. This would afford a more functional form of retention than with a removable appliance.

   b. Provisional splinting in periodontal therapy.

8. Trials for true adhesiveness could be made utilising the featureless areas of prismless enamel, (68)(162) making particular use of deciduous teeth, in both clinical and laboratory studies. It would then be possible to distinguish between mechanical interlocking of adhesive in enamel pores and possible true adhesion to a surface that could be acid etched to produce a reactive layer without creating retentive pores.
CHAPTER 8

SUMMARY

Two systems of direct bonding of orthodontic attachments to enamel were investigated in an in vitro study. Sevriton and Orthomite were the systems used, with clear plastic brackets.

The bonds were formed both with and without recommended intermediate coupling agents and tested for both shear and tensile strength, following evolution of a suitable testing technique.

It was shown that the Sevriton bonds were superior to those of Orthomite and that the coupling agents failed to significantly improve adhesion with either system.

There is reasonable evidence to indicate that the degree of adhesion achieved in the present study should well satisfy the orthodontist's requirement.


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