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A CLINICAL AND IN VITRO INVESTIGATION
OF GLASS Ionomer CEMENT AS AN
ADHESIVE FOR DIRECT ORTHODONTIC BONDING

KENNETH G. MARSHALL, B.D.S. (SYD. UNI.)

A THESIS SUBMITTED IN PARTIAL REQUIREMENT
FOR THE DEGREE OF MASTER OF DENTAL SCIENCE

Department of Preventive Dentistry
Faculty of Dentistry
University of Sydney
Australia
1984
DEDICATION

This thesis is dedicated to my wife, Robyn, who has remained tranquil, supportive and loving through the challenges of the last three years:

To our recently arrived son, Scott, who has added a third dimension to the canvas of life:

And to my mother, Jean Marshall-Coates, BA (Hons), MACE, whose sacrifices and motivation made my early education possible.
ACKNOWLEDGEMENTS

I would like to express my thanks and appreciation to a number of people without whose help, advice and encouragement this project would not have eventuated, or been completed.

My initial interest in glass ionomer cements was stimulated by Keith Powell, Senior Lecturer, Preventive Department, University of Sydney. Further friendly and fruitful discussions with Dr Graham Mount of Adelaide helped to establish a framework upon which this thesis could be commenced.

A substantial portion of my gratitude is extended to Dr Derrick Beech, Director of Australian Dental Standards Laboratory, who allowed me to use the extensive facilities at the ADSL to complete the experimental component of the project. Dr Beech and a considerable number of staff at ADSL provided expertise and support that was broadly based, generously supplied and of a most professionally excellent standard.

Michael Goldman greatly helped with the statistical analysis of the data using the ADSL computer facilities. Photography for all aspects of the project was also provided by the ADSL.

Thanks are also due to my mother, Jean Marshall-Coates, who handled the tedium of proof reading with persistence and good humour; my typist, Pamela Gibbs, whose skill and dexterity crafted the final manuscript; my colleague, Bob Chapman, whose support, comradeship, wit and humorous disposition helped temper the stresses of post-graduate study.

Finally, I would like to thank Professor Keith Godfrey for supervising the overall project and providing constructive advice to bring the thesis to a unified, coherent completion.
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The ability to bond metal orthodontic brackets to enamel has revolutionised fixed appliance orthodontic therapy in recent times (Newman 1973; Whitehill-Grayson 1981). This eventuated as a result of the acid etch revolution fathered by Buonocore in 1955. It was Newman in 1965 who first used the acid etch technique for the direct bonding of orthodontic brackets to teeth with an epoxy derived resin.

The bracket is bonded by mechanical retention at both the enamel-resin interface by resin tags and the bracket-resin interface by a mesh or perforated bracket base (Sheykholeslam & Brandt 1977). The composite resins have no chemical adhesion properties and rely solely on mechanical interlocking for their attachment to enamel (Holtz, McLean, Sced & Wilson 1977). Whilst this technique has produced considerable advantages over the traditional cemented orthodontic band (Brandt, Servoss & Wolfson 1975), there are still some clinical problems (Andreasen & Chan 1981).

The phosphoric acid solution used to etch the enamel surface is both destructive and invasive of enamel (Silverstone 1974; Brown & Way 1978) and lacks biocompatibility with soft tissues.

Decalcification around and under bonding sites can occur during orthodontic treatment (Maijer 1982). This problem is even more accentuated with the cemented orthodontic band (Mizrahi 1977). Plaque will inevitably grow peripheral to and particularly gingival to bonded brackets (Gwinnett & Ceen 1979). Debonding procedures for
resin adhesives can be tedious and difficult with the potential for damage to the enamel surface (Dogon 1982). These problems are paramount when one considers that the bonding of orthodontic brackets is usually restricted to the anterior dentition (Mizrahi 1983).

The development of the polycarboxylate cements by Smith (1968) provided the dental profession with the first truly adhesive material that bonded to both enamel and dentine. Continued research by Wilson and Kent (1972) produced the glass ionomer cements (GIC). These materials were formulated to bring together the silicate and polyacrylate systems thus combining the adhesiveness of the polycarboxylate cements with the mechanical properties and fluoride release of the silicate cements (Wilson 1975). So, the development of glass ionomer cements was an attempt to combine the successful properties of both the dental silicate and polycarboxylate cements (McComb 1982a).

The glass ionomer cements have been improved and refined since 1972. Various manufacturers now supply two types of GIC - a filling or restorative material and a luting cement. Both materials are similar except for the finer powder particle size of the luting materials to allow minimal film thickness (Wilson 1977).

These materials have been used for a diverse array of clinical situations (Mount & Makinson 1982). In many respects, GIC would seem ideally suited for bonding metal orthodontic brackets to enamel. It is adhesive to both enamel and stainless steel (Hotz et al 1977).
There is no need for pre-treatment of enamel with phosphoric acid (Hotz et al 1977). The GIC also possesses an anticariogenic effect with the slow release of fluoride into adjacent enamel (Wilson & Prosser 1982a). It is also feasible that the mesh base now welded or brazed to most types of orthodontic bracket can be dispensed with - allowing an adhesive bond directly to a plain, well-adapted, stainless steel base of the orthodontic bracket. Mizrahi and Smith (1969) have used polycarboxylate cement clinically to bond plain base orthodontic brackets. This would reduce costs in bracket manufacture and possibly facilitate recycling procedures of brackets (Wheeler & Ackerman 1983).

The major obstacle against GIC performing as an adequate orthodontic bonding adhesive will be its mechanical properties, particularly its brittleness in thin sections (Wilson 1977). The glass ionomer cement must be able to withstand the rigours of an approximate two year orthodontic treatment span with the consequent masticatory and orthodontic forces applied to the orthodontic brackets. Bond strength has most frequently been interpreted in vitro as tensile and shear bond strength measurements (Jassem, Retief & Jamison 1981). The measurement of both types of bond strength should give a better picture of clinical performance than either one alone (Beech & Jalaly 1981).

This thesis reports on the development of these cements and about in vitro testing of the capacity of them to serve as orthodontic bonding agents. A range of commercially available filling and luting glass ionomer cements was used to bond both plain and mesh-based
orthodontic buttons to flat enamel surfaces of freshly extracted human teeth. In vitro tensile and shear bond strengths were evaluated. Two clinically successful resin adhesives were evaluated under identical experimental conditions. This hopefully, would provide a bond strength yardstick from which GIC performance could be evaluated.

It has been stated that in vitro experiments do not make it possible to subject materials to conditions identical to those experienced in vivo (Beech 1978). Consequently, a clinical trial was instigated to compare a glass ionomer cement and a resin adhesive for the direct bonding of mesh based orthodontic brackets. The clinical trial would help place the in vitro results into clinical perspective.

From these findings, an attempt will be made to evaluate the clinical viability of GIC as an orthodontic adhesive; which GIC evaluated would be most suited to the task; and whether or not it is feasible to dispense with the mesh base presently employed with present-day stainless steel orthodontic brackets.
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CHAPTER 1  ADHESION TO TEETH - PRINCIPLES AND MECHANISMS

1-1 INTRODUCTION

Adhesion and adhesive have been defined somewhat differently by various authors depending on the contexts in which the terms are used.

In reference to the adhesives industry, Parker and Taylor (1966) define adhesion as the joining together of two dissimilar materials, the adhesive bond being the bond across the adhesive-adherend interface.

Beech (1982) claims that adhesion can be defined in the broadest sense as surface attachment. He refers to other definitions such as the molecular attraction between molecules at an interface.

Buonocore (1981a) discusses the confusion that occurs concerning the terminology relating to adhesion in clinical dentistry. He claims that adhesives are probably the least understood of dental materials. A material is either adhesive or is not adhesive. The descriptive terms truly adhesive or true adhesion are redundant. He defines adhesion as the attraction between atoms and molecules at the surfaces of different materials when these surfaces are brought into contact.

Gwinnett and Smith (1982) attempt to differentiate between the clinical dental terms of bonding and adhesion. The term bonding implies a force by which one substance is held in contact with another. The bonds may be chemical, physical or mechanical. Only the presence of both physical and chemical bonds constitutes adhesion. Strictly therefore, all substances which bond with a substrate may not be adhesive. They may appear to exhibit an adhesive property yet be retained by
sophisticated mechanical means referred to as *mechanical interlocking*.

A similar stance has been taken by the Council on Dental Materials, Instruments & Equipment (1974, 1981) which also defines the terms *adhesive* and *adhesion* in relation to clinical dentistry. An adhesive dental material has no reliance on interlocking effects or the retention form of the prepared cavity. Only two materials - glass ionomer and polycarboxylate cements meet the Council's criteria for adhesive.

Beech (1982) states that the term *bonding* is generally accepted to have the broader connotation of surface attachment. Mechanical interlocking may not be considered as adhesion under the definitions that specify intermolecular attraction. However, the Council on Dental Materials, Instruments & Equipment (1981) notes that the nature of the bond mechanism - physical, chemical, mechanical or a combination - is often difficult to determine.

Smith (1982a) prefers the general term *bonding* to describe all the various mechanisms of surface attachment.

Obviously there exists some difference of opinion as to the mechanisms of adhesion as covered by the above definition. Some authors include both mechanical interlocking and interfacial bonding through molecular attraction whilst others, perhaps the majority, consider only molecular attraction in their understanding of adhesion. Smith (1982a) considers that in practice, both mechanisms probably contribute to the strength of the bonded joint - but one or other predominates. Brauer (1975) states that good dental adhesion does not necessarily imply formation of chemical bonds at the interface since physical forces of
adsorption may be quite adequate.

Adhesion in orthodontics relates to bonding orthodontic attachments directly to enamel surfaces so as to eliminate the need to cement metal bands (Retief 1973a). The advantages and disadvantages of orthodontic bonding have been reviewed by Fields (1982).

Maijer (1982) defines bonding in the orthodontic context as the attachment of a filled or unfilled dental resin to enamel by means of a mechanical or physicochemical process, not only to enamel but also to the orthodontic attachment by means of a mechanical interlock with the bracket base.

It appears that mechanical retention represents the predominant mechanism in the bracket bonding technique (Gwinnett & Matsui 1967). Therefore, resin composites for orthodontic bonding are not adhesive as defined by the Council on Dental Materials, Instruments & Equipment (1981).

The problems of adhesion in dentistry and orthodontics merely reflect those of the wider discipline of Rheology & Adhesive Science (Good 1975). Adhesives have come to play an ever-increasing role in modern technology and industry, from military to domestic uses (De Lollis 1980). Similarly, the development of various dental adhesives has substantially enhanced the effectiveness of dental health care (Eick, Johnson, Froemer, Good & Newman 1972). The Science of Adhesion is a truly multi-disciplined subject (Kinloch 1980), where specific disciplines such as polymer science, surface science and physical chemistry meet, and where they may or may not overlap (Good 1975). The need for a truly adhesive dental material is obvious; the only solution to this
complex problem is a multi-disciplinary approach (Retief 1973a). There have been attempts to carry contemporary knowledge of adhesion over to bio-adhesional problems (Baier, Shaffron, Zisman 1968). There is now an increased understanding of the composition of tooth structure and the mechanism of adhesion to biological substrates, as well as progress in compounding adhesive formulations and basic engineering studies, to improve joint strength in an aqueous environment (Brauer 1975).

A decade ago, the details of adhesion in biological environments, particularly the oral environment, were not fully understood (Brauer 1975). These days the understanding of adhesives, adhesion and mechanisms of bond failure are beginning to come of age (De Lollis 1980).

1-2 GENERAL PRINCIPLES OF ADHESION

The molecular forces involved in adhesion are divided into chemical (primary) and physical (secondary) attractive forces (Retief 1975). These forces are known to vary in magnitude (Buonocore 1981a).

1-2-1 PHYSICAL FORCES OF ADHESION

The physical or secondary molecular attractive forces arise from the Van der Waals forces which include:

(i) London forces - dispersion effect
(ii) Keesom forces - orientation effect
(iii) Debye forces - induction effect (Retief 1975)

The London forces arise from internal electron motions which are independent of dipole moments. These dispersion forces are usually weaker than the polar forces but they are universal and all materials
exhibit them (Kinloch 1980). These dispersion forces play an important part in adhesion (Retief 1975).

The Keesom forces result from the interaction of permanent dipoles in neighbouring molecules of polar compounds (Retief 1975).

The Debye forces take into account the influence of a dipole moment in a molecule that induces an additional dipole moment in a molecule across the interface (Retief 1975).

These physical attractive forces operate through a distance of only a few angstroms (Halpern 1981). As such they are short-range forces, effective over a distance of only one or two atomic diameters (Beech 1982). The wetting and spreading of the adhesive must be sufficiently good to ensure intimate molecular contact of the two phases if physical forces are to suffice (Baier 1970). It is understandable that the adhesive properties of the coronal surfaces of teeth, as well as their adsorption characteristics, are given by the outermost parts of the tooth (Glantz 1977).

Van der Waals forces are more than sufficient to produce bonds stronger than the cohesive strength of most materials used as adhesives (Beech 1982); however alone, they are not sufficient to give long-term bonding in the oral environment (Beech 1978).

1-2-2 CHEMICAL FORCES OF ADHESION

Chemisorption is preceded by physical adsorption (Retief 1973a). Chemisorption gives rise to very strong attractive forces and once established, is not easily reversed (Retief 1975). Adhesion via primary
chemical bonds are optimal for dental adhesion as, unlike physical attractive forces, they are durable in the aqueous environment. Kinloch (1980) states that the additional presence of primary bonding may often increase the measured joint strength and is certainly a necessary requirement for securing environmentally stable interfaces. Although there is considerable indirect evidence emphasising the importance of interfacial chemical bonding (De Lollis 1968; Beech 1972a; Lerchenthal & Brennan 1976), studies which directly confirm its role are scarce (Kinloch 1980).

There are two types of chemical bonds possible with hard dental tissue - ionic and covalent.

Ionic bonds are formed by electrostatic attraction between oppositely charged ions. Unlike physical forces, this is a long range interaction (Beech 1982).

Covalent bonds are formed by electron sharing, most commonly found in organic compounds (Beech 1982). Halpern (1981) believes that covalent bonding offers intriguing possibilities for orthodontic bonding because it enhances adhesive strength without the need for enamel surface alteration such as acid etching.

Mechanical retention represents another important bonding mechanism for dental adhesion. This arises from the mechanical interlocking of the adhesive into surface irregularities. In an oral environment, the physical forces will generally be broken down by penetration of water along the interface, but the mechanical interlocking, if well formed, should resist displacement.
1-2-3 CONTACT ANGLE AND WETTING

The extent to which liquids spread on a solid surface and the adhesion of these liquids define the wetting properties of the solid (Baier, Shafrin & Zisman 1968). It was Zisman, an American surface chemist, who clarified and defined the relationship between the wettability of a surface and its adhesiveness (Glantz 1977).

The extent to which an adhesive will wet a surface depends on, amongst other factors, the contact angle $\theta$ at which the surface of the adhesive meets the adherend surface upon which it rests (Zisman 1963). The contact angle $\theta$ is the one formed between the surface of the adhesive drop and the surface of the adherend upon which it is resting. The contact angle is a useful inverse measure of the wettability of a surface in the sense that small contact angles are obtained with easily wettable surfaces (Baier 1970).

The relationship between contact angle, wetting and adhesion is diagrammatically presented by Figure 1.
Wetting  Adhesion  Contact Angle

FIGURE 1: THE EFFECT OF THE CONTACT ANGLE ON WETTING AND ADHESION
The adhesive completely wets the substrate when the maximum possible number of interfacial contacts are achieved (Brauer 1975). Wetting is a manifestation of the attractive forces between the molecules of the adhesive and the adherend (Retief 1975). Changes in wettability are shown to be dependent on both the atomic constitution and the physical packing of atomic groupings in the outermost surface layer (Baier 1970). Research has shown that for good adhesion complete wetting must be first obtained (Zisman 1975). If good wetting is not achieved, the success of the adhesive bond will be decreased greatly (Eick et al 1972).

Therefore, it is apparent that most investigators in the field of adhesion agree that the wetting process of the adherend by the adhesive is essential for the development of an adequate adhesive bond.

Figure 1 indicates how good wettability means maximum spread of contact with a surface which in turn means a very flat or low angle of contact at the periphery of the wetting agent.

1-2-4 SURFACE TENSION AND SURFACE FREE ENERGY

Surface tension is a direct measurement of intermolecular forces (Kinloch 1980). Molecules on the surface of a liquid or solid are in a one-sided field force pulling them downwards into the bulk of the liquid or solid - this inward force is responsible for the surface tension of solids and liquids (Retief 1975).

The surface tension is dimensionally the same as the surface free energy (Retief 1970a). The surface tension or free surface energy of
the adhesive and adherend play an important role in adhesion (Retief 1983a).

Organic surfaces are low energy surfaces in contrast to inorganic surfaces which are high energy surfaces (Beech 1977). Buonocore (1981a) states that when substances are attracted, there is a loss of surface free energy and a lower energy free state is achieved. A fraction of a monolayer of adsorbed molecules is usually sufficient to give a high energy surface. The same wettability properties as those of a low energy solid are shown to have the same surface constitution (Zisman 1975).

The significance of whether a surface is low or high energy is that the high energy surfaces are more amenable to bonding (Beech 1977). Adhesives readily wet and bond to high energy surfaces and in doing so cause a net reduction in surface free energy (Beech 1977).

The outermost atomic constitution of a material must dictate its initial interactions with its environment (Baier 1970). Such a situation arises when the hydroxyapatite of dental enamel, which is a high energy surface, adsorbs a thin organic surface film (Glantz 1977). The formation of these organic tooth films with their low degrees of adhesiveness probably explains why it is comparatively difficult to adhere materials to natural, untreated tooth surfaces (Glantz 1977). These low energy tooth surfaces, not favourable to bonding, can be activated by etching or polishing thereby promoting the conversion to a high energy surface (Beech 1982).
1-2-5 THEORIES OF ADHESION

The mechanisms of adhesion are still not fully understood and many theories are to be found in the current literature (Kinloch 1980). Much of the confusion undoubtedly arises because test methods commonly employed to measure strengths of adhesive joints are not well suited to theoretical analysis. This inability to measure the interfacial interactions has been the main obstacle to the development of a comprehensive theory of adhesion (Kinloch 1980).

Because of the lack of agreement and lack of understanding, the theories of adhesion have had very little influence on adhesive development. In other words, adhesive development has been empirical in nature (De Lollis 1980).

Theories of adhesion are relatively few in number but the lack in numbers has not necessarily simplified understanding or encouraged agreement (De Lollis 1980).

The four main theories of adhesion are:

1. Mechanical interlocking
2. Diffusion theory
3. Electronic theory
4. Adsorption theory (Kinloch 1980)

These theories have been advanced to fit particular bonding situations but cannot be generalised (Beech 1982). There is no single theory of adhesion that accounts for all the facts (Parker & Taylor 1966).
When one considers the variety of materials and surfaces included in bonded assemblies, it would indeed be strange if one set of rules governed all variables (De Lollis 1980). Ordinary adhesive phenomena involve a number of factors referred to in these theories. Each theory delineates one aspect of adhesion only and to adequately describe the real situation the theories must be combined (Beech 1982).

The use of sophisticated, surface-specific, analytical techniques such as Laser-Raman spectroscopy (Koenig & Shih 1971), X-ray photo-electron spectroscopy (Bailey & Castle 1977) and secondary ion mass spectroscopy (Gettings & Kinloch 1977) have produced definitive evidence that primary interfacial bonding may occur in certain circumstances and may make a significant, indeed vital contribution to the intrinsic adhesion. These new specific surface techniques should provide much interesting information on the nature of interfacial bonding and considerably assist in the identification of mechanisms of adhesion (Kinloch 1980).

1-3  **BONDING TO DENTAL ENAMEL**

1-3-1  **GENERAL PRINCIPLES - THE ORAL ENVIRONMENT**

The factors which influence adhesion to tooth surfaces need consideration. The oral environment is very rigorous and bonds are subject to considerable mechanical and thermal stresses (Beech 1982). Indeed, the oral environment is ideally suited to destruction (Retief 1970b). The problems encountered in the oral environment are numerous, complex and not conducive to maintaining or obtaining adhesion (Retief 1970b). Adhesion to dental enamel has far more stringent requirements than many of those encountered for industrial adhesives (Buonocore 1970a).
As such, many excellent commercial adhesives must be excluded from intra-oral use for they are normally applied under dry conditions at high temperatures and pressures and where toxicity is not of prime importance (Retief 1973a). Under industrial conditions, hours or perhaps days can be allowed for setting (Parker & Taylor 1966). To obtain adhesion in the mouth is a much more difficult task (Retief 1970b).

Patrick (1961) cites several factors that affect bond strength during clinical use. The type of stress to which the joint is exposed, the rate and speeds at which the stresses are applied, the temperature changes and the chemical stresses to which the bond may be subjected, are all factors to be considered.

Wilson and Prosser (1982a) consider that the principal barrier to effective adhesion to dental tissue is water. Both enamel and dentine contain water which cannot be removed without damage to those tissues (Retief 1973a). Therefore, for long term bonding, the adhesive must successfully compete with water at the interface (Beech 1978). A major problem in obtaining adhesion to tooth enamel is the aqueous nature of the oral environment with water usually hindering adhesion (Gwinnett & Smith 1982). A dental adhesive must be capable of bonding to enamel surfaces that may not tolerate complete drying and more importantly, the adhesive must maintain its adhesion in continuous contact with moisture (Buonocore 1970a). The inherent humidity and constant exposure to moisture in the oral environment is one of the greatest problems for dental adhesives (Beech 1982). Under clinical conditions, water is always present on clean enamel and if the adhesive is not able to
react with, dissolve or displace all of the adsorbed water from the surface of the adhesive joint, the resulting bond will be weak (Brauer 1975).

Obviously, long term exposure to moisture, as occurs in the oral environment, requires that the adhesive used must be chemically resistant to decomposition by water (Retief 1973a). Beech (1982) claims that to provide long term retention in the oral environment, then in addition to secondary bond formation, either primary bonds or mechanical interlocking are required.

Retief (1970b) states that for an oral adhesive to be successful, it is required to withstand masticatory forces, thermocycling, rapid pH changes and corrosion whilst in the mouth. Masticatory forces vary momentarily in a patient's mouth (Driessens 1977) and biting stresses may amount to thousands of pounds per square inch (Retief 1970b).

Besides masticatory stresses and rapid temperature changes, an orthodontic bond is subject to additional stresses such as forces induced by the archwires and ligation which can jeopardise the bracket base-enamel interface (Maijer 1982). However orthodontic bonds, in contrast with restorative adhesives, are normally maintained for a period of approximately two years during the active treatment phase. The restorative adhesive must provide long term retention.

A satisfactory adhesive material must take into account the physical and chemical properties of dental hard tissues to which it must adhere (Retief 1973a). As only enamel is relevant to orthodontic
bonding, this review will not dwell on bonding to dentine, although some simple contrasts with enamel adhesion will be made.

1-3-2 THE ENAMEL SURFACE

Special problems exist in obtaining adhesion to tooth enamel (Gwinnett & Smith 1982). Enamel, in contrast to dentine, may be regarded as essentially a single component material (Beech 1982). It has basically an homogeneous, dense, rigid structure composed almost entirely of hydroxyapatite (Beech 1978). Hydroxyapatite, and therefore tooth enamel, has a high energy surface (Beech 1977). Enamel is always covered by an organic layer to minimise its surface energy and this must be removed prior to application of an adhesive (Beech 1977).

The basic structure of enamel consists of rods or prisms about 5μm in diameter extending throughout its full thickness (Beech 1982). Significantly, not all enamel is of the prismatic type (Gwinnett & Smith 1982). A prismless form of enamel commonly occurs in the primary dentition and the cervical third of the permanent dentition (Gwinnett 1967, Ripa & Gwinnett 1967). Such enamel cannot be clinically diagnosed (Gwinnett & Smith 1982). The composition of the enamel surface varies due to abrasion and ion exchange with the contents of the mouth (Beech 1982). Enamel is heterogeneous in structure, changing from tooth to tooth and even in adjacent areas of the same tooth (Retief 1970b).

This gives rise to significant changes in etching patterns which have significant clinical implications in orthodontic bonding (Gwinnett 1981).
Lee & Swartz (1970) described three basic criteria for bonding which Gwinnett & Smith (1982) have emphasised, namely that a substrate should:

1. Be free from surface contamination
2. Be smooth and uniform
3. Have a surface similar to the subsurface with which bonding is to occur.

In the context of the criteria necessary for bonding, enamel surfaces are physically and chemically complex (Gwinnett 1981). It is clear therefore, that enamel surfaces are poor substrates for bonding (Gwinnett 1981). The enamel surface is covered by a surface organic integument called *acquired pellicle* (Dawes, Jenkin & Tonge 1963). The pellicle consists of proteins selectively adsorbed from saliva (Moreno 1975) whose physiological function is to protect the enamel from dietary acids and to reduce friction between teeth and oral mucosa (Rølla 1983). Pellicle is a bacteria-free, organic layer approximately 1-3μm thick (Jenkins 1978) and it differs markedly in structure and composition from the underlying enamel which is 96 per cent mineralised (Gwinnett 1981). This organic surface layer forms a substrate upon which micro-organisms attach and colonise to form dental plaque (Gwinnett 1981).

Removing this acquired pellicle is the first important step in disturbing the integrity of the enamel (Maijer 1982) as the intact tooth surface is generally described as relatively inert and of low energy (Gwinnett & Smith 1982). Acquired pellicle is not removable
by toothbrushing and requires the more vigorous treatment of prophylaxis with an abrasive paste to remove it (Beech 1982). If we want to adhere materials to tooth surfaces, it is possible to do so only when a breakdown of the organic surface films have been obtained (Glantz 1977). The wettability of the enamel varies according to its contamination with these low energy organic films (Brauer 1975).

Tooth surfaces that may appear clinically smooth are seldom so at a microscopic level (Retief 1970b). Ridges, depressions and pits reflect the nature and incremental growth of the tissue (Gwinnett 1981). Abrasion anomalies may also be present (Mannerberg 1960).

Unmodified, natural enamel may be covered by debris, plaque, calculus, prismless layers and pellicle (Beech 1982). A mechanical cleaning of the enamel is an important first step clinically in any bonding procedure (Gwinnett 1981).

Thus enamel (this non-uniform, rough, dirty, moist, low-energy surface) inherently tends to resist potential adhesives (Phillips 1981). In fact, few substances have inherent characteristics that so discourage the attachment of an adhesive (Phillips 1981).

1-3-3 APPROACHES TO OBTAIN ADHESION TO TOOTH STRUCTURE

1-3-3-1 Introduction

Beech (1982) classified the approaches to obtain and improve adhesion to teeth into three distinct categories:
1. The development of new adhesive restorative materials,

2. the use of coupling agents or intermediary layers between restorative material and tooth,

3. modification of tooth surfaces to make them more receptive to bonding.

Lee & Swartz (1970) believe that the logical approach to make the tooth surface more suitable to bonding is to modify the tooth surface itself either by means of surface preparation or reinforcement of the substrate.

However, Beech (1982) believes the first listed approach is undoubtedly the ultimate goal but the more difficult. Beech highlights the conflicting requirements asked of the adhesive material - that short-term it should be hydrophilic for successful competition with the water at the tooth surface; yet hydrophobic long-term to withstand degradation in the aqueous oral environment.

Brauer (1975) surveyed the materials that have been investigated as potential dental adhesives. Bowley (1977) classified these materials into three groups: Adhesives in which hardening occurred by polymerisation, adhesives that harden other than by the action of polymerisation (chelation) and bio-adhesives.

The first group included acrylic resins, epoxy resins, Bowen's resins, cyanoacrylates and polyurethanes. The second group included only polycarboxylate cement to which can be added glass ionomer cement.
Both these cements use polycarboxylate acids to form ionic bonds to calcium in dentine and enamel, becoming insolubilised by cross-linking with added metal ions (Beech 1982). These cements are discussed in detail in the following chapter of the literature review.

Bioadhesives, mainly as the secretions of marine biota, have been investigated for their potential as dental adhesives (Cook 1970; Hillman & Nance 1970). Docking (1967) made reference to the fact that adhesion under wet conditions had already been solved in nature and mentions the investigations of Harrison and Philpott (1966) on bioadhesives. However to date, the exact chemistry and polymerisation of the barnacle secretion remains unknown (Phillips 1981). Moreover, Brauer (1975) has noted previously that the chemical complexity of cements of marine organisms makes it doubtful that such products can be synthesised commercially.

1-3-3-2 Adhesive Materials

Acrylic and Bowens Resins

Two types of direct filling resins are available at present, namely the unfilled acrylic resins and the filled BIS-GMA or composite resin systems (Smith & Peltoniemi 1982). The acrylic resins, based on self-curing acrylics, consist of methylnethacrylate monomer and ultra-fine powder (Whitehill-Grayson 1981). Filled composite resin systems have excellent bond strength, lower water absorption and less polymerisation shrinkage than the acrylic resins (Craig 1981). Composite resins are superior to acrylic resins in most mechanical and physical properties (Jones, McCabe & Spence 1977).
Both acrylic and composite resins have been used successfully in association with the acid etch technique for orthodontic bonding (Maier 1982). Newman (1969a, 1973) provided the initial reports of successful clinical orthodontic bonding using acrylic resin in association with enamel etching.

Maier (1982) claims that orthodontic bonding resins based on BIS-GMA were originally developed to overcome the difficulties experienced with the acrylic resins. However, Beech and Jalaly (1981) found in a recent clinical and laboratory assessment of orthodontic bonding resins that ordinary self-curing acrylic with a 20-50 percent phosphoric acid etchant was the optimum bonding agent in terms of bond strength, clinical effectiveness and ease of post-treatment debonding. Similarly, Sandilands and Edwards (1983) claim great clinical success with Orthomite IIIs, an acrylic orthodontic bonding resin marketed by Rocky Mountain Orthodontics. This acrylic orthodontic bonding system was initially developed by Miura and his co-workers (Miura & Nakagawa 1979; Miura, Nakagawa & Ishizaki 1974).

**Epoxy Resins**

The epoxy resins are natural candidates as adhesives to hard dental tissue as they develop the strongest and most permanent bonds of any known resin type (Lee & Swartz 1970). However as dental adhesives, they cure slowly and bonding is affected by water (Lee 1966). Their reactivity is questionable for biological applications (Lee & Swartz 1970).

Retief, Dreyer and Gavron (1970) used an epoxy resin system to
clinically bond orthodontic brackets. The bonding technique was cumbersome and there was a high number of bond failures.

**Cyanoacrylates**

Another group of polymerising adhesives comprises the cyanoacrylates which consist of liquid monomers that polymerise very rapidly when exposed even to small traces of moisture (Ibsen & Neville 1974).

Leonard, Kulkarni, Brandes, Nelson and Cameron (1966) have written extensively on cyanoacrylates, including their synthesis in high purity.

Alkyl 2-cyanoacrylates have a unique ability to form covalent bonds to protein and have been investigated as intermediary layers between dentine and composite restorative material (Beech 1982). These materials gave generally unsatisfactory bond strengths with enamel which were improved slightly by prior acid-etching (Beech 1972a).

Cyanoacrylates have been investigated for a variety of clinical situations. Leonard (1970) investigated these monomers as tissue adhesives and haemostasis inducing compounds. A number of partially successful clinical trials as fissure sealants have been reported (Cueto & Buonocore 1967; Ripa & Cole 1970; Takeuchi, Shimizu, Kizu, Eto, Nagawora, Ohsawat & Oishi 1971). However, commercial interest in cyanoacrylate sealants waned due to their biodegradability, difficulty of polymerisation in thin films, poor abrasion resistance and the development of more stable resin systems (Gwinnett & Smith 1982).
Newman (1969) investigated the use of cyanoacrylates in relation to orthodontic bonding. He found problems with manipulation, hydrolysis and the fact that the bonds between enamel and orthodontic plastic bracket were too brittle.

Eames & Litvak (1982a) believe that there is little, if any future for the cyanoacrylates as dental adhesives. The bond formed between tooth structure and cyanoacrylate in vivo is at best temporary and predestined to fail because it is degradable in a wet environment. Eames & Litvak (1982a) quote the American Dental Association Materials Council which, believes that (they) cannot be recommended for routine use in dentistry.

Polyurethanes

Polyurethanes have been investigated as both cavity liners (Galligan, Minor & Swartz 1970) and fissure sealants (Rock 1974). Rock found very low retention rates for such a sealant, concluding that it was unsuitable as a permanent caries preventive measure.

The advantages of polyurethanes include their toughness, resilience and abrasion resistance; their disadvantages are mainly their chemical instability and potential toxicity (Gwinnett & Smith 1982).

Potentially useful adhesives such as polyurethanes, cyanoacrylates and epoxy resins are either unable to form effective bonds in competition with water or are hydrodised (Manly 1970). This highlights the fact that moisture is the chief obstacle to the attainment and maintenance of an adhesive bond under oral conditions.
1-3-3-3 Coupling Agents

The second approach, according to Beech (1982), to promote adhesion to tooth structure concerns the use of coupling agents or primers.

Brauer (1975) uses the terms coupling agents and adhesion promoters synonymously. He claims that an adhesion promoter is ideally adsorbed as a monomer layer that will prevent contamination of the solid by organic substances. Smith (1982a) defines a primer or coupling agent as a chemical treatment at a molecular level that produces a surface receptive to bonding. Causton (1982) has described the principle of primers which react with the tissue surface and which must compete with and displace water for durable bonding.

Smith (1982a) claims that most primers are designed to react with the inorganic phase of teeth by:

(a) Complexation with Ca$^{2+}$

(b) Reaction with the PO$_4^{3-}$ or OH$^-$ groups or with bond water in the hydroxy-apatite lattice.

Causton (1982) classified primers for use in dentistry as:

1. N-phenyl glycine derivatives (NPG-GMA)
2. Phosphonate derivatives
3. Silane derivatives
Bowen (1965) proposed N-phenyl glycine derivatives as primers for tooth surfaces. The primer NPG-GMA, the condensation product of N-phenyl glycine and glycidyl methacrylate, has been shown to adsorb strongly to calcium (Mista & Bowen 1977).

Phosphate or phosphonate groups also show strong chelation to calcium (Smith 1982b). Various workers (Farley, Jones & Anbar 1977; Rawls & Cabasso 1982; Causton 1982) have used polymerisable phosphonates as primers or added them to BIS-GMA resins to increase bonding to tooth structure. Improvements in the order of 100 per cent in the bond strength of commercial composites to etched enamel have been reported by Farley and his co-workers (1977).

Silanes are widely used as coupling agents for the treatment of fillers in composite resins. These bond the glass filler particles used in resin composites to the resin polymer matrix (Craig 1981), resulting in substantially improved mechanical properties (Causton 1982).

Bowley (1977) investigated the efficacy of coupling agents (primers) for two direct bonding orthodontic acrylic resins - orthomite and sevitron. In vitro shear and tensile bond-strength tests showed that the coupling agents failed to significantly improve adhesion with either system, using plastic brackets. These results support Beech's opinion (1982). He states: Although a number of primers are available commercially, there is as yet no convincing evidence to indicate that any gives sufficient bonding for reliable long-term retention. Indeed, in the majority of cases there is doubt whether their uses gives any benefit at all. Although it is a reasonable concept it is not particularly effective in practice.
1-3-3-4 Modification of Tooth Surfaces

The third approach to obtain and improve adhesion to teeth is to modify the tooth surfaces to render them more receptive to bonding. Beech (1982) states that this approach can be achieved by the following:

1. Acid etching.
2. Mineralisation or epitaxial deposition of mineral salts.
3. Grafting on to the collagen of dentine.

There has been universal acceptance of acid etching techniques in the use of resin as both a restorative and orthodontic bonding material (Phillips 1981). Most bonding systems use phosphoric acid as the etchant, ranging in strength from 30-65 per cent by weight (Maijer 1982). Gwinnett (1981) claims that etching has a number of effects on enamel including removal of pellicle, increasing the surface area and the enhancement of enamel porosity. Self-curing monomers form secondary bonds and mechanically lock into the pores (Beech 1982). Acid etching, as it relates to orthodontic bonding, will be discussed in greater detail later in the literature review.

Powis, Folleras, Merson and Wilson (1982) attempted to improve the adhesion of GIC to enamel by chemical treatment of the tooth surface prior to bonding. Unlike composite resins, GICs do not require etched surfaces for bonding; rather their requirement is for clean, sound enamel surfaces (Beech 1982). Citric acid pretreatment of enamel has been shown to enhance the GIC bond (Vougiouklakis, Smith & Lipton 1982) but this acid etched enamel is clinically undesirable and unnecessary (Powis et al 1982). Moreover, citric acid can be an
irritant so is not the ideal companion for a bland cement. The study by Powis et al (1982) clearly showed that the use of enamel conditioners can improve the enamel/GIC bond. The untreated enamel surface yielded an enamel/GIC tensile bond strength of 3.18MPa, that with Dodicin, 6.33MPa.

The most effective surface conditions were shown to be solutions containing polyacrylic acid, tannic acid or Dodicin. These substances contain a multiplicity of functional groups capable of hydrogen bonding to tooth material which ensure effective cleaning and wetting of the substrate surfaces. They do not disrupt the surfaces of the enamel unduly and are more effective than are low molecular weight chelating agents such as citric acid and EDTA which dissolve calciferous material and disrupt the surfaces of the enamel. Moreover, Powis et al (1982) conclude that further improvements in the adhesion of GICs to enamel will only be achieved when the cohesive strength of the material is increased. This comment agrees with Vougiouklakis et al (1982), that the weak link in the GIC/enamel bonded joint is the low tensile strength of the cement rather than its poor adhesion to enamel.

There have been numerous attempts using mineralising solutions to increase the adhesion between the polyelectrolyte cements to enamel and dentine (Beech 1973; Levine, Beech, Garton 1977; Causton, Johnson & Daymon 1979). Bond strengths of these cements to dentine are substantially less than to enamel (Beech 1978). The reasons for the wide variation in the effect of these surface treatments is poorly understood (Peddey 1981).
Smith & Cartz (1973) observed specific crystal growth of insoluble calcium salts on the surface of enamel. This observation led to the concept of crystal growth as a retentive mechanism for resins. Subsequently, this concept was further developed by Maijér & Smith (1982) into a crystal bond system for orthodontic brackets. This concept will be discussed in more detail later.

The problem of establishing a permanent adhesive bond with tooth substance, especially dentine, is still most obvious (Smith 1982a). Good adhesion requires substantial cross boundary forces between interlayers of two adjacent phases (Brauer 1975).

The use of biocompatible cleaning protocols coupled with reactive adhesives appears to be the path of future research. The development of adhesive molecules that form complexes with calcium appears promising. Whether such systems will be most successful as primers, polymerisable monomers, or as polyelectrolytes remains to be proved (Smith 1982a).

Brauer (1975) highlights the fact that recent studies of the reactivity and modification of tooth surfaces have provided considerable basic information which will be most useful in solving many of the problems inherent in obtaining adhesion to the heterogeneous tooth surfaces in the aqueous environment encountered in the oral cavity.

Smith (1982a) emphasises that in the final analysis, the success or otherwise of an adhesive system depends on two factors; firstly that it can be used properly under practical clinical conditions and secondly that the adhesive and tooth surface can maintain their integrity.
He continued by commenting that despite the fact that we really do not know the theoretical basis for adhesion to moist enamel, we should continue using an empirical approach to dental adhesive research. This in turn, notes Brauer (1975), requires evaluation of future adhesives by well-designed standardised in vitro testing procedures that correlate with clinical experience.
CHAPTER 2 

POLYELECTROLYTE CEMENTS

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2-1 INTRODUCTION

Wilson (1978) believes that the most important recent development in dental cement technology has been the emergence of a new class of cement - the ionic polymer cement system, where anionic polyelectrolytes are employed as cement-forming liquids. The ionic polymer or polyelectrolyte cements are at present represented by the zinc polycarboxylate of Smith (1968) and the glass ionomer cement (GIC) of Wilson and Kent (1971, 1972). Both cements are notable for being bland towards living tissues (McComb 1982a) and adhesive to enamel, dentine and base metals (Richter, Mitchem & Brown 1970; Hotz, McLean, Sced & Wilson 1977). Thus, stainless steel orthodontic buttons are attached directly to teeth (Wilson 1978). In fact, the Council on Dental Materials of the American Dental Association (1981) considers that only polycarboxylate cement and glass ionomer cements (GICs) meet the Council's criteria of adhesive. This opinion is reinforced by Wilson and Prosser (1982b) who state that chemical adhesion, as opposed to the micromechanical attachment effected by acid etching, has only been achieved by cements based on acidic polyelectrolytes.

Both polycarboxylate and glass ionomer cements use similar liquids - concentrated aqueous solutions of polyacrylic acids (Wilson 1978). The powder used in polycarboxylate cements is principally zinc oxide and some magnesium oxide (Phillips 1982). The powder is especially prepared by heating it alone or with magnesium oxide (up to 10 per cent W/W) to 1100-1300°C for several hours (Wilson 1978). The formulation of the glasses used in GICs are a far more complex topic. Like the dental
silicate cement, GIC employs ground ion-leachable aluminosilicate glasses (Wilson 1980).

The fundamental bonding mechanism of polycarboxylate and glass ionomer cements to tooth structure is the same - the polycarboxylic acids form ionic bonds to calcium in the tooth (Beech 1982). However, Phillips (1982) states that the exact mechanism of the adhesion to the calcium in tooth structure is not entirely understood and remains a subject for speculation.

Smith (1968) developed the polycarboxylate cement in a search for a material which would combine the strength properties of the phosphate system with the biological acceptability of the zinc oxide eugenol system (Smith 1982b).

Wilson and Kent (1972) developed GIC in an attempt to improve upon the performance of the dental silicate cement, for many years the mainstay of aesthetic anterior restorations. There was a need for a filling material that was both bland to living tissues and adhesive to tooth structure (Wilson & Kidd 1978). The GIC combines certain of the favourable features of the silicate cement (fluoride release, high compressive strength, translucency) and zinc polycarboxylate cements (biocompatibility to the pulp and adhesiveness) without having some of their disadvantages (McLean & Wilson 1977a). GIC, in contrast to dental silicate cement, does not have the biological drawbacks of lack of adhesion and irritancy (Wilson & Prosser 1982b).

The polycarboxylate cement is available only as a luting cement.
(Phillips 1982), but has been used clinically as both an orthodontic band cement (Mizrahi 1979) and a direct orthodontic bonding adhesive (Mizrahi & Smith 1969a, 1971). Glass ionomer cement is available both as a filling material and a luting cement (McLean & Wilson 1977b, 1977c; Mount 1981). The clinical uses of GIC have been numerous and varied (Mount 1982), including orthodontic band cementation (Kvam, Broch, Nissen-Meyer 1983).

To date there exist no reports in the literature of the clinical use of GIC as a direct orthodontic bonding adhesive. However, both Owen (1981) and Carlyle, Chamma, Moir and Williams (1978) have undertaken in vitro bond strength tests to evaluate the feasibility of GIC as a direct orthodontic bonding agent.

2-2 POLYCARBOXYLATE CEMENT

2-2-1 INTRODUCTION

It is well known that the polycarboxylate cements display good adhesion to enamel and base metal alloys, especially stainless steel (Smith 1971a). This adhesive capability suggested clinical use in orthodontics for both band cementation (Mizrahi 1979) and the direct bonding of orthodontic brackets (Manning 1971; Mizrahi & Smith 1971).

The polyacrylate cements - polycarboxylate and glass ionomer - possess similar adhesive capabilities and mechanisms (Smith 1982b) but the aluminosilicate glass powder component of GIC has resulted in an improvement in the physical and mechanical properties (McComb 1982a). Therefore, one might expect GIC to be more successful than polycarboxylate-
late cement for orthodontic bonding and band cementation (Owen 1981).

The earlier, extensive clinical and laboratory experiences with polycarboxylate cements (Bertenshaw & Combe 1972a, 1972b, 1976, 1979; Mizrahi 1977a, 1977b, 1979) provide a comparison and perspective as to the potential clinical uses of GIC, particularly in relation to orthodontics. For these reasons, a more detailed account of the composition, physical properties and clinical applications of polycarboxylate cements follow.

2-2-2 COMPOSITION

The composition of the original polycarboxylate cements as developed by Smith (1968) have been modified as these materials have gone through several stages of development since their inception (Smith 1982b). The original cements were based on the reaction between zinc oxide and an aqueous solution of polyacid (Smith 1971b). Information on composition is given by analytical studies by Bertenshaw and Combe (1972a, 1972b, 1976, 1979) and Crisp, Lewis and Wilson (1976a). The continual development of these materials has resulted in many possible variations in the composition of powder and liquid. Bertenshaw and Combe (1979) stressed that due to the continual development of the polycarboxylate cement, their data on nine commercially available cements may not be applicable in all cases to current products.

A number of polycarboxylate cements now contain 4-5% stannous fluoride which affects the setting reaction and improves the manipulation characteristics (Smith 1982b). Phillips (1982) considers that stannous fluoride is the most important additive as it increases the strength of the cement and acts as a good source of fluoride which may well impart
anticariogenic properties to the cement.

Bitner and Weir (1973) and Greenland, Hargreaves, Smith and Beagrie (1974) have demonstrated that significant uptake of fluoride by neighbouring enamel occurs. Smith and Peltoniemi (1982), after reviewing the most recent in vitro evidence, claim that it would appear that polycarboxylate cements containing fluoride are beneficial. However, data to establish definite anticariogenic effects is lacking.

2-2-3 PHYSICAL PROPERTIES

Polycarboxylate cement was the first cement system developed with a potential for adhesion to tooth structure (Smith 1971a). Smith (1982b) states that these cements display good adhesion to enamel and to a lesser extent, dentine as well as to various metal alloys. The bonding of polycarboxylate to such surfaces may exceed the cohesive strength of the material (Beech 1973). A rather wide range of bond strengths to enamel and dentine have been reported (Mizrahi & Smith 1969a; Beech 1972a; Manning 1971; Hotz, McLean, Sced & Wilson 1977). According to Phillips (1982), the differences may be due to the individual test methods.

Bertenshaw and Combe (1979) investigated tensile bond strengths of nine commercially available polycarboxylate cements. The bond strength to enamel ranged from approximately 4-7MPa; the bond strength to dentine 3-5.5MPa. These bond strength values agree with Beech (1978) and Buoncore (1981b) who state that bonding to dentine is not as great as attainable with enamel. This is considered to be due to the lower mineral content of dentine since bonding appears to take
place via the calcium in tooth structure (Beech 1972a).

The importance of calcium to the polyacrylate bond has been well demonstrated by the lower tensile strength of polycarboxylate cement to smooth surfaces of dentine and bone, as compared with enamel (Eden, Craig & Peyton 1970). Likewise, the reduction in bond strength after saliva contamination of the surface, due to the formation of a layer of adsorbed salivary protein (Mizrahi & Smith 1969a).

Smith (1971a) showed in vitro that the de-mineralisation of enamel and dentine surfaces by lactic, citric and 50 per cent phosphoric acid resulted in a decreased bond strength of the polycarboxylate cement. Brauer (1975) supports this statement noting that acid etching of the enamel substrate surface prior to application of the polycarboxylate cement does not increase bond strength. Eden and his co-workers (1970) demonstrated that there was no significant increase of polycarboxylate bond strength to bovine enamel after pretreatment with various etchants, including citric acid, EDTA and phosphoric acid. They noted that the behaviour of the polycarboxylates in respect of acid etching of the enamel was in stark contrast with BIS-GMA resins where etching led to a significant increase in bond strength. Further evidence for the effect of de-mineralisation with similar etchants was obtained by Beech (1973) with dentine bonding. Both Beech (1973) and Peddey (1981) demonstrated an increased bond strength of polycarboxylate to dentine following hypercalcification of the surface with mineralising solutions. Such data suggests that there is little bonding to the proteinaceous surface once it is demineralised.
Smith (1982b) states that bonding of polycarboxylate cements to metal substrates involves specific ion binding through chemical reaction with the surface as shown by experiments with pure metals (Rogers & Griffith 1977a) and known alloys (Mizrahi & Smith 1969a; Ady & Fairhurst 1973; Moser, Brown & Greener 1974).

The bonding of gold appears to be minimal due to its inertness but Rogers and Griffith (1977a) obtained significant bonding to electroplated gold which, according to Smith (1982b), may have been due to microroughness or base metal ions since it was plated on a brass substrate. The bonding of polycarboxylate cement to sand blasted 24 carat gold was demonstrated by Ady and Fairhurst (1973). The 24 hour tensile bond strengths in both cases was about 7MPa.
McLean (1977) has also suggested that adhesive bonding may be obtained by electroplating gold castings with tin and thereby creating a bond through the reaction of carboxyl groups in the cement and tin ions. Phillips (1982) considers that there is a need for further research to determine the validity of this hypothesis.

Higher bond strengths have been obtained with base metal alloys. Jendrensen and Trowbridge (1972) obtained tensile bond strengths of approximately 9MPa for a polycarboxylate cement to stainless steel buttons. Similar results were obtained by Moser and his co-workers (1974). Mizrahi and Smith (1969a, 1968b), using a tensile bond strength evaluation, obtained a bond strength measurement of 6-8MPa between stainless steel buttons and enamel. Moser et al (1974) found similar bond strengths to Jendrensen and Trowbridge (1972) between polycarboxylate cement and a cobalt chromium alloy.
An observation in common with all these investigations was that bond failure with polycarboxylate cements was always cohesive when used to bond stainless steel or cobalt chromium specimens. This indicates that the limiting factor in the bonded assembly was the cohesive strength of the cement. The adhesive bond between the cement and the adherends is stronger than the polycarboxylate cement itself (Mizrahi & Smith 1969b). Smith (1982b) states that the bond strength is limited by the cohesive strength of the cement.

Saito, Sakai, Node and Fusayama (1976) compared the adhesive capabilities of polycarboxylate and zinc phosphate cements to various alloys. They clearly demonstrated that zinc phosphate cements exhibited weak adhesion to metals and that polycarboxylate cement possessed distinct adhesion to varying degrees. Polycarboxylate showed the strongest bonding to a copper alloy followed by nickel, chromium, silver-tin-zinc, silver palladium and type III gold alloy. These results demonstrate that polycarboxylate cements bond well to clean alloy surfaces containing multivalent metal ions.

Smith (1982b) states that the bonding of polycarboxylate cements to present-day dental ceramics is essentially mechanical since they appear too inert for good bonding.

2-2-4 CLINICAL APPLICATIONS

Polycarboxylate cement has been used clinically as both a direct orthodontic bonding adhesive (Mizrahi & Smith 1971) and an orthodontic band cement (Mizrahi 1979).
Mizrahi and Smith (1969b) found that zinc polycarboxylate cement possessed a superior tensile bond strength to silicophosphate, zinc and copper phosphate cements when bonding stainless steel orthodontic buttons to enamel. Manning (1971) compared various cement types for their in vitro bond strength and concluded that it was justifiable to use polycarboxylate cement in a clinical trial as a direct orthodontic bracket adhesive. Clinical trials by Mizrahi and Smith (1971) yielded reasonable success after modification of bracket designs - a 7 per cent bond failure rate over a 6 months treatment period. Mizrahi emphasised the need to prepare and clean the enamel and alloy surfaces properly prior to bracket bonding.

Smith (1982b) has emphasised that the full adhesive potential of polycarboxylate cement can only be realised clinically when proper surface preparation is carried out. Smith (Smith & Maijer 1983a) has claimed that he has successfully used polycarboxylate cement as an orthodontic direct bonding adhesive over a two year treatment period. However, he commented that others have not been able to duplicate his clinical success because polycarboxylate bonding is technique-sensitive. This includes surface preparation, cement mix and placement techniques.

The bond strengths of polycarboxylate cement are less than most direct resin bonding systems (Faust, Grego, Fan & Powers 1978) but are adequate for moving teeth in several orthodontic situations (Mizrahi 1972). However, only under ideal conditions is the bond adequate (Brauer 1975).

Polycarboxylate cements have been assessed in vitro as orthodontic...
band cements. Band retention strengths have been measured by pull-off tests using tensile loads (Smith 1982b). A number of studies (Heagney 1974; Rich, Leisefelder & Hershey 1975; Clark, Phillips & Norman 1977) have compared polycarboxylate and phosphate bonded cements. Smith (1982b) claims that in general, the order of retention seems similar for silicophosphate, phosphate and polycarboxylate cements. The retention values recorded in vitro can vary according to experimental variables such as roughness and cleanliness of various interfaces, effects of band shape, stiffness and surface area, brands of materials used, tooth shape, manipulation of cementing material and the stress system applied to the bonded joint.

Mizrahi (1977a, 1977b, 1979) has assessed the clinical performance of cements in retaining orthodontic bands. The earlier studies used a variety of cement types, the later study (Mizrahi 1979) used only a polycarboxylate cement. His sample size comprised 148 patients (2,920 bands) treated to completion with full fixed appliance therapy using the Begg technique. The percentage of bands recemented, i.e. failure rate, ranged from a maximum of 18 per cent for an experimental formulation to a minimum of 7 per cent for a commercially available polycarboxylate cement (Durelon, Espe). The most commonly recemented bands were the upper central incisors and lower first molars. Mizrahi (1977a, 1977b) analysed in detail the contributory causes of failure and considered that the incidence of loose bands will rise as treatment periods are extended.

Along with studies by Wisth and Bergenkreutz (1974) and Clark et al (1977) it is apparent that for the three cement types assessed -
zinc phosphate, zinc polycarboxylate and silicophosphate, failure rates of 5-10 per cent appear to develop over a one to two year period. Smith (1982b) notes that the results of these clinical studies do not correlate with laboratory data for bond strength and the physical properties of these cements. This is most probably due to excessive stress exerted on the bands by the patients and due to the accuracy of fit and cementation by the orthodontist.

Mizrahi (1981, 1982) by selectively combining conventional orthodontic bands (molars and premolars) with direct bonding (canines and incisors), achieved an overall failure rate of only 4.7 per cent. This study was carried out on 100 patients using a combination of 822 conventional bands cemented to maxillary and mandibular molars and premolars and 1194 brackets bonded directly to maxillary and mandibular canine and incisor teeth.

Mizrahi, Cleaton-Jones and Austin (1981), as a result of studying the effect of surface contamination on band retention, suggest that bands cemented with polycarboxylate cement are retained by the luting action of the cement and not by chemical adhesion to both the enamel and stainless steel band. More importantly, the study demonstrated that surface contamination of the enamel by dry saliva and liquid paraffin reduced the retention of the bands cemented by polycarboxylate whilst those bands cemented by zinc phosphate and silicophosphate were unaffected. These findings confirm yet again the clinical necessity for proper surface preparation for optimum adhesion when using polycarboxylate for orthodontic bonding or band cementation.
Smith (1982b) lists the main advantages of polycarboxylate cements as low irritancy, adhesion to tooth substance and alloys, with strength, low solubility and film thickness comparable with zinc phosphate cements. The disadvantages are more critical manipulation (proper F/L ratio for optimum properties), lower compressive strength and possibly greater visco-elasticity than zinc phosphate cements, the short working time of some materials and the need for clean surfaces to utilise the adhesion potential.

Although zinc polycarboxylates adhere to enamel surfaces, the usefulness of these cements is limited by their physical and mechanical properties (Brauer 1975).

Recent commercial developments of the polycarboxylate cements have led to some improvements including the additional advantage of leachable fluoride (Smith & Maijer 1983a). Wilson (1978) claims that the concept of polyelectrolyte adhesive materials is capable of considerably further development and exploitation. One successful exploitation of the polyacrylate system is the glass ionomer cement (GIC) developed by Wilson & Kent (1972).

2-3 **GLASS IONOMER CEMENT**

2-3-1 **INTRODUCTION**

The glass ionomer cement (GIC) developed by Wilson and Kent (1972) and subjected to further development by others (Kent & Wilson 1973, Crisp & Wilson 1974a, 1974b) is a hybrid of silicate and polycarboxylate cement (Am. Dent. Assoc. 1979). Smith (1968) substituted the phos-
phoric acid of the zinc phosphate cement system with polyacrylic acid
to synthesise the polycarboxylate cements. Similarly, Wilson and Kent
(1972) created the glass ionomer cements by replacing the zinc oxide
powder of the polycarboxylate cement with an ion leachable alumino-
silicate glass. Hence, GIC was formulated by bringing together the
silicate and polyacrylate cement systems (Wilson 1975) in an attempt
to combine the successful properties of both cement types (McComb
1982a). Theoretically, GIC should possess desirable characteristics
of both systems (Maldonado, Swartz & Phillips 1978).

Wilson and Kidd (1978) provide an account of the development of
GIC with particular reference to its clinical considerations and
compare these to the present day composite restoratives.

The glass ionomer cements have been in general clinical usage in
Europe since 1975 and were introduced into the North American market
in 1977 (Am. Dent. Assoc. 1979). The original GIC was ASPA, intro-
duced to the profession in Australia by John McLean in 1976 (McLean
& Wilson 1977a, 1977b, 1977c) and marketed by A.D. International Pty
Ltd. It has recently been superseded by Chemfil, marketed by the same
company and subsequently the G.C. Company of Tokyo, Japan produced and
marketed Fujit Type II in 1978. The Espe Company of Munich, West
Germany, released capsulated Ketac glass ionomer cement in 1981 and
Ketacem, a luting version shortly thereafter. The Shofu Company of
Kyoto, Japan began marketing Hy-bond glass ionomer cement at the

Although GIC was originally envisaged as a filling material, a
version for luting was subsequently developed (Wilson, Crisp, Lewis &
McLean 1977).
The glass ionomer cement represents the first step in an advance towards a truly biocompatible restorative material (Wilson & Prosser 1982a). The presently available commercial GICs are undergoing continual development and improvement (Smith & Maijer 1983a).

2-3-2 COMPOSITION

Both the restorative material and luting version of GIC are normally dispensed as a powder liquid in a two-pack presentation (Phillips 1982). Ketac (Espe) is available as a capsulated material where the powder/liquid ratio is fixed (Mount 1982). The GIC system consists of the following constituents (Crisp, Ferner, Lewis & Wilson 1975):

1. A poly-alkenoic acid, commonly a homo- or copolymer of acrylic acid;

2. an ion leachable aluminosilicate glass to provide ions to cross-link the polyacid chains;

3. water, as a reaction medium;

4. tartaric acid to improve working and setting characteristics.

A typical glass ionomer cement formulation is shown in Table 1.
TABLE 1: COMPOSITION OF A GLASS Ionomer cement

<table>
<thead>
<tr>
<th>POWDER</th>
<th>LIQUID</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fusion mixture composition</td>
<td>Liquid components</td>
</tr>
<tr>
<td>(% m/m)</td>
<td>(% m/m)</td>
</tr>
<tr>
<td>SiO₂ 29.0</td>
<td>Poly(acrylic acid/itaconic acid) 47.5</td>
</tr>
<tr>
<td>Al₂O₃ 16.6</td>
<td>Tartaric acid 5.0</td>
</tr>
<tr>
<td>CaF₂ 34.2</td>
<td>Water 47.5</td>
</tr>
<tr>
<td>Na₃AlF₆ 5.0</td>
<td></td>
</tr>
<tr>
<td>AlF₃ 5.3</td>
<td></td>
</tr>
<tr>
<td>AlPO₄ 9.9</td>
<td></td>
</tr>
<tr>
<td>Maximum particle size</td>
<td></td>
</tr>
<tr>
<td>ASPA IV (filling) 45 μm</td>
<td></td>
</tr>
<tr>
<td>ASPA IV A (luting) 20 μm</td>
<td></td>
</tr>
</tbody>
</table>

The cement liquid is an aqueous solution of polyacrylic acid or is a copolymer of acrylic and itaconic acid. Figure 2 illustrates the molecular structure of both liquids.

FIGURE 2: Molecular structure of (a) polyacrylic acid (b) a copolymer of acrylic and itaconic acids
In current clinical formulations, the copolymer of acrylic acid and itaconic acid, on account of its low viscosity and stability when in aqueous solution, is preferred to polyacrylic acid (Wilson 1977). The copolymer may also be freeze-dried and incorporated into the powder to form the so-called water setting versions of GIC (Prosser, McLean & Wilson 1978). Such a product is Chemfil (A.D. International) which has the dehydrated polyacrylic acid already incorporated into the powder and the liquid used is distilled water (Mount 1982). The elimination of the viscous polyacid solution from the system yields a more satisfactory mix as cement pastes formed from such packs are easily workable and very fluid (Wilson & Prosser 1982a).

In addition to the acrylic acid-itaconic copolymer, the liquid also contains a small amount of tartaric acid in the range of 5 per cent (Crisp & Wilson 1976). Tartaric acid is a low molecular weight chelating agent which improves the working and setting characteristics (Wilson, Crisp & Ferner 1976; Prosser, Jerome & Wilson 1982).

Crisp, Lewis and Wilson (1979) examined the effect of tartaric acid on the properties of glass ionomer cements. They found that there is an optimum concentration for the tartaric acid in the cement-forming liquid for the attainment of the best set of cement properties. The tartaric acid isomer used was also found to be important and indicated that this additive plays a structural role in the set cement.

The itaconic acid reduces the viscosity of the liquid and also makes it more resistant to gelation (Crisp, Ferner, Lewis & Wilson, 1975).
The powder used in GICs are ion leachable glasses belonging to a family of calcium aluminosilicates (McLean & Wilson 1977a). All are prepared by fusing the constituents in the forms of oxides, fluorides and phosphates to the appropriate temperature and then shock-cooling the melt (Kent, Lewis & Wilson 1979). The glass frit is then finely ground to have a maximum particle size of 45μm for filling materials or 20μm for the luting agents (Wilson & Prosser 1982a).

Practical glasses used in dental compositions are based on SiO₂ – Al₂O₃ – CaF₂. This three-component glass produces all other, more complex ionomer glasses for dental use (Wilson 1980). Fluorides act as a flux and serve to reduce the fusion temperatures (Wilson 1978).

These fluorine-containing glasses always form the basis of formulations used in dentistry because a number of advantages are obtained (Wilson 1980). They are:

1. The workability and setting characteristics of the cement pastes are improved.

2. The strength of the set cements is increased.

3. The cements are rendered more translucent.

4. Release of fluoride from the cement restoration imparts a cariostatic property to adjacent tooth enamel.

The formulation of GICs can vary widely as illustrated by the vast variety of aluminosilicate glasses (Kent, Lewis & Wilson 1979) and polyacids (Crisp et al 1980) that have been prepared. This allows
considerable scope for development as suitable variations in
formulation can alter the properties of GIC such as setting time,
translucency, strength and resistance to moisture (Wilson & Prosser
1982b).

2-3-3 **SETTING REACTION**

When the glass powder and aqueous solution of polyacrylic acid
are mixed together, an immediate reaction occurs - the paste which
forms rapidly hardens into a solid mass bound by a polyclay gel
(Phillips 1982).

Wilson (1977) claims that the reaction may be represented as an
acid-base reaction between polymeric substances:

\[
\text{Glass (base)} + \text{Polyacid} = \text{Polysalt gel} + \text{Silica gel}
\]

(POWDER) (LIQUID) (MATRIX) (PARTICLE COATING)

Thus the acid (the liquid) reacts with a base (the powder) to form
a salt which, because of its polymeric nature, acts as a binding matrix.
No toxic monomers, initiators or activators are involved in the setting
reaction, unlike the setting of composite resins (Wilson 1977).

The chemical reaction taking place during the setting and
hardening of GIC has been elucidated by both monitoring the soluble ion
contents of cement pastes (Paddon & Wilson 1976; Crisp et al 1975) and
recording changes in the infra-red spectra (Crisp, Pringuer, Wordleworth
& Wilson 1974; Wilson, Paddon & Crisp 1979; Belton & Stupp 1980;
Prosser, Richards & Wilson 1982).
Although the setting chemistry appears to have been well documented, Cook (1983a) claims that the kinetics and thermodynamic equilibria involved in the setting reaction of GIC do not appear to be well understood. Cook (1983b) also claims that the chemical and infra-red measurement techniques used so far have shortcomings. He consequently used a selective chemical degradative technique to show that both Ca\(^{2+}\) and Al\(^{3+}\) are liberated in the early stages of the setting reaction, suggesting both are responsible for the initial set.

The cement-forming reaction takes place in a number of overlapping stages (Crisp & Wilson 1974a, 1974b; Crisp et al 1974; Wilson 1978; Wilson & Prosser 1982a):

1. The acid attack on the glass when ion leaching occurs, that is the extraction of ions from the glass by the acid.

2. The initial precipitation and gelation - the precipitation of cations and anions as salts.


Figure 3 shows a schematic representation of the setting process for a glass ionomer cement.
The GIC undergoes a prolonged setting reaction when compared with other dental restorative materials (Smith 1982b). Only recently has the clinical significance of these three stages in setting been clearly understood (Mount & Makinson 1978, 1982; Mount 1981, 1982).

Most evident from these studies is that the clinical manipulation of GIC is technique sensitive and that the newly mixed cement must be protected from moisture contamination by a varnish whilst the GIC matures and hardens during the first hour (Mount & Makinson 1982). Clearly, the clinical implications of this prolonged reaction must be considered if optimum results are to be obtained with GIC (Mount 1982).
When the glass powder and polyacrylic acid solution are mixed, an immediate reaction occurs (Wilson 1978). Metal ions are extracted from the particles of glass and form insoluble metal salts that lead to gelation. Subsequently, hardening and hydration proceed (Mount & Makinson 1982). Clinically, the initial gelation is identified as the setting time (Mount 1982) which is approximately 4-5 minutes from the commencement of mix (Stokes 1980). In the early stages, the presence of cross-links, mainly of calcium ions, results in a set material of low strength and stiffness with a high plastic flow (Mount 1981).

The initial set appears to result from the formation of a calcium polysalt; the aluminium salt forms later contributing to the hardening and increase of rigidity in the aging cement (Wilson & Prosser 1982a). At this early stage the setting reaction of GIC is vulnerable to contamination by moisture (Mount 1981). Part of the art of formulating GICs lies in reducing this period of vulnerability to a minimum (Wilson & Prosser 1982a). Over the next twenty four hours an aluminium polycarboxylate gel is formed (Mount 1981), the precipitation action is completed and the cement becomes stable (Crisp & Wilson 1974b). However, hydration reactions appear to continue (Wilson, Paddon & Crisp 1979), the GIC becoming stronger and less plastic as it ages (Mount & Makinson 1982). Consider that the initial 60 minutes after mixing are the most critical in terms of maintaining control over the cement environment so as to optimize clinical success.

Crisp, Jennings and Wilson (1978) measured the temperature rise of a number of setting dental cements and they showed that the GICs
exhibited the lowest temperature rise during the setting reaction whilst zinc phosphate demonstrated the greatest rise.

Barry, Clinton & Wilson (1979) studied the microstructure of a glass ionomer cement using electron microscopy and related it to the setting processes. The cement-forming glass was found to be phase-separated with its structure dependent on its thermal history. Phase structure was found to affect the setting reaction as the acid attack on these glasses is selective and most pronounced at the interface between the droplet and main phases. In the setting process, there is rapid leaching of calcium ions from this interface followed by a slower release of aluminium (and also calcium) from the main glass phase. In the fully hardened cement, it was found that aluminium polyacrylate predominated over calcium polyacrylate in the matrix.

Figure 4 shows a diagrammatic representation of a set glass ionomer cement.

![Diagram](image)

**Figure 4:** A diagrammatic representation of a set glass ionomer cement
The salt hydrogel that is formed in the aqueous phase of the cement acts as a matrix that binds the partly reacted glass particles together. The larger particles have an unattached glass core surrounded by an outer zone of silica gel, the zone from which ions have been removed by acid attack.

Wilson (1977) believes that the graded structure between the reinforcing particle and matrix acts as a stress-relieving buffer. Wilson and Prosser (1982a) consider the microstructure of the set GIC as superficially akin to a composite filling material. The glass particles act as a filler but unlike a composite material, the filler particles have actually taken part in the reaction.

The chemical nature of the matrix consists of calcium and aluminium polyacrylates (Wilson & Prosser 1982b). Infra-red studies (Crisp et al 1974) indicate that while calcium links are purely ionic, those of aluminium have some covalent nature. Fluoride is presumed to adopt the role of ligand to the metal ions (Wilson 1974).

Wilson and Prosser (1982b) claim that the molecular structures in the GIC have yet to be identified. However, they have postulated as to these molecular structures, based on the co-ordination and valency requirements of $\text{Al}^{3+}$ and $\text{Ca}^{2+}$ (assumed to be six).

Understanding the setting reaction of GIC forms a basis for identifying the mechanisms by which this cement can adhere permanently to reactive or polar substrates such as enamel apatite under moist conditions (Wilson & Prosser 1982a).
If dentine is excepted, GIC can only bond to surfaces with which it reacts chemically and which provide cations (FDI 1979). According to Wilson and Prosser (1982b), an initial wetting of the substrate surface by the freshly mixed cement paste is due to hydrogen bonding, followed by further hydrogen bonding, then followed by ionic bonding (metal ion bridges) in the set, mature condition. This situation is diagrammatically illustrated in Figure 5 and Figure 6.

**FIGURE 5**: Diagram depicting the structure of the glass ionomer cement. The solid black particles represent unreacted glass particles surrounded by the gel (dotted structure) that form when Al$^{+++}$ and Ca$^{++}$ ions are leached from the glass as a result of attack by the polyacrylic acid. The Ca$^{++}$ and Al$^{+++}$ form poly salts with the COO$^{-}$ groups of the polyacrylic acid to form a cross-linked structure. The carboxyl groups react with the calcium of the enamel and dentine.
ENAMEL

Apatite Surface

- - - - - - - H⁺ - - - - - - OOC
Polyacid chain

- - - - - - - M²⁺ - - - - - - OOC
Hydrogen bond (wetting)

- - - - - - - Ionic bond (set condition)

COLLAGEN (Dentin)

Polyacid chain

- - - - - - - COO⁻ - - - - - - M²⁺ - - - - - - OOC
Ionic bond

Collagen backbone

NH₂ - - - - - - - H⁺ - - - - - - OOC
Side-chains

FIGURE 6: The diagram illustrates postulate mechanisms for the adhesion of polycarboxylate and glass ionomer cement to enamel and dentine.

The upper diagram shows a suggested mechanism whereby the fresh, mobile cement paste wets and adheres to the enamel apatite surface by hydrogen bonds provided by free carboxylic acid groups. As the cement reaction proceeds, most of these hydrogen bonds, as represented by the arrow, are replaced by metal ions to give metal ion bridges that provide adhesion for the cement to the enamel.

The lower diagram illustrates possible mechanisms of adhesion between those cements and dentine. Collagen contains some branch chains that terminate in carboxylic acid groups and others that terminate in amino groups. The former can link to the cement mass by metal ion bridging, while the latter groups bond by hydrogen bridges (Wilson 1975).
Adhesion would appear to result from electrostatic attraction with cations sandwiched between negatively charged oxide surfaces and COO⁻ groups in the cement (FDI 1979).

Wilson (1975) has suggested that wetting of substrate surfaces by a freshly prepared glass ionomer cement paste is promoted by the presence of hydrophilic COOH (carboxyl) groups that can form hydrogen bonds to the hydroxyapatite in enamel or the pendant amine (NH₂) or carboxyl groups of collagen in dentine. This ability to form hydrogen bonds enables the GIC to wet, adapt and adhere to the polar surfaces of enamel apatite, dentine or base metals (Wilson & Prosser 1982a). Hence, adequate fluidity of the mixed GIC paste is essential to allow sufficient, available carboxyl groups necessary for this initial wetting process and subsequent adhesion (Smith 1982b). The more fluid materials for cementing show better bonding than the filling materials because of more available reactive liquid (Smith 1982a).

As the reaction proceeds, hydrogen bonds to the substrate which form the initial attachment. These are then replaced by metal ion bridges of the type: Enamel apatite O⁻ ... M²⁺ ... COO⁻ - cement (Wilson & Prosser 1982a).

These cations (M²⁺) may be derived either from the cement or the substrate - enamel, dentine or oxide - surfaces of base metals (Wilson & Prosser 1982b). The importance of calcium to the enamel-polyacrylate bond has been well documented by both chemical evidence (Mizrahi & Smith 1969a; Smith 1971b; Beech 1972a) and clear visual evidence (Eick et al 1972; Abramovich, Kalyza & Ribas 1977). Accor-
ding to Phillips (1982), the mechanism of adhesion to enamel by GIC is through the reaction of the carboxyl groups of the polyacrylic acid with the calcium in the tooth structure (see Fig. 6).

Both Beech (1973) and Wilson (1974) consider that the final bond is of ionic nature. However, recent work by Wilson, Powis and Prosser (1981) has suggested that the polyanion chains diffuse into the surface of the enamel and displace phosphate ions.

Mount (1981) states that during the first five minutes of the setting reaction, a calcium polycarboxylate gel is formed, providing the initial adhesion to tooth structure. The setting reaction continues for at least 24 hours by which time the adhesion can be considered mature.

For other multivalent ions such as Cu$^{2+}$, Al$^{3+}$ and Fe$^{3+}$ which may be present in dental alloys or chemically treated tooth surfaces, a slower rate of reaction, but stronger complex formation and specific site binding, appears to occur (Crisp, Prosser & Wilson 1976).

The mode of GIC adhesion to dentine is the subject of some controversy and conflicting results. Dentine contains both hydroxyapatite and collagen, Wilson (1974) claiming that attachment to both components is possible. However, studies by Beech (1973) indicated that adhesion declined as the dentine surface was demineralised by acids, results which support the view that adhesion is solely to the apatite constituent of dentine. However, Vougiouklakis, Smith and Lipton (1982) have shown that retention of GIC cervical restorations
on dentine may involve both adhesion and micromechanical interlocking through tubular penetration. Wilson and Prosser (1982b) conclude that GIC adhesion must be attributed to the interaction of the polar groups associated with the polymeric carboxylic acids with the polar surfaces of the substrates.

Because attachment of GIC is via ionic and polar bonds, the release of fluoride and its uptake by enamel is facilitated. The intimate molecular contact between adhesive and adherend facilitates fluoride ion exchange with the hydroxyl ions in the apatite of the surrounding enamel (McLean & Wilson 1977a, 1977b).

2-3-4 PHYSICAL PROPERTIES

2-3-4-1 Fluoride Release

Glass ionomer cements contain a high concentration of fluoride and are expected to be beneficial in terms of decreasing the enamel solubility and increasing the resistance to recurrent caries and enamel decalcification (Am. Dent. Assoc. 1979). Mount (1981) claims that apart from the adhesive potential of GIC, its ability to leach fluoride ions represents GIC's other major clinical strength. Increased enamel, dentine and cementum fluoride levels have been demonstrated (Wessenberg & Hals 1980a; Retief, Bradley, Denton & Switzer 1982; Smith, Simonsky & Lux 1982).

Fluoride tends to displace the hydroxyl group from enamel apatite which apparently increases the resistance of enamel to dissolution (McClundie & Murray 1972). The surface energy of apatite is also
reduced, making it more difficult for dental plaque to adhere to the enamel surface (Jendresen & Glantz 1980). It is also possible that the fluoride alters the metabolic activity of the plaque formed at the margins of a restoration (Norman, Mehra, Swartz & Phillips 1972). As the attachment of GIC to enamel is via ionic and polar bonds (Am. Dent. Assoc. 1979), the release of fluoride and its uptake by the enamel is facilitated. This intimate molecular contact facilitates exchange of fluoride ions with the hydroxyl ions in the apatite of the surrounding enamel (McLean & Wilson 1977a, 1977b; Crisp, Lewis & Wilson 1976c).

Barry, Miller and Wilson (1973) have shown that the matrix of GIC contains sheathed droplets of calcium fluoride which confers the ability to leach fluoride ions into surrounding tooth enamel (Crisp, Lewis & Wilson 1976c).

There have been a number of in vitro investigations (Forsten 1977; Kidd 1978; Maldonado, Swartz & Phillips 1978; Hotz 1979; Wessenberg & Hals 1980b) and in vivo trials (McLean & Wilson 1977b; Williams & Winter 1976; Williams, Price & Winter 1978) concerned with fluoride release from GIC and its possible anticariogenic effect on enamel. Dental silicate, silicophosphate and GIC all contain fluorine as an essential constituent (Wilson 1978) and have the ability to release fluoride steadily under aqueous conditions (de Freitas 1968; Crisp, Lewis & Wilson 1976c). According to Wilson and Prosser (1982b), GIC is the most effective in this respect.

Forsten (1977) has found a higher amount of fluoride release
from a GIC (ASPA) than from a silicate cement. Forsten also stated that there is an increase in the fluoride content of enamel which had been in contact with ASPA for a period of two weeks. He concluded that it may be justified to expect a cariostatic effect from the GIC.

Kidd (1978) used an artificial caries technique to assess the cariostatic effect of GIC restorations. He concluded, like Forsten, that glass ionomer cements exert a cariostatic effect on human enamel.

Maldonado, Swartz and Phillips (1978) showed in vitro that greater fluoride release occurred from GIC than from silicate and that the solubility of enamel, in apposition to the GIC, was reduced 52 per cent. The authors felt that the GIC should be as effective as silicate cement in inhibiting secondary caries.

Hotz (1979) has also demonstrated fluoride release from GIC and showed that demineralisation around GIC restorations was markedly reduced compared with fluoride-free filling materials. Hotz considered that the released fluoride may be responsible for the cariostatic properties of the material.

Swartz, Phillips, Clark and Norman (1979) used an in vitro enamel biopsy technique to demonstrate substantial increases of fluoride content in enamel adjacent to the GIC restorations. Significant amounts of fluoride uptake by the enamel occurred, both adjacent to and at some distance from the GIC, thereby decreasing the enamel
solubility. Further investigations by Swartz, Phillips, Clark, Norman and Potter (1980) and Peltoniemi (1978) indicate that GIC restorations may well provide an anticariogenic effect for the entire tooth, not just the enamel immediately adjacent to the GIC. Both surface and subsurface enamel biopsies showed a considered increase in fluoride content. Peltoniemi (1978) concluded that GIC may have potential as a long term topical fluoride agent.

Another two recent studies (Tveit & Gjerdet 1981; Retief, Bradley, Denton & Switzer 1982) support these earlier findings that GIC releases fluoride and that the enamel uptake, even at distances from the cement, can be considerable.

Sadowski, Retief and Bradley (1983) compared the enamel fluoride uptake from a zinc phosphate cement, a zinc phosphate cement containing 5 per cent stannous fluoride and a silicophosphate cement. A number of extracted teeth had orthodontic bands cemented with the above cements. These teeth were then individually suspended in synthetic saliva for 12 weeks at 37°C. The results indicated that there was a nett loss of fluoride from enamel beneath the orthodontic bands cemented with the non-fluoride-containing zinc phosphate, while the enamel beneath the zinc phosphate containing 5 per cent stannous fluoride and silicophosphate cement acquired significant amounts of fluoride.

The enamel which had acquired fluoride from the fluoride-containing cements is apparently more resistant to artificial caries attack.

When one views the results of this study (Sadowski, Retief &
Bradley 1983) in perspective with the previously mentioned investigations (Tveit & Gjerdet 1981; Kidd 1978; Forten 1977), it is not unreasonable to expect a similar anticariogenic effect from GIC, when used clinically, for orthodontic band cementation (Kvam, Broch & Nissen-Meyer 1983; Mollenhauer 1984) or bonding (Owen 1981).

A number of clinical trials have now indicated that there is little evidence of secondary caries about GIC restorations (Plant, Shovelton, Vliestra & Wartnaby 1977; Camara, Osborne, Swartz & Phillips 1978; Lawrence 1979; Saito 1977). In addition, a comparison of various fissure sealants suggests that the GIC has a cariostatic effect in vivo (Williams & Winter 1976; Williams, Price & Winter 1978).

The ability of GIC to leach fluoride and exert a cariostatic effect on enamel has major clinical implications for its use in orthodontic band cementation and for the bonding of brackets. The decalcification of enamel surfaces under orthodontic bands (Smith 1981) and around the bases of bonded brackets (Gorelick, Geiger & Gwinnett 1982) is well documented. GIC could be expected to act as a long term topical fluoride agent (Peltoniemi 1978), its effect being exerted on the entire tooth (Swartz et al 1979) well beyond the enamel beneath the orthodontic bands and adjacent to the bases of the bonded brackets. In terms of present-day knowledge, there exists little clinical rationality for the use of non-fluoride containing orthodontic cements (Smith & Maijer 1983a).

Cranfield, Kuhn and Winter (1982) reviewed the studies which have attempted to analyse the rate of fluoride release from GICs. Noteworthy was the total lack of agreement amongst the cited investigators
(Forsten 1977; Maldonado et al. 1978; Crisp, Lewis & Wilson 1976c; Tveit & Gjerdet 1981). Of crucial importance from a clinical point of view is the longevity of the fluoride release. The vigorous release of fluoride from GIC in the early periods, perhaps at a rate greater than the enamel is capable of taking up, is of relatively little value; while any suggestion that the release might dry up after a fraction of the expected lifetime of the restoration would be most disturbing. Causton (1981) has recently suggested that fluoride release from GIC ceases after about 6 months. Cranfield, Kuhn and Winter (1982) concluded that the nature of the process controlling fluoride release is not established and that the clinical importance of Causton's (1981) statement be confirmed or refuted by further studies.

Clinical trials to establish the ability of GIC to prevent or reduce enamel demineralisation beneath cemented orthodontic bands are still forthcoming. Such clinical evidence would obviate the need for prebanding enamel coatings presently being studied and promulgated (Tillery, Hembree & Weber 1976; Hughes, Hembree & Weber 1979; Younis, Hughes & Weber 1979; Zachrisson 1978b).

2-3-4-2 P:L Ratio

There is general agreement that the powder:liquid (P:L) ratio (g/ml) of GIC is critical if optimum properties are to be achieved (Crisp, Lewis & Wilson 1976b; Phillips 1982). In fact, the most common failures of GIC are due to incorrect proportioning and mixing of the liquid and powder (Am. Dent. Assoc. 1979).

The powder:liquid ratio used for a filling material is usually
in the range of 3:1 (Phillips 1982).

For luting, a P:L ratio of 1.5:1 is recommended (Mount & Makinson 1978; McCabe, Jones & Wilson 1979).

For fissure sealing, the P:L ratio should be 1.5:1 for initial fissure penetration, then 3:1 for the subsequent surface material (Mount & Makinson 1978; Williams & Winter 1976). However recently, Fuji ionomer Type III has been introduced specifically as a fissure sealant which may supersede the previous recommendation.

Crisp, Lewis and Wilson (1976b) concluded that GICs used for fillings should be mixed at the highest possible P:L ratio in order to produce cement pastes which have rapid hardening rates, high strengths and the greatest resistance to the effect of moisture. The qualifying consideration is that of working time which is reduced as the P:L ratio increases. The required working time will depend on clinical considerations appertaining to the treatment being given. However, the FDI status report on on glass ionomer cements (1979) warns that excess powder (high P:L ratio) will reduce the amount of free polyacid available which is so essential for the production of the chemical bond.

As with other types of cements, reduction in P:L ratio produces a decrease in physical properties (Phillips 1982). P:L ratio is an important parameter that affects both strength and erosion of GIC (Irie, Onozawa, Kawamura, Isogai, Nakai & Hashimoto 1978).
A low P:L ratio causes crazing (FDI 1979), increases the solubility and lowers the resistance of the set cement to abrasion (Mount 1981). In addition, working time increases with decreasing P:L ratio (Crisp, Lewis & Wilson 1977).

Both the speed of set and cement strength increase as the P:L ratio is increased, together with resistance to aqueous attack and in the case of luting GIC, the film thickness (Wilson & Prosser 1982b). Thus, for a filling material, the thickest mix compatible with adequate rheological properties is called for. In the case of a luting cement, a lower consistency has to be used in order to achieve adequate flow (Wilson & Prosser 1982b).

2-3-4-3 Working and Setting Time

Stokes (1980) demonstrated that the working time (WT) for a GIC was approximately one minute and the setting time (ST), four minutes from the commencement of mix. Mount (1982) has subsequently formulated a clinical protocol for the handling of these cements. He has recommended:

- A mixing time of 30 seconds.

- A working time of 45 seconds.

- Rest for initial set after four minutes. However, due to the prolonged setting time, the material should be protected for the next 40-60 minutes by a coat of waterproof varnish. Final set is achieved after about 24 hours.

This protocol corresponds closely to the working time determined
for a filling and luting GIC by an oscillating rheometer technique (McCabe, Jones & Wilson 1979). However, they commented that the initial set or working time assessed by this method may well be longer than if it were assessed clinically. These same investigations clearly demonstrated that the WT of a GIC can be increased by mixing on a cooled glass slab without increasing the setting time. The limiting temperature is dew point because condensation can affect the resultant cement. Recently, a rapid setting GIC known as Fuji Type II-F has been released for restoration of deciduous teeth (Telford 1983).

The initial set (working time) is due to the formation of calcium polycarboxylate (Crisp et al 1974), the final set to aluminium polycarboxylate formation (McLean & Wilson 1977a). The latter process may continue for a year or more providing maximum surface hardness (Crisp, Lewis & Wilson 1976b).

The powder composition affects setting time, the main variable being the $\text{Al}_2\text{O}_3:\text{SiO}_2$ ratio - the setting time increases into a peak ratio level above which it decreases (Kent, Lewis & Wilson 1979). Chelating comonomers are used to decrease the setting time (Wilson, Crisp & Ferner 1976).

Ideally, a GIC should combine a maximum working time with a minimum setting time, allowing for a sharp set (Wilson 1978). The clinically short working time of GIC limits its usefulness for band cementation relative to zinc phosphate cement (Kvam et al 1983).
2-3-4-4 Solubility and Disintegration

GIC is particularly susceptible to attack by water during its initial setting process (McLean & Wilson 1977a). Therefore, it is necessary to coat all accessible margins of the restoration to protect the cement from premature exposure to moisture (Mount 1981). It is possible that newer formulations may be more resistant to the aqueous oral environment (Phillips 1982); indeed, part of the art of formulating GICs lies in reducing this early period of vulnerability to a minimum (Wilson & Prosser 1982a).

The early (24 hour) solubility of GIC in water is greater than either silicate or polycarboxylate cement (Phillips 1982). Like silicate, the initial solubility is probably due to the leaching of intermediary products (Crisp, Lewis & Wilson 1976c). However, when tested in vitro, the GIC tends to be more resistant to acid attack than silicate (McLean & Wilson 1977a). Mitchem and Gronas (1978) have demonstrated in vivo that less surface loss occurs from GIC than from other types of dental cement.

Resistance to early contamination by moisture varies considerably between different GICs and is related to sharpness of set and speed of hardening which has been improved (Wilson & Prosser 1982a).

Water solubility increases with decreasing P:L ratio and the amount of material which can be water-leached is a reflection of setting time (Crisp, Lewis & Wilson 1976b).

McCabe, Jones and Wilson (1979) found that the solubility of GIC
increases significantly with agitation.

The tendency for early cement dissolution following moisture contamination necessitates good saliva control during the cementation of orthodontic bands, particularly with hypersalivating adolescents. They may well indicate the use of antisialagogues for optimal results (Zachrisson 1977a) although one could envisage the orthodontic band acting as a protective matrix during the setting phase of the GIC.

2-3-4-5 Strengths

The transverse, compressive and tensile strengths of GIC have been determined by various in vitro investigations (McCabe, Jones & Wilson 1979; McLean & Wilson 1977a; Crisp, Lewis & Wilson 1976a). These strengths have been compared with silicate, polycarboxylate and zinc phosphate cements (Wilson 1976).

McLean and Wilson (1977a) report that half the 24 hour compressive and tensile strengths are developed within one hour, which has been confirmed by others (Crisp, Lewis & Wilson 1976a). Crisp et al (1976a) report a considerably greater compressive strength in one year old specimens, probably the result of increasing cross-links with time. Specimens stored in water reached a maximum strength whereas those in paraffin did not. The clinical environment in near-neutral oral fluids is somewhere between these two storage conditions.

McCabe, Jones and Wilson (1979) evaluated the strength of a GIC luting and filling material (ASPA). The transverse strengths were 11-15MPa, somewhat lower than a silicate cement and considerably less
than a composite resin (85MPa) assessed under similar conditions (Jones, McCabe & Spence 1977).

Wilson & Prosser (1982a) tabulate the 24 hour compressive and diametrical tensile strengths of both glass ionomer filling and luting cements. The filling materials have almost twice the tensile strength of luting cements because of the higher P:L ratio. However, the compressive strength of the filling material is only slightly greater than that of the luting cement. As with all types of cements, reduction of P:L ratio produces a decrease in physical properties (Wilson 1978).

The glass ionomer luting cements show high compressive strengths but lower tensile strengths than zinc polycarboxylate cements (Jones, Sutow, Peacocke & Milne 1979). The hardness of the two types of cements is similar (Crisp, Lewis & Wilson 1976b). Although the P:L ratio and early compressive strength of GIC are less than silicate cements, the setting time, tensile strength and solubility in water are comparable (Am. Dent. Assoc. 1979).

Wilson (1978) indicates that the 24 hour tensile strength of silicate, polycarboxylate and GIC are similar but greater than zinc phosphate cement. However, silicate has the highest 24 hour compressive strength, with GIC greater than both polycarboxylate and zinc phosphate cements.

Compressive strength increases with increasing P:L ratio (Crisp, Lewis & Wilson 1976b). Both compressive and tensile strengths in-
crease with increasing polyacid concentrations (Crisp, Lewis & Wilson 1977) and molecular weight (Wilson, Crisp & Abel 1977). The tensile strength is highest when the liquid contains poly (acrylic, itaconic) acids (Crisp & Wilson 1974b). Both Mount (1982) and McCabe et al (1979) stress that the tensile strength of GIC is low, thus the material should not be used in clinical situations where high stresses are applied.

2-3-5 CLINICAL APPLICATIONS

Recommended clinical usages for GIC have been described in several studies (McLean & Wilson 1977b, 1977c; Mount 1981; Telford 1983) but the earlier applications were primarily concerned with the restoration of cervical erosion lesions (McLean & Wilson 1977c; Wilson & Kidd 1978; Lawrence 1979) and the sealing of pits and fissures (McLean & Wilson 1974; Williams & Winter 1976). Subsequent clinical experience and published reports (Mount & Makinson 1978, 1982; Mount 1981, 1982; Williams & Winter 1981; Kvam et al 1983; Telford 1983) suggest other functions based on the chemical marginal seal (Fuks, Hirschfield & Grajower 1982) and fluoride release (Cranfield et al 1982) provided by these materials, neither of which is available with composite resin.

According to Mount (1982), the major limitation to clinical success is a lack of tensile strength (Lipton & Smith 1977), as with all cements, which means that GICs are rather weak in thin sections and need to be supported by remaining tooth structure. This requirement for bulk means that the material should be used with a minimal thickness of 1mm (McLean & Wilson 1977c) and in areas not subject to occlusal loading that may produce shear forces (Mount 1981). These provisos virtually define the clinical contra-indications for GIC.
McComb (1982a) has cited these as restoration of Class IV and Class II cavities, restoration of large areas of labially exposed enamel and restoring large cusp areas.

GIC has been recommended for a wide range of clinical situations. These could be listed as:


7. Repair of defective gold inlay/crown margins (Mount 1982).


The restoration of erosion lesions, commonly found in mature adults (Sognnaes, Wolcott & Xhonga 1972) has always posed problems as the removal of a considerable amount of healthy tooth structure is undesirable. GIC has proven clinically to be an appropriate material for such restorations (Flynn 1979; Low 1981; Mitchell 1982). McLean & Wilson (1977c) have reported a clinical retention rate of 91 per cent over three years and Mount and Makinson (1978), a retention rate of over 99 per cent in one year. All the authors stress the importance of careful clinical technique when placing these restorations. Mitchell (1982) claims that the more recent Chemfil (A.D. International) has improved the lack of abuse tolerance evident with the earlier GIC (ASPA).

The American Dental Association Council of Dental Materials, Instruments and Equipment (1981) has outlined minimal acceptable clinical performances for an adhesive restorative such as GIC. This equates to a minimum of a 95 per cent retention rate over a period of six months using a sample of not less than 50 restorations. Retention should not be reliant upon undercutting, pinning or the geometric
design of the prepared tooth.

The adhesive character of GIC means that it can be used to fill deciduous teeth with the minimum of drilling and therefore, trauma (Telford 1983). Plant, Shovelton, Vliestra & Wartenby (1977) restored deciduous teeth with GIC, the clinical trials indicating that GIC handled well, but the setting time was considered excessive. These restorations were again assessed some 6-13 months after placement by Vliestra, Plant, Shovelton and Bradnock (1978) - a high success rate with 75 per cent of the restorations remaining intact. Telford (1983) claims that GIC is particularly useful for the restoration of pulpotonised deciduous molars which tend to be brittle and prone to cusp fracture. There is a growing awareness amongst the rank and file practising general dentist that indeed, GIC offers some unique advantages over the composite resin restorative materials (Miller-Yardley 1979; Millman 1979).

The nature of GIC suggests that it meets the requirements for a fissure sealant (Gwinnett & Smith 1982). It adheres to tooth structure and acts as a permanent fluoride gel releasing topical fluoride (Peltoniemi 1978), the assumption being that the adhesion will seal pits and fissures from the oral environment and that the high level of leachable fluoride from the GIC has affected the enamel in the region of the pit so as to render it less susceptible to carious attack (Gwinnett & Smith 1982). A number of clinical trials (McLean & Wilson 1974; Williams & Winter 1976, 1981) have indicated that GIC is effective for this purpose. The newer luting GICs are more effective as fissure sealants due to their flow and fine grain size (FDI 1979).
Wilson and Prosser (1982b) claim that GIC is as good as the best resin-based sealant for fissure sealing. However, long term clinical studies by Williams and Winter (1981) and Ripa (1980) clearly indicate the superiority of BIS-GMA based fissure sealants relative to GIC. Lacefield, Reindl and Retief (1982) have shown that GIC exhibits a high marginal leakage. These studies may question the rational use of GIC in lieu of resin-based sealants.

In addition to their usefulness as restorative materials and fissure sealants, GICs have been recommended as potential luting agents (Wilson, Crisp, Lewis & McLean 1977; Reisbick 1981; Bogopolsky 1981). Although these fine-grained, luting GICs are of considerable interest, the American Dental Association Status Report (1979) recommends that these materials be used cautiously until clinical trials have established their permanency. There may be significant differences in behaviour between these luting materials because of differences in composition (Smith 1982b). According to McLean and Soed (1976), GIC is particularly suitable for the luting of porcelain inlays, crowns and bridges because retention can be significantly improved by bonding porcelain to tin-plated platinum foil. However, Grieve and Glyn-Jones (1981) have recently shown that tin-plating gold inlays did not confer any additional benefits on the sealing properties of these materials.

It has been recommended by the American Dental Association Council on Dental Materials and Devices (1978) that crowns be vented when cemented with GIC to ensure a well-sealed restoration. Krupp, Caputo, Trabert and Standlee (1979) have shown that GIC offers no advantage over zinc phosphate or carboxylate cements for post retention.
However, McComb (1982b), in contrast to these earlier findings, demonstrated that a GIC luting agent (Fuji ionomer Type I) significantly improved the retention of cast-gold inlays when compared with a zinc phosphate and silicophosphate cement. Zmener (1981) has undertaken preliminary experiments cementing endosseous endodontic implants with GIC. McComb (1982b) comments that together with the advantages of low film thickness, high compressive and tensile strengths, high resistance to creep, fluoride leach, resistance to acid attack and chemical bonding, the glass ionomer luting cements show a potential advance over present cements.

Finger (1983), in a thorough and comprehensive laboratory assessment of GIC as a luting agent, concluded that the cements evaluated (Chembond, Fuji ionomer and Ketac-Cem) were clinically acceptable luting materials. The GICs were assessed relative to zinc phosphate and silicophosphate luting cement. Reisbick (1981) has also concluded that Chembond possessed physical and working qualities compatible with clinical practice.

Clinically, the major criticism of GICs have been opacity and tendency to chalkiness with early moisture contamination (Causton 1981). The more recently developed GICs have now been made sufficiently translucent to match tooth enamel (Wilson & Prosser 1982b). Early moisture contamination can be readily avoided by controlling the environment of the GIC after initial setting with an immediate application of waterproof varnish (Mount 1981). Within an hour of placement, the GIC will be sufficiently resistant to absorption of water to remain undamaged if the varnish is lost (Crisp, Lewis & Wilson 1980).
Further criticism of GIC has come from Smales (1981) who found that ASPA (an early version of GIC) retained the most plaque and had the roughest surface when compared with a range of other restorative materials including composite resin. Smales and Joyce (1978) previously demonstrated that ASPA possessed poor abrasion resistance when compared with a composite resin (Concise). These shortcomings have been largely overcome with the more recently introduced GICs (Smith & Maijer 1983a).

Dijken, Meurman and Järvinen (1980), using an electron microscope, have clearly demonstrated that GIC (ASPA) has a far rougher surface texture than microfilled composite resins after finishing procedures using fine abrasives. These findings support the later studies by Smales (1981).

Kvam, Broch and Nissen-Meyer (1983) have assessed the clinical suitability of GIC for the routine cementation of orthodontic bands by a comparison with zinc phosphate cement in a controlled clinical trial. They concluded that a lack of good manipulative characteristics (short setting time, moisture susceptibility and difficulty in removing excess cement) limits the use of GIC for routine band cementation. However, both McCabe, Jones and Wilson (1979) and Stokes (1980) have emphasised the advantages of using a chilled glass slab for cement preparation as this will prolong the working time without unduly extending the setting time. In contrast, the liquid must be kept at room temperature because chilling increases the viscosity (Mount 1981).
Kvam and his colleagues (1983) also found that GIC was less soluble than zinc phosphate cement in the mouth. These results agree with previous clinical studies on the solubility of GIC luting agents by Mitchem and Gronas (1978, 1981). Kvam and his co-workers have recommended GIC for the cementation of appliances that are to remain in place for an extended period of time, or are under exceptional strain, and for cementation of bands to teeth with unusual crown morphology. It is noteworthy that neither the type (filling or luting), brand nor P:L ratio of the GIC used in this study was mentioned.

Mollenhauer (1983) usually cements upper molar bands with GIC if he selects headgear therapy as he has found that there is less band dislodgement compared with zinc phosphate cement. This supports the opinion of Kvam et al (1983) that GIC is more suited to the cementation of appliances under particular mechanical strain. Telford (1983) states that where stainless steel crowns or orthodontic bands are required in children's dentistry, GICs are excellent luting agents.

Goto, Atachi and Otani (1981) have indicated that there exists a definite relationship between the film thickness of a GIC luting agent and the degree of retention of a crown. When the cement film thickness was reduced below a critical value, the retention increased markedly and the cement was not affected by the environment. This would indicate the necessity for closely adapted orthodontic bands or bonding bases with subsequent minimal cement thickness, maximum retention and resistance of the GIC to the oral environment.

Similarly, Mesu (1979) demonstrated that a thin layer of luting
GIC (20μm) was found to be far less susceptible to degradation by lactic acid than a thicker cement film (100μm). This again emphasises the clinical importance of well adapted orthodontic bands and bonding bases to provide a minimal cement film thickness.

The following aspects of technique have been recommended in handling the glass ionomer cement (Wilson, Crisp, Lewis & McLean 1977; McLean & Wilson 1977b, 1977c; Am. Dent. Assoc. 1981; Mount & Makinson 1978; Mount 1981, 1982): Dispense the powder and liquid precisely, do not expose the dispensed liquid to the atmosphere prior to mixing, mix on a refrigerated glass slab using chilled powder to prolong working time, avoid contamination of the cement by water during and after placement by using a matrix before set and a coating of varnish after set, avoid contamination tooth surfaces and use a clean surface with a suitable conditioner. Failure of the adhesive bond occurs if cavity cleansing is inadequate or if contamination is allowed; if the cement is applied too late when its surface gloss has disappeared, or if there is carelessness in removing the matrix or in finishing.

According to Mount (1982), the GICs in their present form are probably the first phase of a new restorative system. In addition, abrasion resistance is adequate for the currently recommended applications but the compressive strength and tensile strength must be upgraded before these materials are adequate for the restoration of occlusal surfaces. Further, improvements in the adhesion of GIC to tooth material will be achieved only when the cohesive strength of the material is increased (Powis, Polleras, Merson & Wilson 1982).
It is clear that long term clinical trials are required to establish the viability for the multiplicity of uses to which GIC has been applied (Am. Dent. Assoc. 1979).

2-3-6 **BIOCOMPATABILITY**

The biological evaluation of GIC, due to the recent development of these cements, is still being compiled and only a small number of studies have been reported to date (McComb 1982a). GIC represents the first step in an advance towards a truly compatible material and is amongst the least irritant of all dental cements as it contains preformed polymers rather than monomers - setting is by ionic cross linking and not by polymerisation (Wilson & Prosser 1982a). Both the silicate cement and composite resins suffer from the disadvantage that before fully hardened, they contain irritant substances - either strong acids or unsaturated monomers and polymerisation agents (Wilson & Prosser 1982a).

The biological compatibility of GIC has been assessed by three distinct approaches (Helgeland 1982); pulp and cell culture studies along with clinical trials. The majority of these studies has been involved with the effects of GIC on human or animal dental pulps. The major studies to date are:

2. **Cell culture studies** (Dahl & Tronstad 1976; Kawahra, Imanishi & Oshima 1979).


The available evidence from in vivo and in vitro tests (Tobias et al 1978; Yakushiji et al 1979; Pamijor et al 1981) including clinical experience with cervical restorations (Mount & Makinson 1978; Garcia & Charbeneau 1981) suggests that pulp and gingival response to GIC is likely to be mild. McComb (1982a) agrees that tissue compatibility appears good, especially if measures are taken to protect the pulp where the lesion is deep. Kawahra and his colleagues (1979) recommended GIC for wide use as a biomaterial in the medical field, despite the fact that human in vivo testing was not fulfilled in this study.

Helgeland (1982) notes that different methods for in vitro evaluation of the biocompatibility for dental cements differ widely in experimental design - it is therefore expected that varied results will be obtained. Different brands of GIC may also differ in composition (Phillips 1982), as for example fluoride content, which may be of importance for the cytotoxicity observed (Helgeland 1982).

According to Smith (1982c), any effects arising from differences in fluoride release between brands and the possible effects of the aluminosilicate glass ingredients and tartaric acid concentration have yet to be clarified. He therefore states that additional in vitro, in vivo and clinical evidence is required to assess the biocompatibility
of GICs. Martin (1980) also stresses that long-term clinical studies are necessary before any assertion that GIC is completely bland to the pulp can be made.

The tissue compatibility (Dahl & Tronstad 1976) and lack of gingival response to GIC (García, Caffesse & Charbeneau 1981) further vindicate the clinical use of this polyacrylate cement in orthodontics. Indeed, Davidson, Sheinis and Shepherd (1982) stress the importance of biocompatibility of orthodontic bonding adhesives (similarly band cements) as they may come into contact with tissues other than the enamel of the patient's tooth. These tissues may include skin, oral mucosa and gingiva in routine bonding and banding procedures. The patient, orthodontist or chairside assistant may be contacted directly by any or all of these orthodontic adhesive or band cement components. Similarly, Terhune, Sydiskis and Davidson (1983) have demonstrated in vitro toxicity of orthodontic bonding sealants.

2-3-7 CONCLUSION

At this time, the exact role(s) that GIC may ultimately fulfil in dentistry and orthodontics is unknown. The cement has a number of characteristics that are clinically attractive. It is adhesive under oral conditions to enamel (bond strength 4MPa), dentine (bond strength 3MPa) and base metals including stainless steel (Wilson 1980). They do not bond to the inert surfaces of noble metals and porcelain (McLean & Wilson 1977a). Adhesive bonding almost equals the tensile strength of GIC (Wilson & Prosser 1982a), therefore future improvements in bond strength will occur only when the cohesive strength of the cement is increased (Powis et al 1981).
GICs are translucent and can be made to match tooth enamel optically (McLean & Wilson 1977a). They are also considered to be bland to pulp (Plant 1977; Rowe 1977; Kawahara et al 1979) as well as biocompatible (McComb 1982a).

Significantly, fluoride release from the cement protects adjacent enamel against caries (Swartz et al 1980) and possible demineralisation beneath loosened orthodontic bands and around bracket bases (Tveit & Gjerdet 1981). In fact, orthodontic band cementation and bonding with GIC could well represent a vital component in a comprehensive preventive programme during the duration of fixed appliance therapy. Numerous investigators (Zachrisson 1973, 1976, 1978a, 1978b; Hirschfield 1978) have shown that low concentration fluoride mouth rinses used daily during orthodontic treatment can be a highly effective caries preventive measure. GIC can be considered akin to a topical fluoride agent (Peltoniemi 1978).

The GICs have the drawback of being weaker than composite resins (Naijer 1982) and in demanding more skill in preparation and manipulation (Mount 1982). The use of polyelectrolyte based materials in dentistry has much to recommend it, as aqueous-bonded, hydrophilic materials seem more appropriate in biological systems than the hydrophobic organic polymers (Smith 1982a).

Belton & Stupp (1980) consider that due to a number of shortcomings, the clinical use of GIC is limited, despite its attractive qualities. They believe that further research is needed to maximise the chemical adhesion in the oral environment over long periods of
time, upgrade the mechanical strength and decrease the sensitivity of the setting process to moisture.

The most recent generation of GICs have seen marked improvements in handling characteristics and properties since the materials were introduced (Smith & Maijer 1983a). The eventual scope of useful applications for GIC in both general dentistry and orthodontics will be dependent upon further improvement in formulations and a more complete definition of its clinical performance (Phillips 1982).
### CHAPTER 3  

**COMPOSITE RESIN BONDING SYSTEMS IN ORTHODONTICS**

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CHAPTER 3 COMPOSITE RESIN BONDING SYSTEMS IN ORTHODONTICS

3-1 INTRODUCTION

The combination of acid-etching and bonding has led to dramatic changes in the practice of orthodontics (Fields 1982). Since the introduction of the acid-etch technique (Buonocore 1955) and its adaptation to orthodontic bonding (Newman 1965a, 1965b), the literature has indicated the numerous clinical and research studies on the subject. The simple yet innovative acid-etch bonding technique has been adopted, refined and applied to meet particular needs of various dental disciplines (Gwinnett 1982).

In 1979, Gorelick found in a survey that 93 per cent of the respondent orthodontists were bonding orthodontic attachments as a clinical routine. The traditional orthodontic band is being used less frequently on anterior teeth. Changes have been rapid in the relatively short history of orthodontic bonding (Newman 1982). Resin bonding is now being used for the placement of attachments and various types of retainers (Zachrisson 1977b), including resin build-ups that address tooth size and shape problems encountered during orthodontic treatment (Fields 1982). Although direct bonding is only in its infancy, the technique has had a tremendous impact on orthodontic treatment (Maijer 1982).

Successful bonding in orthodontics has been described as multifactorial (Smith & Maijer 1983a), requiring a meticulous clinical technique, a satisfactory adhesive system and compatible bracket design.
Both direct and indirect bonding techniques have been developed and proved to be clinically effective (Schimann 1978). Various types of orthodontic attachments are available - plastic, metal, metal reinforced plastic or ceramic (Maijer 1982) along with different bases - mesh, sintered or solid undercut (Rogers & Griffith 1977; Maijer & Smith 1981; Smith & Maijer 1983b). The demands of the new era of lingual orthodontics on resin bonding systems are onerous and have yet to be fully assessed (Scholz & Swartz 1982).

The arrival of bandless orthodontics has not been without clinical problems. Bond failure rate can be unacceptably high if strict clinical technique is not followed (Wertz 1980). Debonding - the removal of the bonding resin from the enamel surface following treatment - is still very much a problem for the orthodontist (Artun & Bergland 1984). The debonding and subsequent clean-up procedure at the end of treatment can be tedious and time-consuming (Brobakken & Zachrisson 1981). Present bonding techniques have the potential to induce iatrogenic effects including damage, decalcification and staining of the enamel surface (Zachrisson 1978a, 1978b; Jones 1980; Ceen & Gwinnett 1980a, 1981; Bennett, Shen & Waldron 1984).

This review will discuss the use of resin bonding in respect of the placement of orthodontic attachments and the critical variables related to this procedure. Those areas of orthodontic resin bonding that are presently causing clinical concern will be highlighted. Hopefully, this may be viewed in perspective relative to the merits and shortcomings of GIC as a potential orthodontic bonding adhesive.
3-2 DEFINITION - ADVANTAGES AND DISADVANTAGES OF BONDING

Maijer (1982) has defined bonding in the orthodontic context as the attachment of a dental resin to enamel by means of a mechanical or physicochemical process. Orthodontic bonding involves attachment of the filled or unfilled dental resin, not only to enamel but also to the orthodontic attachment by means of a mechanical interlock.

Initially, the bonding of orthodontic attachments was heralded for its numerous advantages (Slodov & Armbrrecht 1974; Brandt, Senoss & Wolfson 1975; Reynolds 1976; Sheykholeslam & Brandt 1977). According to Fields (1982), these advantages are specific for different treatment phases. During the initial placement of the appliance there was less patient discomfort since separation and band seating were eliminated. Arch length was not increased by band material. Partially erupted teeth can be bonded and aberrant tooth shape did not result in difficult banding and post-attachment position. These advantages resulted in less chair time for the dentist and patient. Bonding also eliminates the need for a large inventory of bands (Maijer 1982).

During the active treatment phase, the appliance was more aesthetically appealing than the conventional band. With the lack of band material, interproximal radiographs can easily detect incipient interproximal decay (Fields 1982). In the event that the brackets come loose, the patient is immediately aware of this and can notify the office, whereas sticky bands will often go undetected by the patient until he returns for his regular appointment.

Both Fields (1982) and Reynolds (1976) claim that bonded
attachments provide better access for cleaning, especially at the gingival margin. Plaque will inevitably grow peripheral to and particularly gingival to bonded brackets (Gwinnett & Ceen 1979). The presence of bonded brackets or orthodontic bands is unlikely to have any bearing on plaque growth. However, Zachrisson (1977a) has shown that flossing interproximally with brackets in place is possible, particularly if aids are used. This proximal access must be weighed against the loss of the interproximal caries protection mechanisms afforded by a well-fitted cemented band (Zachrisson 1978a). Consequently, Zachrisson (1978a) routinely bands all first molars.

When treatment is completed, there is no band space to close and tooth size problems are more easily addressed by stripping (Barrer 1975; Tuverson 1980; Vickers 1982) or resin build-ups (Fields 1981) since interproximal areas are accessible.

Several disadvantages soon emerged although Brandt et al (1975) found it difficult to enumerate these, claiming that perhaps attention to technique detail and bond failure were the only two noteworthy problems. Fields (1982) notes, with the benefit of subsequent clinical experience, a more extensive array of problems. Careful isolation of teeth during bonding was necessary to eliminate salivary contamination. Moisture control also made the bonding of partially erupted teeth exceedingly difficult. Some bonding agents were not sufficiently strong while others would not bond to polycarbonate brackets. Better access for cleaning did not necessarily guarantee better hygiene, especially if resin was allowed to extend beyond the bracket base. Gwinnett (1982) stresses that every attempt should be made to feather the margins at the
bracket base periphery to minimise plaque accumulation.

Finally, there is lack of agreement (Gwinnett & Gorelick 1977; Zachrisson 1977b; Whitehill-Grayson 1981; Retief & Denys 1979) concerning the best method for removal of resins from the tooth surface following treatment. There is little understanding of the ramifications of any remaining resin (Fields 1982). In addition, depending on the bonding material used, debonding time can be significantly greater than debanding time (Maijer 1982).

Many of the problems are being tackled with the development of new bonding techniques (Maijer & Smith 1979), the assessment of debonding instrumentation (Zachrisson & Artun 1979; Bennett, Shen & Waldron 1984) and rebonding procedures (Perry 1980). It also appears that the recycling of orthodontic attachments is now a clinical reality (Buchman 1980; Moore 1984).

3.3 ENAMEL PREPARATION

The effectiveness of orthodontic bonding depends on several parameters which include (Dogon 1981): The material used to clean the surface of the tooth before etching; the effectiveness of the etchant; the chemical and physical nature of the tooth enamel itself; the area of the surface of the enamel etched; and the properties of the resin materials used to provide the sealing and attachment of the bracket to the teeth.

Hence, in preparing for the mechanical bond between enamel and orthodontic attachment, it is necessary to clean the enamel surface
with pumice (Maijer 1982). This cleaning process eliminates the acquired pellicle which consists of salivary proteins and which acts as a protective film that minimises changes in acidity in the enamel environment (Gwinnett 1976). The importance of the dental prophylaxis prior to etching has been emphasised (Miura, Nakagawa & Ishizaki 1973; Gwinnett 1981). It has been clearly demonstrated that maximum bond strengths are obtained only when a prophylaxis was given followed by acid conditioning (Gwinnett 1982).

3-3-1 ETCHING

Following prophylaxis, etching of the enamel surface takes place. Buonocore (1981b) defines *Proper Acid Etching* as the achievement of maximum potential bonding area per unit of surface enamel.

Phosphoric acid in the form of a gel or solution is considered the present etching agent of choice (Gwinnett 1981). A host of scientists have investigated the action of etchants (Gwinnett 1981; Aranè 1974; Retief 1973). According to Buonocore (1981b), phosphoric acid in concentrations ranging from 30-50 per cent seem not only to be favoured but also optimal for achieving long-lasting bonding in clinical situations. Buonocore's personal preference, based on more than ten years of laboratory and clinical evaluations, is for a 50 per cent by weight phosphoric acid solution buffered with 7 per cent zinc oxide. Soetopo, Beech and Hardwick (1978) found that maximum enamel/resin bond strength was obtained with a phosphoric acid concentration of 10-30 per cent. These results conflict with Gwinnett (1982) who cites studies by Rock (1974) and Mitchem and Turner (1974) that indicate that the lowest acceptable concentration of phosphoric acid
is approximately 30 per cent. Chow and Brown (1973) have shown that with concentrations lower than 30 per cent an insoluble crystalline reaction product may form on the enamel surface and occlude the porosities important to resin retention. Soetopo et al (1978) refer to these reaction products as deposition of primary and secondary calcium phosphates. Currently available commercial bonding systems contain phosphoric acid solutions or gels in concentrations ranging from 37-65 per cent (Gwinnett 1982).

When acid solutions are applied to clean enamel surfaces, dissolution of the large inorganic component occurs (Gwinnett & Smith 1982) which in turn, gives rise to a significant increase in surface area (Dogon 1981). Such dissolution is irregular and occurs at preferential sites related to the cores or peripheries of enamel prisms (Gwinnett 1971).

The preferential dissolution pattern which results may be influenced by enamel crystallite orientation (Sharpe 1967; Nichol, Judd & Ansell 1973), the presence of organic material at various prism sites (Johnson, Poole & Tyler 1971), the presence of trace elements in diffusion pathways (Poole & Johnson 1967), the time of exposure to acid (Wichwire & Rentz 1973), the concentration of the acid (Gwinnett 1971) and the type of acid solution used (Ohsawa 1972).

The acid conditioning process creates or enhances existing enamel porosity (Gwinnett & Smith 1982). A resin of proper surface tension, viscosity and affinity for the high energy etched enamel is attracted into and fills these voids in the enamel to produce the so-called
resin tags intimately attached to the enamel (Buoncore 1981b). It is these tags that provide a mechanical interlock resulting in long term retention in the moist oral environment (Beech & Soetopo 1977; Soetopo et al 1978).

Recent studies have indicated that resin tag lengths of 150-200 microns and in some cases up to 300 microns exist (Xu Heng-Chang, Wang Tong & Wang Hungying 1983).

Buoncore (1981b) has defined the ideal etch as one that consistently produces the highest and most durable bond strengths in both laboratory and clinical tests. He claims that the length and number of tags and the extent of prism dissolution required to achieve this are not fully known at present. Factors which may influence the depth and pattern of etching are the presence of prismless enamel (Ripa, Gwinnett & Buoncore 1968) and fluoride treatments (Sheykholeslam, Buoncore & Gwinnett 1972). Bond strengths may be detrimentally affected when fluoride agents are applied before (Lee, Stoffey, Orlowski, Swartz, Ocumpaugh & Neville 1972) or after (Sheykholeslam et al 1972) etching treatments. One fluoride treatment, stannous fluoride, applied after etching may enhance bonding (Low, von Fraunhofer & Winter 1977).

An ideal etch is obtained by a combination of three variables (Bates, Retief, Jamison & Denys 1982):

1. Optimum phosphoric acid concentration
2. Duration of etch
3. Washing time
Brandt et al (1975) believed that improper etching was probably the most single important factor causing bond failure. However, Dogon (1981) believes that contamination of the etched enamel surface by water, saliva, blood or any other agent before placement of the resin is the chief cause of bond failure. Failures at the resin enamel interface are a good clinical indicator of contamination of the etched enamel surface prior to bonding (Gwinnett 1982).

A properly etched surface has a frosty, whitish appearance (Dogon 1981) and in orthodontics, the complete facial surface should be etched (Silverman & Cohen 1975; Zachrisson 1978b). A range of etching and wash times have been recommended for clinical success (Soetopo et al 1978; Williams & von Fraunhofer 1977; Silverstone & Dogon 1976; Beech & Jalaly 1980; Bates et al 1982). According to Gwinnett (1982), 10-15 seconds per quadrant seems generally acceptable clinically. Washing for twice that time should be done when a gel is used. However, studies by Bates et al (1982) showed that the tensile bond strengths to enamel surfaces etched for 30, 60 and 120 seconds were not significantly different. They claim that only a 5 second wash was required after acid etching to maintain bond strength. Saliva contamination of etched enamel surfaces was effectively eliminated by a 5 second wash without resorting to re-etching of the enamel surface.

Maijer (1982) warns of the potential dangers of phosphoric acid etchant. This acid is strongly acidic and if it is allowed to run on to the gingival or touch the lip it may give rise to a severe burn of these soft tissues. For this reason, some orthodontists prefer gel
etchants whereby placement control is enhanced.

Finally, strict moisture control is imperative to prevent contamination of the etched high-energy enamel surface. A rubber dam is preferred although cotton rolls, triangular absorbent pads, evacuators of several designs and antisialogues may be considered as alternatives (Kumar 1976; Borges & Prucha 1977; Zachrisson & Brobakken 1978). Proteins from saliva to the etched enamel form an organic film that cannot be removed by water (Gwinnett 1982). Whether the contaminant is dried or remains wet significantly affects bond strength (Normati, Fuller & Deney 1980).

3-3-2 SEALANTS

There is considerable debate about the use and indication of sealants in orthodontic bonding. As most composite orthodontic resins are too viscous to penetrate deeply in between friable projections created by acid etching, it has been recommended by Dogon (1975) that an unfilled BIS-GMA material be used to make a positive seal between the etched enamel and the composite. Dogon (1975) has determined that the viscosity of the resin is of major importance to the penetrability of the material into the etched enamel. In addition, Maijer (1982) notes that the working time of many resins used in orthodontics is quite short and may not allow the composite resin matrix to flow and wet the etched enamel porosities. Hence, the effectiveness of the acid etching may be partially negated.

Studies investigating the improved adhesion when a sealant is used in connection with a composite resin lack agreement (Bishara,

Zachrisson (1978b) identifies four distinct advantages for using sealants in orthodontic bonding, each one of them, according to Zachrisson, important enough alone to merit its use:

1. Caries protection.

2. Increased bond strength.

3. Moisture control is no longer extremely important once the sealant is applied to the etched surface.

4. The debonding is facilitated since less adhesive is needed.

Newman (1981) is in complete accordance with Zachrisson (1978b) when outlining his routine clinical bonding procedure for orthodontic attachments. Newman (1981) stated that the use of the primer (sealant) tended to enhance adhesion and decrease decalcification. The low viscosity of the primer allows for maximum wetting and mechanical interlocking into the etched enamel and the primer bonds chemically to the adhesive. Zachrisson (1978b) believed that there were no disadvantages or risks in using sealants as long as the excess polymerised sealant contacting the gingiva was removed using a scaler.

However, recent findings have brought much equivocation on the role of the sealant in orthodontic bonding. Auto-polymerised
sealants used on smooth surfaces adjacent to bonded brackets have been shown to inadequately cover the enamel. As such, they do not protect the enamel against decalcification and therefore formation of white spots (Gwinnett & Ceen 1981; Zachrisson, Heimgard, Raytor & Mjor 1978).

Maijer (1982) concurred with these findings, noting that a clinical study currently in progress (Maijer, Karod & Smith 1982) indicated that placing of sealants did not necessarily eliminate the possibility of decalcification. If the sealant was placed without the protection of a composite resin, it would probably be brushed off within 1-6 months. Maijer (1982) concluded that at the time, it was still not clear whether microleakage was directly related to decalcification under the bracket.

Zachrisson (1978b) discussed the future development of sealants and believed that with further experimentation, improved sealants would probably be developed. He conceded that inhibition of polymerisation of thin films by contamination with air remained a problem, particularly when interproximal sealing for caries prevention was the goal.

Fields (1982), in contrast with Zachrisson (1978b), states unequivocally that an unfilled sealer is not necessary to achieve acceptable bond strength and does not afford protection for demineralisation adjacent to the bracket base.

Further controlled clinical studies are obviously necessary to
clarify the status of sealants in orthodontic bonding.

3-4 RESIN COMPOSITE BONDING SYSTEMS

The two basic types of polymeric dental resins currently employed in orthodontic bonding are the acrylic and diacrylic resins (Whitehill & Grayson 1981). The acrylic resins, based on self-curing acrylics, consist of methyl methacrylate monomer and ultra fine powder. The diacrylic resins, the so-called BIS-GMA or Bowen's resin, like the acrylic resins also occur in filled or unfilled form. These filled diacrylate resins are known commonly as composite resins (Smith & Peltoniemi 1982). They have excellent bond strength, lower water absorption and less polymerisation shrinkage than the acrylic resins (Maijer 1982). The disadvantage of composite resins is that they do not bond to plastic brackets as do the acrylic resins (Reynolds 1976).

The composite resin materials available for orthodontic bonding contain a variable percentage of inorganic filler particles (Smith & Maijer 1983a). They are classified as lightly filled (20-30 per cent filler) or heavily filled (60-76 per cent filler) (Gwinnett 1982); the heavily filled composites have an increase in physical properties and are recommended for posterior bonding (Gwinnett 1982).

Smith and Maijer (1983) have analysed a number of currently available; commercial composite resin orthodontic bonding adhesives. They have categorised concise (3M), Unite (Unitek) and Autotach (Caulk) as heavily filled composite resins (60-75 per cent of filler
particles), whilst Endur (Ormeo), Dynabond (Unitek) and Monolok (Rocky Mountain) are lightly filled (20-30 per cent of filler). The newer, lightly filled materials contain considerably less filler particles than those composite resins used for restorative purposes (Smith & Maijer 1983a). From a clinical viewpoint, the heavily filled resins like Concise (3M) provide less bond failures and are more suitable for posterior bonding (Jenner 1982). However, they are more difficult to debond and can cause tremendous iatrogenic damage to the enamel surface (Smith & Maijer 1983a).

Orthodontic resins have different requirements from the conventional restorative resins (Maijer 1982). The resin should not have the same hardness as conventional restorative materials since it needs to be removed in two years (Faust, Grego, Fan & Powers 1978). However, the material must have adequate strength to resist a variety of force modes used in orthodontic treatment (Keizer, Ten Cate & Arends 1976).

Maijer (1982) and Brandt et al (1975) have discussed the requirements of an ideal orthodontic bonding adhesive. The adhesive should be aesthetically pleasing and should not stain. Ideally, it should bond to both metal and plastic brackets. The resin system should have a variable setting time so that it may be used for both direct and indirect bonding techniques. It should be strong enough to resist masticatory and appliance forces yet be readily debondable causing no iatrogenic damage to enamel. The remaining resin tags in the outer enamel should not stain. Rebonding procedures should be readily accomplished and the material must be biocompatible. Maijer (1982)
believes none of the bonding systems presently available meet all of the above requirements.

Fluoride-containing acrylic resins are available for restorative purposes and have been adapted to orthodontic bonding with limited success (Newman 1969). A significant uptake of fluoride from enamel from such an adhesive system containing monofluorophosphate has been demonstrated (Newman & Cimasoni 1971).

Forsten and Paunio (1972) have measured both the fluoride contents of several commercially available composite resins and the fluoride uptake from these materials. One such composite, TD71 (Dental Fillings, England) had a higher fluoride content than the silicate cements tested. Unfortunately, there are no studies using fluoride-containing composites available for orthodontic bonding. Smith and Peltoniemi (1982) state that the nature and duration of fluoride release from composites is unclear. Although the fluoride-containing composite resin appears attractive in theory, evidence relating to the practicality of such a material is lacking.

It should be noted that resins of different composition and strength exhibit acceptable clinical performance (Alexandre, Young, Sandrik & Bowman 1981). The lower limit and type of bond strength necessary for acceptable clinical performance are unknown (Fields 1982).

The orthodontic resins can be divided on the basis of their curing systems into two categories:
1. The UV, white light or command-cure systems.

2. The autopolymerisation or self-cure system.

3-4-1 UV AND WHITE LIGHT CURE SYSTEMS

The use of ultraviolet light as an external activator of polymerisation was introduced into restorative dentistry in the early 1970s (Buonocore 1970b). This concept was soon adapted for orthodontic bonding (Slodov & Armbrecht 1974; Silverman & Cohen 1974; Garn 1976). The early popularity of this system was due to the fact that the orthodontist possessed unlimited working time, thus enabling him to position attachments accurately prior to resin activation. According to Maijer (1982), there are many problems associated with UV cured materials such as the possibility of obtaining non-uniform cure due to variations in resin thickness and UV intensity. Exposure to extended periods of UV radiation can be hazardous to the skin, oral mucosa and eyes (Mills, Lytle, Andersen, Hellman & Bochstahler 1976).

At present, UV curing systems have fallen into slight disfavour (Maijer 1982); they were popular with polycarbonate (Cohl, Green & Eick 1972) and perforated brackets, but the inaccessibility of light to the resin under mesh-back brackets has turned most clinicians towards autopolymerised resins (Fields 1982). A development which overcomes some of their disadvantages is the introduction of visible light activated materials and it seems likely this may have orthodontic applications (Smith & Maijer 1983a). Visible light appears to provide greater depth and rapidity of polymerisation even through enamel (Smith & Maijer 1983a).
3-4-2 SELF-CURING SYSTEMS

The self-curing or autopolymerising systems are presented as either liquid/liquid, liquid/powder or paste/paste forms (Maijer 1982). One part of the system contains the initiator (usually benzoyl peroxide) and the other part contains the accelerator, often a tertiary amine (Smith & Maijer 1983a).

An advantage of self-curing resins is that provided the two parts of the system are mixed properly, uniform curing will occur regardless of the thickness of the material used (Maijer 1982). Self-curing systems based on the old powder/liquid system are basically unfilled polymethyl methacrylate systems while the composite filled systems are usually in paste/paste form (Whitehill-Grayson 1981).

The polymethyl methacrylates were the first resins to be used routinely in orthodontic bonding (Reynolds 1976), having been specifically modified for use as an orthodontic adhesive. Such a material was developed by Miura et al (1974) and marketed as Orthomite IIs (Rocky Mountain Corp.) in North America. These acrylic resins are extremely effective with the use of plastic brackets as they react physically with the back of the polycarbonate bracket (Crabb & Wilson 1971). Newman, Snyder & Wilson (1968) have described the advantages of the polymethyl methacrylate system. However, Maijer (1982) notes that methyl methacrylate is volatile and has a very strong, objectionable odour including a marked polymerisation shrinkage.

To overcome some of the difficulties with acrylic resins,
materials based on BIS-GMA have been developed (Gwinnett 1982). BIS-GMA type resins are usually extremely viscous and require thinning for optimal use as an orthodontic adhesive (Artun & Zachrisson 1982). The BIS-GMA orthodontic resins require sufficient viscosity in the uncured state to hold the brackets in place, hence the need to use a two-part system (Maijer 1982). A low viscosity sealant is placed in position to give adequate penetration into the etched enamel and following this, the BIS-GMA resins to which filler particles have been added to give additional strength, are used to bond the bracket (Smith & Maijer 1983a). The BIS-GMA resins do not bond to polycarbonate (plastic) brackets and this is a definite disadvantage (Reynolds 1976).

Until recently, all bonding materials based on BIS-GMA self-cure systems have required the mixing of two pastes on a pad (Belostoky 1982). Now, several companies have introduced bonding resins in which two pastes need not be mixed but will polymerise or cure when one paste touches the other (Fried & Newman 1983). These IBS-GMA adhesive systems are known as No-Mix or Cure-on-Touch bonding adhesives in contrast with mix systems (Scholz 1983).

The non-polymerising property of these materials, unless contact occurs, permits the use of a preloaded bracket delivery system. This allows the orthodontist and assistant to work independently of each other during the bonding procedure, unlike the mixing systems where an assistant is essential (Belostoky 1982).

Although the no-mix materials were originally designed for direct bonding, they have been adapted successfully for indirect bonding in lingual orthodontics (Scholz & Swartz 1982).
DECALCIFICATION ABOUT BONDING SITES

One of the major concerns in orthodontic bonding is the risk of decalcification about or under the bonded orthodontic brackets (Maijer 1982). In fact, according to Phillips (1980b), decalcification around the bracket pad is the only significant problem that remains in orthodontic bonding. One of the arguments put forward in orthodontics for the use of bonded brackets instead of metal bands is the greater ease of oral hygiene and reduced risk of decalcification (Williams 1982). Bonded brackets do not completely solve the problem however and plaque still accumulates. This has been well demonstrated on brackets and on some of the resins used to bond them, even in patients with good oral hygiene (Gwinnett & Ceen 1979).

Enamel decalcification or white spot formation has been described as occurring in two locations relative to the bonded bracket - either under, or about, the bracket pad (Williams 1982). The relative incidence of each has not been documented.

A few studies have reported the overall incidence of white spot formation during banding and bonding procedures. A 33 per cent incidence in 30 banded maxillary lateral incisors has been reported (Hirschfield & Johnston 1974). In a comparison of banded and bonded cases, a comparable incidence of white spot lesions was found (Gorelick, Geiger & Gwinnett 1982). The highest incidence was on the maxillary central incisor of approximately 17 per cent for banded and 21 per cent for bonded teeth. By segment, the highest incidence was on bonded molars and premolars (15.3 per cent). According to Maijer (1982), no long term data is available on the incidence of decalcifi-
casion seen in orthodontic bonding.

Decalcification about the bonded bracket follows a U-shaped configuration bordering the cervical portion of the bonded area, sometimes extending into the embrasures (Gwinnett 1982). Bonded brackets do not prevent the problems created by poor oral hygiene, with accumulation of plaque and food debris peripherally and gingivally to the bracket base, producing a potential for decalcification (Gorelick 1977).

Decalcification occurring under bonded brackets is not frequently seen and is probably due to failure or microleakage at the enamel/resin interface allowing percolation of oral fluids and cariogenic bacteria (Silverman, Cohen & Gwinnett 1979).

Sealants and glazes are applied in anticipation of preventing white spot formation (Gwinnett 1982). However, there exists some controversy as to how clinically effective this procedure might be (Gwinnett & Ceen 1981). It has been shown that the incidence of enamel decalcifications (acquired white spots) is far lower with bonding than banding providing a sealant is used (Zachrisson 1978b). However, Ceen and Gwinnett (1981) found that auto-polymerised sealants repeatedly failed to cover the enamel and protect against white spot formation. Silverman, Cohen and Gwinnett (1979) believe that white spots occur with or without a sealant having been applied to the etched enamel.

The prevention of enamel decalcification and caries during
orthodontic treatment lies within a comprehensive preventive programme (Harvey & Powell 1981; Newman 1981). This includes office and home fluoride therapy (Zachrisson 1978a; Stratemann & Shannon 1979), oral hygiene measures and dietary control (Harvey 1980). Clinical studies are urged to seek the best combination of home and office fluoride therapy for the orthodontic patient (Gwinnett 1982). The role that sealants may play in this overall preventive strategy has yet to be fully determined. Zachrisson (1977a) believes that the combination of sealants and regular fluoride supplementation are the greatest aids to the prevention of white spot enamel lesions.

Harvey and Powell (1981) have outlined fluoride application procedures for the treatment (remineralisation) of post-treatment enamel decalcifications. These procedures outline home and surgery fluoride application regimes for white spot lesions on both anterior and posterior teeth. Due to the possible pigmentation of lesions by stannous fluoride, this agent is recommended only for posterior teeth.

Obviously, as clinicians, it behoves us to watch for decalcification and caries during orthodontic treatment as etching does in fact remove a hard fluoride-rich outer layer of enamel (Maijer 1982).

3-6 **BOND FAILURE RATES AND REBONDING**

Acid etch bonding or orthodontic attachments to the labial and lingual surfaces of teeth is generally adequate for clinical service (Smith & Maijer 1983b). However failures do occur, the percentage of failed bonded attachments being defined as Bond Failure Rate (Gwinnett 1982). The bond failure rate has been analysed as either the overall failure rate or failure rate for individual teeth (Mizrahi 1983).
Bonded bracket failure rates are often described in a broad clinical sense as either *clinically acceptable* (Mizrahi 1982) or *clinically unacceptable* (Artun & Bergland 1984). No one has defined the limitations of these terms with respect to bond failure rates.

Numerous clinical trials have attempted to evaluate bond failure rates for various types of orthodontic resin adhesives (Mizrahi 1983). These include both autopolymerised (Cavina 1977) and UV-cured resin composites (Garn 1976) utilising both direct (Zachrisson 1977a) and indirect bonding techniques (Zachrisson & Brobakken 1978). Only one study has compared both lingual and labial bond failure rates (Geiger, Gorelick & Gwinnett 1983). However, Alexander and his colleagues (1982) did conclude, without providing specific bond failure rate data, that bracket retention rates for lingual bonding (indirect technique) were as reliable as with properly bonded labial brackets.

Unfortunately, direct and simplistic comparisons between the various clinical studies cannot be made due to the numerous clinical and experimental variables involved (Fields 1982). Therefore, the interpretation of the results requires full cognizance of these variables.

Mizrahi (1983) has compared a large number of reported clinical studies that have quoted success or failure rates using direct bonding techniques (see Table 2). He records the clinical variables as the number of patients treated, number of brackets bonded, time period of treatment (months) and type of direct bonding adhesive (acrylic, lightly filled and heavily filled resin, polycarboxylate cement). The reviewer (K.G. Marshall) has included a more recent
<table>
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<th>Author</th>
<th>Patients (No.)</th>
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<th>Failure Rate (%)</th>
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<td></td>
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<td>(Nuva Seal)</td>
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<td>1000</td>
<td>Primer plus composite (Lee Unique)</td>
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<td>5</td>
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<tr>
<td>Mizrahi (1982)</td>
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<td>1194</td>
<td>Composite resin (?)</td>
<td>± 7</td>
<td>4-6</td>
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<td>276 ling.</td>
<td>Sealant plus composite resin (Concise)</td>
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<tr>
<td></td>
<td></td>
<td>812 labi.</td>
<td></td>
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study in Table 2 - Geiger et al (1983) who compared bond failure rates of both labial and lingual attachments. This should provide a broader perspective of bond failure rates with regards to earlier direct bonding techniques.

There are many clinical variables not mentioned by Mizrahi (1983) that can exert a profound effect on bond failure results. These include such factors as the bonding technique (Phillips 1980b), moisture and salivary control (Gorelick et al 1978), occlusal factors (Geiger et al 1983), bracket base design (Smith & Maijer 1983b), bracket base area, contour and adaptation to enamel surfaces (Maijer & Smith 1981) or whether brackets were bonded to teeth of patients with or without known histories of fluoride supplements (Artun & Bergland 1984). Some studies also suffered the disadvantages of operator variability, where more than one orthodontist bonded attachments (Geiger et al 1983). Mizrahi (1983) also considers that the reliability of many of the earlier studies may be questioned on the grounds of inadequate sample sizes and the short duration of the studies.

Hence, on contemplation of the above variables, it is not surprising that such a wide range of bond failure rates, both overall and individual, have been reported in the literature (Lee et al 1974; Zachrisson 1977; Wertz 1980).

However, despite the shortcomings inherent in bond failure rate studies, they have in a collective sense provided an overall idea of the clinical effectiveness of orthodontic bonding techniques. The
term, *clinically acceptable*, as used in the literature (Gwinnett 1982), still remains vague. One could however, deduce from the literature that clinically acceptable bond failure rates for anterior teeth range from approximately 5-10 per cent (Zachrisson & Brobakken 1978; Mizrahi 1982, 1983; Geiger et al 1983) and for posterior teeth, 10-30 per cent (Zachrisson 1976; Gorelick 1977; Newman 1978; Artun & Bergland 1984). Mizrahi (1983) concluded that the lowest failure rates, both overall and for individual teeth, could be achieved by selectively combining the clinical use of conventional bands (posterior teeth - molars and premolars) and directly bonded brackets (anterior teeth - canines and incisors). The results of his clinical study showed an overall failure rate of 4.7 per cent which corresponds well with lowest failure rates reported previously (Zachrisson 1976, 1977a; Newman 1978).

Despite the variability between the individual clinical studies cited in Table 2, there does emerge a number of consistent trends and reasons for bond failure. These include:

1. A higher individual and overall failure rate for posterior teeth compared with anterior teeth (Gorelick et al 1978; Silverman et al 1979; Geiger et al 1983). Reasons for this have been noted as:

   - poor access
   - poor moisture control
   - inadequate etch
   - occlusal force
   - irregular buccal surface anatomy requiring careful adaption of bracket base for minimal adhesive film thickness for maximum
2. Proper bonding technique is critical to obtaining optimal bond success (Gorelick et al. 1978; Phillips 1980a).


4. Bonds that survive early period are apt to remain throughout treatment (Cavina 1977; Zachrisson 1977a).

5. Mesh bases provide a better bond success rate than perforated bases (Geiger et al. 1983).

6. Occlusal force, particularly traumatic occlusion can be an important factor of bond failure (Cavina 1977; Newman 1978).

7. Indirect bonding techniques are as successful as direct techniques (Zachrisson & Brobakken 1978).

8. Labial and lingual failure rates are similar (Alexander et al. 1982; Geiger et al. 1983).

9. Bracket base size has gradually decreased as adhesive systems, bonding techniques and bracket base design have improved (Smith & Maijer 1983b). This has allowed a more aesthetic bracket and consistently closer adaption to the enamel surface to be bonded (Phillips 1980b; Fields & Proffit 1983).
10. Ligation and placement of initial archwires can occur within approximately five minutes of bonding last the bracket (Gwinnett 1982; Artun & Zachrisson 1982).

11. Site of clinical bond fracture invariably at resin/enamel interface (Fields 1982), probably due to poor technique (Gwinnett 1982).

12. Rebonding procedures are clinically feasible and successful (Zachrisson 1977a; Perry 1980). This has been substantiated with in vitro studies on rebond bracket strength (Jassem, Retief & Jamison 1981).

In addition to the above factors, Zachrisson (Gorelick et al 1978) believes that in his experience there are four major sources of clinical bond failure. They are:

1. Moisture contamination;

2. disturbances during resin setting period;

3. poor adaptation of bracket to tooth surface (i.e. too thick a layer of adhesive);

4. too heavy a pull during ligation.

Both Gorelick (Gorelick et al 1978) and Gwinnett (1982) discuss bonding in terms of a chain, with failures occurring at different links. The cause of failure at each link interface may vary. The chief causes of bond failure at the enamel interface are salivary
contamination, inadequate prophylaxis and improper etching. The chief causes within the adhesive are movement during polymerisation and delayed bracket placement so the adhesive has begun to set, using the adhesive after its shelf life has expired and tying the archwire prematurely. The main cause for bond failure at the adhesive/bracket interface is not forcing the adhesive through the mesh and not pressing the bracket firmly against the tooth.

There does not appear to be a direct relationship between bond strength per se and clinical bond performances as measured by bond failure rates (Beech & Jalaly 1981; Fields 1982). The strength of the resin adhesives appears adequate whilst the clinical technique appears to be the critical factor (Gorelick et al 1978; Phillips 1980a). However, a number of options for improving the clinical success rate of orthodontic bonding have been proposed. These are:

1. Increase the bond strength of the adhesive, thereby eliminating cohesive-type failures within the bulk of the adhesive (Newman 1978). However, this will increase debonding problems and not necessarily improve bond success rate (Beech & Jalaly 1981).

2. Improve bracket base design, thereby eliminating resin/bracket failures (Smith & Maijer 1983b).


As new adhesive materials appear and as the present bonding systems improve along with careful recognition of technique details,
we may expect an improvement of both the overall and individual bond failure rates in the future.

3-7 DEBONDING

Orthodontic debonding involves the removal of the bonded orthodontic attachments and resin from the enamel surfaces at the completion of the active appliance phase of treatment. The enamel surfaces should then be restored as closely as possible to the original anatomical and topographical features of the tooth (Silverman et al 1979). Ideally, the enamel surface should be no more susceptible to pathology than at the pretreatment time (Fields 1982).

Orthodontic debonding can be considered a three stage process:

1. Bracket removal
2. Resin removal
3. Polishing the enamel tooth surface (Phillips 1980b)

Debonding procedures have proven to be time consuming, tedious and uncomfortable for both orthodontist and patient with currently available methods (Gorelick 1977). Debonding has become of major concern to the orthodontic profession with the advent of heavily-filled resin composites (Gwinnett 1982). The differences in the ease of debonding can be related to the type of resin. The most difficult resin to remove is the heavily-filled composite, followed by the lightly-filled and then unfilled resin (Newman 1978).

The aim of orthodontic bonding is to combine optimal bond strength
with quick and easy debonding whilst causing no iatrogenic damage to the enamel surface (Artun & Bergland 1984). Both Phillips (1980a) and Zachrisson (Gorelick et al 1978) believe that the question of minimal, adequate bond strength remains unresolved. In fact, bond strength requirements vary in different regions of the mouth (Zachrisson 1977a). The situation seems to be that the stronger the adhesive, the more difficult it is to remove (Gorelick et al 1978). However, if it became necessary to make a choice between easier debonding and optimal bond strength, the latter would certainly be the choice of most clinicians (Artun & Bergland 1984). Certainly, in terms of disruption to clinical routine, lost chairside time and practice public relations, most orthodontists would opt for maximum bond strength (heavily-filled resins) with the consequence of an increased debonding tedium (Smith & Maijer 1983b).

Early recommendations for debonding procedures were developed from those to remove bands and cement (Gwinnett 1982). This was an ill-conceived approach and resulted in unwarranted iatrogenic damage caused as the result of inadequate instrumentation (Gwinnett & Gorelick 1977). Both Gwinnett and Gorelick (1977) and Burapavong and colleagues (1978) have looked critically at tissue response to various debonding methods. Basically, the debonding armamentarium can be categorised as:

1. Hand instruments
2. Rotary instruments
3. Ultrasonic devices (Gwinnett 1982)
3-7-1 BRACKET REMOVAL

There exists general agreement that this is best achieved with the use of ligature cat cutters, using a shearing force with the blades held flat against the tooth surface (Zachrisson 1977a; Newman 1978; Zachrisson & Artun 1979; Phillips 1980b). Normally, the bracket comes away at the resin/bracket base interface leaving the bulk of the remaining resin attached to the enamel surface (Gorelick 1977).

3-7-2 RESIN REMOVAL

Opinions as to the best procedure for the removal of the remaining attached resin varies in detail from authority to authority although the overall concepts are similar (Gwinnett & Gorelick 1977; Gorelick 1977; Brown & Way 1978; Newman 1978; Zachrisson 1978b; Burapavong et al 1978; Retief & Denys 1979; Zachrisson & Artun 1979).

The bulk of the attached resin is effectively reduced with the use of a high-speed, multi-bladed finishing bur in a dry, well-illuminated field (Gwinnett 1982). When a bur is used, the removal of the resin is stopped just short of the enamel surface (Maijer 1982). The procedure is then completed with a green rubber wheel and pumice (Gwinnett & Gorelick 1977). A final pumice polish has been strongly recommended to restore natural enamel features (Burapavong et al 1978). According to Maijer (1982), the use of large rubber wheels as recommended by Gwinnett and Gorelick (1977) produces a lot of heat, is offensive to the patient and potentially dangerous to the pulp. An alternative procedure with an impregnated cup (Shofu greenie) using a mixture of fine flour pumice and gel toothpaste produced an extremely smooth enamel surface (Maijer & Smith 1978). An additional procedure following pumicing, with the use of graded abrasive 3M softlex discs, has been recommended by Phillips (1980b) for an optimal enamel surface.
finish.

Ultrasonic scalers have been found to be slow and ineffective in removing heavily filled resin (Gwinnett & Gorelick 1977).

An important consideration in debonding is the initial bonding procedure. A minimal amount of composite resin placed at the beginning of the bonding procedure, and careful removal of any excess, eliminates any large amount of clean-up post-treatment (Maijer 1982).

3-7-3 IATROGENIC DAMAGE

Bonding and debonding procedures may produce iatrogenic damage to enamel surfaces (Zachrisson 1978a, 1978b). This includes the formation of white spots (Gwinnett & Caen 1981) as well as enamel loss (Pus & Way 1980), cracks (Jones 1980), scratches (Retief & Denys 1979) and staining (Ceen & Gwinnett 1980a).

The formation of white spots or the decalcification of enamel surfaces has been discussed previously.

A number of studies has attempted to quantify enamel loss due to acid etching and debonding procedures (Fitzpatrick & Way 1977; Zachrisson & Artun 1979; Gaberoglio & Cozzani 1979). There is a lack of agreement as to the exact extent of this enamel loss.

The enamel surfaces usually bonded are estimated to have a thickness of 1,500-2,000 microns (Sicher & Bhaskor 1972).

Fitzpatrick and Way (1977) found that approximately 55 microns of
enamel (3 per cent of total enamel thickness) was lost during etching, bracket placement, bracket removal and clean-up when using a UV cured system. Brown and Way's (1978) results showed a 42 micron enamel loss for the highly-filled self-curing resins. These two results appear to confirm each other. Zachrisson and Artun (1979) claim a loss of only 5 microns. Their analysis is based on measuring the height of perihymata and demonstrating their presence with stereo scanning electron micrographs. It would appear that enamel loss judged by tooth surface morphology is a misleading method of assessment which gives an impression of very limited enamel loss (Pus & Way 1980). Maijer (1982) has also questioned the accuracy of such a method.

Possibly one of the most disturbing features of bonding and debonding procedures is that the outer enamel layer, so carefully fluoridated by so many topical applications and fluoride rich (Koch & Friberger 1971), is completely lost. Even prior to debonding procedures, acid-etch treatment has caused a previous additional enamel loss (Shey & Brandt 1982). Although this form of iatrogenic damage does occur, careful finishing of the enamel after debonding does not damage the tooth structure to a great degree (Maijer 1982). However, as it is impossible to precisely gauge the resin/enamel interface during debonding, it is understandable that some enamel will be removed (Gwinnett 1982). These facts would strongly suggest that fluoride application after orthodontic bonding is indicated to enhance remineralisation and reduce susceptibility to caries (Lehman, Davidson & Duijsters 1981).

Caspersson (1977) found that resin was present in all debonded
enamel surfaces which he studied when assessed by scanning electron-microscopy. Recent evidence (Dietrich 1981; Xu Heng-Chang et al 1983) indicates that resin tags may be present at depths from 100-300 microns into the enamel. This is far greater than the etch depth of 10-25 microns estimated elsewhere (Maijer 1982). If conservative methods are utilised for debonding so as to return the enamel surface to a smooth state, it is unlikely when considering studies by Fitzpatrick and Way (1977) and Brown and Way (1978) that all the resin tags will be removed. According to Fields (1982), the long-term ramifications of the resin remaining in these surfaces remains unknown.

Other iatrogenic damage claimed to have been caused by debonding includes enamel cracks and fractures (Gwinnett & Gorelick 1977; Jones 1980; Dietrich 1981). A less common but nonetheless dramatic form of iatrogenic damage can result from bracket corrosion (Ceen & Gwinnett 1980a). Crevice corrosion products from type 304 stainless steel brackets can leach into the enamel surface causing small black spots (Smith & Maijer 1983b). Some companies (Ormco) are now producing their brackets from type 316L stainless steel to prevent this corrosion potential.

Despite the possible iatrogenic problems discussed, Zachrisson (1977a) has shown that for periods up to twelve months, no accumulation of stain or discoloration was noted on the enamel surfaces following bracket removal. Undoubtedly, longer term clinical studies are required to substantiate these observations.

As orthodontists, we should not allow the loss of surface enamel
due to bonding and debonding techniques to burden our professional consciences. Enamel removal due to occlusal equilibration (Casullo 1983) and enamel stripping (Vickers 1982) are considered acceptable clinical procedures. However, we should be acutely aware of the extent of the problem and attempt to minimise it at all times.

3-8 CONCLUSION

At the present time, the technology of orthodontic bonding remains in rapid flux and it is likely that the precise techniques used in the future will not be the same as those in current use (Fields & Proffit 1983). Indeed, aesthetic hydroxyapatite brackets with self-adhesive bases, eliminating the need for enamel etching, now appear on the clinical horizon (Kameda 1982). The continued development and improvement of intrinsically adhesive cements such as glass ionomer cement, will continue to provide possible options to the present-day acid-etch/resin bonding techniques (Maijer & Smith 1979; Owen 1981).

The bonding of orthodontic attachments to teeth has been well documented in the literature with both the advantages and disadvantages having been fully elucidated (Dogon 1981). However, the euphoria evident from early clinical bonding successes (Silverman, Cohen, Gianelly & Dietz 1972) has given rise to the sober realisation that bonding does possess the inherent capacity for iatrogenic damage to dental tissues (Zachrisson 1978a, 1978b). Debonding procedures and post-treatment, white spot (decalcification) lesions appear to be the greatest clinical thorns (Phillips 1980a). These problems are presently being addressed (Ceen & Gwinnett 1980b, 1981).
The present clinical vogue encompasses the use of both mix and no-mix diacrylate resins (Artun & Zachrisson 1982; Fried & Newman 1983). These may be lightly or heavily filled composite resins (Silverman, Cohen & Gwinnett 1979). There exists general agreement that the heavily filled composite resins such as Concise (3M) and Unite (Unitek) possess greater bond strength but they are more difficult to debond and finish when compared with the unfilled and lightly filled resins (Gorelick, Masunaga, Thomas & Zachrisson 1978; Smith & Maijer 1983a). The concomitant use of sealants or primers still remains controversial (Gwinnett & Ceen 1981).

The UV light-cured resin system underwent a brief period of popularity in the early 1970s (Cohi et al 1972). However, the health problems to both patient and orthodontist from UV light exposure led to the development of the white light command cure system (Smith & Maijer 1983a). The potential use of the white light system, with its 6mm curing depth through tooth structure, has yet to be fully assessed for orthodontic bonding (Maijer 1982).

The majority of orthodontists favour a direct bonding technique (Gorelick 1979). However, poor access, variable lingual anatomy and the need for precise bracket height location have made the indirect method the technique of choice for lingual orthodontics (Alexander, Alexander, Gorman, Hilgers, Kurz, Scholz, Smith 1982). Lingual orthodontics undoubtedly places exacting demands on available bonding techniques (Paige 1982).

Bond failure rates have been clinically assessed with both
lingual and labial attachments (Geiger, Gorelick & Gwinnett 1983) as well as direct and indirect techniques (Zachrisson & Brobakken 1978). The term, *clinically acceptable*, as applied to bond failure rates has not been defined. However, a review by Mizrahi (1983) indicates that overall failure rates for conventional bands and directly bonded brackets are comparable. By combining the two techniques, an overall bond failure rate of 4.7 per cent was obtained. This figure appears to represent the clinical optimum for present day acid etch/resin bonding techniques.

The causes of bond failure appear to be multifactorial (Keizer et al 1976), having been identified as the nature of enamel itself, poor technique, the adhesive system and attachment retentivity of the bracket base design (Gwinnett 1982; Smith & Maijer 1983b). It appears that the weak link is operator technique (Phillips 1980a). However, Smith and Maijer (1983b) believe that if further improvement in the mechanical retention of resin to the bracket base can be obtained, then the bond strength would be limited only by the cohesive strength of the resin. Bond failures do not indicate inadequate bond strength *per se* as the weak link clinically in the bonded assembly is often the resin/bracket base interface (Maijer 1982).

Present materials available have sufficient bond strength but removal of the bonding adhesives remains difficult and unsatisfactory (Maijer 1982). Presently, both in vitro and in vivo studies are underway to adjust bond strengths to the tooth surface within an acceptable range for prolonged treatment and convenient debonding (Dogon 1981). Less traumatic debonding forces would yield a higher percentage of undamaged brackets that would be suitable for recycling.
(Phillips 1980b; Higgins 1982). Ideally, a bonding material is needed that can be removed by chemical reversal or use of a non-toxic solvent (Maier 1982).

The profession continues to experience a wide range of bracket base types which are undergoing development and refinement (Gwinnett 1982). The concept of a ceramic sintered porous base with the capability of fluoride release is a fascinating concept (Smith & Maier 1983b), combined with plethora of bracket types and resins available.

It is understandable that this rapid evolution, some say revolution, has caused confusion (Silverman, Cohen & Gwinnett 1979).

Orthodontic bonding techniques are now being expanded and adapted into other areas of orthodontic need. A variety of bonded retainer designs have been well described in the literature (Reinhardt, Deneyh & Chan 1979; Artun & Zachrisson 1982; Jones 1982b; Meyers & Vogel 1982). Expansion and Herbst appliances made of metal framework and acrylic overlays are being bonded over the entire coronal tooth surfaces (Phillips 1980a; Howe 1982; Howe & McNamara 1983). The versatility of bonded resin techniques has also been adapted to orthodontic tooth size or shape problems (Fields 1982; Tuverson 1980).

The extent to which composite resin bonding systems in orthodontics can be utilised seems only to be limited by the imagination and ingenuity of the orthodontist. Future research will undoubtedly overcome many of the present limitations with innovative techniques and better ad-
hesives (Maijer 1982). Such recent innovations are the concept of

*rystal bonding* (Maijer & Smith 1979; Artun & Bergland 1984), the
bonding of attachments to porcelain crowns in adult orthodontics
(Chassemi-Tary 1979) and the development of a new adhesive resin,

4-Meta, will bond chemically to both enamel and stainless steel
without the need for phosphoric acid-etching (Mogi 1982). We are
certainly past the state of cautious optimism with bonding and into
the realm of everyday application (Silverman et al 1979). However,
the ultimate and optimum orthodontic bonding adhesive is still not
in hand (Halpern 1981).
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CHAPTER 4

IN VITRO STUDIES ON ORTHODONTIC BOND STRENGTHS

4-1 INTRODUCTION

Over the last twenty years, many different test methods have been devised to examine the bond strength between enamel and dental adhesives (Mizrahi & Smith 1969a; Lee, Swartz & Culp 1969; Keizer et al 1976; Lopez 1980). The test methods presently in use for evaluating orthodontic adhesives have been modified, according to Paxton (1983), from previous restorative adhesive tests to suit the particular needs of orthodontic bonding.

Bond tests are capable of statistically ranking the bonding abilities of dental adhesives (Eden, Craig & Peyton 1970). Bond strength tests have also been widely used in orthodontics to assess the effects of the variables which affect bond strength, including the adequacy of the acid etch technique (Bates et al 1983), the particular bonding agent employed (Guzman et al 1980), the nature of the force system applied to the bond interface (Thanos, Munholland & Caputo 1979) and the bracket base design (Smith & Maijer 1983b).

The tooth-adhesive-bracket complex has now been extensively investigated using in vitro orthodontic bond strength studies (see Table 3). However, comparisons between these studies is difficult because of the multiplicity of variables, the lack of standardised testing and the pace at which new products appear on the market (Wheeler & Ackerman 1983). Indeed, Keizer et al (1976) believe that the considerable differences reported in both clinical and laboratory trials for direct orthodontic bonding are not surprising since orthodontic bonding
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<tr>
<td>1. Hanson et al 1983</td>
<td>1 (Concise) 1a</td>
<td>Metal 1a, 1c</td>
<td>3rd molars premolars 1a</td>
<td>Tensile Kg/mm²</td>
<td>3 hours exposed to air</td>
<td>- Tensile BS - Mesh Vs sintered bases - Immersion time</td>
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<td>2. Smith &amp; Maijer 1983b</td>
<td>2 BIS-GMA 1a Acrylic 2</td>
<td>Metal 1a, 1c, 2</td>
<td>-Human enamel 1a 1b</td>
<td>Tensile Kg/cm²</td>
<td>24 hours water 37°C</td>
<td>- Base types - Mesh Vs sintered - Adhesives</td>
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<td>3. Wheeler &amp; Ackerman 1983</td>
<td>1 BIS-GMA 1a</td>
<td>Metal 1a</td>
<td>Premolars 1a</td>
<td>Tensile lbs</td>
<td>?</td>
<td>- Recycling BS - Mesh</td>
</tr>
<tr>
<td>4. Aguirre et al 1982</td>
<td>1 BIS-GMA 1a</td>
<td>Metal 1a</td>
<td>Premolars 1a</td>
<td>Shear Kgf</td>
<td>?</td>
<td>- Indirect Vs Direct - Shear BS - Clín. Vs Lab. time</td>
</tr>
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<td>5. Naslia &amp; Chen 1982</td>
<td>17 BIS-GMA 1a</td>
<td>Metal 1a, 1c</td>
<td>Maxillary incisors 1a</td>
<td>Shear lbs</td>
<td>24 hours room temp. 100% humidity</td>
<td>- Recycling process - Shear BS</td>
</tr>
<tr>
<td>6. Alexander et al 1981</td>
<td>3 BIS-GMA 1a</td>
<td>Metal 1a</td>
<td>Premolars 1a</td>
<td>Shear lbs</td>
<td>24 hours 27 days</td>
<td>- Adhesives - Shear - Interface failure</td>
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</table>

ADHESIVE:  
1a. BIS-GMA or acrylic resin  
1b. BIS-GMA or acrylic resin = UV light-cure  
2. Acrylic (methylmethacrylate) resin  
3. Glass ionomer cement (GIC)  
4. Polyacrylate cement (PCC)

ORTHODONTIC ATTACHMENT: Metal bracket with -  
1a. Mesh base  
1b. Perforated base  
1c. Miscellaneous base type: Photoetched (5, 10); Solid undercut (18); polymeric coating (17); sintered porous metal (1, 2); sintered ceramic (2)  
2. Metal lingual button with mesh base  
3. Plastic bracket  
4. Plastic lingual button

SUBSTRATE:  
1. Human enamel - (a) Natural curved tooth surface (b) Flat enamel surface prepared with abrasives  
2. Bovine enamel - (a) Natural curved tooth surface (b) Flat enamel surface prepared with abrasives  
3. Plastic
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<td>-Tensile &amp; Shear BS</td>
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<td>Acrylic 2</td>
<td></td>
<td></td>
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<td>8. Jassem et al 1981</td>
<td>1</td>
<td>Metal</td>
<td>Maxillary incisors</td>
<td>Tensile Shear MPa/m²</td>
<td>24 hours thermocycle + room temp., 37°C water</td>
<td>-Tensile Vs Shear BS</td>
</tr>
<tr>
<td></td>
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<td>-Bond Vs Rebond BS</td>
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<td>(Sealant)</td>
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<td>-Thermocycling</td>
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<td></td>
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<td>1a</td>
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<td>-Bracket bases</td>
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<td>1b, 1c</td>
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<td></td>
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<td>-Weld spots</td>
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<td></td>
<td>Metal</td>
<td></td>
<td></td>
<td></td>
<td>-Mesh type</td>
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<tr>
<td>9. Maier &amp; Smith 1981</td>
<td>1</td>
<td>Metal</td>
<td>Premolars</td>
<td>Shear Kgf</td>
<td>24 hours water 37°C</td>
<td>-Sealant</td>
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<tr>
<td></td>
<td>BIS-GMA 1a (Dynabond)</td>
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<td>-Bracket bases</td>
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<td></td>
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<td>-Weld spots</td>
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<td>1a, 1c</td>
<td></td>
<td></td>
<td></td>
<td>-Mesh type</td>
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<tr>
<td>10. Dickinson &amp; Powers 1980</td>
<td>2</td>
<td>Metal</td>
<td>Human enamel</td>
<td>Tensile Kg/m²</td>
<td>24 hours water 37°C</td>
<td>-Tensile BS</td>
</tr>
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<td>BIS-GMA 1a</td>
<td></td>
<td></td>
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<td>-Bracket base area</td>
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<td></td>
<td></td>
<td>Metal</td>
<td>Plastic 3</td>
<td></td>
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<td>-Mesh size</td>
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<tr>
<td></td>
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<td>1a, 1b, 1c</td>
<td></td>
<td></td>
<td></td>
<td>-Spot welding</td>
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<td>11. Hirce et al 1980</td>
<td>1</td>
<td>Metal</td>
<td>Premolars</td>
<td>Shear inch/ounces</td>
<td>12 days distilled water</td>
<td>-Fluoride and Shear BS</td>
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<td></td>
<td>BIS-GMA 1a (Endur)</td>
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<td></td>
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<td>Metal</td>
<td>3rd molars</td>
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<td></td>
<td>1a</td>
<td></td>
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<tr>
<td>12. Lopez 1980</td>
<td>1</td>
<td>Metal</td>
<td>incisors</td>
<td>Shear Kg/cm²</td>
<td>-24 hours -30 days</td>
<td>-Bracket bases</td>
</tr>
<tr>
<td></td>
<td>BIS-GMA 1a (Endur)</td>
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<td></td>
<td></td>
<td>Distilled water 37°C</td>
<td>-Base size</td>
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<td></td>
<td>Metal</td>
<td></td>
<td></td>
<td></td>
<td>-Immersion time</td>
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<td></td>
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<td>1a, 1b, 1c</td>
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</tbody>
</table>

**ADHESIVE:**
1. BIS-GMA or diacylate resin
2. Acrylic (methylmethacrylate) resin
3. Glass ionomer cement (GIC)
4. Polyacrylate cement (PAC)

**ORTHODONTIC ATTACHMENT:**
1. Mesh base
2. Perforated base
3. Miscellaneous base types: Photoetched (5, 10); solid undercut (10); polymeric coating (17); sintered porous metal (1, 2); sintered ceramic (2)
2. Metal lingual button with mesh base
3. Plastic bracket
4. Plastic lingual button

**SUBSTRATE:**
1. Human enamel - (a) Natural curved tooth surface
   (b) Flat enamel surface prepared with abrasives
2. Bovine enamel - (a) Natural curved tooth surface
   (b) Flat enamel surface prepared with abrasives
3. Plastic
<table>
<thead>
<tr>
<th>STUDY</th>
<th>ADHESIVE No. &amp; Type</th>
<th>ORTHODONTIC ATTACHMENT</th>
<th>SUBSTRATE</th>
<th>LOADING MODE AND BOND STRENGTH (BS) UNITS</th>
<th>SPECIMEN AGE AND STORAGE</th>
<th>TEST VARIABLE(S)</th>
</tr>
</thead>
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<tr>
<td>13. Thamos et al 1979</td>
<td>5 BIS-GMA 1a</td>
<td>Metal 1a, 1b</td>
<td>Human enamel 1a</td>
<td>-Tension</td>
<td>30 minutes saline soltn</td>
<td>-Mesh Vs perforated bases</td>
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<td></td>
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<td></td>
<td>-Shear</td>
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<td>-BS</td>
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<td></td>
<td></td>
<td>-Torsion</td>
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<td>14. Carlyle et al 1978</td>
<td>2 BIS-GMA 1a</td>
<td>Metal 1b</td>
<td>Human teeth 1a</td>
<td>Shear Newtons</td>
<td>'5 days 100% humidity</td>
<td>Shear BS</td>
</tr>
<tr>
<td></td>
<td>GIC 3</td>
<td></td>
<td></td>
<td>room temp.</td>
<td></td>
<td>Resin &gt;&gt; GIC</td>
</tr>
<tr>
<td>15. Faust et al 1978</td>
<td>13 BIS-GMA 1a, 1b</td>
<td>Metal 1a, 1b Acrylic 2</td>
<td>Maxillary incisors 1a</td>
<td>Tensile lb/m²</td>
<td>24 hours distilled water</td>
<td>-Diametric tensile strength</td>
</tr>
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<td></td>
<td>PCC 4</td>
<td>Plastic 3</td>
<td></td>
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<td>37°C</td>
<td>-Tensile BS</td>
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<td></td>
<td></td>
<td>-Adhesives</td>
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<td>16. Moir &amp; Dogon 1978</td>
<td>1 BIS-GMA 1a</td>
<td>Metal 1a</td>
<td>Premolars 1a</td>
<td>Shear lbs</td>
<td>Thermo cycling in water</td>
<td>Variations of Concise bonding</td>
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<td>(Concise)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>system</td>
</tr>
<tr>
<td>17. Reynolds &amp; Fraunhofer</td>
<td>4 BIS-GMA 1a</td>
<td>Metal 1a, 1b, 1c</td>
<td>Premolars 1a</td>
<td>Tensile kg</td>
<td>3 hours water 20°C room</td>
<td>Bracket bases</td>
</tr>
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<td>1977</td>
<td>Acrylic 2</td>
<td>Plastic</td>
<td></td>
<td></td>
<td>temp.</td>
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</tr>
<tr>
<td>18. Rogers &amp; Griffith 1977</td>
<td>7 BIS-GMA 1a</td>
<td>Metal 1c</td>
<td>Human enamel 1a</td>
<td>Tensile MPa</td>
<td>24 hours 100% humidity</td>
<td>-Tensile BS</td>
</tr>
<tr>
<td></td>
<td>Acrylic 2</td>
<td></td>
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<td>37°C</td>
<td>-Adhesive</td>
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<tr>
<td></td>
<td>GIC 3</td>
<td></td>
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<td></td>
<td>-Bracket base</td>
</tr>
<tr>
<td></td>
<td>PCC 4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>(solid undercut)</td>
</tr>
</tbody>
</table>

ADHESIVE: 1a. BIS-GMA or diacrylate resin
1b. BIS-GMA or diacrylate resin = UV light-cure
2. Acrylic (methylmethacrylate) resin
3. Glass ionomer cement (GIC)
4. Polycarboxylate cement (PCC)

ORTHODONTIC ATTACHMENT: Metal bracket with -
1a. Mesh base
1b. Perforated base
1c. Miscellaneous base type: Photoetched (5,10); solid undercut (18); polynem coating (17); sintered porous metal (1, 2); sintered ceramic (1)
2. Metal lingual button with mesh base
3. Plastic bracket
4. Plastic lingual button

SUBSTRATE: 1. Human enamel - (a) Natural curved tooth surface
(b) Flat enamel surface prepared with abrasives
2. Bovine enamel - (a) Natural curved tooth surface
(b) Flat enamel surface prepared with abrasives
3. Plastic
<table>
<thead>
<tr>
<th>STUDY</th>
<th>ADHESIVE No. &amp; Type</th>
<th>ORTHODONTIC ATTACHMENT</th>
<th>SUBSTRATE</th>
<th>LOADING MODE AND BOND STRENGTH (BS) UNITS</th>
<th>SPECIMEN AGE AND STORAGE</th>
<th>TEST VARIABLES(S)</th>
</tr>
</thead>
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<tr>
<td>19. Johnson et al 1976</td>
<td>7</td>
<td>Metal 1a</td>
<td>Bovine incisors 2a</td>
<td>Shear lbs</td>
<td>-24 hours -1 month -3 months saline solution</td>
<td>Adhesive -Immersion time</td>
</tr>
<tr>
<td>20. Keizer et al 1976</td>
<td>5 BIS-GMA 1a Acrylic 2</td>
<td>Plastic 3</td>
<td>Bovine enamel 2b</td>
<td>Shear Kg/cm²</td>
<td>1 hour</td>
<td>Shear BS of adhesives</td>
</tr>
<tr>
<td>21. Moser et al 1976</td>
<td>5 BIS-GMA 1a, 1b Acrylic 2 GIC 4</td>
<td>Screw into Adhesive</td>
<td>Incisors 1b</td>
<td>Tensile Kg/cm²</td>
<td>-24 hours -7 days -30 days Artificial Saliva 37°C</td>
<td>Saline primer -Storage time</td>
</tr>
<tr>
<td>22. Reynolds &amp; Fraunhofer 1976a</td>
<td>8 BIS-GMA 1a Acrylic 2</td>
<td>Metal 2</td>
<td>Premolars 1a</td>
<td>Tensile Kg</td>
<td>3 hours water 22°C Room temp.</td>
<td>Gauze mesh size</td>
</tr>
<tr>
<td>23. Reynolds &amp; Fraunhofer 1976b</td>
<td>9 BIS-GMA 1a Acrylic 2 PCC 4</td>
<td>Metal 2</td>
<td>Premolars 1a</td>
<td>Tensile Kg</td>
<td>3 hours 1 week 1 month 6 months Water 22°C Room temp.</td>
<td>Adhesives -Water immersion -Acid etchant</td>
</tr>
</tbody>
</table>

ADHESIVE: 1a. BIS-GMA or diacrylate resin 1b. BIS-GMA or diacrylate resin + UV light-cure 2. Acrylic (methylmethacrylate) resin 3. Glass ionomer cement (GIC) 4. Polycarboxylate cement (PCC)

ORTHODONTIC ATTACHMENT: 1a. Metal bracket with - 1b. Perforated base 1c. Miscellaneous base type: Photoetched (5, 10); solid undercut (18); polymeric coating (17); sintered porous metal (1, 2); sintered ceramic (1)

SUBSTRATE: 1. Human enamel - (a) Natural curved tooth surface (b) Flat enamel surface prepared with abrasives 2. Bovine enamel- (a) Natural curved tooth surface (b) Flat enamel surface prepared with abrasives

3. Plastic
is a complicated problem. The final results are influenced by several factors.

In vivo bond failure rate studies have enabled investigators to define quantitatively the success rate of bonded orthodontic attachments under clinical conditions (Mizrahi 1983). In vivo studies also help establish the general clinical practicality of the bonding adhesive (Lee et al 1974). On the other hand, in vitro bond strength tests attempt to analyse the variables that influence the bond strength of the tooth-adhesive-bracket assembly under ideal conditions in the laboratory (Beech & Jalaly 1980).

According to Alexandre, Young, Sandrik and Bowman (1981), orthodontic bond strength tests have always been a matter of some controversy. Principally, the different test methods used by various investigators make the comparison of results difficult, even though the individual results may be valid. Similarly, Lopez (1980) states that the lack of standardisation in testing methods makes the comparison of results complicated.

Smith (1982d) has highlighted the dilemma of attempting to design physical test methods that emphasise the reproductibility of the method for standardisation purposes, rather than creating test conditions that closely resemble the clinical situation. Consequently, making clinical assumptions on the basis of in vitro bond strength tests can be hazardous (Loeys et al 1982). Many different types of bond tests have been proposed and used to determine bond strengths of composite resins (Williams, De Vries & Despain 1973). Up to six types of adhesion tests
may be carried out - shear, tensile, impact, cleavage, bending and peel (Patrick 1961). The more recent bond strength tests on restorative resins have used either shear (Yedid & Chan 1980; Boyer, Chalkley & Chan 1982) or tensile test modes (Zidan, Asmussen & Jorgensen 1980). Mizrahi and Smith (1969a) believed that analysis of the forces applied to an orthodontic bracket, cemented directly to the crown of the tooth, indicated that the three major stresses developed within the cement bond were tensile, shear and a form of peel stress.

However, it appears that the type of force which most accurately simulates clinical stress on the orthodontic attachment is still not clear, as many investigators use either tensile or shear loading alone (Dickinson & Powers 1980; Mascia & Chen 1982).

The majority of orthodontic bond tests have evaluated either composite (BIS-GMA) resins or acrylic resins attached to etched enamel (Faust, Grego, Fan & Powers 1978; Beech & Jalaly 1981). In addition, polycarboxylate cement (Mizrahi & Smith 1969b); Reynolds & von Fraunhofer 1976b) and glass ionomer cement (Carlyle et al 1978) have been evaluated as orthodontic adhesives using laboratory bond tests.

Numerous experimental variables have complicated the comparison of various orthodontic bond strength studies. Testing models, storage time, storage media, substrate preparation, bracket design and the adhesive are but some of the parameters involved (Fields 1982). Even the effect of thermocycling of resins has been shown to affect bond strength (Bishara, Khowassah & Oesterle 1975; Jassem, Retief & Jamison 1981).
Estimates only have been made as to the minimal bond strength necessary to withstand the forces of orthodontic treatment. The validity of 29Kg/cm² as suggested by Newman (1969a) is not clear. Both Keizer et al (1976) and Smith and Maijer (1983b) cite the requirement for the maximum force exerted on a bracket to be about 30Kg/cm². However, Phillips (1980a) believes that the minimal suitable bond strength, be it tensile, shear or torsion, remains an unanswered question.

Most in vitro bond strength failures occur at the mesh base/resin interface, whilst most of the clinical failures are at the resin/enamel interface (Fields 1982); this is probably due to poor isolation and moisture contamination during in vivo bonding (Jassem et al 1981). Stable positioning of the bracket during bonding (Zachrisson 1977a) and a thin layer of resin help to ensure maximum bond strength (Buonocore 1963).

The bond fracture site has been described by Retief (1975) as occurring at a number of interfaces. Gwinnett (1982) has further redefined these fracture sites as they relate to the enamel/adhesive/bracket complex. These fracture sites have been assessed visually, under high magnification and with scanning electron microscopes (Eick et al 1972; Retief 1975; Alexandre et al 1981; Artun & Berkland 1984). The object of these investigations is to examine the fractured surfaces of these experimental bonds in order to obtain a better understanding of the mechanism of failure (Retief 1975).
As the resin/bracket base interface is the most common failure site during in vitro bond tests, many attempts have been made to maximise the mechanical retention between base and resin (Lopez 1980). This has involved altering the resin viscosity (Moin & Dogon 1978), varying the mesh size (Reynolds & Von Fraunhofer 1976a) and using alternative base retentive mechanisms (Rogers & Griffith 1977a; Smith & Maijer 1983b).

Sintered porous base coatings have provided promising increases in tensile bond strengths when compared with mesh base brackets (Hanson, Gibbon & Shimizu 1983). Clinical trials are yet to substantiate these laboratory tests.

Bond strength tests have been used to evaluate many of the variables involved in the enamel/adhesive/bracket bonded unit (Moin & Dogon 1978).

This review will attempt to highlight and elucidate the multiplicity of factors that are relevant to the design, investigation and interpretation of orthodontic bond strength tests. Many of the shortcomings of bond strength studies have been recognised for years (Eden et al 1970), yet it is still apparent that even the most recent studies struggle with two fundamental problems (Williams et al 1973):

1. The test or method by which the strength of an adhesive can be most reliably and meaningfully measured.

2. The interpretation of the test data in terms of strength of the bond on particular applications.
These two problems, combined with the multitude of test variables, inevitably lead to a lack of agreement on key issues between various investigators.

4-2 EVALUATION OF BOND STRENGTH TESTS - BACKGROUND

4-2-1 EXPERIMENTAL DESIGN

The evaluation of orthodontic adhesives necessitates the development of a well-designed, standardised, in vitro test procedure that closely correlates with clinical experience (Brauer 1975). In most cases, there has been a poor definition and analysis of these clinical conditions (Smith 1982d). In addition, most dentists have not taken into consideration the mechanical aspects of the matter, particularly with regard to the test jig design (Paxton 1983). Consequently, according to Paxton (1983), much time has been wasted pursuing test methods which did not provide reliable results. The lack of standardisation amongst orthodontic bond strength tests is obvious when various studies are compared (Lee et al 1974; Reynolds & Von Fraunhofer 1977; Hanson et al 1983).

Faust et al (1978) commented that when testing the bond strengths of direct bonding cements, a choice must be made to measure the bond between the cement and enamel or the strength of the enamel/cement/bracket unit. In the former test, an orthodontic bracket is generally not used; instead, a screw is embedded within the cement to allow the system to fail either at the enamel/cement interface or within the cement (Eden et al 1970). The latter test is more of a clinical test to determine which interface (bracket/bracket, bracket/cement or
cement/enamel) is the weakest.

Measurement of bond strengths to tooth structure is the principal method used to study adhesives and tooth surface pre-treatments (Beech 1978). Invariably, in orthodontics, these have involved the acid etch technique and direct bonding adhesive resins (Hirce et al 1980).

The results of orthodontic bond strength tests are said to be influenced by the following factors (Keizer, Ten Cate & Arends 1976):

1. The adhesive strength between enamel and adhesive;

2. the mechanical properties of the adhesive;

3. the adhesion between adhesive and bracket;

4. the mechanical properties of the bracket material;

5. the dimensions of the bracket wings, these being the point of contact for the application of the test force.

The overall experimental design of orthodontic bond strength tests can be considered to encompass the following features:

1. Design, suitability and mechanical aspects of the test rig (Paxton 1983);

2. substrate selection and preparation (Lee et al 1974);

3. type of direct bonding adhesive (Faust et al 1978);
4. orthodontic attachment base type and size (Gwinnett 1982; Fields & Proffit 1983);

5. Test mode - tensile, shear, torsion (Thanos et al 1979);

6. storage conditions (Alexandre et al 1981);

7. test specimen age (Beech & Jalaly 1981);

8. test load rate (Smith 1982d).

Eden, Craig and Peyton (1970) developed a tensile test for evaluating the bond strength of direct filling resins to etched enamel. They concluded that a tensile test method should be simple, inexpensive and demonstrate an ability to produce results with a minimum of variability. These comments are still relevant to contemporary orthodontic bond strength tests (Hanson et al 1983; Smith & Maijer 1983b). The information gleaned from bond strength tests should aid the orthodontist in the selection of a direct bonding adhesive and give definition to the parameters that may influence the clinical success of the direct bonding technique (Faust et al 1978).

Paxton (1983), after analysing a number of strength tests for both fissure sealants and composite resin restorative materials, concluded the following:

1. The search for a test method to determine the bond strength between dental adhesives and enamel should be approached scientifically.
2. Both jig and specimen preparation must be as simple as practical. This enables rapid processing of test samples.

3. The test jig must possess a self-aligning capacity to ensure that only tensile forces act on the bonded joint. The force applied must be uniaxial, directed along the central axis of the test specimen.

4. The bond area should be constant and easily calculated. This allows the completion of the equation -

\[
\text{bond strength} = \frac{\text{max. tensile load}}{\text{area of bond}}
\]

5. An attempt should be made to obtain a uniform, flat enamel surface for use as a test site. Paxton (1983) believes that the irregular shape of the bonded tooth surface is the major factor contributing to a wide variation in results.

6. In order to measure shear bond strength, the bond surface of the tooth must be flat and aligned parallel to the shear force.

7. Of the three types of forces - tensile, shear and compressive - tensile forces are probably the easiest to design around.

Paxton (1983) considers that the poor reproductibility of bond strength results is primarily due to a complete lack of regard for mechanics and lack of a flat, uniform enamel bond test site. This
view-point is in contrast to some of the more biologically orientated, dental investigators who identify enamel heterogeneity as the major source of variable bond strength data (Aguirre et al 1982).

Low, Davies and Von Fraunhofer (1976) have also suggested six requirements that should be fulfilled by a suitable tensile bond strength test method. They are as follows:

1. Maintain a constant area of attachment of adhesive to tooth surface;

2. maintain a constant pressure of application of the adhesive material, preferably simulating conditions during clinical use of the material;

3. eliminate the use of a butt joint, or where the butt joint is used, maintain a constant thickness of adhesive at the joint;

4. apply the tensile stress directly at the adhesive-enamel junction during tensile testing;

5. the test method should be simple and easy to operate so that a large number of specimens can be tested over a short period of time;

6. the specimens must be capable of being easily subjected to conditions simulating those in the mouth whilst under storage.

When one considers the requirements of a suitable bond strength test as outlined by both Low et al (1976) and Paxton (1983), it be-
comes clearly evident that many of recently published orthodontic bond strength studies do not stand up to scientific scrutiny.

For instance, the majority of studies in Table 3 bond orthodontic brackets to variable, convex enamel tooth surfaces (Dickinson & Powers 1980; Hirce, Sather & Chao 1980; Mascia & Chen 1982; Wheeler & Ackerman 1983); many of these brackets have curved bases which, according to Paxton (1983), makes it impossible to apply a uniaxial shear or tensile load. Hanson, Gibbon and Shimizu (1983) are one of the few studies that admit their tensile tests or pull tests contain both shear and tensile components in varying proportions.

Many studies make direct comparisons with other orthodontic bond strength studies with little regard for the differences in experimental design (Faust et al 1978; Dickinson & Powers 1980). Hanson et al (1983) noted the variables within their own study that made cross-comparison with other bond strength studies hazardous. These were:

1. Jig design and test method;
2. adhesive type - they used a heavily-filled resin (Concise);
3. a three hour specimen age.

Similarly, Maijer and Smith (1981) note that their shear bond strength values were lower than those of Lopez (1980) due to the latter's use of a highly-filled resin adhesive (Auto-Tach) and to differences in experimental arrangement.
The details of the experimental design and testing jigs are inadequately defined in some studies (Aguirre et al 1982). Similarly, Wheeler and Ackerman (1983) failed to mention both specimen age and storage conditions prior to testing. Retief (1975) was unable to compare his bond strength data with the manufacturer of cosmic bond resin restorative as they failed to specify under what experimental conditions their test specimens were prepared and stored prior to testing.

The majority of recent bond strength studies have used metal edgewise brackets or lingual buttons bonded to labial or buccal surfaces of freshly extracted human teeth (Moin & Dogon 1978; Jassem et al 1981).

Some studies have used plastic (polycarbonate) brackets in bond strength tests (Faust et al 1978). Under tensile loads, the plastic brackets have distorted and fractured as the bond strength has exceeded the bracket strength (Reynolds & Von Fraunhofer 1977). It is presumably for this reason most reported bond strength studies with plastic brackets have involved shear type tests (Newman 1969a; Keizer et al 1976).

Smith (1982d) considers that the rate of machine crosshead movement can influence bond strength results. This statement is interesting in view of the vast variations of loading rates employed by recent orthodontic bond strength studies. For instance, Smith and Maijer (1983b) use a machine crosshead speed of 5mm/min to assess tensile bond strength, which is a tenfold increase over 0.5mm/min as
used by Rogers and Griffith (1977a). Similarly, Moin and Dogon (1978) use a crosshead speed of 0.2 in/min to assess shear bond strength, which is a tenfold increase over 0.02 in/min as used by Johnson et al (1976) and Jassem et al (1981). The effect which this experimental variable - crosshead speed - may have on bond strength values appears to be unknown.

Another experimental variable is specimen age. Short term tests after 1 or 7 days may not reflect long term strength maintenance because of phase changes, water sorption and hydrolytic decomposition (Smith 1982d). Storage for several months at 37°C in water often shows deterioration not experienced at earlier times (Reynolds & Von Fraunhofer 1976b).

No consensus exists as to the long term retention values for direct bonding orthodontic adhesives. Some authors report an increase in retention (Low & Fraunhofer 1976), some report a decrease (Johnson, Hembree & Weber 1976) whilst others report no change (Khowassah, Bishara, Francis & Henderson 1975). A number of studies have demonstrated changes in bond strength depending on the humidity and temperature (Khowassah et al 1975; Bishara, Khowassah & Oesterle 1975; Jassem et al 1981).

On assessment of Table 3, it is most apparent that there exists a vast variation between studies concerning specimen age (storage and time). Reynolds and Von Fraunhofer (1976a, 1976b, 1977) routinely assessed bond strength after 3 hours as, in their opinion, this period approximated the first conscious application of force by a
patient. On the other hand, Thanos et al (1979) completed bond
strength tests after 30 minutes as this was a practical time for the
orthodontist to activate the appliance. Mascia and Chen (1982) kept
the specimens for 24 hours after bonding so as to maintain consistent
bond strength values relative to other studies (Retief et al 1970);
Khowassah et al 1975; Dickinson & Powers 1980). The differences in
storage time of bonded specimens complicates the comparison of
various bond strength studies.

There exists a distinct lack of consistency between various
bond strength studies. There needs to be a scientifically designed
test rig using standardised conditions for test preparation and
storage (Paxton 1983). Many of the recent orthodontic studies are
not cognisant of many of the variables that can affect their bond
strength data. Certainly these in vitro tests have distinct limita-
tions. Without careful experimental design these limitations can
become so great as to make the bond strength data meaningless (Paxton
1983).

4-2-2 SUBSTRATE

The vast majority of orthodontic bond strength studies use
freshly extracted human teeth as enamel substrates (see Table 3). A
wide variety of human teeth have been used including incisors (Mascia
& Chen 1982), premolars (Wheeler & Ackerman 1983) and molars (Hanson
et al 1983). Often the choice of substrate is determined by the
availability of teeth, so much so that both animal teeth (bovine
enamel) and plastic cylinders have been used as alternatives (Johnson,
Many investigators favour the large, flat labial surfaces of maxillary central incisors as a bond site (Faust et al 1978; Jassem et al 1981). Premolars extracted for orthodontic purposes, provide the largest and readily available source of enamel (Wheeler & Ackerman 1983). Unfortunately, the buccal surfaces of premolars are extremely variable and convex (Fields & Proffit 1983). No manufacturer can design a bracket base that consistently matches enamel surface contour in posterior teeth (Phillips 1980a). Hence, the close adaptation of bracket base to enamel and the control of adhesive thickness is difficult. Adhesive thickness is known to affect bond strength (Silverman, Cohen & Gwinnett 1979). The closer the adaptation of the bracket base, the thinner the adhesive film and the stronger the bond (Buonocore 1963; Retief 1970).

Accordingly, Paxton (1983) claims that the preparation of a flat, smooth enamel surface is mandatory to provide reliable bond strength data. However, the majority of the studies cited in Table 3 ignore this aspect of bond site preparation. Mascia and Chen (1982) used the labial surfaces of maxillary incisors to minimize the variability of tooth anatomy and bracket position.

Some investigators have used both bovine and human enamel within the same study (Lee et al 1974; Smith & Maijer 1983b). According to Lee et al (1974), the results obtained with bovine enamel were fully comparable with adhesion to human teeth. Bovine enamel was selected for this study due to availability and uniformity of these animal teeth.

Eden et al (1970) have previously used bovine enamel as a model
dental substrate in order to rank the bonding ability of adhesives. They concluded that bovine enamel was found to preserve the rank order of bonding abilities of dental resins tested against human enamel. In addition, they noted that a far greater test variability is to be expected from the more inherently variable biological substrates, such as enamel, compared with a metal substrate, such as aluminum. The variations seen in resin/tooth bonds were due to the inherent variability of the biologic substrates used rather than the test method itself. Paxton (1983) on the other hand, believes that bond strength variability is due to the test method itself.

Dickinson and Powers (1980) used plastic cylinders as substrates because central incisors were difficult to obtain. The plastic cylinders appeared to be an acceptable model because like enamel substrate, in vitro bond failures were observed most frequently at the base/adhesive interface.

Aguirre et al (1982) actually bonded orthodontic brackets in vivo, extracted the premolar teeth and then mounted them for in vitro bond strength tests. They did however, acknowledge that extraction procedures may have affected the bond strength.

Only two studies (Beech & Jalaly 1980; Artun & Bergland 1984) referred to the previous fluoride history of the teeth they used as substrates. However, they did not discuss the potential effect this may or may not have on the bond strength results. Hirce et al (1980) have shown that topical fluoride application after etching procedures achieved the benefits of fluoride uptake without changing the bond
strength of the resin adhesive to the enamel structure. The great individual variation of both unetched and etched enamel surfaces have been clearly demonstrated by electron microscopy studies (Watson 1972).

The diverse media in which teeth have been stored prior to test preparation shows little standardisation. Teeth have been stored in a variety of storage media including distilled water (Lopez 1980), water (Reynolds & Von Fraunhofer 1976a), alcohol (Jassem et al 1981), 30 per cent saline solution (Johnson et al 1976), 10 per cent formalin (Moser, Marshall & Green 1979) and an aqueous solution of thymol (Alexandre et al 1981).

The effect that such media may have on surface enamel appears to have received scant attention in the literature. Obviously, in the interests of standardisation of orthodontic bond strength studies the type, source, preparation and storage of the enamel substrate should be rationalised, particularly as the enamel substrate is fundamental to bond tests.

4-2-3 TENSILE, SHEAR AND TORSION TESTS

The majority of orthodontic bond strength studies cited in Table 3 use only a single test mode, either tensile or shear, to evaluate the enamel/adhesive/bracket unit. According to Beech and Jalaly (1981), a bonded bracket in clinical use is subjected to forces from many directions, but all are resolvable into components at right angles (tensile) and parallel (shear) to the tooth/bracket interface. However, there does not appear to be a simple relationship between shear and
tensile bond strength. Both Beech and Jalaly (1981) and Thanos et al (1979) found that there existed a far greater variability associated with the shear data than with the tension data. In addition, the various test systems (bracket base and adhesive) generally ranked differently depending upon the test method utilized. These results would tend to reinforce Beech and Jalaly's (1981) opinion that both tensile and shear tests are required to provide a greater indication of clinical performance than either one alone.

A number of studies have used shear tests alone (Keizer et al 1976; Moin & Dogon 1978; Hirce et al 1980; Aguirre et al 1982) and others have used tensile tests only (Reynolds & Von Fraunhofer 1976a, 1976b, 1977; Dickinson & Powers 1980; Wheeler & Ackerman 1983). Only a limited number of studies have compared tensile and shear bond strength measurements (Jassem et al 1981) and only one study cited in Table 3 has compared tensile, shear and torsion tests simultaneously (Thanos et al 1979). There appears little agreement as to the optimum bond strength test.

The problem with a tensile test is that the force is transmitted through the body of the adhesive and partial cohesive failure, rather than interfacial failure, often occurs (Short, Hembree & Knight 1976). The consequent variation from specimen to specimen may obscure the interfacial bond strength (Beech & Jalaly 1980). In the determination of shear bond strength (Causton, Samara-Wickrama & Johnson 1976), the maximum force is exerted along the interface and in practice, a more reproducible interfacial fracture is observed, with much less cohesive failure (Mitchem & Turner 1974).
Mascia and Chen (1982) initially experimented with tension, shear and torsion loads to test bond strength. They concluded that only the shear mode gave consistently good results due to the variables of tooth and bracket positioning. Eden et al (1970) state that shear tests require relatively simple equipment for specimen preparation, conditioning and testing whilst providing a relatively high degree of reliability.

Maijer and Smith (1981) used a shear test mode to show that weld spots, used to attach mesh to the bracket base, reduced the available retentive surface area which may become critical in very small bracket bases.

Thanos et al (1979) determined bond strengths using tension, shear and torsion loading modes. They believed that it is not sufficient to select an adhesive on the basis of retention from one test alone; rather, the results of tension, shear and torsion tests should all be considered. During testing in torsion, a large number of bracket wings became distorted or fractured before removal of the bases from the teeth. A similar distortion and fracture of plastic brackets was noted by Keizer et al (1976). Hence, bracket systems incompatible with the test mode used invalidate test results (Reynolds & Von Fraunhofer 1977). Shear test modes place strain at the adhesive/bracket junction, so there is rarely any problem with bracket deformation. The torsional test apparatus of Thanos et al (1979) and the tensile test of Reynolds and Von Fraunhofer (1977) place a more direct strain on the bracket material.
Reynolds and Von Fraunhofer (1977) warn against using bond strength data in isolation as the only yardstick for the clinical choice of an orthodontic bonding adhesive. Other factors, such as handling properties, viscosity, cleanliness, cost, general availability and storage life can be equally as important. Likewise, Coury et al (1982) state that GIC/enamel shear bond strength studies cannot fully predict the clinical behaviour of these cements because of less controllable clinical variables.

There appears to be no consensus as to the optimal test mode for orthodontic bond strength studies. The greatest emphasis on bond strength measurement has been on tensile bond strengths (Beech & Jalaly 1980). However, there are many disadvantages inherent in tensile tests (Eden et al 1970; Williams, Von Fraunhofer & Winter 1974). Shear tests appear to provide more reliable data (Mascia & Chen 1982). Thanos et al (1979) have shown that an adhesive system (bracket base and adhesive) cannot be selected on the basis of one test (tension, shear or torque) alone as the various systems generally ranked differently depending upon the test method utilized. Moreover, bond strength data is not always a direct indicator of clinical success (Smith 1982d). In fact, laboratory bond strength tests have significant weaknesses (Artun & Bergland 1984).

4-3 BOND STRENGTH

There appears to be poor agreement amongst authors as to the definition of the terms, tensile and shear bond strength and tensile and shear strength. Whilst in some studies the authors do in fact, clearly define these terms (Dickinson & Powers 1980; Alexandre et al
1981), others do not (Smith & Maijer 1981; Mascia & Chen 1982). Consequently, these terms are used loosely and interchangeably within the literature. This confusion of terminology is highlighted by the variety of force units by which bond strength has been expressed (see Table 3).

Dickinson and Powers (1980) define tensile bond strength as the tensile force required to debond the base (Kg), divided by the nominal area of the base (mm²) to obtain bond strength.

Alexandre et al (1981) define shear bond strength as the load applied parallel to the bracket/adhesive/enamel interfaces necessary to cause failure, divided by the area of the adhesive under the bracket. In the same study, the term shear strength is expressed as the load (lbs) needed to dislodge the bonded bracket from the enamel substrate.

Wheeler and Ackerman (1983) use the term tensile bond to refer to the force in pounds required to dislodge the bracket from the tooth. No attempt is made to relate this dislodgement load to the area of the bracket base.

Lopez (1980) converts shear strength (lbs) to shear bond strength (psi) to ascertain if the size of the base significantly influences the mean shear strength. Thanos et al (1979) describe tensile bond strength as the force in pounds (lbs) required to dislodge the bond under tension. Likewise, Moin and Dogon (1978) use the term shear strength to describe the force in pounds required to disrupt the bond. Moin and Dogon (1978) do not use the term shear
bond strengths.

Therefore, it appears that the term, shear or tensile bond strength, relates the force to dislodge the bracket with the surface area of the base. Whereas, shear and tensile strength, describes the force (Newtons) or load (pounds or kilograms) to dislodge the bonded bracket.

Paxton (1983) defines tensile bond strength with the equation

\[
\text{Tensile bond strength} = \frac{\text{max. tensile load}}{\text{area of bond}}
\]

Retief (1975) notes that bond strengths obtained previously have been expressed in lb/in\(^2\) (psi) or Kg/cm\(^2\). During tensile load tests, force and not mass is measured and it would therefore be more correct to express bond strength in N/mm\(^2\) (Newtons per square millimetre) or MPa (Megapascals). The conversion can be readily obtained

\[
1 \text{ Kg/cm}^2 = 0.0981 \text{ N/mm}^2
\]

\[
\text{IN/mm}^2 = 1 \text{ MPa}
\]

Unfortunately, Retief's (1975) recommendation has been largely ignored by recent North American orthodontic bond strength studies (Dickinson & Powers 1980; Aguirre et al 1982; Smith & Maijer 1983b).

Bracket base area has been shown to have an influence on shear and tensile strength (Cavina 1977; Lopez 1980; Maijer & Smith 1981). Hence, it would seem logical to express bond strength data in terms of MPa rather than as a dislodgement force of kilograms or pounds.
This would facilitate standardisation of future studies and eliminate the need for tedious conversions in order to compare bond strength values. Table 3 lists bond strength data as Kg/cm² (Lopez 1980; Smith & Maijer 1983b), Kg/mm² (Faust et al 1978), MPa (Rogers & Griffith 1977a, 1977b; Beech & Jalaly 1981), MN/m² (Jassem et al 1981) and inches/ounce (Hirce et al 1980). This situation certainly does not facilitate ready reference and comparison between studies.

Of fundamental importance in vitro bond strength data is its relevance to the clinical situation (Moin & Dogon 1978). This relates particularly to what constitutes an acceptable, minimum in vitro bond strength (tensile and shear) relative to clinical orthodontic needs.

Maijer and Smith (1983b) state that from a functional viewpoint there are two major requirements for a directly bonded attachment:

1. The mean bond strength under functional stresses such as tensile, shear, torque and peel should be well above the minimum required to resist fracture;

2. The range of fracture strengths that is experienced under such stresses should be as small as possible. Acceptable clinical direct bonding techniques should demonstrate not only a high mean bond strength but also a narrow distribution about the mean.

Lopez (1980) states that the mean bond strength required of an adhesive is difficult to determine since occlusal loads vary enormously. To make an assessment of the force transmitted to an
individual tooth would be difficult. However, he claims that a maximum tensile value of 60-80Kg/cm² would appear reasonable for clinical requirements.

Storey and Smith (1952) used a force of 5.9 Newtons to stimulate tooth movement. Bowley (1977) related this force value to shear bond strength data. The lowest, single shear bond strength value obtained in his study was 13 Newtons (Mean 57N ± 44). This value was more than twice that of the force (4.9N) used by Storey and Smith (1952). Bowley (1977) therefore concluded that his test adhesive (Orthomite) was more than adequate for clinical service.

Reynolds and Von Fraunhofer (1976b) comment that great variability is generally found in all studies of tensile bond strengths of orthodontic adhesives. Therefore, statistical data in the form of mean values is of limited use to the clinician. Consequently, they present the minimum and maximum fracture loads as the worst and best performance that can be expected under clinical conditions. Unfortunately, Reynolds and Von Fraunhofer (1977) make no attempt to identify bond strength values adequate for clinical bonding.

Snijder, Wilson, Newman and Semen (1967) selected the average bond strength minus three standard deviations as a criterion of reliability. Keizer et al (1976) later modified this criterion to the average bond strength minus two times the standard deviation.

Careful control of technique factors such as acid etch technique, the resin system, attachment retentivity and moisture contamination
are important in reducing the standard deviation (Smith & Maijer 1983b). Keizer et al (1976) state that orthodontic adhesives should aim not only for higher average bond strengths but also for a smaller standard deviation. Both Keizer et al (1976) and Smith and Maijer (1983b) cite a requirement for the maximum force exerted on bracket to be about 30Kg per square cm (30Kg/cm²). This is half the value as estimated by Lopez (1980). Beech and Jalaly (1981) believe that there is no simple relationship between bond failure in vivo and bond strength. It is reasonable to expect that bond strengths above a certain minimum, at which good clinical performance is obtained, will not produce any improvement in failure rate.

Obviously, attempting to calculate a clinically adequate bond strength value from in vitro bond tests is extremely difficult. According to Phillips (1980a), the minimal suitable bond strength remains an unanswered question. The answer lies in a properly conducted clinical trial, over a full treatment period, on a sufficient number of patients to statistically validate the study (Artun & Bergland 1984).

The large variability of in vitro bond strength data has prompted considerable comment in the literature (Williams et al 1974; Guzman et al 1980; Jassem et al 1981; Smith & Maijer 1983b). A large variability in bond strength values casts doubt about the test method reliability and is often expressed statistically as the coefficient of variation (CV) where -
\[ \text{CV} = \frac{\text{SD}}{\bar{x}} \times 100 \quad \text{SD} = \text{standard deviation} \]
\[ \bar{x} = \text{mean} \]
\[ \text{CV} = \text{coefficient of variation} \]

(Eden et al 1970)

A small standard deviation relative to mean bond strength results in a small coefficient of variation and reflects greater reliability. According to Eden et al (1970), a small coefficient of variation for a resin-aluminium combination implied that the larger coefficients of variation seen in resin-enamel bonds were due to the inherent variability of biological substrates used rather than the test method itself.

Paxton (1983) disagrees with this statement as she believes the test method itself is the primary problem. According to Paxton (1983), coefficients of variation in excess of 40 per cent invalidate the bond strength data.

Brauer and Huget (1972) reported coefficients of variation in tensile bond strengths or composite resins to etched enamel ranging from 15-50 per cent. Coefficients of variation of the same order have been reported by Moser et al (1976) for tensile bond strength and Keizer et al (1976) for shear bond strength for orthodontic bonding resins.

Reynolds and Von Fraunhofer (1976a, 1976b) indicate that great variability of bond strength values is found in all orthodontic bond
strength studies, although the coefficient of variation never exceeded 40 per cent in their study. They propose two possible reasons for this phenomenon:

1. Within the test jig, although a universal coupling was used, it is possible that the stress exerted on the bond was not purely tensile in character.

2. There may have been areas of weakness within the filled resin, which together with the presence of voids, would affect the strength of the bond.

Rogers and Griffith (1977a) believe that the high degree of variation noted with glass ionomer cement indicates that either the test method or the material in question.

Alexandre et al (1981) believe that the high degree of variation found in their data may be due to several factors such as:

1. Minor misalignment of the testing jig;

2. uneven adhesive film thickness;

3. voids within the adhesive;

4. inaccurate proportions of base and catalyst;

5. degree of extent of etch on enamel surface.

Jassem et al (1981) attempted to reduce the introduction of
forces other than tensile or shear during the testing procedure. They claim it is impossible to eliminate these forces entirely.

Guzman et al (1980) state that the variability of in vitro bond strength data is due to differences in resin composition, including the size, type and amount of filler particles. The handling characteristics, such as mixing and working time, may contribute to variation in vitro and certainly to in vivo use. Clinical variations can be caused by patients' saliva or different operators.

Williams, Von Fraunhofer and Winter (1974) believe undoubtedly that a number of clinical variables account for the high coefficients of variation. These include the degree of drying of the in vitro enamel surfaces, the adhesive constituents, crystal orientation at the enamel surface and the degree of wetting of the enamel by the adhesive. These variables are inherent in the system and cannot be controlled.

It would appear that the variability of bond strength data, particularly tensile tests, can be due to four major factors:

1. The test method (jig) itself (Jassem et al 1981; Causton 1982).

2. The heterogeneity of enamel (Williams et al 1974; Aquirre et al 1982).


4. The adhesive (Rogers & Griffith 1977a; Guzman et al 1980).
The term, *bond strength*, has been defined and clarified relative to bond strength studies. The variety of bond strength units presently employed in the orthodontic literature have been highlighted and a need for standardisation recognised. Previous attempts to quantify a bond strength value that is clinically adequate have been reviewed. In addition, the possible causes for the variability of bond strength data, so typically inherent in these studies, have been identified.

**CONCLUSION**

This review has highlighted and discussed the features of in vitro orthodontic bond strength tests, the shortcomings of which have long been recognised (Williams et al 1973). The design and fabrication of test rigs which will produce pure tensile and shear dislodgement forces have been difficult to achieve (Retief 1973). The test mode employed should eliminate all other forces other than the test force required (Beech & Jalaly 1981). The adequacy of the design features, especially the mechanical aspects of many test methods, has been seriously questioned (Paxton 1983).

There exists no standard test procedure for orthodontic adhesive bond strength evaluation or for the bracket/adhesive/enamel bonded unit (Johnson et al 1976). Such a standardised test should possess a scientifically designed test jig and involve variables that most ideally duplicate both functional (masticatory) and appliance forces encountered during orthodontic treatment (Smith 1982d). Moreover, the size and preparation of the test specimens should reflect clinical manipulation. The lack of uniformity between tests makes it difficult
to compare results from different studies (Lopez 1980). The expression of bond strength in an array of both imperial and metric units compounds the problem.

There exists no agreement as to whether shear or tensile tests are most representative of the clinical situation (Jassem et al 1981). Thanos et al (1979) believe that shear, tensile and torsion tests together best define clinical conditions.

Buonocore (1981) believes that in vitro bond tests should not be considered valid unless specimens have been previously stored in water or artificial saliva for long periods, including stress by thermocycling. De Lollis (1980) notes that interpretation of laboratory experiments, which must of necessity attempt to isolate experimental adhesive variables, must also of necessity be limited in their application.

Beech and Jalaly (1981) stress that laboratory evidence is only suggestive of clinical performance as there exists no simple relationships between clinical bond failure and in vitro bond strength. Bond strength tests do allow adhesives to be ranked according to bond strength under ideal conditions (Eden et al 1970). According to Guzman et al (1980), if bond strength is the most important factor in selecting an adhesive, then the data does allow the orthodontist a choice of stronger or weaker cements, if other important variables such as cost and ease of manipulation are assessed.

Bond strength tests have been useful in assessing the effects of
variables which influence orthodontic bond strength. These include the acid etch technique (Bates et al 1982), the bonding agent (Faust et al 1978) and bracket base design (Hanson et al 1983). There appears to be no agreement as to whether the variability of bond strength data (coefficient of variation) is due to the test method (Paxton 1983), the innate, biological heterogeneity of the enamel substrate (Alexandre et al 1981) or the adhesive itself (Rogers & Griffith 1977a).

Smith and Maijer (1983b) appear correct in their statement that the optimal adhesive should possess a high bond strength which exhibits a narrow range about a mean. The validity of 30Kg/cm² as the maximum force exerted on a bracket (Keizer et al 1976) is questionable.

An awareness of the usefulness and limitations of orthodontic bond strength studies should allow the clinician to interpret results intelligently. Future refinements and standardisation of such studies should produce data that is less variable and more valid relative to the clinical situation.

In a recent study, Artun and Bergland (1984) investigated crystal bonding in orthodontics using students as test volunteers. In this way, the shortcomings intrinsic to in vitro bond strength evaluation were avoided. Perhaps this investigative approach to orthodontic bonding will become more prevalent in the future.
The aim of this study was to evaluate the suitability of GIC as an adhesive for direct bonding of orthodontic stainless steel brackets. This evaluation comprised:

1. Measuring tensile and shear bond strength in vitro of a representative range of GICs.

2. Comparing the suitability of plain and mesh backed stainless steel brackets.

3. Undertaking a clinical trial using GIC as a bonding adhesive in order to place the results in clinical perspective.

The results were to be assessed in relation to both the clinical performance and in vitro bond strength of two conventional resin adhesives.
CHAPTER 6

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6-3 LINGUAL BUTTONS
CHAPTER 6

6-1 GLASS Ionomer Cements

6-1-1 GLASS Ionomer Cements for Filling

These GICs are used primarily for restorative purposes. Fuji-ionomer II was selected as representative of filling cements available. This cement has been available for some years so that it has been subjected to wide clinical usage.

Fuji-ionomer Type II F is a more recently introduced material which the manufacturers claim has been developed for the treatment of deciduous teeth and the filling of simple cavities. Its major claimed difference is its quicker setting time relative to other filling cements available.

6-1-2 GLASS Ionomer Cements for Luting

Representative samples of four luting glass ionomer cements were selected for this study. These materials are recommended as luting agents for crowns, bridges and inlays. The manufacturers also claim they can be used as a lining material under both amalgam and composite resin restorations. The single, major difference between luting and filling glass ionomer cements is the smaller glass powder particle size of the luting cements that allows a minimum film thickness adequate for cementing purposes.

There are however, differences between the powder and liquid
components amongst the four luting cements selected. Fuji (I) and Hybond-C (H) are both similar in that the liquid component contains the viscous polyacrylic acid. Both Ketac-cem and Aquacem have incorporated the polyacrylic acid into the powder in a dry form. The Aquacem liquid is distilled water and the Ketac-cem liquid is distilled water and tartaric acid. The incorporation of the polyacrylic polymer into the powder provides a cement mix with less initial viscosity and a clinical feel more like that of the traditional zinc phosphate cement to which dentists are more accustomed. Details of all GICs used in the study are shown in Tables 4 and 5.

6-1-3 POWDER:LIQUID RATIO

The powder:liquid ratio used for each glass ionomer cement is indicated in Table 4. Hybond-C (H), Ketac-cem, Aquacem (A) and Fuji Type I (I) were used according to P:L ratios recommended by the manufacturers. Fuji Type I was also used at a higher P:L ratio (an increased P:L ratio from 1.3:1.0 to 1.8:1.0) to evaluate the effect of changing P:L ratio would have on tensile and shear bond strength. The P:L ratio of Fuji Type II was reduced from the manufacturer's recommended 2.2:1.0 to 1.8:1.0. This was due to the author's subjective assessment that the higher P:L ratio was too thick and viscous for bonding of orthodontic lingual buttons.

All powders and liquids of the glass ionomer cements were weighed on a Mettler pan-top scale (Plate 1) for each individual mix of cement immediately prior to the bonding of the orthodontic lingual buttons to the prepared enamel surfaces. The cements were mixed on a clean, dry glass slab at room temperature (23 ± 2°C) using a plastic spatula. Mixing time was always between 45-60 seconds, as recommended by the manufacturer.
<table>
<thead>
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<th>NAME</th>
<th>CODE</th>
<th>MANUFACTURER</th>
<th>BATCH</th>
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<tr>
<td>1. Fuji-ionomer Type II</td>
<td>II</td>
<td>G-C Dental Indust. Corp., Tokyo, Japan</td>
<td>Liquid: 050811</td>
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<td></td>
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<td>Powder: 230711</td>
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<td></td>
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<td>Powder: 230411</td>
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<td></td>
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<td>7. Monolok</td>
<td>M</td>
<td>Rocky Mountain</td>
<td>041382 Etch 020882</td>
</tr>
<tr>
<td>8. Right-on</td>
<td>R</td>
<td>T.P. Laboratories, La Forte, Indiana</td>
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II, II F - Glass Ionomer Type II (restorative material)
I, H, K, A - Glass Ionomer Type I (luting & lining)
M, R - Resin Composites (orthodontic bonding)
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<th>COMPOSITION</th>
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<td>Fuji-ionomer Type II F</td>
<td>II F</td>
<td>1.7:1</td>
<td>LIQUID: polyacrylic and tartaric acid</td>
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<td>1.3:1</td>
<td>POWDER: aluminosilicate glass</td>
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<td>1B</td>
<td>1.8:1</td>
<td>LIQUID: polyacrylic and tartaric acid</td>
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<td>Hy-Bond glasionomer C</td>
<td>H</td>
<td>1.5:1</td>
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<tr>
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<td>Ketac-Cem</td>
<td>K</td>
<td>3.4:1</td>
<td>POWDER: aluminosilicate glass and &quot;~lyacrylic acid</td>
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<td>Aquacem</td>
<td>A</td>
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<td>POWDER: aluminosilicate glass and polyacrylic and tartaric acid</td>
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PLATE 1: METTLER PAN-TOP SCALES FOR MEASURING P:L RATIOS
Both Monolok and Right-on are lightly filled composite resins manufactured specifically for orthodontic bonding (Plate 2).

**Plate 2: Orthodontic Bonding Resin Composites Used in Study**

They are similar in that they are a no-mix self-cure system using an unfilled resin primer that is brushed on to the etched enamel and mesh base immediately prior to placement of the filled resin and the orthodontic bracket. The author has used both materials clinically with satisfactory results.

The etch solutions for both Right-on and Monolok were 50 per cent phosphoric acid solutions used for a 60 second etch. The enamel surface was then washed copiously with water and dried with air from a dental chip syringe.
Details of all the materials used in this study are summarised in Tables 4 and 5 and illustrated in Plates 2 and 3. All the materials were coded for easy reference and batch numbers recorded. The materials can be classified into three main groups in which they have been discussed above.

PLATE 3: GICs USED IN THE STUDY

6-2-1 GLASS Ionomer Cements for Filling
- Fuji-ionomer Type II (II)
- Fuji-ionomer Type II F (II F)

6-2-2 GLASS Ionomer Cements for Luting
- Fuji-ionomer Type I (F I)
- Hybond Glasionomer-C (H)
- Ketac-cem (K)
- Aquacem (A)
6-3 LINGUAL BUTTONS

Two types of lingual orthodontic buttons were used in this study:

(1) Plain base stainless steel button (Dentaurum) with a base diameter of 3.0mm (Plate 4).

(2) Mesh based button (T.P. Laboratories No. 224-112) with a base diameter of 3.2mm (Plate 4).

PLATE 4: ORTHODONTIC LINGUAL BUTTONS MESH & PLAIN BASED

The base diameters were measured using a Mitutoyo Profile Projector PV600 floor model. A random sample of ten buttons of each type was measured and a mean value to one decimal place calculated (see Appendix 2). This information was necessary to determine bond
strength values (see Appendix 3). The comparative differences in base topography can be seen in Plates 5 and 6.

**PLATE 5: BASE OF PLAIN BUTTON (DENTAURUM)**

**PLATE 6: BASE OF MESH BUTTON (T.P)**
It is evident that the mesh base has been spot welded and not brazed into position. The surface of the plain button base is obviously not totally flat - there is a slight concavity with a dimple in the central region.
7-1 PREPARATION OF TEST SPECIMENS

The labial or buccal surfaces of freshly extracted human teeth with sound, intact, restoration-free enamel surfaces were used in this study. All teeth had been stored in distilled water following extraction. The roots were removed and the crowns embedded in the ends of dental stone cylinders (Investo Flintstone Water:Powder Ratio 0.24) using screw connected sectional brass moulds (Plate 7).

**PLATE 7: BRASS SECTIONAL MOULDS FOR SPECIMEN PREPARATION**

Flat enamel surfaces were obtained by abrading the crown surface under a stream of water on silicon carbide paper, finishing with grade 600. The cylinders containing the enamel specimen were held in a metal 'X' block to ensure that a perfectly flat tooth surface at right
CHAPTER 7

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angles to the sides of the stone cylinder was produced (Plate 8).

130 teeth were prepared in this manner and the cylinder specimens clearly numbered (Plate 9).
Self-adhesive PVC tape was used to mask off a circular area of a diameter of either 3.0mm (plain base dentaurum button) or 3.2mm mesh base T.P. button. Colour coded PVC tape was used so that the 3.0mm diameter and 3.2mm diameter specimens were not confused with each other prior to cementation of the button.

Immediately prior to bonding with glass ionomer cement, or resin, the exposed enamel surface was cleaned with pumice and water then dried with air using a dental chip syringe.

A stainless steel lingual button, either plain or mesh based, connected to a short, twisted, orthodontic ligature wire was cemented to the exposed enamel surface circumscribed by the PVC tape. The button was cemented immediately after completion of mixing the cement.

The ten minute test specimens were allowed to set on the laboratory bench at room temperature (23 ± 2°C) after commencement of mixing prior to testing.

The 24 hour test specimens were allowed to set on the laboratory bench for 10 minutes after commencement of cement mix prior to storage at 100 per cent humidity at 23 ± 2°C.

7-2 MEASUREMENT OF TENSILE BOND STRENGTHS

The tensile bond strength was measured at either 10 minutes or 24 hours after the commencement of mixing the specimens, using a 5T Shimadzu Autograph (Plate 10).
PLATE 10: TESTING MACHINE, 5T SHIMADZU AUTOGRAPH

The specimen cylinder was carefully loaded into a metal tube which was then locked into position on the testing machine by a cross pin. The twisted ligature wire attached to the lingual button was connected by means of a chain to the load cell (Plates 12 & 13).
PLATE 12: TENSILE TEST RIG
CONNECTED TO LOAD CELL

PLATE 13: TENSILE TEST RIG
CLOSE UP
All specimens were tested in tension to bond failure at a cross head speed of 1mm/min. with a chart speed of 10mm/min. using a 200N load cell. The only variable was the chart full scale which was calibrated to either 40 Newtons or 100 Newtons. The tensile force which caused separation of the button from the enamel surface (bond failure) was measured off the recording chart in Newtons (N) (Plate 11). These values were later converted into bond strength values (MPa) (Appendix 3).

PLATE 11: TESTING MACHINE, RECORDING CHART
7-3 MEASUREMENT OF SHEAR BOND STRENGTHS

The specimen was clamped into a metal 'X' block using the jig shown in Plate 14. The blade of the shear jig was carefully orientated adjacent to the button as shown in Plate 15.
The whole jig assembly was then locked firmly into position by creating a strong magnetic field within the heavy metal platform upon which the shear jig assembly sat. With the rigid blade screwed into the load cell, using a cross head speed of 1mm/min., the shearing force was exerted at the tooth/button base junction. Chart speed was 50mm/min. and chart full scale to either 100 Newtons or 200 Newtons.

Both 10 minute and 24 hour specimens were tested under identical experimental conditions. Specimens were stored under identical conditions as for the tensile tests.

A minimum of 6 specimens were tested for each experiment for both tensile and shear bond strengths. In some instances, the specimens were damaged loading them into the metal tube. However, there were never less than five specimens tested for any one experiment (see Table 26).

7-4 ASSESSMENT OF BOND FAILURE SITES

The enamel surface and button base were assessed without magnification immediately following fracture of the adhesive joint in order to evaluate the bond failure site. The fracture sites not readily visualised without magnification were assessed under a binocular microscope. This assessment was completed for all bond strength tests, tensile and shear.

The following code was developed in an attempt to describe as simply and meaningfully as possible the various locations and types of fracture sites:
A. Fracture at button base/cement or resin interface (adhesive failure).

B. Fracture within the cement or resin composite (cohesive failure).

C. Fracture at the cement or resin composite/enamel interface (adhesive failure).

It was not uncommon to witness a bond failure that was a combination of both adhesive and cohesive failure. In this instance the bond failure site was designated as $A + B$ or $A + B + C$ without attempting to quantify the various contribution of each site. Some of the bond failure types are illustrated in Plates 16-19.
PLATE 17: PLAIN BASE 'A + B' AND 'B' (COHESIVE) FRACTURE SITES

PLATE 18: ENAMEL SURFACE 'A' AND 'C' FRACTURE SITE
PLATE 19: ENAMEL SURFACE 'A + B' AND 'B' (COHESIVE) FRACTURE SITES

7-5 SETTING CHARACTERISTICS

Each glass ionomer cement used in this study was evaluated using an oscillating rheometer (Plates 20 & 21) at the powder:liquid ratio designated in Table 6.

PLATE 20: CLOSE-UP OSCILLATING RHEOMETER CEMENT PLACEMENT BETWEEN UPPER AND LOWER PLATEN
This enabled the determination of:

1. Initial set time (working time)

2. Final set time (setting time)

according to the method outlined in Appendix 4.

This method was originally developed for the evaluation of resin-based restorative materials. All rheometry tests were conducted at room temperature (23°C ± 2°C), chart speed was 3cm/min. Plate 21 shows the typical inverted wine glass shape of a rheometer trace. The determination of working and setting time from such a trace is described in Appendix 4.
CLINICAL TRIAL

A limited clinical trial was commenced in early 1983 when four patients had orthodontic brackets bonded with Fuji Type I (P:L ratio 1.8). Only the six upper anterior teeth had brackets bonded with GIC. The lower six anterior teeth had brackets bonded with resin composite (Monolok). The posterior teeth had orthodontic bands cemented using the same GIC as for bonding.

Two patients underwent Edgewise mechano therapy using Rocky Mountain triple control brackets. One case was uniarch treatment using high-pull headgear with anterior J hooks (Plates 22 & 23).

The other two patients underwent Begg mechano therapy using T.P. 256-500 brackets. The base area of the Begg brackets was less than the Edgewise brackets.

All brackets were cemented into position after the labial enamel surface had been pumiced, washed and dried. The brackets were then kept dry for 10-15 minutes before placement of initial archwires. No varnish was used to protect the excess GIC about the periphery of the bracket base.

The bond failure rate was assessed over an 8 months period. The sites of bond failure were also noted.
PLATE 22: EDGewise BRACKETS BONDED WITH FUJI TYPE II

PLATE 23: BRACKETS AND BONDS CEMENTED WITH FUJI TYPE II
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CHAPTER 8

RESULTS

The shear and tensile bond strengths (MPa) determined for the orthodontic adhesives used in this study are given in Table 6. These results are further represented as coloured bar graphs in Figures 7 & 8.

In broad terms, the tensile mean bond strength of GIC is similar to resin (3-5MPa) for mesh brackets at 24 hours. This is in contrast to shear bond strength where the resins are significantly stronger (14MPa) than GIC (8MPa) for mesh brackets.

In addition, the mean shear bond strengths of both GIC and resins were two to three times greater than tensile bond strength for mesh brackets at 24 hours. However, no precise relationship between shear and tensile bond strengths was apparent.

Of the two resins assessed in this study, Monolok (M) and Right-on (R), the former displayed consistently higher mean bond strengths. In fact, Monolok had the highest mean bond strength values of all materials tested. In general terms, Right-on was similar in tensile strength, but somewhat stronger in shear, than the strongest GIC tested.

The 24 hour bond strengths of GIC were at least twice that of the 10 minute values, indicating that the setting reaction was far from complete at this earlier time.
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The 10 minute test specimens were used to indicate the clinical feasibility of placing initial archwires following the bonding of orthodontic brackets. Ten minutes was considered a clinically realistic time span for initial archwire ligation. Both Right-on (R) and Monolok (M) have proved to be clinically successful in this regard by the author.

The 24 hour bond strength values represent somewhere near the maximum strength that the orthodontic brackets would attain clinically. These later bond strength values must be capable of withstanding in vivo appliance and masticatory forces over a two-year, active treatment period.

The results of this study were also assessed in the following terms:

8.1 A comparison of plain and mesh base bond strengths. It was postulated that GIC could form strong, chemical bonds directly to the bracket base, thereby eliminating the need for the mesh.

8.2 A comparison of bond strength values between the GICs and the resins (Monolok and Right-on).

8.3 The effect of P:L ratio on bond strength.

8.4 The optimum GIC in terms of the highest bond strength values.

8.5 The relationship between tensile and shear bond strength.
8.8 A limited clinical trial.

8-1 COMPARISON OF PLAIN AND MESH BASE BOND STRENGTHS

Bond strength values of GIC were compared to determine differences between plain and mesh based attachments. The data was assessed statistically using a two-way analysis of variance. The results are summarised in Table 7.

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</tr>
<tr>
<td>------------------------</td>
</tr>
<tr>
<td>10 min.</td>
</tr>
<tr>
<td>24 hour</td>
</tr>
<tr>
<td>SHEAR</td>
</tr>
<tr>
<td>10 min.</td>
</tr>
<tr>
<td>24 hour</td>
</tr>
</tbody>
</table>

HS - highly significant \( p < 0.001 \)
S - significant \( p < 0.01 \)
PS - probably significant \( p < 0.05 \)
NS - not significant \( p > 0.05 \)

It is evident that mesh base brackets provide stronger shear bond strength but weaker tensile bond strength, compared with a plain base bracket at 24 hours after cementation.
If one considers tensile bond strength, it would appear that the clean, stainless steel metal surface of the plain base has formed a strong, chemical bond with the GIC. The presence of the mesh has in some way interfered with this process. Perhaps the mesh has acted as a physical barrier, preventing the cement from completely reaching the base. The resultant bond has therefore been mechanical and/or partially chemical rather than totally chemical in nature. This situation is reflected in the cohesive type failures of plain base buttons under tensile loads (see Results 8-7).

In contrast to this situation, the shear results indicate that the mesh has reinforced the bond against shear loads. This again is reflected in the part cohesive/adhesive bond failures of mesh base buttons under shear loads (see Results 8-7), whilst the plain bases exhibited adhesive type failures at the cement/base interface.

These results do not clearly indicate whether the mesh base can be eliminated from the orthodontic attachment, particularly if shear bond strength is considered more critical than tensile bond strength relative to clinical performance. The results of the limited clinical trial should indicate whether the bond strength of a mesh base bonded bracket is adequate for clinical service.

8-2 COMPARISON BETWEEN GLASS Ionomer AND RESIN BOND STRENGTHS

A comparison between the mean bond strengths of GIC and the resins was statistically evaluated using students' $t$ tests. Only bond strength values obtained using mesh bases were compared.
Comparisons between GIC and resins were made for tensile and shear bond strengths at 10 minutes and 24 hours. The data was assessed in the following terms:

1. A comparison between pooled data of GIC (II, I-1.3, I-1.8, H, K, A) and pooled data of resins (M & R).

2. A comparison between pooled data of GIC and individual resins (M & R).

3. A comparison between individual GICs and individual resins.

**TENSILE BOND STRENGTH**

The comparison between GIC and resin tensile bond strengths is presented in Table 8. This information is also graphically illustrated in Figure 9.

At 10 minutes, the results indicate that the resins are significantly stronger than the GICs. On an individual basis, no GIC was stronger than a resin. However, Fuji-ionomer Type I P:L 1.8 (I-1.8) had an equivalent 10 minute tensile strength to Right-on (R). Surprisingly, Right-on (R) was significantly weaker (2MPa) than Monolok with a tensile strength of 5MPa.

These results suggest that only Fuji-ionomer Type I (P:L 1.8) possesses a sufficiently strong 10 minute tensile bond strength to allow early ligation of archwires. Monolok could withstand more vigorous ligation procedures than Right-on at 10 minutes. On this basis, Monolok could be recommended for rebonding procedures.
<table>
<thead>
<tr>
<th>Bond Strength Comparisons</th>
<th>Significance</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 minutes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pooled resins &amp; pooled GIC (M, R) (II I-1.3 I-1.8 H K A)</td>
<td>HS</td>
<td>Resins &gt; pooled GIC</td>
</tr>
<tr>
<td>M &amp; Pooled GIC</td>
<td>HS</td>
<td>M &gt; pooled GIC</td>
</tr>
<tr>
<td>M &amp; I-1.8</td>
<td>HS</td>
<td>M &gt; I-1.8</td>
</tr>
<tr>
<td>R &amp; Pooled GIC</td>
<td>PS 1 tail test</td>
<td>R &gt; pooled GIC</td>
</tr>
<tr>
<td>R &amp; I-1.8</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>24 hours</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pooled resins &amp; pooled GIC (M, R) (II I-1.3 I-1.8 H K A)</td>
<td>HS</td>
<td>Resins &gt; pooled GIC</td>
</tr>
<tr>
<td>M &amp; Pooled GIC</td>
<td>HS</td>
<td>M &gt; pooled GIC</td>
</tr>
<tr>
<td>M &amp; I-1.8</td>
<td>HS</td>
<td>M &gt; I-1.8</td>
</tr>
<tr>
<td>R &amp; Pooled GIC</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>R &amp; I-1.8</td>
<td>NS</td>
<td></td>
</tr>
</tbody>
</table>

HS - highly significant ($p > 0.001$)
S - significant          ($p > 0.01$)
PS - probably significant ($p > 0.05$)
NS - not significant      ($p < 0.05$)
At 24 hours, a similar pattern of tensile bond strength was evident. The resins were significantly stronger than the GICs. However, on an individual basis there was no significant difference between Right-on and both Fuji Ionomer Type I (P:L 1.8) and the pooled GIC data. As previously, Monolok was stronger than Right-on and GICs.

Using Right-on as a yardstick for adequate clinical bond strength, it would seem apparent from this study that Fuji ionomer Type I (P:L 1.8) would provide adequate tensile bond strength for the purposes of orthodontic bonding.

**SHEAR BOND STRENGTH**

The comparison between GIC and resin shear bond strengths is presented in Table 9. This information is graphically illustrated in Figure 10.

At both time intervals, the two resins collectively provided significantly stronger shear bond strength than the GICs (I-1.3, I-1.8). This bond strength differential is clearly reflected in Figure 10.

The shear strength differential was not as pronounced on an individual basis. There was no significant difference between Right-on and Fuji ionomer Type I (P:L 1.3) at 24 hours, with a similar relationship between Right-on and Fuji-ionomer Type I (P:L 1.8) at 10 minutes.
<table>
<thead>
<tr>
<th>SHEAR</th>
<th>BOND STRENGTH COMPARISON</th>
<th>SIGNIFICANCE</th>
<th>COMMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 Minutes</td>
<td>(1) Pooled resins &amp; pooled GIC (M + R) (I-1.3, I-1.8)</td>
<td>HS</td>
<td>Resins &gt; pooled GIC</td>
</tr>
<tr>
<td></td>
<td>(2) M &amp; pooled GIC</td>
<td>HS</td>
<td>M &gt; pooled GIC</td>
</tr>
<tr>
<td></td>
<td>(3) R &amp; pooled GIC</td>
<td>S</td>
<td>R &gt; pooled GIC</td>
</tr>
<tr>
<td></td>
<td>(4) R &amp; I-1.8</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>24 Hours</td>
<td>(1) Pooled resins &amp; pooled GIC (M + R) (I-1.3, I-1.8)</td>
<td>S</td>
<td>Resins &gt; pooled GIC</td>
</tr>
<tr>
<td></td>
<td>(2) R &amp; I-1.8</td>
<td>PS</td>
<td>R &gt; I-1.8</td>
</tr>
<tr>
<td></td>
<td>(3) R &amp; I-1.3</td>
<td>NS</td>
<td></td>
</tr>
</tbody>
</table>

HS - highly significant  \( (p < 0.001) \)
S - significant \( (p < 0.01) \)
PS - probably significant \( (p < 0.05) \)
NS - not significant \( (p > 0.05) \)
These results, when considered collectively for both tensile and shear bond strength, tend to indicate that GIC can provide only marginally sufficient bond strength necessary for clinical success. This situation should reflect clinically in a higher bond failure rate for GIC than the resin bonding agents. Certainly, in terms of bond strength, Monolok appeared to provide the greatest safety buffer for clinical success.

8-3 EFFECT OF POWDER:LIQUID RATIO ON BOND STRENGTH

The change in P:L ratio and its effect on bond strength is shown in Table 10. The differences between the two P:L ratios of I-1.3 and I-1.8 were statistically assessed using Student t Tests. Figure 11 graphically illustrates the effect P:L ratio had on mean tensile and shear bond strengths.

<table>
<thead>
<tr>
<th></th>
<th>SIGNIFICANCE</th>
<th>COMMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>TENSILE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 Minute: Mesh</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>Plain</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>24 Hour: Mesh</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>Plain</td>
<td>S</td>
<td>I-1.3 &gt; I-1.8</td>
</tr>
<tr>
<td>SHEAR</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 Minute: Mesh</td>
<td>HS</td>
<td>I-1.8 &gt; I-1.3</td>
</tr>
<tr>
<td>Plain</td>
<td>PS</td>
<td>I-1.8 &gt; I-1.3</td>
</tr>
<tr>
<td>24 Hour: Mesh</td>
<td>NS</td>
<td></td>
</tr>
<tr>
<td>Plain</td>
<td>NS</td>
<td></td>
</tr>
</tbody>
</table>

HS - highly significant (p < 0.001)
S - significant (p < 0.01)
PS - probably significant (p < 0.05)
NS - not significant (p > 0.05)
If one considers just the mesh base results, the only difference between the two powder:liquid ratios was at 10 minutes shear where the higher P:L ratio was significantly stronger. There was no significant difference for mesh bases at 24 hours for either tensile or shear tests between the two different P:L ratios.

The plain base results showed that at 24 hours tensile the lower P:L ratio (1-1.3) had a significantly greater (p < 0.001) tensile bond strength. At 10 minutes shear the higher P:L ratio probably had a significantly greater (p < 0.05) shear bond strength. There were no other significant differences with plain bases between the two P:L ratios.

The only advantage the lower P:L ratio (1.3) offers is an increased plain base tensile bond strength at 24 hours. This is most probably due to the formation of strong chemical with the plain stainless steel base. The increased availability of free carboxyl groups for bonding, due to reduced P:L ratio, could explain this phenomena.

Overall, there appeared to be little advantage in using the lower P:L ratio for orthodontic bonding with either mesh or plain base.

8.4 THE OPTIMUM GIC

The optimum GIC for orthodontic bonding was assessed on the basis of the highest tensile and shear bond strengths at both 10 minutes and 24 hours. Mesh and plain base bond strength values were assessed separately.
An analysis of variance was used to determine any statistically significant difference in bond strengths between the GICs.

<table>
<thead>
<tr>
<th>TENSILE</th>
<th>MATERIALS</th>
<th>SIGNIFICANCE</th>
<th>COMMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 mins</td>
<td>II I-1.3  I-1.8 H K A</td>
<td>HS</td>
<td>II I-1.3 I-1.8 K &gt; H A</td>
</tr>
<tr>
<td>24 hrs</td>
<td>II I-1.3  I-1.8 H K A</td>
<td>HS</td>
<td>II I-1.3 I-1.8 H K &gt; A</td>
</tr>
<tr>
<td>SHEAR</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 mins</td>
<td>I-1.3 I-1.8</td>
<td>HS</td>
<td>I-1.8 &gt; I-1.3</td>
</tr>
<tr>
<td>24 hrs</td>
<td>I-1.3 I-1.8</td>
<td>NS</td>
<td>No significant difference</td>
</tr>
</tbody>
</table>

8-4-1 MESH BASE

The results shown in Table 11 indicate that in terms of maximum tensile strength, four (4) GICs provided optimal performance. These materials were Fuji-ionomer Type II, Fuji-ionomer Type I (P:L 1.3 & 1.8) and Hybond.

In terms of shear bond strength, both Fuji-ionomer Type II and Hybond were not tested. Therefore, the results for tensile and shear bond strength with mesh base indicate that Fuji-ionomer Type I (P:L 1.8) is the GIC of choice for orthodontic bonding.

8-4-2 PLAIN BASE

At 10 minute tensile there were highly significant differences (p < 0.001) between the materials. II-F was significantly stronger than any of II, I-1.3, I-1.8, H, K and A. Also, A was shown to have
a significantly weaker bond strength than the rest of the GICs tested. Aquacem was considered totally inadequate for orthodontic bonding due to its delayed setting time and negligible bond strength levels at 10 minutes, despite its high tensile strength at 24 hours.

<table>
<thead>
<tr>
<th>TABLE 12: THE OPTIMUM GIC, PLAIN BASE.</th>
</tr>
</thead>
<tbody>
<tr>
<td>MATERIALS</td>
</tr>
<tr>
<td>TENSILE</td>
</tr>
<tr>
<td>10 mins</td>
</tr>
<tr>
<td>24 hrs</td>
</tr>
<tr>
<td>SHEAR</td>
</tr>
<tr>
<td>10 mins</td>
</tr>
<tr>
<td>24 hrs</td>
</tr>
</tbody>
</table>

HS - highly significant (p < 0.001)
S - significant (p < 0.01)
PS - probably significant (p < 0.05)
NS - not significant (p > 0.05)

Fuji ionomer Type IIIF is designed as a rapid setting GIC for restorative purposes in pedodontics, where moisture control and working conditions can be difficult. The tensile bond strengths of this material when compared at 10 minute and 24 hours (see Fig. 12) support the manufacturer's claim. There is no significant difference between the 10 minute and 24 hour tensile bond strengths. This is in stark contrast to all other GICs, particularly Aquacem, where the setting reaction is only partially complete at 10 minutes. However, Fuji ionomer Type IIIF (II-F) appears to pay a price for its early high strength in terms of a reduced 24 hour strength level. In fact, IIIF
has the lowest mean tensile bond strength (3.2MPa) at 24 hours of all GICs tested. II-F also produces the lowest mean shear bond strength (4.2MPa) for a plain base attachment (see Fig. 13).

Excepting Fuji-ionomer Type II-F, all the GICs tested for tensile strength with plain bases showed a significant increase in bond strength between the 10 minute and 24 hour tests.

The results of shear bond strength evaluation with plain base attachments (Table 14), indicate that for both test times only I-1.8 showed a significantly higher bond strength at 10 minutes.

Therefore, of the materials tested for both tensile and shear bond strengths with plain bases, II, I-1.3 and I-1.8 had the highest bond strengths. These three GICs would be the materials of choice for orthodontic bonding if using a plain base bracket.

It is interesting to note that both II and I-1.8 have identical P:L ratios and are in fact similar materials, the only difference being the smaller powder particle size of I-1.8.

When one considers the tensile and shear bond strengths for both plain and mesh bases, it appears that overall I-1.8 is the material of choice.

8-5 RELATIONSHIP BETWEEN TENSILE AND SHEAR BOND STRENGTHS

The relationship between tensile and shear bond strengths for both mesh and plain bases at 24 hours was investigated. Mean tensile
and shear bond strengths were compared using student t tests.

Results for mesh base showed that shear bond strength for both GIC (I-1.3, I-1.8) and resin composites (M, R) were significantly greater (p < 0.001) than tensile bond strengths (see Table 13).

This relationship is graphically illustrated in Figure 14.

The mean tensile bond strength of GIC is approximately 75 per cent of resin composites.

The mean shear bond strength of GIC is approximately 58 per cent of the resin composites.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>SIGNIFICANCE</th>
<th>COMMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>I-1.3</td>
<td>HS</td>
<td>Shear &gt; Tensile</td>
</tr>
<tr>
<td>I-1.8</td>
<td>HS</td>
<td>Shear &gt; Tensile</td>
</tr>
<tr>
<td>M</td>
<td>HS</td>
<td>Shear &gt; Tensile</td>
</tr>
<tr>
<td>R</td>
<td>HS</td>
<td>Shear &gt; Tensile</td>
</tr>
</tbody>
</table>

Results for plain base indicated that shear bond strength was not significantly greater (p > 0.05) than tensile bond strengths for all GICs evaluated (see Table 14).

However, Aquacem showed a reverse trend to the rest of the plain and all of the mesh base materials by recording a higher mean tensile
bond strength than shear bond strength.

The mean tensile and shear bond strengths for the plain base GICs are illustrated in Figure 15.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>SIGNIFICANCE</th>
<th>COMMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>NS</td>
<td>Mean tensile and shear bond strengths not significantly different</td>
</tr>
<tr>
<td>II-P</td>
<td>NS</td>
<td>Mean tensile &gt; Mean shear bond strength</td>
</tr>
<tr>
<td>I-1.3</td>
<td>NS</td>
<td>Reverse trend:</td>
</tr>
<tr>
<td>I-1.8</td>
<td>NS</td>
<td></td>
</tr>
</tbody>
</table>

These results suggest that mesh improves the shear strength of the attachment when GIC is used as an orthodontic adhesive. On this basis, there appears to be little clinical indication for the use of plain base brackets.

8-6 THE SITE OF BOND FAILURE

The bond failure sites are recorded in Appendix 1, Tables 1-25, for both glass ionomer cements and the resins. An attempt was made to determine the mode of failure quantitatively in Table 15.
I **TENSILE**

(1) **Glass Ionomer Cements**

The glass ionomer cements under tensile load (mesh and plain base) showed a range of complete or partial adhesive and cohesive bond failures. The majority of bond failures were either partially or completely cohesive, the exception being 24 hour mesh where there were no complete cohesive failures; instead, the bond failures were predominantly adhesive at the mesh/cement interface (61% Type A).

(2) **Resin Composites**

The resin composites under tensile load showed only adhesive bond failures...

At 10 minute tensile the adhesive bond failure was predominantly at the mesh/resin interface (67% Type A).

At 24 hour tensile, by which time the resin composite had completely set, bond failure was adhesive predominantly at both the mesh/resin and resin/enamel interfaces (60% Types A & C).

This is in contrast to GIC which showed a mainly cohesive type bond failure.
# TABLE 15

<table>
<thead>
<tr>
<th>BOND TEST INTERFACE</th>
<th>TYPE OF BOND FAILURE</th>
<th>GIC (mesh)</th>
<th>GIC (plain)</th>
<th>RESIN (mesh)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>TENSILE</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 min.</td>
<td>A</td>
<td>9</td>
<td>23</td>
<td>67</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>38</td>
<td>18</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>24</td>
<td>35</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>A + B</td>
<td>5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>A + C</td>
<td>11</td>
<td>6</td>
<td>33</td>
</tr>
<tr>
<td></td>
<td>B + C</td>
<td>13</td>
<td>18</td>
<td>-</td>
</tr>
<tr>
<td><strong>TENSILE</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>24 hr.</td>
<td>A</td>
<td>61</td>
<td>32</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>-</td>
<td>28</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>19</td>
<td>9</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>A + B</td>
<td>11</td>
<td>21</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>A + C</td>
<td>9</td>
<td>10</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>B + C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>SHEAR</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 min.</td>
<td>A</td>
<td>43</td>
<td>94</td>
<td>62</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>-</td>
<td>-</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>43</td>
<td>3</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>A + B</td>
<td>14</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>A + C</td>
<td>-</td>
<td>3</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>B + C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>SHEAR</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>24 hr.</td>
<td>A</td>
<td>9</td>
<td>83</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>55</td>
<td>4</td>
<td>69</td>
</tr>
<tr>
<td></td>
<td>A + B</td>
<td>18</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>A + C</td>
<td>18</td>
<td>13</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>B + C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Mode of bond failure: Type A, metal/GIC or resin interface (adhesive)

Type B, within GIC or resin (adhesive)

Type C, GIC or resin/enamel interface (adhesive)
II SHEAR

(1) GIC

The GIC under shear load showed almost entirely adhesive type bond failures.

The plain base showed only adhesive bond failure, predominantly at the metal/cement interface (10 minutes 94% Type A; 24 hours 83% Type A).

The mesh base showed mainly adhesive bond failure (10 minutes 43% Type A, 43% Type C; 24 hours 9% Type A, 55% Type C).

However, in contrast to plain base, there was also some partial cohesive bond failure (10 minutes 14% Type A & B; 24 hours 18% Type A & B).

(2) Resin Composite

The resin composites under shear load showed almost entirely adhesive type bond failures.

At 10 minutes, bond failure occurred mainly at the mesh/resin interface (62% Type A). There were a small number of cohesive bond failures (8% Type B).

At 24 hours, the main fracture site was at the resin/enamel interface (69% Type C).
III GLASS Ionomer CEMENT AND ETCHING

Aquacem was used to cement mesh based buttons to both untreated and etched enamel (37% phosphoric acid in 60 seconds). Both were tested at 24 hours for tensile bond strength.

The untreated enamel showed adhesive bond failure consistently at the cement/enamel interface (Type C).

The acid etched enamel however, showed adhesive bond failure consistently at the mesh/cement interface (Type A).

The clear distinction between Aquacem Type A and Type C bond failure sites is recorded in Plate 16.

It was noteworthy that the mean tensile bond strengths were similar (see Appendix 1, Tables 1-25) and not significantly different at the 95 per cent confidence level (p > 0.05).

These results indicate that GIC bond strength is not enhanced by acid etching the enamel. This confirms that the GIC/enamel bond is primarily chemical, not micromechanical in nature.

8-7 SETTING TIME

The time required to reach initial set and final set for all GICs used in this study was determined according to the method outlined in Appendices 4A and 4B. The results are shown in Table 16.
TABLE 16: SETTING TIME OF GICs

<table>
<thead>
<tr>
<th>GIC</th>
<th>FUJI II</th>
<th>II-F</th>
<th>I-1.3</th>
<th>I-1.8</th>
<th>H</th>
<th>K</th>
<th>A</th>
</tr>
</thead>
<tbody>
<tr>
<td>P:L ratio</td>
<td>1.80</td>
<td>1.70</td>
<td>1.30</td>
<td>1.80</td>
<td>1.50</td>
<td>3.40</td>
<td>3.30</td>
</tr>
<tr>
<td>Initial Set (mins/sec)</td>
<td>2.55</td>
<td>1.40</td>
<td>3.10</td>
<td>2.15</td>
<td>2.00</td>
<td>6.00</td>
<td>3.35</td>
</tr>
<tr>
<td>Final Set</td>
<td>11.55</td>
<td>6.15</td>
<td>13.50</td>
<td>10.10</td>
<td>10.10</td>
<td>11.00</td>
<td>14.30</td>
</tr>
</tbody>
</table>

The time for initial set ranged from 1 minute 40 seconds to 6 minutes. Ketac-cem was the slowest by a considerable margin and Fuji II-F the quickest. The mean for initial set was approximately 3 minutes as measured from commencement of mix.

It is interesting to note that the two slowest materials to reach initial set, Ketac-cem and Aquacem, are both water/powder formulations where the viscous polyacrylic acid has been incorporated into the powder.

The time for final set ranged from 6 minutes 15 seconds to 14 minutes 30 seconds. Aquacem had the slowest final set and Fuji II the quickest. The mean for final set was approximately 9 minutes 30 seconds.

The results show that for Fuji I, increasing the P:L ratio decreased both initial and final setting time.

An increase in the P:L ratio from 1.3 to 1.8 resulted in an approximate 40% decrease in time for both initial and final set.
It could be broadly stated that for the GICs tested, the mean working time (initial set) was 3 minutes and setting time (final set), 9 minutes 30 seconds.

8.8 RESULTS OF CLINICAL TRIAL

GIC BONDING

During the 8 months that the clinical evaluation of direct bonding orthodontic brackets was carried out, an overall failure rate of 27.5 per cent was observed. This represented a total of 7 failed brackets from the original 24 bonded. Four of these were T.P. 256-500 brackets and three were R.M. triple control brackets (see Table 15).

None of the brackets failed between the initial bonding appointment and the next appointment some 406 weeks later. All failures occurred later during treatment and appeared to be a result of masticatory forces.

All of the failures occurred at the cement/mesh base interface.

The failed brackets were rebonded using Fuji Type II (P:L ratio 2.0) after the remaining GIC had been debonded and the enamel pumiced. None of the rebonded brackets has failed subsequently during the period of this clinical evaluation.
**TABLE 17: RESULTS OF CLINICAL TRIAL GIC**

<table>
<thead>
<tr>
<th>BRACKET TYPE</th>
<th>BRACKET NO.</th>
<th>FAILURE NO.</th>
<th>% FAILURE RATE</th>
<th>TIME OF FAILURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>T.P. (Begg)</td>
<td>12</td>
<td>4</td>
<td>30.0</td>
<td>2, 5, 7 mo</td>
</tr>
<tr>
<td>R.M. (Edgewise)</td>
<td>12</td>
<td>3</td>
<td>25.0</td>
<td>3, 5 mo</td>
</tr>
</tbody>
</table>

**RESIN BONDING**

The same patients had orthodontic brackets bonded on to their lower anterior teeth using an orthodontic resin (Monolok, Rocky Mountain). This totalled only 18 brackets as one patient received upper arch treatment only.

Over an 8 month period, only two brackets failed - one Begg and one Edgewise (see Table 18). This represented an overall bond failure rate of 12.5 per cent over the period of the trial.

Both failures occurred at the enamel/resin interface within the first two months of treatment.

These brackets were rebonded and remained intact for the duration of the clinical evaluation.

**TABLE 18: RESULTS OF CLINICAL TRIAL RESIN**

<table>
<thead>
<tr>
<th>BRACKET TYPE</th>
<th>BRACKET NO.</th>
<th>FAILURE NO.</th>
<th>% FAILURE RATE</th>
<th>TIME OF FAILURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>T.P. (Begg)</td>
<td>12</td>
<td>1</td>
<td>8.3</td>
<td>2 mo</td>
</tr>
<tr>
<td>R.M. (Edgewise)</td>
<td>6</td>
<td>1</td>
<td>16.5</td>
<td>1 mo</td>
</tr>
</tbody>
</table>
If the failure rates of the resin bonded and GIC bonded brackets are compared, it is obvious that the GIC failure rate (27.5%) is more than twice that of the resin bond failure rate (12.5%).

The high failure rate of the GIC may be partially due to early inexperience by the clinician (K.G.M.) as the rebonded brackets remained in situ. It may also reflect the inferior bond strength values of Fuji Type II as compared with Monolok, as indicated by the in vitro bond strength studies.

The bond failures of the resin bonded brackets occurred early in the treatment period, those of the GIC bonded brackets mainly after two months.

Noteworthy was that all GIC bond failures occurred at the cement/mesh interface. The GIC remained tenaciously attached to the enamel. This indicated that the strength of the enamel/GIC bond was adequate whilst the GIC/mesh interface remained the weak link.

In contrast, the brackets bonded with resin both failed at the enamel/resin interface.
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<tr>
<td>9-2 IN VITRO STUDY</td>
<td>220</td>
</tr>
</tbody>
</table>
9-1 CLINICAL TRIAL

The clinical trial in this study was limited in both terms of sample size (42 bonded orthodontic brackets) and duration (8 months). According to Mizrahi (1983), for such a study to be valid it should involve a minimal sample size of 600 brackets and span the complete, fixed appliance treatment period.

Few studies in fact, meet these stringent requirements (Zachrisson 1976, 1977; Gorelick 1977; Newman 1978; Delmuth 1981). Regardless of the limitations inherent within the present study, it did provide a comparison between the bonding performance of the GIC and resin under clinical conditions.

The resin used in this study was Monolok which had the greatest tensile and shear bond strength of all materials tested in the laboratory. The cement used was Fuji-ionomer Type II (P:L 2.0), a filling type GIC. The same GIC, at a slightly lower P:L ratio (1.8), was assessed in vitro for tensile strength only. Its tensile bond strength was comparable to Fuji-ionomer Type I (P:L 1.8).

It should be noted that the GIC-bonded brackets were cemented only on maxillary incisors and canines, whereas the resin-bonded brackets were placed on the mandibular anteriors.

Brackets bonded to maxillary anterior teeth are known to provide the lowest bond failure rates (5-7%) due to the ease of access for
bonding and reduced occlusal forces (Newman 1978). On the other hand, brackets bonded to mandibular anteriors display a higher bond failure rate (11%) due to increased masticatory forces, especially in deep bite situations (Gorelick 1977).

The bond failure rate of approximately 12% for the resin bonded brackets agreed closely with the studies cited by Mizrahi (1983). However, the GIC bond failure rate of 28% was considered clinically unacceptable.

It was interesting to note that the resin-bonded brackets failed early in the treatment period or not at all. This agrees with previous findings by Zachrisson (1977) and Newman (1978). In contrast, all the GIC bond failures occurred after two months of treatment. This demonstrated that bond strength deteriorated with time. This may have been due to moisture contamination of the GIC, as no protective layer of varnish was applied to the bracket periphery after the attachment was bonded. Mount and Makinson (1978) have stressed that GIC exposed to the oral environment should be protected by a varnish layer to avoid contamination with water.

The brackets that were rebonded with GIC remained intact for the remainder of the clinical trial. This suggested that GIC bond failure may be due in part to early clinical inexperience as GIC is known to be technique sensitive (Mount 1981). In fact, Zachrisson (1977) has stressed that the clinical bonding procedures can be as important as bond strength in terms of reducing failure rates.
Both the resin-bonded brackets failed at the enamel/resin interface. This indicates possible moisture contamination of the etched enamel (gingival fluid, saliva), forces exceeding the bond strength (sudden impact forces of mastication, occlusion or trauma), inadequate adaptation of bracket to the tooth surface providing an uneven or thick adhesive layer, or individual variation (hypersalivation, differences in enamel composition and response to etching) (Retief 1974). Most clinical bond failures for resin adhesives occur at the enamel/resin interface (Fields 1982).

In contrast, all GIC-bonded brackets failed at the cement/mesh interface. All brackets bonded with GIC appeared to fail as a result of shear-type masticatory forces. The high bond failure rate could reflect the low in vitro shear bond strength of GIC relative to Monolok. After bond failure, the GIC remained tenaciously attached to the enamel surface. This indicated that adhesive failure at the cement/mesh interface was the weak link in the enamel/cement/bracket complex. The remaining GIC was not as difficult to remove as resin. The debonding and clean-up process was readily achieved by scraping off the remaining GIC and then polishing the enamel surface with 3M Softlex discs. A highly polished enamel surface was achieved in a minimum of chairside time.

9-2 IN VITRO STUDY

In this present study, the tensile bond strength obtained for GIC at 24 hours ranged from approximately 3-5.5MPa. These results are similar to other studies by Peddey (1977), Rogers and Griffith
(1977b), Negm, Beech and Grant (1982). Powis and his colleagues (1983), using a luting cement (ASPA IV), reported a GIC tensile bond strength to enamel of 3.2MPa. Their experimental design was similar to this present study. However, with the judicious use of enamel surface conditions (polyacrylic acid, tannic acid and dodicin), these investigators were able to increase the tensile bond strength to approximately 7MPa. Powis and his colleagues (1983) concluded that the GIC tensile bond strength was limited by the intrinsic tensile strength of the cement as failures were cohesive in nature. Owens (1981) arrived at a similar conclusion when he stated: *It becomes clear that the limitation of this system is not the adhesive bond but more so the cohesive strength of the material.*

Bowen and Cobb (1983) have also recently reported a GIC/enamel tensile bond strength of 7.2MPa. This value agreed closely with Powis and his co-workers (1983).

In the present study, tensile bond failures of plain base attachments were 49 per cent cohesive or mixed cohesive in nature. The balance of failures were adhesive. The plain base tensile strength at 24 hours was significantly greater than mesh base. This suggested that the cohesive strength of GIC was a limiting factor for the tensile bond strength attainable with this cement. The upper limit of GIC tensile bond strengths in their present state of development appears to be about 7MPa (Bowen & Cobb 1983; Powis et al 1983).

If one considers that the mean tensile bond strength of the two resins in this study was approximately 5MPa, it would appear that GIC if used judiciously, has satisfactory tensile bond strength for use as
an orthodontic bonding adhesive.

The resin tensile bond strength in this study was low compared with other published results. Beech and Jalaly (1981), using a similar experimental method, evaluated a number of orthodontic bonding resins. The mean tensile bond strengths at 24 hours ranged from 7-11MPa. Rogers and Reynolds (1977b) used an innovative bracket-base design for the evaluation of a variety of resin and cement orthodontic adhesives. The tensile bond strengths were 20-30MPa for the highly-filled resins. These investigators noted that the bond strength obtained for the acid etch, BIS-GMA and acrylic resins were far in excess of the GIC (5MPa) and polycarboxylate cement (5.2MPa) tested.

Other studies have indicated resin tensile bond strengths ranging from 10-17MPa (Hotz et al 1977, Jassem et al 1981, Owen 1981).

A number of factors may explain the relatively low resin tensile strength values in this study relative to previous investigations. Firstly, both Monolok and Right-on are lightly-filled resins (Smith & Maijer 1983a) which are known to have less strength than the heavily-filled resins such as Coniase (Jassem et al 1981). Secondly, tensile bond strength is known to be affected by mesh size (Reynolds & Von Fraunhofer 1976a; Dickinson & Powers 1980) and spot welds which attach the mesh to the bracket base (Maijer & Smith 1981). The mesh bases in this study were spot welded and of a coarse mesh type (see Plate 6). All these factors may have contributed to a reduced bond strength although it should be noted that in vitro failures were adhesive at either the resin/mesh interface or the enamel/resin interface.
The GIC shear bond strengths obtained in this study were about 5MPa for plain base and somewhat higher, at 7-9MPa, for mesh base attachments. The resin shear bond strength at 24 hours was considerably higher at 14MPa.

Carlyle et al (1978) made the only other study which has compared the shear bond strength of an orthodontic resin with a GIC (luting type, ASPA IVA). They concluded that the shear bond strength of the resin (Nuva Seal/Nuva Tach) was significantly greater than the GIC. This conclusion agreed with the present study, but the extent of the difference in shear strength was more exaggerated (Carlyle et al 1978). The resin shear strength (4.2MPa) was more than four times that of the GIC shear strength (0.9MPa). However, the test load in this study was applied to the bracket by means of a stainless steel orthodontic wire loop ligated into the slot. In the author's opinion (KGM), this was not a true shear-type force as compared with the method used in the present study. Additionally, the test specimens were stored for 120 hours at 100 per cent humidity which may have allowed moisture to affect the GIC strength.

Certainly, the 0.9MPa GIC shear bond strength, as reported by Carlyle and his colleagues (1978), was low relative to this and other studies. Cowry et al (1982) found a shear bond strength of 3.8MPa for GIC bonded to enamel whilst Negm et al (1982) reported a shear strength value of 1.6MPa. However, both the latter studies used only a drop of GIC on the enamel, thereby eliminating any influence of a cement/bracket base interface.
In the Carlyle et al (1978) study, all bond failures occurred at the GIC/enamel interface. This agreed with the present study where 55 per cent of failures occurred at the same site. This information is in contrast with the clinical trial where all the GIC-bonded bracket failures were at the cement/bracket interface. The in vitro results were not able to indicate clinical experience. This highlights the difficulty of making an assumption concerning clinical success from laboratory results. Zachrisson (1977) has stated that laboratory testing of bond strength is most difficult to correlate to expected clinical results and only long-term clinical evaluations of large sample populations may give clinically valid data.

The resin shear bond strengths obtained in this study were similar to previous investigations (Lopez 1980; Jassem et al 1981). Boyer et al (1982) used both lightly and heavily-filled resins to indicate the effect of particle concentration on shear bond strength. The range of shear bond strength values obtained were 14-26MPa. This correlated closely to the shear strength of 14MPa obtained with the lightly-filled resins used in this study.

The question which invites discussion must be: Do the GICs possess sufficient in vitro bond strength to indicate clinical success as a direct orthodontic bonding adhesive?

It is reasonable to expect that bond strengths above a certain minimum, at which good clinical results are obtained, will not produce any improvement in bond failure. Possibly of equal importance to a high bond strength value is the variability about the mean (Smith &
Maijer (1983b). Reynolds and Von Fraunhofer (1976b) consequently presented the minimum and maximum fracture loads as the worst and best performance expected under clinical conditions. Bowley (1977) followed a similar line of reasoning, using the force value required to move a tooth (5.9 Newtons) as a guide to minimal shear bond strength. Both Keizer et al (1976) and Smith and Maijer (1983b) cite 2.9MPa (30Kg/cm²) as the maximum force exerted on a bracket under clinical conditions.

Lopez (1980) considered that 5.8MPa–7MPa (60–80Kg/cm²) represented a lower threshold shear bond strength value for satisfactory clinical performance. According to Newman (1965), the orthodontic force applied to brackets was approximately 1MPa. He stated that a load of approximately 3MPa was the maximum which probably occurred under clinical conditions. Miura et al (1971) used an acrylic resin as an orthodontic adhesive, which yielded clinically satisfactory results over a two year period. This acrylic resin had a shear bond strength of 5.1MPa. This value agreed closely with Lopez's (1980) assessment of a clinically adequate in vitro shear bond strength of 5.8–7.8MPa.

From the above it may be concluded that the GIC tensile bond strength, approximately 3–5MPa, is sufficient for orthodontic bonding. This conclusion is reinforced by a comparison with the clinically-proven resin tensile bond strength of 5MPa.

The GIC shear bond strength of 7–9MPa with mesh base attachments likewise appears sufficient for adequate clinical performance. An
unacceptable bond failure rate could indicate that shear bond strength, rather than tensile strength, is a more accurate in vitro indicator of clinical performance.

The weaker shear bond strength of GIC (7-9MPa) relative to resin (14MPa) may be critical in terms of clinical success. The high bond failure rates for GIC-bonded brackets suggest a minimal acceptable shear bond strength of between 7 and 14MPa. Within this range of shear bond strengths there appears to be a critical value for a clinically acceptable performance. In fact, this shear bond strength range is not a great deal above 5.8-7.8MPa (60-80Kg/cm²) as recommended by Lopez (1980).
CHAPTER 10  

SUMMARY AND CONCLUSIONS

The bond strengths of a range of commercially available GICs were investigated using plain and mesh based orthodontic buttons attached to human enamel. Tensile and shear bond strengths were evaluated at both 10 minutes and 24 hours after the commencement of mixing. Two resin orthodontic adhesives were assessed under identical experimental conditions for mesh base attachments only. These two resins, Monolok and Right-on, had previously proved to be clinically successful in the hands of the author (KGM). Therefore, the resins were used as an in vitro bond strength yardstick against which GIC bond strength could be measured.

A clinical trial was instigated so as to assess the in vivo suitability of GIC as a direct orthodontic bonding adhesive. This clinical trial was limited in terms of sample size and duration. The findings of the controlled clinical study should help place the laboratory tests into appropriate context.

All the data was statistically analysed and the following conclusions were drawn:

10.1 In vitro there appeared to be no clinical advantage to be gained by eliminating the mesh base when GIC was used as an orthodontic bonding adhesive. Mesh based brackets provided a significantly greater 24 hour in vitro shear bond strength than plain base brackets.
10.2 The in vitro study suggested that the tensile bond strength of GIC when compared with resin was sufficient to indicate likely success as a direct orthodontic bonding adhesive. However, the shear bond strength of GIC was significantly less than that of the resin adhesives.

10.3 An increase in the P:L ratio of Fuji-ionomer Type I from 1.3 to 1.8 will increase the early shear bond strength of mesh base brackets whilst decreasing both the working and setting times. This should be advantageous for orthodontic bonding.

10.4 On the basis of optimum tensile and shear bond strength, the luting type GIC, Fuji-ionomer Type I (P:L 1.8), could be recommended as a direct bonding orthodontic adhesive for future controlled clinical studies. According to Mizrahi (1983), this clinical trial should include a sample size of at least 600 brackets and extend over a full treatment period.

10.5 Coefficients of variation ranging from 15-55 per cent were observed in the bond strength data of this study. This variability of data was similar to many previous bond strength studies. As there was rigorous control of all stages of the experimental procedure, it appears that heterogeneity of the enamel substrate, rather than the adhesive or test method, was the major cause of this variability.

10.6 The relationship between tensile and shear bond strength differed for mesh and plain base attachments. For plain base,
there was no difference (p > 0.05) between GIC tensile and shear bond strength. However, for meshbase brackets, both resin and GIC shear bond strengths were significantly greater (p < 0.001) than tensile strength.

10.7 The mean working time for the GICs tested was 3 minutes which was considered adequate for the purposes of orthodontic bonding. However, the mean setting time of 9 minutes 30 seconds was considered excessive for an orthodontic adhesive. The prolonged setting time of GIC increases the susceptibility of the cement to moisture contamination and precludes early ligation procedures.

10.8 The use of GIC as an orthodontic bonding adhesive demands careful attention to clinical manipulation and handling. A cold slab technique is recommended for multiple placement of orthodontic brackets. This allows the clinician sufficient working time to carefully place and align the brackets on a number of teeth with a single cement mix. A capsulated GIC, suitable for orthodontic bonding, would simplify bracket placement and guarantee a consistency of cement mix. A capsulated GIC would also ensure an optimal P:L ratio. The GIC flash extruded peripheral to the bracket base should be trimmed away with a sharp probe before the cement sets. The remaining exposed GIC should be immediately coated with a protective varnish layer.

10.9 Fuji-ionomer Type II-F had both the shortest working and setting time. However, the advantage of early strength appeared to be negated by a reduced tensile strength at 24 hours relative to
other GICs tested. The water-mix GICs, Ketac-cem and Aquacem, were considered to be totally unsuitable as orthodontic bonding adhesives due to their prolonged setting time. In addition, their reduced viscosity permitted excessive bracket drift when the orthodontic attachments were positioned on the enamel surface.

10.10 Noteworthy was the fact that early GIC bond strength values were approximately half their 24 hour levels. This reflected the prolonged and overlapping reactions that occurred during cement formation. A considerable increase in cement bond strength was apparent over the first 24 hours. This would suggest extreme patient care of GIC bonded fixed appliances over the initial 24 hours.

10.11 The clinical study indicated that GIC was technique sensitive and possessed inadequate bond strength for orthodontic treatment. The clinical study also suggested that shear, rather than tensile bond strength, was more crucial to clinical performance. The high bond failure rate may be due to insufficient shear bond strength, early clinical inexperience or lack of a protective varnish layer.

10.12 It is more likely that further development of GICs will take place as these cements are still in their infancy. Continued research may be able to increase the cohesive strength of these cements to equal that of the resin adhesives. Such improvements should enable the clinical realisation of the major advantages offered by GIC as a direct orthodontic bonding adhesive. These
advantages are:

1. That there is no need to acid etch enamel as GIC is adhesive.

2. That the leaching of fluoride from GIC is anticariogenic and prevents decalcification about bonded brackets.

3. That the bonding of orthodontic attachments becomes a rapid and simplified prophylaxis-then-cement procedure.
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<th>Author(s)</th>
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APPENDIX 1

EXPERIMENTAL DATA
<table>
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<tr>
<th>CEMENT P:L RATIO</th>
<th>TIME INTERVAL BASE TYPE</th>
<th>LOAD AT FAILURE NEWTONS</th>
<th>BOND STRENGTH MPa</th>
<th>MEAN BOND STRENGTH MPa</th>
<th>S.D.</th>
<th>FRACTURE SITE</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>10 MINUTE</td>
<td>(1) 4.4</td>
<td>0.55</td>
<td></td>
<td></td>
<td>A + B</td>
</tr>
<tr>
<td>FUJI TYPE II</td>
<td>MESH</td>
<td>(2) 7.3</td>
<td>0.91</td>
<td></td>
<td></td>
<td>B + C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(3) 14.8</td>
<td>1.84</td>
<td></td>
<td></td>
<td>B + C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(4) 7.0</td>
<td>0.87</td>
<td></td>
<td></td>
<td>A + B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(5) 14.7</td>
<td>1.83</td>
<td>1.32</td>
<td>0.61</td>
<td>B + C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(6) 15.3</td>
<td>1.90</td>
<td></td>
<td></td>
<td>B</td>
</tr>
<tr>
<td>1.8:1.0</td>
<td>10 MINUTE</td>
<td>(1) 4.4</td>
<td>0.62</td>
<td></td>
<td></td>
<td>A + B + C</td>
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<tr>
<td></td>
<td>PLAIN</td>
<td>(2) 20.6</td>
<td>2.91</td>
<td></td>
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<td>B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(3) 11.5</td>
<td>1.63</td>
<td></td>
<td></td>
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<td>8.83</td>
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<td>(10) 71.2</td>
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APPENDIX 2

CALCULATION OF BASE BONDING AREA

A. BASE BONDING AREA

Ten mesh and plain based buttons were selected randomly and their diameter measured to the third decimal point using a Mitutoyo Profile Projector FV600 floor model. The readings provided a mean figure to the nearest decimal point.

<table>
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<th>Mesh</th>
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<td>2.960</td>
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<td>2.993</td>
<td>3.197</td>
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<tr>
<td>1.993</td>
<td>3.183</td>
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</table>

\[ \bar{x} = 2.991 \quad 3.191 \]

SD 0.012 0.008

Diameter of plain button base = 3.0mm
Diameter of mesh button base = 3.2mm
Area of base = \( \pi r^2 \)

1. Plain button = 3.14 x (1.5)² = 7.068mm²
2. Mesh button = 3.14 x (1.6)² = 8.042mm²
APPENDIX 3

BOND STRENGTH CALCULATION

BOND STRENGTH = FORCE PER UNIT AREA

= \frac{FORCE \ (Tensile \ or \ shear \ load \ to \ cause \ bond \ failure)}{AREA \ OF \ BUTTON \ BASE}

= \frac{NEWTONS}{mm^2}

= \frac{MN}{m^2}

= MPa \ (Megapascal)
APPENDIX 4A

METHOD FOR DETERMINING INITIAL SET TIME

A1 SCOPE. This Appendix sets out the method for determining the initial set time of Type I resin-based dental restorative materials.

A2 PRINCIPLE. The time interval from the start of mixing to a stage at which the viscosity and elastic modulus of the material increase rapidly is determined by an oscillating rheometer.

A3 APPARATUS. An oscillating rheometer, which records graphically or instrumentally the viscosity changes in the setting material, is required.

A suitable instrument which comprises the following is illustrated schematically in Fig. A1:

The main housing (A) is mounted and can rotate in a ball race (B), which has very little friction. The lower platen (C), 10 mm in diameter, is held in the main housing by two grub screws (D) and a knurled screw (E), with the upper platen (M), 10 mm in diameter, in position on its guide, a distance of 1 mm above. The main housing and the lower platen are driven in an oscillatory motion by means of a 10 r/min electric motor attached to an eccentric wheel (F) which is connected to arm (G) by means of two balanced and tensioned coil springs (H). The maximum rotary movement of arm (G), when no material is on the platen, is 1° 12' ± 12'. This rotary movement is measured by means of a transducer (I), the output from which is fed to a centre zero microammeter or, alternatively, to a suitable automatic recording apparatus.

The instrument is checked by attaching a fine nylon thread to hook (J). A weight having a mass of 10.2 g is attached to the other end of the thread which passes over a PTFE bearing. Hence a force of 0.1 N is applied at right-angles to arm (G) at point (J) which is 54 mm from the centre of platen (C); this is equivalent to a torque of 5.4 N mm. When this calibration torque is applied, the deflection on the microammeter should equal the deflection (i.e. half of the total swing) obtained when the instrument is running with no material on the platen.

A4 PROCEDURE. Prepare the test material in accordance with the manufacturer's instructions. Start timing from the start of mixing. Place a sufficient amount of the mixed material on platen (C) and lower the upper platen (M) on its guide screw into position 60 s after the start of mixing, ensuring that the material fully contacts both platens but does not significantly exude from the edges of the platens. Maintain the water circulating through M at 23 ± 1°C.

The transducer output should be fed into a suitable automatic recording apparatus giving a continuous trace such as that shown in Fig. A2. Measure from the commencement of mixing, the time at which the amplitude of oscillation is less than or equal to 95 percent of the original amplitude. This is the initial set time.

A5 REPORT. The initial set time shall be reported to the nearest 5 s.
Fig. A1. SCHEMATIC REPRESENTATION OF OSCILLATING RHEOMETER
APPENDIX 4B

METHOD FOR DETERMINING FINAL SET TIME

B1 SCOPE. This Appendix sets out the method for determining the final set time of Type I resin-based dental restorative materials.

B2 PRINCIPLE. The time interval from the start of mixing to the stage at which the elastic modulus has reached a certain high level is determined by an oscillating rheometer.

B3 APPARATUS. The rheometer apparatus described in Paragraph A3 of Appendix A is required.

B4 PROCEDURE. Carry out the procedure described in Appendix A, circulating water at 37 ± 1°C through the platen (M). The water shall have been circulating for at least 1 h prior to starting the test.

From the curve obtained (see Fig. B1), record the final set time as the time at which the amplitude equals 0.5 percent of the original amplitude.

B5 REPORT. The final set time shall be reported to the nearest 5 s.

Fig. B1. TYPICAL RHEOMETER TRACE OBTAINED DURING THE MEASUREMENT OF FINAL SET TIME

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Fig. A2. TYPICAL RHEOMETER TRACE OBTAINED DURING THE MEASUREMENT OF INITIAL SET TIME