

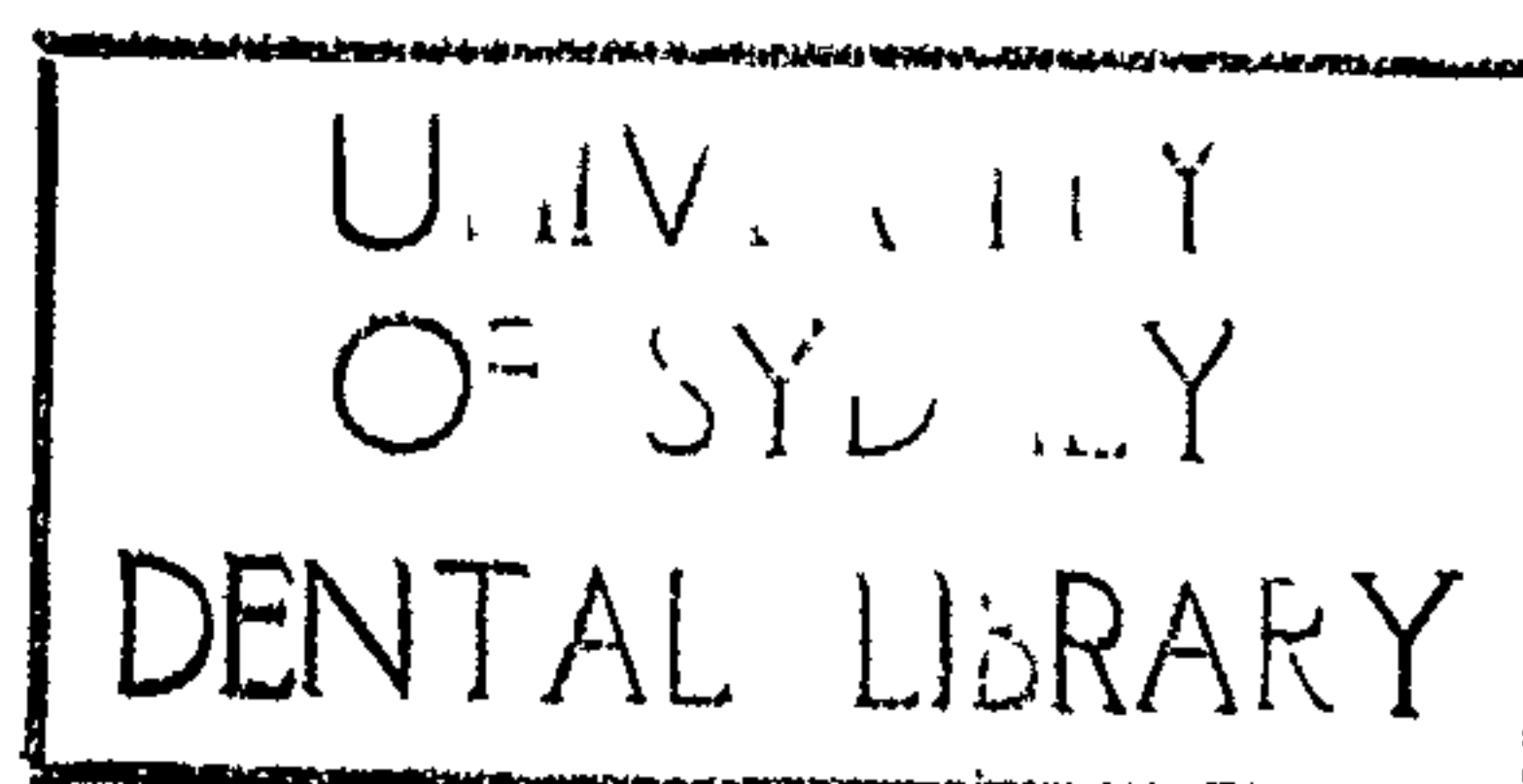
# REDUCED ETCHING TIMES:

A clinical study of orthodontic bracketing  
using a reduced etching time  
in a group of Sydney school-children

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## ABSTRACT

A clinical study was undertaken on a group of 17 Sydney school-children, using a reduced etching time in the bonding of brackets for routine Begg LWT. A 20-second-etch was found to give similar retention rates to those achieved with a normal 60-second-etch over an observation period of 6-8 months.

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## DEDICATION

*A Dedication to my Wife*

To whom I owe the leaping delight  
That quickens my senses in our wakingtime  
And the rhythm that governs the repose of our sleepingtime,  
The breathing in unison

Of lovers whose bodies smell of each other  
Who think the same thoughts without need of speech  
And babble the same speech without need of meaning.

No peevish winter wind shall chill  
No sullen tropic sun shall wither  
The roses in the rose-garden which is ours and ours only

But this dedication is for others to read:  
These are private words addressed to you in public.

T.S. Eliot Collected Poems 1909-1962 (Faber & Faber)

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# 1 INTRODUCTION

From the dental and orthodontic literature it seems that reduced etching times should produce adequate conditions for the successful bonding of orthodontic brackets. This clinical study was undertaken to see whether Begg brackets bonded to teeth etched for 20 seconds would be retained as successfully during routine treatment as those bonded subsequent to a conventional 60 second etch. It should be stressed that this study was conceived to simulate as much as possible a standard, routine clinical situation; one that might apply in any orthodontic situation, except that, as the time of etch was the critical variable, this was given over to greater scrutiny in this study (ie measured to the second).

A number of studies have been carried out showing that shorter etch times provide a similar if not identical etching pattern to the more conventional 60 second etch (Brännström & Nordenvall, 1977; Barkmeier, Gwinnett & Shaffer, 1985; Barkmeier, Gwinnett & Shaffer, 1986).

A superior etching pattern, in terms of greater surface irregularities as viewed by SEM, has been reported by Nordenvall, Brännström & Malmgren (1980) and Brännström, Malmgren & Nordenvall (1982) for a reduced etch time.

Reported *in vitro* bond strength differences between reduced and normal etch times are not statistically significant according to a number of authors (Beech & Jalaly, 1980; Barkmeier, Gwinnett & Shaffer, 1985; Barkmeier, Shaffer & Gwinnett, 1986). In contrast, Main et al (1983) reported a 21% reduction in *in vitro* bond strengths for fissure sealants using a reduced etch time, although the recorded bond strength was still high. Mardaga & Shannon (1982) also recorded some

reductions in *in vitro* bond strengths (44.6% for a 20 second etch) for the bonding of orthodontic buttons.

Stephen et al (1982) found higher retention rates for fissure sealants using a reduced etch time, although Eidelman, Shapira & Houpt (1984) found no statistically different rates of retention in their study (20 versus 60 seconds).

Carstensen (1986a) recorded similar retention rates of edgewise brackets for a 15-20 second or 30-35 second etch time. This was initially after determining that a 30-35 second etch trial produced bracket failure rates lower than some of those reported in the literature.

Greater loss of gross enamel has been shown to occur with a 60 second etch compared to a 20 second etch (Barkmeier, Gwinnett & Shaffer, 1985). Shey & Brandt (1982) noted that enamel loss is proportional to etch time.

It seemed reasonable, therefore, to postulate that reduced etch times could lead to reduced enamel loss, yet essentially the same underlying histologic and morphologic changes, resulting in clinically acceptable bonding and retention rates for orthodontic brackets during the usual treatment period.

It would be necessary to conduct a study along the lines of Fitzpatrick & Way (1977), Brown & Way (1978) or Pus & Way (1980) to determine if there is less enamel removed after debonding and subsequent clean-up when a 20 second etch time is employed. Further studies could be carried out subsequent to debonding comparing the relative stain uptake between a 20 second etch group and a control group

etched for 60 seconds. These considerations are important ones, especially as they reflect the concerns expressed by many dentists and orthodontists concerning iatrogenic treatment effects. However, they cannot be addressed satisfactorily in this study, and they await further consideration.

Bonds that survive the early period are apt to remain throughout treatment (Zachrisson, 1977) and bonds that fail, generally fail early, that is, within a few weeks (Gorelick, 1977). This study was undertaken over 6 to 8 months to ensure that the period of observation was adequate.

The following literature review attempts to provide an overview of the nature of enamel; the concepts of adhesion and the special problems posed by the oral environment; the acid-etching and subsequent bonding to enamel; and clinical failure of orthodontic attachments. Following this is an explanation of the clinical study of reduced etching times applied in the direct bonding of Begg brackets to a group of Sydney school-children undergoing routine orthodontic fixed-appliance therapy at the United Dental Hospital of Sydney (fluoridated water supply since 1968).

## 2 ENAMEL

Human dental enamel is a unique tissue. Enamel is the most highly calcified or highly mineralised tissue in the human body (Scott & Symons, 1974 p188; Ten Cate, 1985 p198). It is also the hardest of the body's tissues (Scott & Symons, 1974 p188; Sheykholeslam & Brandt, 1977). Its density decreases from the surface of the enamel to the dentino-enamel junction, and from the incisal edge to the cervical margin (Scott & Symons, 1974 p188). Enamel is extremely hard due to its high mineral content, and is comparable to mild steel although it is quite brittle (Ten Cate, 1985 p198). Enamel is thickest over the working surfaces, that is, occlusally and incisally (up to 2.5 mm), reducing to a fine or feathered edge cervically (Scott & Symons, 1974 p188; Ten Cate, 1985 p198).

Due consideration must be given to the chemical composition and structure of enamel, as enamel is the substrate to which we bond our orthodontic attachments. Acid etching removes a shallow layer of enamel, removing with it plaque, surface and subsurface cuticles and chemically inert crystallites in the surface enamel. In addition to this, the remaining subjacent enamel is rendered histologically porous (Silverstone, 1975). The fourth part of this literature review will deal with the preferential etching of enamel prisms that results from topical phosphoric acid application. The high-energy surface so-created is the indispensable basis for the micromechanical bonding, and perhaps the adhesion, that secures the bonded orthodontic appliance in such an unforgiving environment as the mouth.

## 2.1 CHEMICAL COMPOSITION

### 2.1.1 Inorganic

Mature enamel contains a high proportion of inorganic salts, approximately 96% by weight (Scott & Symons, 1974 p188; Osborn & Ten Cate, 1976 p97; Ten Cate, 1985 p198) The generally agreed structure is that of apatite, specifically, hydroxyapatite (crystalline calcium phosphate):  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$  (Scott & Symons, 1974 p190; Ten Cate, 1985 p198). Other ions may be present due to their incorporation or adsorption during the formation process, for example: sodium, magnesium, carbonate, strontium, lead and fluorine (Scott & Symons, 1974 p189; Ten Cate, 1985 p198).

Scott & Symons (1974 p190) report that the mineral of enamel is three times the mass of the organic portion and thus this inorganic component is approximately 86% by volume.

The water content of the enamel is approximately 12% by volume. It exists loosely bound between the crystallites or firmly bound as hydration shells (Scott & Symons, 1974 p190). The water content of the enamel diminishes during the process of enamel maturation (Osborn & Ten Cate, 1976 p101).

### 2.1.2 Organic

Scott & Symons (1974 p188) and Ten Cate (1985 p198) both state that the remaining 4% by weight of enamel is made up of water and organic material.

This organic content is difficult to assess according to Scott & Symons (1974 p189) because it is both small in absolute quantity, and,

during extraction, likely to be contaminated by the adjacent cementum or dentine. This organic fraction is 2% by volume or 0.4 to 0.8% (permanent enamel) or 0.5 to 0.9% (deciduous enamel) by weight. Further they state that this organic fraction is often concentrated as tufts or lamellæ. The organic content comprises soluble and insoluble proteins, peptides and citric acid. The soluble and insoluble proteins occur in approximately equal amounts and are a function of the demineralisation of enamel by ethylene diamine tetra-acetic acid (EDTA) or weak organic acids. Foetal enamel has as much as 19% organic content, and, as it matures, remarkable changes take place. There is a considerable loss of proline and histidine and this suggests their importance in enamel formation and so the term *Amelogenins* has been used to describe that fraction of the organic matrix containing them. Foetal enamel is unlike both the keratins and collagen in its amino acid content.

Osborn & Ten Cate (1976 pp98-101) state that the nature of the organic component is not fully understood. Whilst the proportions of the constituent amino acid residues are well known, the configuration of the resultant proteins is not. The very small quantities make isolation difficult. The amino acid ratios are unique to enamel; there is an unusually high proportion of proline (25%). The presence of histidine, lysine and arginine in a ratio of 3:1:1 distinguishes these proteins from the eukeratins (ratio of 1:4:12 respectively), as does the very small amount of cystine, which is approximately a quarter the amount found in the keratins. Further, the protein mix changes during maturation, so that the final organic matrix of enamel not only has less protein, but protein of a different composition.

Osborn & Ten Cate (1976 p100) propose that the large numbers of proline residues suggest the presence of a relatively unstable

polypeptide, and that the structure of the organic component is amorphous and gel-like rather than an oriented, fibrous assembly. Within this distinctive thixotropic matrix, apatite crystals develop.

Ten Cate (1985 p198) states that a fine, lacy network of organic material can be seen to exist between the densely packed crystallites when viewed via the scanning electron microscope. This material is largely proteinaceous with some polysaccharide content. High molecular weight proteins (enamelins) persist in mature enamel and are tightly bound to the apatite crystallite surfaces, occupying all of the intercrystallite spaces.

## **2.2 ULTRASTRUCTURE**

### **2.2.1 Crystallites**

The primary structural unit of the enamel is the crystal or crystallite (Meckel, Griebstein & Neal, 1965a). The chemical composition of these is generally accepted to be hydroxyapatite (Johansen, 1964; Meckel, Griebstein & Neal, 1965a; Glas & Nylen, 1965; Helmcke, 1967; Orams, Zybert, Phakey & Rachinger, 1976; Ten Cate, 1985 pp201-202), although Palamara, Phakey, Rachinger and Orams (1980) have also found whitlockite and possibly octacalcium phosphate present in some large crystals in surface enamel. Scott (1965) states that there is no evidence of a bundle or network of fibrils within the enamel crystals.

Crystallite shape has been described as lath-shaped or irregular hexagonal (Johansen, 1964) or long and lath-like (Meckel, Griebstein & Neal, 1965a). Nylen (1967) described generally long rods with hexagonal, yet highly irregular cross-sections and Scott & Symons (1974 p191)

described slightly flattened hexagonal rods of fairly uniform size. Orams et al (1976) demonstrated close-packed long laths in longitudinal section and hexagons in cross section.

The observations of crystallite size vary widely. Johansen (1964) measured a width range of 500 to 1000 Å and lengths up to 10 000 Å. Glas & Nylen (1965) noted hexagonal pencils with diameters of 200 by 800 Å of indeterminate length. Orams (1966) observed widths which varied from 1200 Å down to 100 Å. Scott & Symons (1974 p191) report an average width of 400 Å, an average thickness of 250 Å and state that the length of the crystallite may be 1600 to 10 000 Å.

### 2.2.2 Prisms

The shape of the enamel prism or rod is a subject of some conjecture. Lester (1964) notes a variety of rod shapes including hexagonal or round (least common), through very irregular, to polygonal/oval to arcade shaped (most common). Meckel et al (1965a; 1965b) contend that the most frequently observed cross-sectional shape is the keyhole. It has a head of approximately 5 µm in diameter, a tail 5 µm long (giving an approximate overall length of 9 µm). Longitudinal sections viewed also with the scanning electron microscope (SEM) corroborate this structural description.

Hardwick, Martin & Davies (1965) state that the prism appearance varies considerably, but that the most typical was the main body of a rod enclosed by a horseshoe-shaped sheath with intra-rod material flowing out into a tail. Further, Hardwick (1965) made some observations on the variability of shape of enamel prisms, finding square shaped prisms, arcade/horseshoe shaped prisms and horseshoes which failed to touch (the tail therefore broadening). He reported the

conclusion that the keyhole shape cannot be accepted as the generalised form.

Long, rather irregular half-cylinders is the description given by Glas & Nylen (1965), which had extended from the flat surface a narrow fin, the inter-rod substance.

Nylen (1967) attempted to reconcile seemingly opposing views by stating that whilst the arcade-shaped prism is accepted as the most prevalent type, the arcade designates the prism sheath and the prism in toto is, in transverse section, a keyhole. Swancar, Scott & Njemirovskij (1970) conclude that the arcade-shaped rod is the most plausible and versatile concept of prism structure. Osborn & Ten Cate (1976 p110) feel that the two conflicting descriptions of enamel structure, viz, keyholes with no interprismatic regions or circular prisms separated by interprismatic regions, are related solely to differences in terminology. They state that there is no disagreement about the structure being described, and further, both descriptions require the construction of imaginary lines to complete the prism borders. However, they feel it is less confusing, more consistent and probably more realistic to describe human enamel prisms as roughly circular in continuity with the interprismatic region below. Ten Cate (1985 p201) feels that the keyhole analogy does not adequately account for all structural arrangements of enamel. He agrees the width of the rod is of the order of 5  $\mu\text{m}$ .

Meckel et al (1965a) made three-dimensional models of enamel blocks using extruded prisms which were keyholes in cross-section. They made sections at various angles to the two reference planes of head-tail direction and prism long axis. From this they predicted the

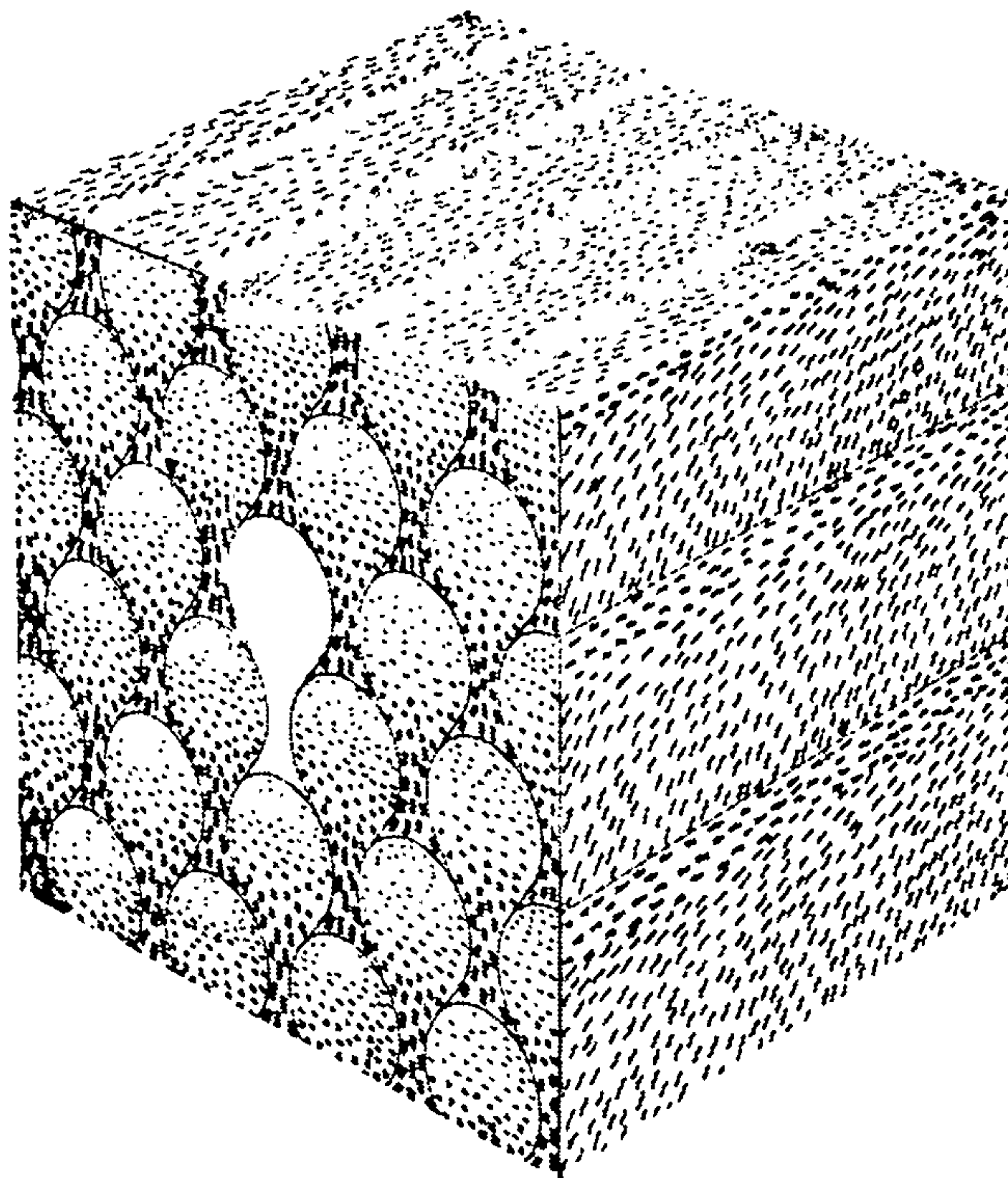
SEM appearance of real sections of enamel cut at those same angles. They conclude that many observations of enamel structure are therefore compatible with their postulated model including the common arcade, and that this "keyhole" is the basic structure. Swancar et al (1970) whilst disagreeing about the nature of the prism shape, agree that the cross-sectional angulation does influence the appearance of the prism outlines within the section.

The orientation of the prisms appears to be such that the head lies occlusally and the tail cervically (Meckel et al, 1965a,b; Glas & Nylen, 1965; Helmcke, 1967).

Crystallite orientation within the prism has been reported by a number of authors. Poole & Brooks (1961) observed a gradual change in crystallite orientation from one side of the prism to the other. Glas (1962) observed that the mean direction of the crystallites within the prism was towards the cervical. Ripa, Gwinnett & Buonocore (1966) and Gwinnett (1966, 1967) also observed that the mean direction of the outer poles of the crystallites within the prismatic enamel points cervically.

Meckel, Griebstein & Neal (1965a,b) describe the crystallite orientation for the keyhole in relation to the prism's long axis. In the front (occlusal) half of the head, the crystallites are parallel to the long axis. As the tail is approached the crystallites display a gradual change in direction, until within the tail the crystallites assume a nearly perpendicular arrangement to the prism axis. Within the tail they fan out from the prism midline. Helmcke (1967) states that the prism axis is eccentrically placed, lying to the occlusal side of the prism.

Crystallite deviation is greater in a sagittal plane than in a



Cardboard model of dental enamel structure.

**Griebstein (1965), Discussion of 4th session.  
In: Tooth Enamel, Its composition, properties and  
fundamental structure. (Eds: Stack & Fearnhead) p 190.  
John Wright & Sons, Bristol**

mesiodistal plane according to Glas (1962), who reported an average deviation of crystallites (from the prism axis) of 30 to 40°, with a maximal deviation of 73°. Hardwick et al (1965) also noted a change in orientation of crystallites between body and tail. Griebstein (1965) states that crystallites may lie at an angle of up to 70° to the prism long axis. Meckel et al (1965a) report the average angulation as about 30° with 60 to 70° in the tail region. Glas & Nylen (1965) agree; the crystallites being more nearly parallel to the long axis within the rod itself, whilst those in the fins deviated by up to 40 to 45°. Gwinnett (1966, 1967) observed a similar spread of crystallites within the prism as did Helmcke (1967).

### **2.2.3 Interprismatic Enamel**

Meckel et al (1965a,b) concluded that there is no evidence for the existence of a separate interprismatic material; all crystallites could be assigned to a specific prism. Hardwick et al (1965) regarded the "interprismatic cement" as the tails of the next layer of rods. Helmcke (1965) states that rods and interprismatic substance are structurally linked together, are identical in both organic and inorganic make-up and differ only in the direction of their structural elements. Glas & Nylen (1965) determined that both rod and inter-rod regions are mineralised to the same degree. Orams (1966), likewise, could not demonstrate a separate interprismatic substance.

Gustafson & Gustafson (1967) observe that the interprismatic substance can be abundant or partially or totally absent. Most likely, they say, there are no differences, at least quantitatively, between prism core, sheath and interprismatic substance. Helmcke (1967) reiterates that there is no special interprismatic substance. Swancar et al

(1970) contend that the core of the prism is confluent with, and similar to, the interprismatic material. Osborn & Ten Cate (1976 p110) also feel no difference exists between prism core and interprismatic enamel.

#### **2.2.4 Prism Sheaths**

Prism sheaths have been reported to contain a relatively higher content of organic material (Johansen, 1964; Glas & Nylen, 1965; Gustafson & Gustafson, 1967; Scott & Symons, 1974 p194; Ten Cate, 1985 p201). Lester (1964) reports of a matrix of submicroscopic fibrils, often divisible into inter-rod substance, rod sheaths and rods themselves. Rod and inter-rod areas also were noted to exhibit a feltwork of fibrils by Scott, Ussing, Sognnaes & Wyckoff (1952) and Johansen (1964). The matrix was found to be uniformly distributed through rod and inter-rod areas (Glas & Nylen, 1965).

The prism borders are readily apparent because of sudden changes in orientation of crystallites at the junctions of adjacent prisms. From this arises the characteristic herringbone pattern (Meckel et al, 1965a,b; Nylen, 1967; Ten Cate, 1985 p201). Further, Meckel et al (1965a) reject the contention that mineral-free, organic-rich gaps (so-called sheaths) are present: none was found in their SEM study. Orams (1966) found no continuous structure that could be described as a distinct sheath. The observed spaces, he concluded, could be artefact or acid-soluble organic sheaths lost during etching for preparation.

The width of the prism sheath has been variously reported as 1000 to 1500 Å (Boyde, 1965), 1000 to 2000 Å (Glas & Nylen, 1965; Scott & Symons, 1974 p194) and 0.1 µm at most (Osborn & Ten Cate, 1976

p113).\*

### 2.2.5 Surface Layer

The perikymata are horizontal, rib-like structures long considered to be the termination of the Retzius lines at the surface (Gustafson & Gustafson, 1967 p112; Osborn & Ten Cate, 1976 p115).

Newman & Poole (1974) noted great variation in surface appearance. The spacing and amplitude of the perikymata may vary from region to region; and, whilst bands of pitted enamel most commonly alternate with smooth, there may be areas which are either all smooth or all pitted (with or without perikymata). The small pits in the troughs of the perikymata were observed to be the ends of rods and the smooth areas, aprismatic enamel. Sometimes frequent and widespread outcrops or depressions were seen that were larger than the prism ends (to the order of 5 to 60  $\mu\text{m}$ ).

A number of authors have remarked on the presence of aprismatic or prismless enamel on the surface of both deciduous and permanent teeth (Ripa et al, 1966; Gwinnett, 1966, 1967; Nylen, 1967, Newman & Poole, 1974; Ten Cate 1985 p202). The incidence has been reported as 100% and 70% for deciduous and permanent enamel, respectively (Ripa et al, 1966). The width of the prismless enamel has been reported as approximately 30  $\mu\text{m}$  (Ripa et al 1966; Gwinnett, 1966; Ten Cate, 1985 p202). The crystallite orientation of the prismless enamel differs in relation to the mean crystallite orientation of the underlying enamel in deciduous teeth by approximately 27° (Ripa et al, 1966) and 33° (Gwinnett, 1966). This is not so in permanent enamel; the crystallite

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\*0.1  $\mu\text{m}$  is equal to 1000 Å.

orientation being within the range of spread of the underlying enamel crystallites (Gwinnett, 1967). The crystallites have a preferred, parallel arrangement which is closely packed and approximately perpendicular to the enamel surface (Ripa et al, 1966; Gwinnett, 1966, 1967; Nylen, 1967; Newman & Poole, 1974; Ten Cate, 1985).

Ripa et al (1966) found three striking features of prismless enamel to be the difference in crystallite orientation, the absence of prism markings and fine, surface-parallel laminations. This prismless enamel is more radiodense than the underlying enamel (Gwinnett, 1966, 1967) and Nylen (1967) suggests this is due to the absence of prism sheaths and the closer packing of crystallites.

Palamara et al (1980) examined the surfaces of unerupted teeth and discovered a primary enamel cuticle of no apparent structure, free from micro-organisms and with a thickness of 0.5 to 1  $\mu\text{m}$ . Under this cuticle the crystalline surface is composed of small ( $\sim 10\text{ nm}$ ) loosely packed crystals. Some big crystals ( $\sim 500\text{ nm}$ ) were found among the small ones and in the cuticle. They postulate that this layer is lost following emergence and the coarser, sub-surface layer is exposed to form the new surface typical of erupted teeth.

Brudevold (1960), in listing the concentration gradients of a number of minerals, states that more fluoride is present in surface enamel. Speirs (1971) agrees that the highest concentration is in the outer 20  $\mu\text{m}$ . Scott & Symons (1974) allow that surface enamel is different (cf subsurface enamel) in that it has 5 to 10 times more fluoride, it is harder, it is less soluble, it has a higher concentration of carbohydrate and it may have areas of prismless enamel.

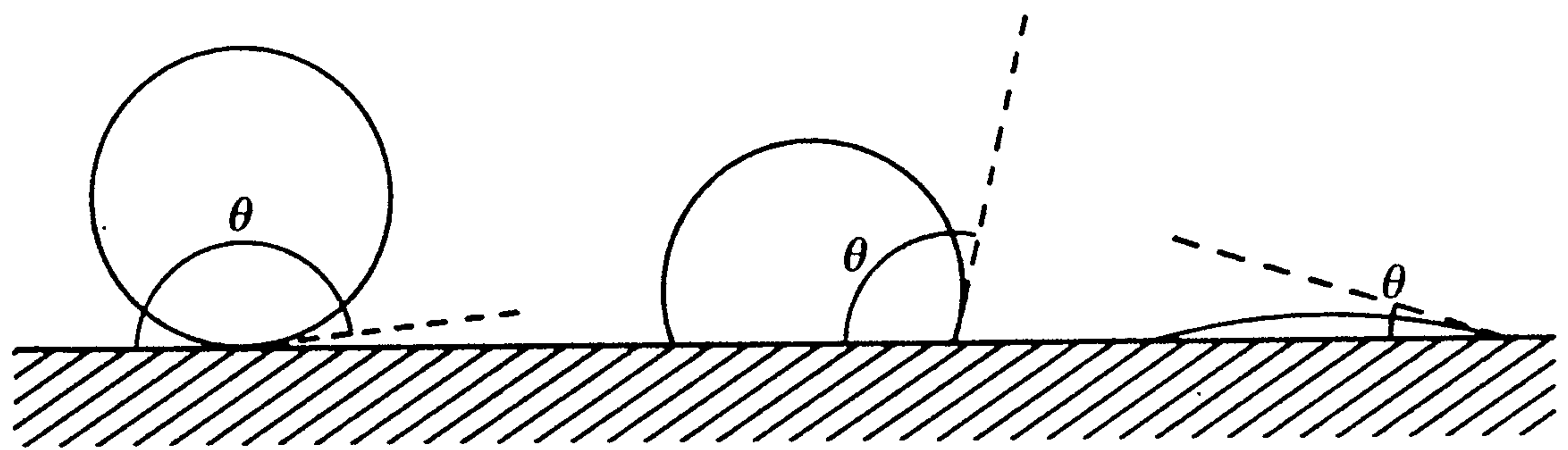
### 3 ADHESION AND BONDING

Adhesion in its broadest sense is simply surface attachment (Beech, 1977). It may be further defined as the forces of attraction between atoms or molecules across an interface, that is, at the surfaces of different materials when they are brought into contact (Buonocore, 1963, 1975a; Phillips, 1973; Beech, 1982; Smith, 1982). Adhesion is dependent on interfacial contact because the molecular forces of attraction do not operate beyond a few Ångström units. This range has been defined as 7 Å (Phillips, 1973), 3 to 4 Å (Retief, 1975a), or 2 Å according to Gwinnett & Smith (1982). Adhesion does not refer to any other means of joining; and the term "true adhesion" is a tautology (Buonocore, 1975a). Smith (1975) states that a minority of authors would consider "adhesion" to be comprised of both mechanical interlocking and interfacial molecular attraction, although he and others would disagree, and call this phenomenon "bonding".

An adhesive is a material which unites, joins or attaches itself to other substances; and commonly used synonyms are cement, glue and bonding agent. An adherend is the surface to which an adhesive is bonded - generally a solid to which a liquid adhesive is attached (Buonocore, 1975a).

Cohesion is the same molecular attraction involved in adhesion but involves like molecules instead of unlike molecules (Buonocore, 1975a).

Only the presence of physical and chemical bonds constitutes adhesion; the term "bonding" is more general and can encompass micromechanical interlocking also (Gwinnett & Smith, 1982). Buonocore (1975a) refers to bonding as a general term describing the joining, uniting or attaching of adhesives to a substrate (eg enamel). Bonding is



NON-WETTING

WETTING

SPREADING

Contact angle  $\theta$  in the different situations of (a) non-wetting, (b) wetting and (c) spreading.

From Driessens (1977), Chemical adhesion in dentistry.  
Int Dent J 27:317-323

an observable fact which does not specify the means of attachment, that is, adhesion or mechanical retention.

### 3.1 WETTING

A fundamental requirement (for adhesion) is the establishment of intimate interfacial contact between the adhesive and the substrate or adherend (Huntsberger, 1965), that is, good wetting must take place (Eick, Johnson, Fromer, Good & Neumann, 1972; Beech, 1982). For the adequate reaction of adhesion to occur, the fluid adhesive must wet and spread on the adherend surface to achieve the requisite molecular closeness (Smith, 1982).

At an atomic level, solid surfaces are not smooth, and it is very difficult to get them to adhere to one another. Molecular attraction is virtually negligible when surface molecules are separated by more than a few Å. One way to overcome this is to use a liquid adhesive (Phillips, 1973; Buonocore, 1973; Driessens, 1977). If solid surfaces were atomically smooth and flat they would spontaneously adhere when brought into contact. Further, a liquid adhesive will bond to an adherend surface or unite two solid surfaces only if it is able to wet the surface of the materials; and this wetting is a manifestation of the attractive forces between the molecules of the adhesive and the adherend (Buonocore, 1975a; Retief, 1975a), so that, when these attractive forces are high, wetting will occur (Buonocore, 1975a). As a general principle, the greater the affinity between the adhesive and the substrate, the greater the wetting will be (Driessens, 1977).

Wetting may thus be described as the process of obtaining molecular nearness or establishing interfacial contact; and maximal wetting is the state in which all of the interfacial contacts possible

have been established (Buonocore, 1963, 1975a; Huntsberger, 1965).

### 3.2 CONTACT ANGLE

Wetting can be described in geometric terms related to a wetting contact angle (Gwinnett & Smith, 1982). This is the angle formed by adhesive and adherend at their interface; it demonstrates the extent to which the adhesive will wet the adherend (Phillips, 1973). The contact angle can be influenced by certain properties, mainly of the substrate (adherend) surface. It is an inverse measure of the relationship between the free surface energy of the solid and liquid in contact (Glantz, 1969). The contact angle is an inverse measure of wettability and the cosine of this angle is an obvious direct measure of this relationship (Brauer, 1975; Retief, 1975a).

The phenomenon of wetting is associated with the existence of a small or zero contact angle. The stronger the attraction, the smaller will be the wetting angle (Buonocore, 1963). Driessens (1977) states that when the contact angle ( $\theta$ ) is large, no wetting is observed, but when it is a few degrees only, spreading occurs.

If the contact angle is small, the adhesive will wet the adherend surfaces and the molecular forces of adhesion will operate along the entire surface and strong adhesion will result (Retief, 1975a). When  $\theta$  equals zero, the liquid will spread spontaneously over the solid surface (Brauer, 1975). Phillips (1973) states that if the molecules of the adhesive are attracted to the molecules of the adherend as much or more than they are to themselves, the liquid adhesive will spread completely over the surface of the solid and no angle will be formed.

For a given adherend, several adhesives may show zero contact

angles yet have different degrees of attraction for the adherend. These differences in attractive forces cannot be ascertained on the basis of zero contact angles as it is obvious that this is the smallest angle that can be measured (Buonocore, 1975a).

Buonocore (1975a) also notes that some liquids will spread over a surface appearing to form small contact angles, thus presenting the same appearance produced by substances that have a strong molecular attraction for one another. In fact there is an absence of strong molecular attraction due to the extremely low cohesive forces (ie low surface tension) of the liquid. Huntsberger (1965) argues that zero contact angles are not necessarily associated with either the extent of wetting or maximal rates of wetting. He makes the point that it does not require a zero contact angle to get things to adhere strongly, one to the other.

Retief (1978) maintains that a small contact angle indicates wetting of the adherend surface resulting in increased bond strength whilst a large contact angle demonstrates the converse. Beech (1982) proposes that complete wetting occurs when the contact angle is zero or when  $\cos \theta$  is equal to one.

### **3.3 FORCES OF ADHESION**

Phillips (1973) outlines three types of bonds:

- (1) primary interatomic bonds;
- (2) secondary interatomic bonds;
- (3) metallic bonds.

Only the first two will be considered here (as they relate to adhesion).

Primary or chemical bonds are of two types, ionic or covalent. The secondary, physical bonds are also known as van der Waals forces. These

include the effects of both fluctuating and permanent dipoles. Hydrogen bonding is one of the most important examples of permanent dipole interactions. Physical forces of adhesion are weaker than chemical forces.

Buonocore (1975a) classifies the physical forces of adhesion as:

(1) van der Waals forces, consisting of:

(a) Debye forces due to the existence of induced dipoles;

(b) Keesom forces due to permanent dipoles;

(c) London forces, which are due to the nonpolar dispersion effects resulting from random electronic or atomic motion, and are the strongest of the three forces.

(2) Hydrogen bonds are a special dipole-dipole interaction being greater than the other van der Waals forces yet considerably weaker than those involved in the formation of chemical bonds.

He describes the chemical forces of adhesion as:

(1) ionic bonds (electron transfer);

(2) covalent bonds (electron sharing).

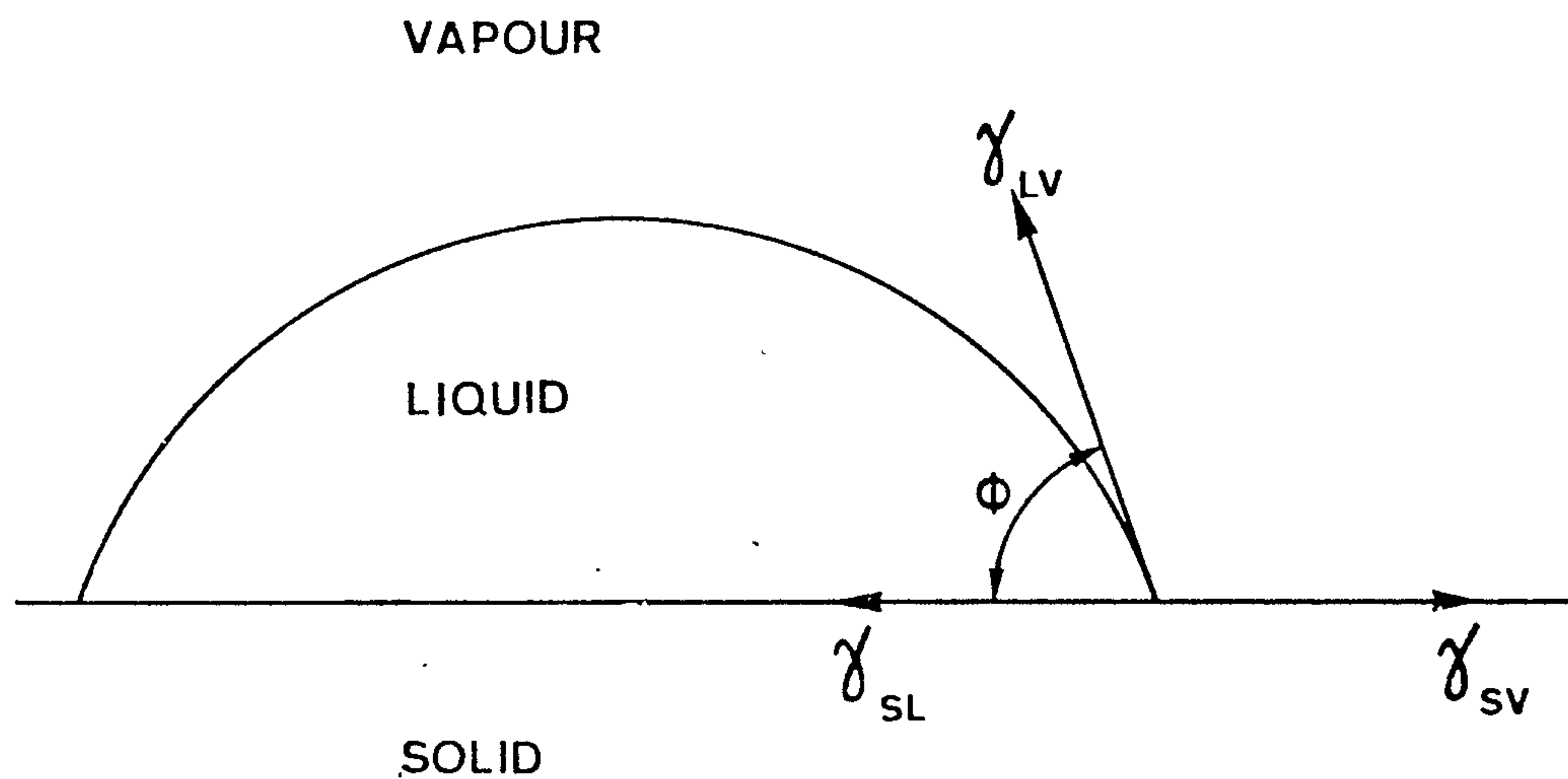
Retief (1975a) describes the forces of adhesion in a similar way to Buonocore (1975a), although he describes hydrogen bonding as a type of primary or chemical force.

Glantz (1969) likewise mentions primary chemical bonds but describes the physical forces somewhat differently, subdividing them into electrostatic forces, van der Waals-dispersion forces, hydrogen bonds and pi-adducts. Glantz (1977) classifies the attractive forces as:

(1) long-range (electrostatic and dispersion forces);

(2) short-range (chemical bonds);

(3) intermediate-range (hydrogen bonds).



Contact angle ( $\Phi$ ) between a liquid (L) and a plane solid (S) surface.  $\gamma_{LV}$ ,  $\gamma_{SV}$  and  $\gamma_{SL}$  denotes the free surface energies (or the surface tensions) of the interfaces between the liquid and the saturated vapour (V), the solid and the saturated vapour, and the solid and the liquid respectively.

**From Glantz (1977), Adhesion to teeth.  
Int Dent J 27:324-332**

Beech (1977) states that the weaker van der Waals forces are by nature intermolecular as opposed to the intramolecular primary bonds in which electron sharing or exchange takes place. Beech (1982) reports that hydrogen bonding may be both intra- and intermolecular and is intermediate between the forces of physical adsorption and the forces of chemisorption. He states that the physical forces are effective only over a distance of one or two atomic diameters, varying as the inverse of the seventh power of the distance separating the molecules. Ionic (chemical) forces vary likewise, but only as the inverse of the second power of the distance.

Mechanical retention can be brought about by the interlocking of the adhesive and adherend due to either natural or artificially created micro-irregularities on the adherend surface (Buonocore, 1975a). In addition to mechanical and secondary bonding forces, chemical reaction may subsequently occur between reactive groups in the adhesive and adherend, and may possibly provide increased joint strength and greater chemical and water resistance (Buonocore, 1973). For adequate penetration of the irregularities by the adhesive, good wetting is required (Beech, 1982). Water is a strongly polar compound (Buonocore, 1973) and in an aqueous environment like the mouth, physical forces will generally be broken down by penetration of water along the interface, but the mechanical interlocking, if well formed, should resist displacement (Beech, 1982).

### **3.4 SURFACE FREE ENERGY**

The energy at a solid's surface is greater than in its interior, because the outermost atoms or molecules are not attracted equally in all directions (Phillips, 1973). Surface tension (most commonly

associated with liquids), is related to the forces of cohesion and is the result of the inward attraction of molecules at the surface by molecules in the interior (Buonocore, 1975a). To extend the surface area against this inward pull requires energy (to do work) – the surface free energy. As a result of this tendency to contract, the surface of a liquid acts as if it were in a state of constant tension and the surface tension is a force acting equally at right angles to all points on the surface (Beech, 1982). More energy will be expended in extending a surface of high surface tension than one of low surface tension (Buonocore, 1975a).

Generally speaking the harder the surface of a solid and the higher the melting point, the higher its free surface energy and hence its adhesiveness. The converse is also true. (Glantz, 1977). There is a mathematical relationship between the contact angle and the surface tensions or energies of a liquid adhesive on a solid adherend at equilibrium (Glantz, 1969; Driessens, 1977; Beech, 1982). Low energy surfaces are generally not favourable to bonding (Beech, 1982).

### **3.5 PROPERTIES OF THE ADHESIVE AND ADHEREND**

The establishment of a proper adhesive joint is affected by a number of properties of both the adhesive and the adherend and their interaction.

The extent to which a liquid will wet a surface depends on the viscosity of the liquid, the topography of the solid surface, the free energy of the surface and the contact angle formed between adhesive and adherend (Zisman, 1963 cited by Retief, 1978; Glantz, 1969; Eick, Johnson, Fromer et al, 1972). The cleanliness of a surface is critical as an adsorbed film may lower the surface energy (behaving like a material of low surface energy), adversely affecting wetting and hence adhesion

(Phillips, 1973).

### **3.5.1 Viscosity**

Viscosity is the property of resistance to flow; an internal friction (Retief, 1975a). Viscosity describes consistency, according to Buonocore (1975a), and a thick material cannot be expected to flow readily over a surface nor to fill surface irregularities as rapidly or to the same extent as a more fluid adhesive. Therefore viscosity can interfere with the establishment of maximal wetting despite a strong attraction existing between a viscous adhesive and an adherend. Incomplete wetting is conducive to the entrapment of air at the base of pores, crevices and capillaries.

Buonocore (1975b) reported some disadvantages of a too viscous adhesive. He found a quantitative and qualitative decline in tag formation of a dental resin (adhesive) on etched enamel (adherend). The tags were foreshortened and fewer in number; in other words wetting was incomplete. The expected effects would be a weaker bonding due to the decreased surface area of attachment; the formation of areas of stress concentrations due to the presence of voids; and microleakage due to the less intimate and weaker attachment.

The viscosity of the adhesive makes wetting a time-dependent process. The greater the viscosity, the longer is the time required for spreading and attainment of mechanical equilibrium; and therefore, a low viscosity adhesive is imperative for good wetting (Driessens, 1977).

### 3.5.2 Polarity

Polarity in a molecule is due to the existence of distinct regions of positive and negative charges. Hydroxyl ( $-OH$ ), carboxyl ( $-COOH$ ), cyano ( $-CN$ ) and amino ( $-NH_2$ ) are strongly polar groups which confer polarity on a molecule; and the more of these groups present, the greater the polarity. Hydrogen bonding can be considered as being due to the strong electrostatic forces between or within polar substances. In theory, the strongest bonds should be formed between polar substances. It is obvious that, as water is a strongly polar material, adsorption to other polar substances is favoured (for example, enamel) and therefore militates against a good bond (Buonocore, 1975a).

Certain chemical groups have an effect on surface energy and therefore adhesiveness. Those that decrease surface energy are  $-CH_3$ ,  $-CH_2-$  and  $-F$ . Those groups that increase surface energy are  $-OH$ ,  $-SH$ ,  $-COOH$  and  $-NH_2$  (Beech, 1982). Polarity and surface free energy (of an adherend) are directly related; therefore the wettability of a substance is also directly related to its polarity (Buonocore, 1975a).

### 3.5.3 Surface tension of the adhesive

Other things being equal, a relatively free-flowing liquid adhesive with a high surface tension would tend to fill the surface irregularities more rapidly than one with a lower surface tension, because the high surface tension acts to reduce the surface area after the adhesive has wet the substrate (Buonocore, 1975a).

### 3.5.4 Surface free energy (SFE) of the adherend

Wettability is determined by the surface chemical properties of the outermost atoms in the exposed atom groups on the solid surface, and

this is true of a high energy surface covered with a (low energy) organic film (Glantz, 1969). Adsorbed water generally decreases the surface energy of the adherend - one monolayer at room temperature can convert a high energy surface to a low energy one. As more water is adsorbed, the more the surface resembles that of bulk water in respect of its critical surface tension (Beech, 1982). It is axiomatic that the SFE of the solid must be greater than the SFE of the liquid for suitable wetting to occur. This is another way of saying that the attraction of the liquid adhesive molecules for those of the adherend should be greater than the mutual attraction of the adhesive molecules (Buonocore, 1975a). Further, most adhesives will readily wet and bond to high energy surfaces (Beech, 1977) which have considerably greater surface free energies than liquids (Buonocore, 1975a).

### **3.5.5 Surface topography**

Surface roughness can determine the extent of wetting (Brauer, 1975). Eick, Johnson, Fromer et al (1972) showed that the topography of the adherend, in this case tooth structure, does play an important role in the formation of an adhesive bond as the rugosity may affect wetting. Some roughness may promote wetting through capillary pressure effects whilst too much may act as a barrier, allowing the formation of air pockets. Jendresen, Glantz, Baier & Eick (1981) found that increased roughness of a solid surface will increase its wettability if the liquid in contact with a plane surface of that solid forms an angle of less than  $90^\circ$ . The converse is also true.

### 3.6 TOOTH STRUCTURE

The free surface energy of both enamel and dentine is partially built up by polar forces and/or hydrogen bonds (Glantz, 1977). Adhesion in dentistry is made difficult because of the aqueous environment; the presence of water is detrimental both before and after bonding (Buonocore, 1963). Smith (1975) has stated that under practical clinical conditions where water is always present, polar and hydrogen bond forces are not strong enough for durable bonding and chemical reaction at the interface is necessary. As noted previously, water is a strongly polar compound (Buonocore, 1973) and in an aqueous environment like the mouth, physical forces will generally be broken down by penetration of water along the interface, but the mechanical interlocking, if well formed, should resist displacement (Beech, 1982). Buonocore (1975a) has called water the "arch enemy" of adhesion since it is so strongly attracted to most substances due to its polarity and capacity for hydrogen bonding.

A number of authors have noted that the incorporation of fluorine into enamel reduces wetting and its consequent adhesiveness (Phillips, 1973; Glantz, 1977; Beech, 1982).

### 3.7 ORAL BIOFILMS AND ADHESION

Tooth enamel tends to be rendered a low energy surface by virtue of its reaction with a variety of adventitious constituents during its exposure to the oral environment (Buonocore, 1975a). Oral organic films form very quickly, in a matter of seconds, on clean solid surfaces to a detectable thickness of the order of 10 nm (Glantz, 1977). Jendresen & Glantz (1980) found similar low energy surface films on untreated enamel surfaces *in vivo* over a wide age range and noted a high degree

of stability in both film formation and maintenance as shown by the reproducibility of contact angle measurements with test liquids.

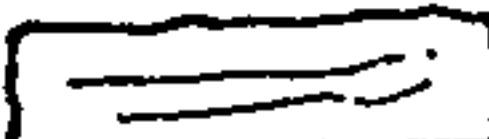
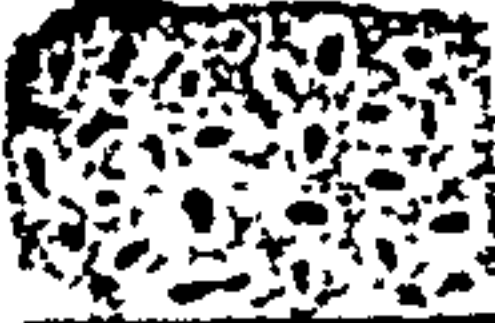
|                             | PRIMARY CUTICLE<br>(Nasmyth Membrane)                                               | STRUCTURES ACQUIRED AFTER ERUPTION                                                   |                                                                                       |                                                                                       |                                                                                       |
|-----------------------------|-------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|
|                             |                                                                                     | CUTICLES                                                                             |                                                                                       | STAINED PELLICLE                                                                      | PLAQUE                                                                                |
|                             |  |  |  |  |  |
| APPROXIMATE THICKNESS       | 1-5 $\mu$                                                                           | 1-3 $\mu$                                                                            | 0.2 $\mu$                                                                             | 1-10 $\mu$                                                                            | 5-200 $\mu$                                                                           |
| STAINING WITH BASIC FUCHSIN | CELLS STAIN RED                                                                     | ?                                                                                    | PURPLISH RED                                                                          | LITTLE OR NONE<br>NATURALLY BROWNISH                                                  | BRIGHT RED                                                                            |
| CALCIFICATION               | OBSERVED IN CELLS ONLY                                                              | ?                                                                                    | ?                                                                                     | YES                                                                                   | YES                                                                                   |

FIG. 1. Diagrammatic representation of organic films encountered with enamel surfaces.

From Meckel (1965), The formation and properties of organic films on teeth. Arch. Oral Biol. 10:585-597

## 4 ACQUIRED SURFACE FILMS

A number of authors have reported surface biofilms on teeth and have attempted to explain their origins. As noted in the discussion on adhesion, the exposed surface molecules will determine the adhesiveness of a surface, especially if it is more than a monolayer in thickness. The importance of the enamel surface, its acquired surface films and the observed phenomena of adhesion will become apparent when the bonding of orthodontic brackets to tooth enamel is considered along with the prerequisite acid etching of the enamel surface.

Meckel (1965) discovered by electron microscope (EM) examination the presence of three acquired organic cuticles. On the enamel surface are two of these: the surface cuticle (SC) of approximately 0.2  $\mu\text{m}$  and the stained pellicle (1–10  $\mu\text{m}$ ). There is also a subsurface cuticle (SSC) of 1–3  $\mu\text{m}$ , consisting of a system of closely interwoven fibrils deposited within the surface layer of slightly damaged enamel. These films resist removal except by abrasion or enamel dissolution (by acid), and in the case of SSC the latter method only will totally remove it. The formation of SSC on slightly damaged enamel has been demonstrated.

Meckel (1965, 1968) concludes that these films derive from the saliva and that their role is protective in that their presence modifies enamel surface behaviour in solubility studies, and in the mouth they presumably retard the attack of acids on the teeth. Meckel (1968) states that the bacteria-free surface cuticle and stained pellicle is constantly reformed.

Jendresen, Glantz, Baier & Eick (1981) also concluded that an organised and specific biological mechanism exists; designed to quickly protect and maintain the hard, mineral tooth surface by acquiring a

relatively low state of surface free energy. This is achieved through the adsorption of a biological film, the acquired pellicle. Moreno (1975) lends credence to the contention that the pellicle has a protective function. He found that the pellicle acted as an ionic, permselective membrane, retarding the transport of ions substantially. His results suggest that this action is related to a specific combination of salivary proteins and their concentrations, and to undefined structural changes occurring after their adsorption.

Leach (1967) proposes that the most likely composition of the bacteria-free, acquired pellicle, is salivary glycoprotein. This formation from salivary components is not dependent on either bacteria or dietary components.

The adsorption of salivary proteins onto the enamel surface is the simplest explanation for the formation of the acquired pellicle according to Hay (1967), and is distinguished from plaque by its resistance to toothbrushing and its freedom from micro-organisms. Hay (1973) reported that hydroxyapatite and dental enamel powders selectively adsorb 7 parotid salivary components, and these were either proteins or peptides.

Armstrong & Hayward (1968) describe a pellicle complex with an outer scalloped edge, composed of lysed and whole bacteria, bacterial cell walls and debris, pellicle matrix and the dendritic fibrils of the subsurface cuticle. The pellicle is envisaged as forming by the settling and growth of micro-organisms along the tooth surface which elaborate extracellular enzymes causing the collapse and local deposition of salivary mucoproteins. This, of course, conflicts with the 'selective adsorption' school.

Mayhall (1970) found that the formation of experimental pellicles from saliva was a selective process, reporting that *in vivo* and *in vitro* pellicles were similar in composition, indicating that all the necessary constituents were present in saliva and that bacteria or bacterial products were not essential.

Armstrong (1971) distinguished plaque from pellicle by stating that plaque is removed by vigorous scrubbing whilst the pellicle is tenaciously attached to the tooth and can be removed by the demineralisation of the enamel surface subjacent to it. He describes a pellicle structure as a 1-3  $\mu\text{m}$  thick amorphous matrix with a characteristic scalloped outer edge in which were embedded bacterial debris and cell wall structures. However, this pellicle was only found on teeth that had been erupted for some considerable period of time and was not found in teeth that had been cleaned of previously formed integuments and then exposed to the oral environment for varying short periods (days to weeks). These latter teeth appeared to have surface cuticle structures, with an overlying amorphous bacteria-free tenuous structure which later became colonised. He postulates that these structures (along with an occasionally observed SSC) were of salivary (protein) origin.

Sönju & Rölla (1973) support the view that the pellicle is formed as a result of selective adsorption of acidic proteins. Bacterial cell wall components were not detected in their analyses. The formation rate varies (on pumiced teeth, *in vivo*), with the greatest formation in the first 60 minutes, with little further formation between 60 and 90 minutes.

In an SEM study, Diedrich (1981), found a 5 second saliva or mucosa

contact on etched enamel resulted in partial coverage from salivary glycoproteins.

## 5 ACID-ETCHING

Acid etching of enamel to enhance adhesion was originally conceived as a means to decontaminate the surface; there is a metallurgical precedent for this. The retention of acrylic resin was in fact enhanced after etching with an 85% solution of phosphoric acid for 30 seconds (Buonocore, 1955).

Newman (1965) concluded that pumicing and etching with 40% phosphoric acid for 60 seconds enhances joint strength of attachments bonded to this treated surface.

Acid etching of tooth enamel renders it more suitable for bonding by increasing the surface area of the enamel through the opening up of microspaces and porosities, and by increasing its surface free energy and thus its wettability, thereby facilitating resin penetration (Buonocore, 1973; Retief, 1978).

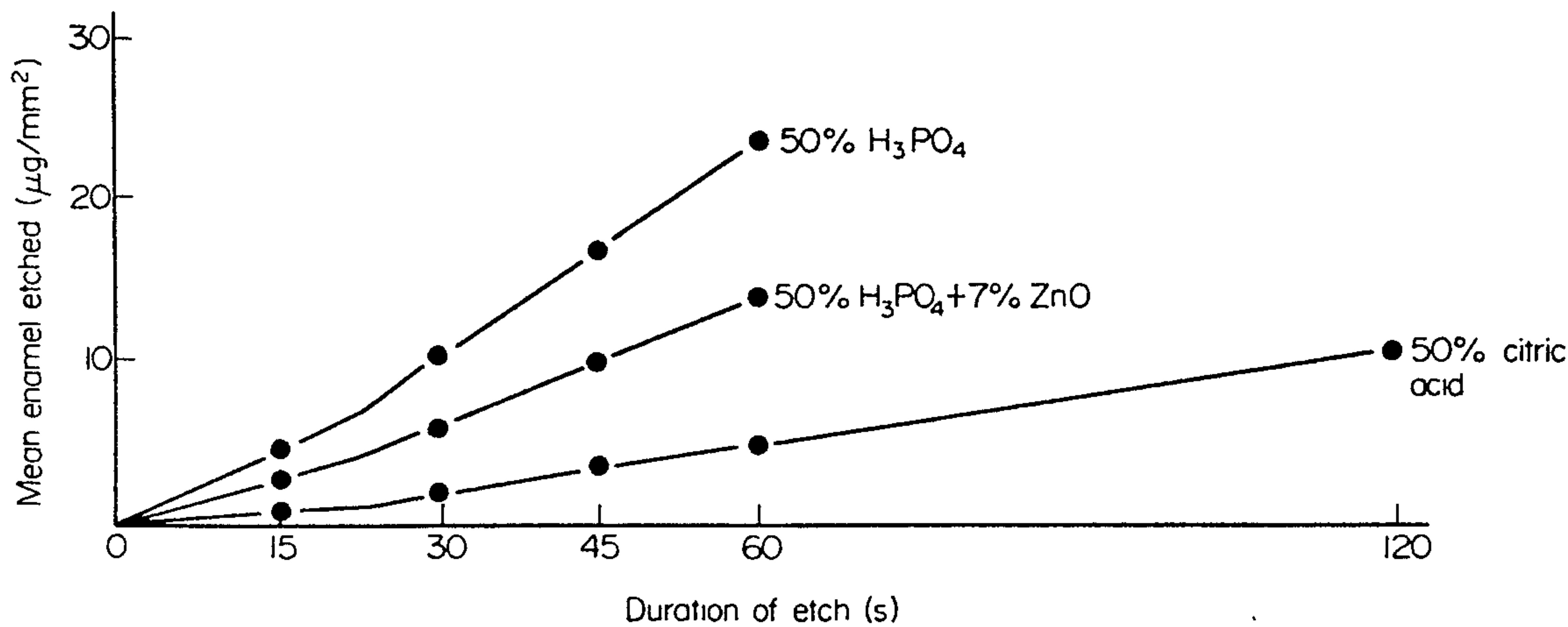
### 5.1 ETCHANT TYPE AND CONCENTRATION

Buonocore (1955) initiated his work into acid etching using an 85% solution of phosphoric acid. Preparations of phosphoric acids in the range 35 to 60 per cent, are the most commonly used in acid etching (Maijer, 1982). Gwinnett (1982) states that currently available commercial systems of phosphoric acid solutions or gels contain concentrations between 37% and 65%. In 1975, Reynolds stated that the most commonly used etchant was 50% phosphoric acid (sometimes buffered).

Chow & Brown (1973) show that a concentrated phosphoric acid solution would dissolve considerably less apatite than would a more dilute solution, as the early formation of  $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$  seems to

Rate of etch of enamel surfaces exposed to three etching solutions

| Etching procedure                             | No. of teeth etched | Enamel etched ( $\mu\text{g}/\text{mm}^2$ ) |                |                |                |                |
|-----------------------------------------------|---------------------|---------------------------------------------|----------------|----------------|----------------|----------------|
|                                               |                     | 15 s                                        | 30 s           | 45 s           | 60 s           | 120 s          |
| 50% $\text{H}_3\text{PO}_4$                   | 10                  | $4.3 \pm 1.2$                               | $10.0 \pm 2.3$ | $16.4 \pm 3.2$ | $23.1 \pm 3.8$ | —              |
| 50% $\text{H}_3\text{PO}_4$ + 7% $\text{ZnO}$ | 10                  | $2.5 \pm 0.4$                               | $5.7 \pm 0.7$  | $9.4 \pm 1.1$  | $13.4 \pm 1.5$ | —              |
| 50% citric acid                               | 10                  | $0.8 \pm 0.02$                              | $1.9 \pm 0.4$  | $3.1 \pm 0.2$  | $4.6 \pm 1.1$  | $10.1 \pm 1.8$ |



Rate of etch of three conditioning solutions.

From Retief (1974a), A comparative study of three etching solutions: effects on contact angle, rate of etching and tensile bond strength. J Oral Rehab. 1:381-390

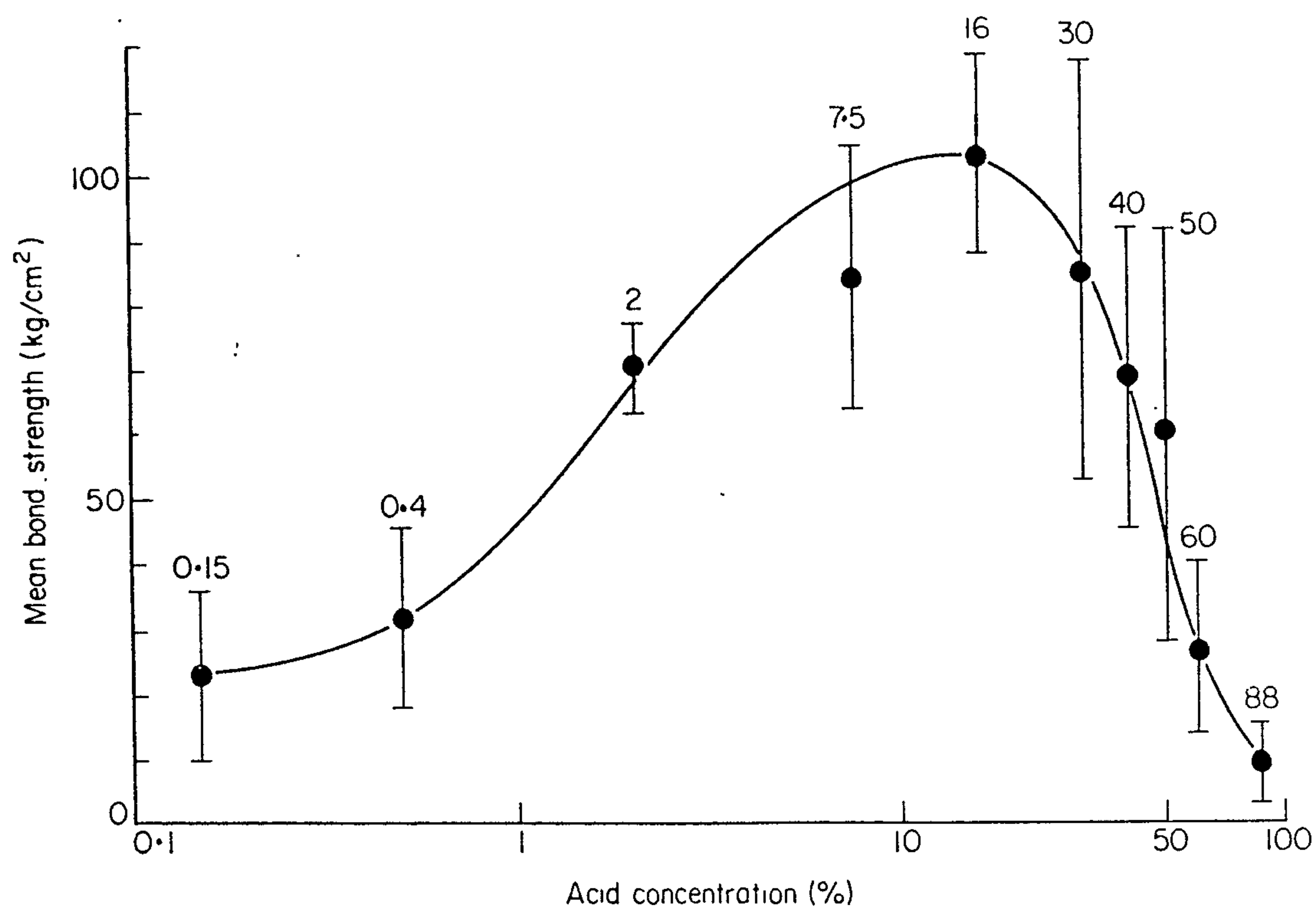
protect the enamel from excessive dissolution when the more concentrated solution is applied. Because of its high solubility in water, the  $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$  would be completely washed away in the clinical situation, thus leaving a clean surface available for bonding. Solutions containing less than approximately 27% phosphoric acid were unacceptable due to the production of insoluble dicalcium phosphate dihydrate which remains as a surface contaminant.

Silverstone (1974), in comparing 5 etchants, found that the most retentive conditions for a sealant were in the use of phosphoric acid in the range 20-50% with 30% proving to be the most effective single agent.

Retief (1974a) compared 50% phosphoric acid (with and without 7% ZnO) and 50% citric acid. The 50%  $\text{H}_3\text{PO}_4$ /7% ZnO etchant produced the lowest contact angle (with uncured epoxy resin) and the highest bond strength. The straight 50% solution produced the greatest etching of the enamel surface in terms of  $\mu\text{g}/\text{mm}^2$  of enamel lost.

A citric acid (50%) etchant had the mildest etching action compared to two phosphoric acid solutions (50% vs 50%+7% ZnO) which produced similar etching patterns (Retief, 1975b). It was found that the citric acid etch led to significantly less adhesive penetration, compared to the tag-like projections for phosphoric acid etching.

Galil & Wright (1979) found that 5 distinct etching patterns were produced regardless of the acid concentration used. Their etchants were two commercial 50% etchants; a commercial 50% etchant with 7% ZnO; a commercial 37% etchant; and a laboratory prepared 37% solution of phosphoric acid.



**Plot of mean tensile bond strength of Restodent (kg/cm<sup>2</sup>) vs concentration (%) of phosphoric acid used for etching. Vertical lines indicate standard deviation.**

**From Soetopo, Beech & Hardwick (1978), Mechanisms of adhesion of polymers to acid-etched enamel: effect of acid concentration and washing on bond strength.  
J Oral Rehab. 5:69-80**

Brännström, Nordenvall & Malmgren (1978) report that one minute etching with an acid gel (50% phosphoric) produced the same effect as an acid liquid (37% phosphoric) for the same period.

Brännström, Malmgren & Nordenvall (1982) could find no differences between a 37% solution or a 50% gel in patterns produced on young permanent teeth etched for 15 seconds.

Phosphoric acid (50% solution) pretreatment enhances the tensile strength of attachment of acrylic to enamel by some 2800% at 1 week over an unetched control (Laswell, Welk & Regenos, 1971).

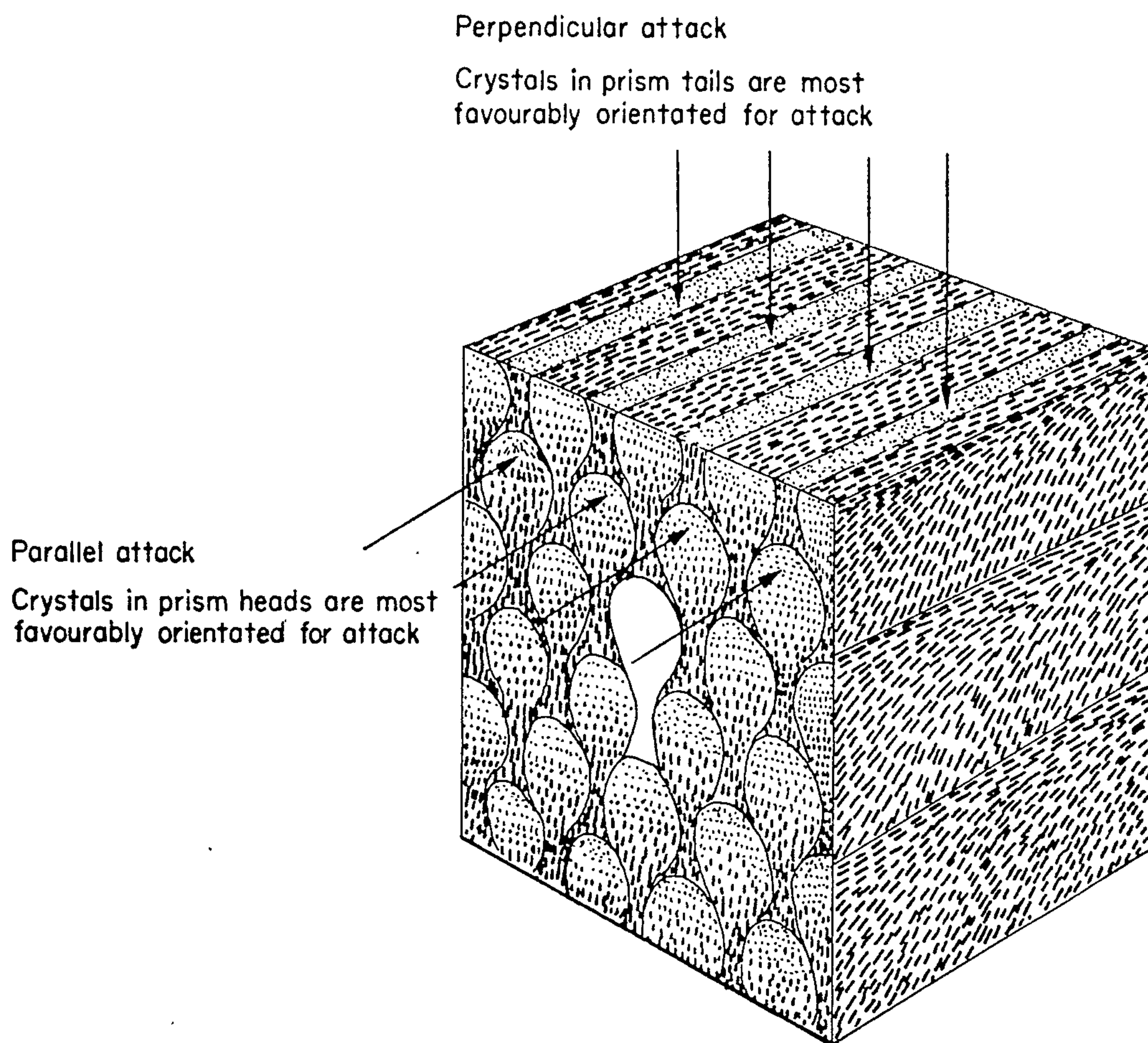
Lee, Phillips & Swartz (1971) also reported significantly increased resin bonding to bovine enamel etched for 60 seconds with a 50% phosphoric acid solution.

Rock (1974) reported a finding of substantially increased tensile strengths of fissure sealant bonds (to human enamel, *in vitro*) using a 30% solution of phosphoric acid rather than a 50% solution. For one resin this increase was of the order of 55%.

Nelson, Till & Hinding (1974) reported similar bond strengths of acrylic resin to enamel, using 25% or 50% phosphoric acid as an etchant and significantly lower bond strengths when a 75% solution was utilised.

Soetopo, Beech & Hardwick (1978) tested composite resin tensile bond strengths to enamel using different solutions of phosphoric acid, and found that a 20% solution gave the highest value.

After testing phosphoric acid concentrations in the range 10-70%, Gottlieb, Retief & Jamison (1982) found that the mean tensile bond



Illustrating the arrangement of crystals in enamel and their behaviour in parallel and perpendicular attack.

**From Sharpe (1967), Influence of the crystal orientation in human enamel on its reactivity to acid as shown by high resolution microradiography. Archs Oral Biol 12:583-591**

strengths of a composite resin bonded to etched enamel was not significantly different for the range 10-60%, but was significantly lower for a 70% solution.

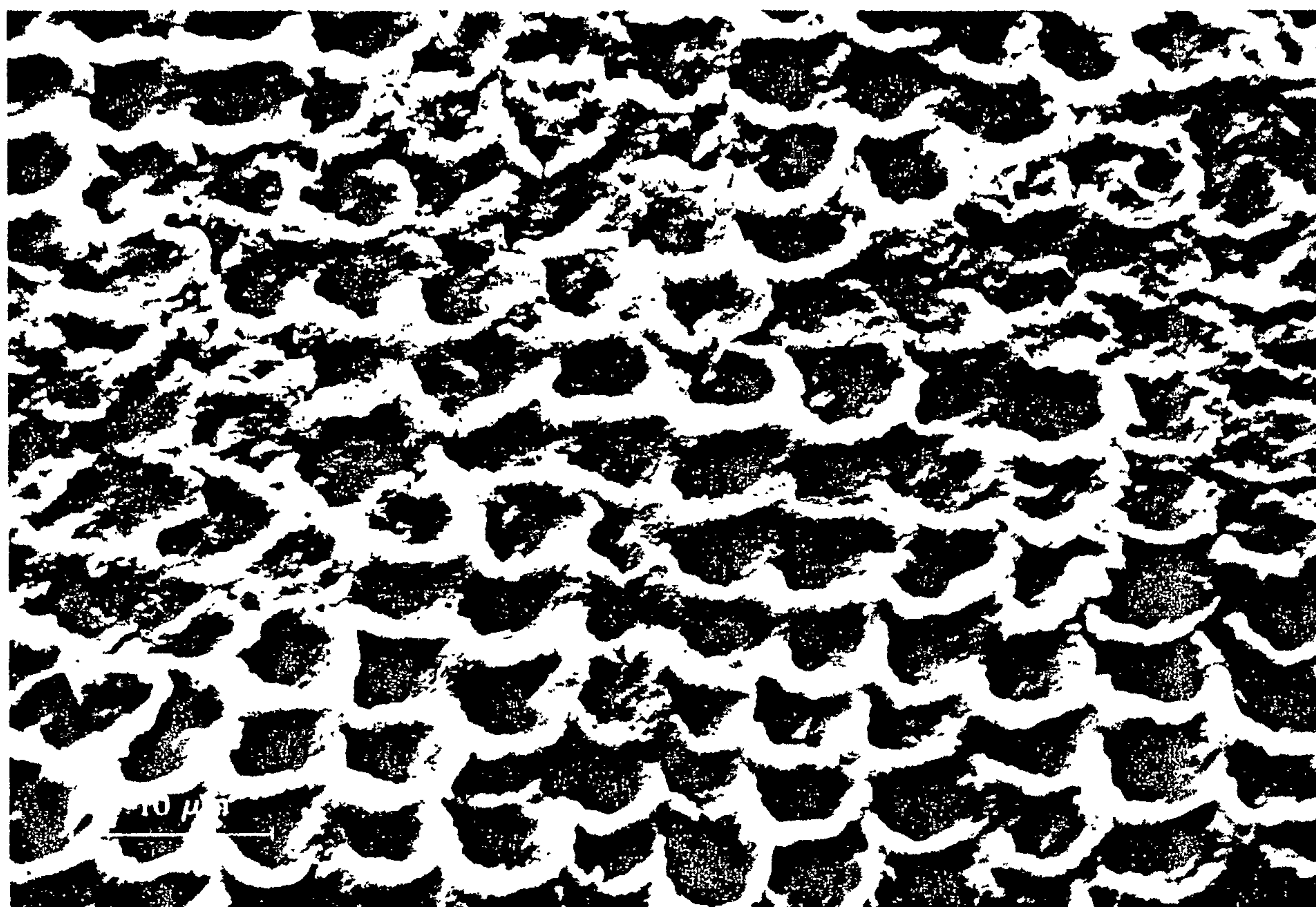
Zidan & Hill (1986) report that mean bonding strengths of composite resin bonded to enamel etched with a 2, 5 or 35% solution of phosphoric acid were not significantly different (some 2 kg/mm<sup>2</sup>). A 0.5% solution gave a high, but significantly reduced, bond strength.

## 5.2 ETCHING PATTERNS

Sharpe (1967) has demonstrated a honeycomb appearance on etching of enamel parallel to prism long axes. A different pattern is produced on etching the enamel perpendicular to the prism long axis, and this is explained by crystallite orientation and crystallite anisotropy.

Poole & Johnson (1967) reported that etching with formic, lactic or hydrochloric acid preferentially removed prism cores (honeycomb appearance) in transverse sections of enamel and that EDTA preferentially removed prism peripheries. They note that surfaces prepared in a plane perpendicular to the prism direction (transverse section) were more seriously damaged than those cut in a plane parallel to prism direction (longitudinal section). They conclude that it is crystallite direction along with variations in composition of the enamel structure that contributes to this difference in etching.

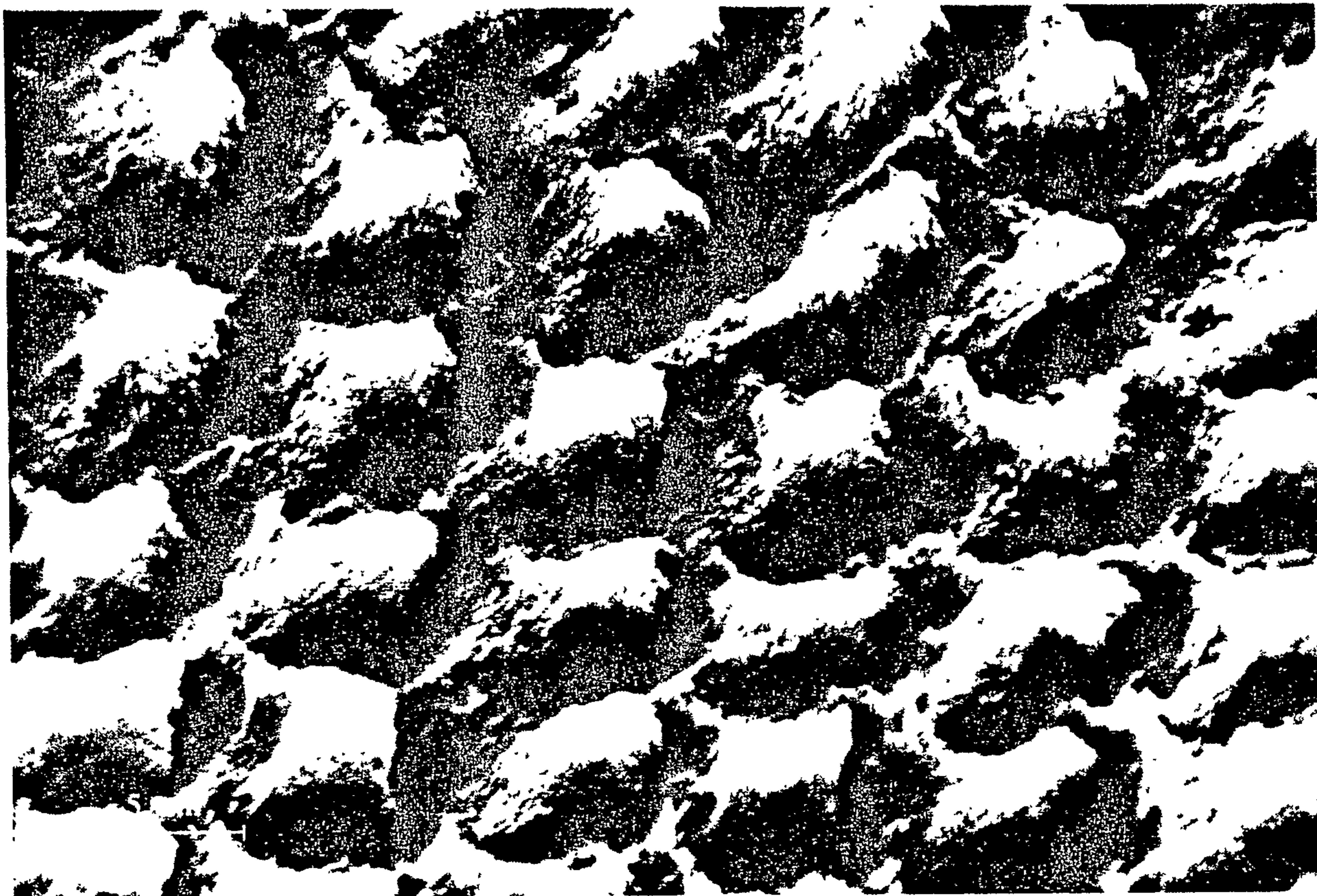
Gwinnett (1971a) described resinous tags left after enamel was demineralised subsequent to the bonding of various resins to acid etched enamel surfaces. These replicas showed areas which could be smooth or rough, that is, showing many irregularities. The resinous structures resemble either finger-like, cone-shaped or crater-shaped phenomena.



Scanning electron micrograph of an enamel surface that has been etched for 60 seconds with 30 per cent phosphoric acid. The etch is seen to be evenly distributed over the entire enamel surface. A Type 1 etching pattern is seen in which prism centres have been removed preferentially.

**From Silverstone (1975), The acid etch technique: in vitro studies with special reference to the enamel surface and the enamel-resin interface.**

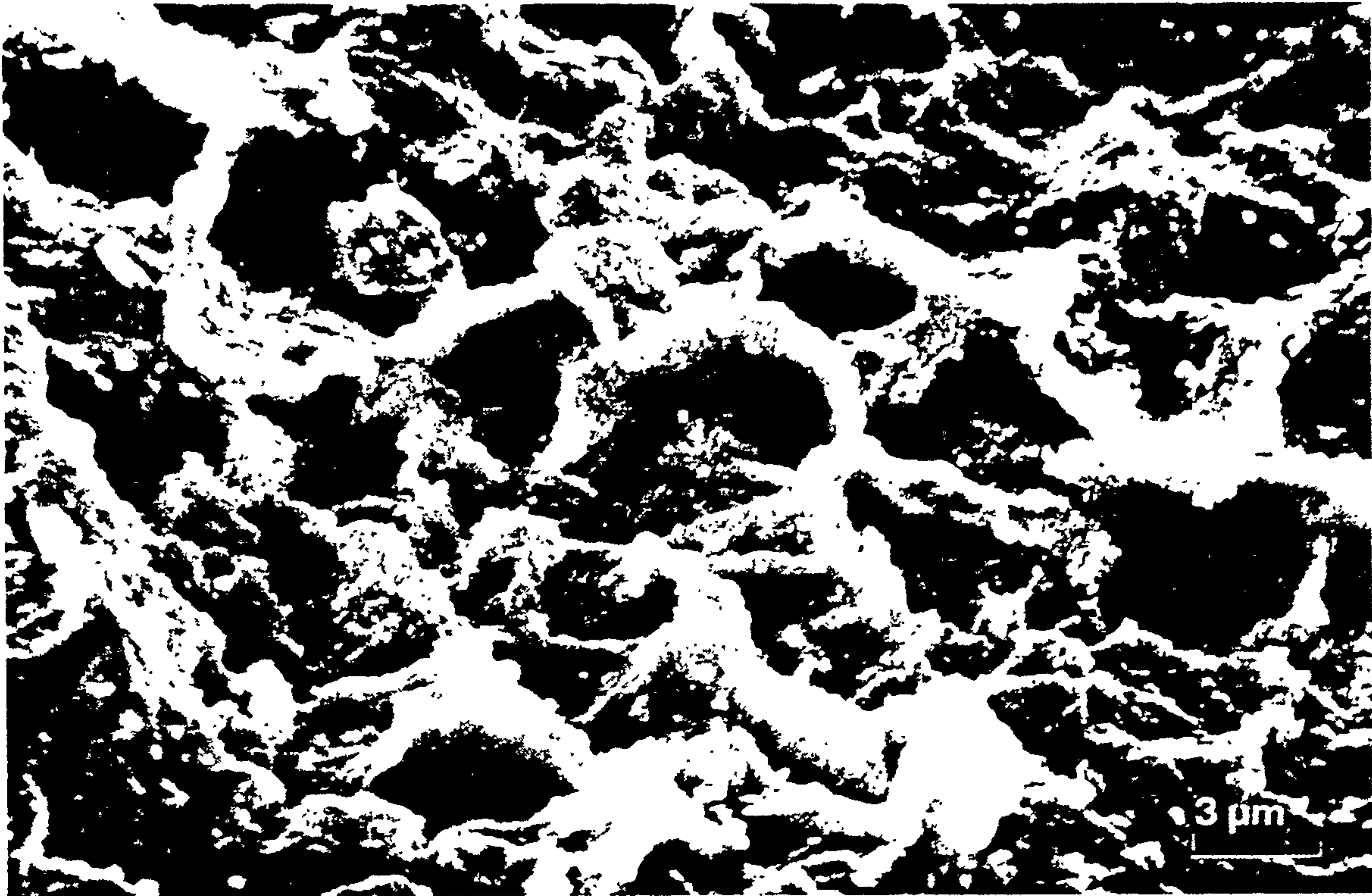
**In: Proceedings of an International Symposium on the Acid Etch Technique (Eds: Silverstone & Dogon) pp 213-229  
North Central Pub. Co., St Paul, Minnesota**



Scanning electron micrograph of an enamel surface which has been exposed to 40 per cent phosphoric acid for 60 seconds. In this case, the etching pattern is one in which there has been a preferential removal of prism peripheries, termed a Type 2 etching pattern.

**From Silverstone (1975), The acid etch technique: in vitro studies with special reference to the enamel surface and the enamel-resin interface.**

**In: Proceedings of an International Symposium on the Acid Etch Technique (Eds: Silverstone & Dogon) pp 213-229  
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Scanning electron micrograph of an enamel surface exposed to 30-percent phosphoric acid for 1 min. In this, areas can be seen showing a type 1 etching pattern in which there is a preferential removal of prism cores. However, adjacent regions show a type 2 etching pattern in which the reverse pattern can be seen. In addition, in many areas the pattern of surface damage is difficult to relate to prism structure. Such a field is referred to in this study as a type 3 etching pattern.

**From Silverstone, Saxton, Dogon & Fejerskov (1975),  
Variation in the pattern of human dental enamel examined by  
scanning electron microscopy.  
Caries Res. 9:373-387**

The cone-shapes predominated, and were irregular and varied in both height and width. Etching having a preferential effect on prism cores would result in cones of resin, while preference for prism peripheries would produce crater-like characteristics. There is an obvious and tremendous increase in surface area. This, together with the actual penetration of resin into the enamel substructure, greatly enhances retention of the resin, if only from a mechanical standpoint.

Preferential loss of tissue results in the creation of a variety of etching patterns which include the preferential loss of material from the rod core and rod peripheries (Gwinnett, 1971b). The most common finding was that of extensive loss of prism core material while the peripheral zone remains relatively intact. Retief (1973) also reported preferential etching of enamel with 50% phosphoric acid for one minute. The central portions of prism heads were dissolved to a greater extent than the periphery, thus producing a honeycomb appearance. This is the so-called type 1 pattern (Silverstone, Saxton, Dogon & Fejerskov, 1975)

Less frequently, the reverse of this phenomenon was observed in which the core remained intact while the periphery was lost, the type 2 pattern (Silverstone, Saxton, Dogon & Fejerskov, 1975). This etch pattern occurs less frequently than type 1 (Silverstone, 1975)

The type 3 pattern is a more random pattern, which has areas of both types 1 and 2, and areas in which the pattern cannot be related to prism morphology (Silverstone, Saxton, Dogon & Fejerskov 1975). This pattern comprises the least common of the phenomena of topographical change. The surface appeared either pitted, or smooth and relatively featureless with no clear prism delineation. This smooth or pitted appearance was not related to the presence or absence of prismless

enamel (it can occur on the surface of prismatic enamel also) but more likely to differences in intrinsic solubility, as the 3 etching patterns, referred to above, can all be seen in the one section of enamel (Gwinnett, 1971b).

The etching pattern can vary in appearance, distribution and depth of etch. These variations can occur not only from tooth to tooth, or surface to surface, but also from site to site on a single tooth surface. (Retief, 1973; Silverstone et al, 1975; Jørgensen, 1975; Diedrich, 1981).

Silverstone, Saxton, Dogon & Fejerskov (1975) conclude that no one specific pattern is produced by acid etching and that such differences are difficult to explain on the basis of variation in chemical composition and crystallite orientation (as has been suggested by Poole & Johnson, 1967).

Silverstone (1975) reports that all three types of etching pattern occurred in the range 20-60% (phosphoric acid concentration), yet only type 3 occurred for 5-15% and 70-80% phosphoric acid concentrations.

Galil & Wright (1979) agree with Silverstone (1975) and Silverstone et al (1975) regarding three types of etching patterns, finding types 1 and 2 (75% frequency on the coronal third) and type 3 (80% frequency on the middle third) of buccal surfaces of permanent teeth. On the cervical third (75% frequency) they demonstrated both type 4 (pitted) and type 5 (flat and smooth).

Ripa et al (1966) reported that of the 70% of permanent teeth found to have prismless enamel, two-thirds of these had prismless enamel covering the gingival third. Arakawa, Takahashi & Sebata (1979) noted

the appearance of cervical enamel on the buccal surface of human premolars after etching. It showed a pitted appearance with no prism delineation, whereas the middle and occlusal thirds did show the characteristic prism-end structures. Increased etching times, or enamel grinding, did not alter the character of this pattern.

Diedrich (1981) described four etching patterns, the first three essentially the same as Silverstone's (1975). He found the type 3 pattern in areas of prismless enamel, the surfaces of recently erupted teeth and on the cervical third of older teeth. The fourth pattern was an irregular build-up of prism groups, yielding a starlike or fernlike pattern.

### **5.3 CONTRALATERAL SYMMETRY**

Jørgensen (1975a,b) demonstrated that pairs of contralateral teeth showed a very similar etch pattern symmetrically along the midline. No difference in the pattern was observed in enamel etched with either a 35%, 37% or 50%(+ZnO 7%) phosphoric acid solution for 60 seconds.

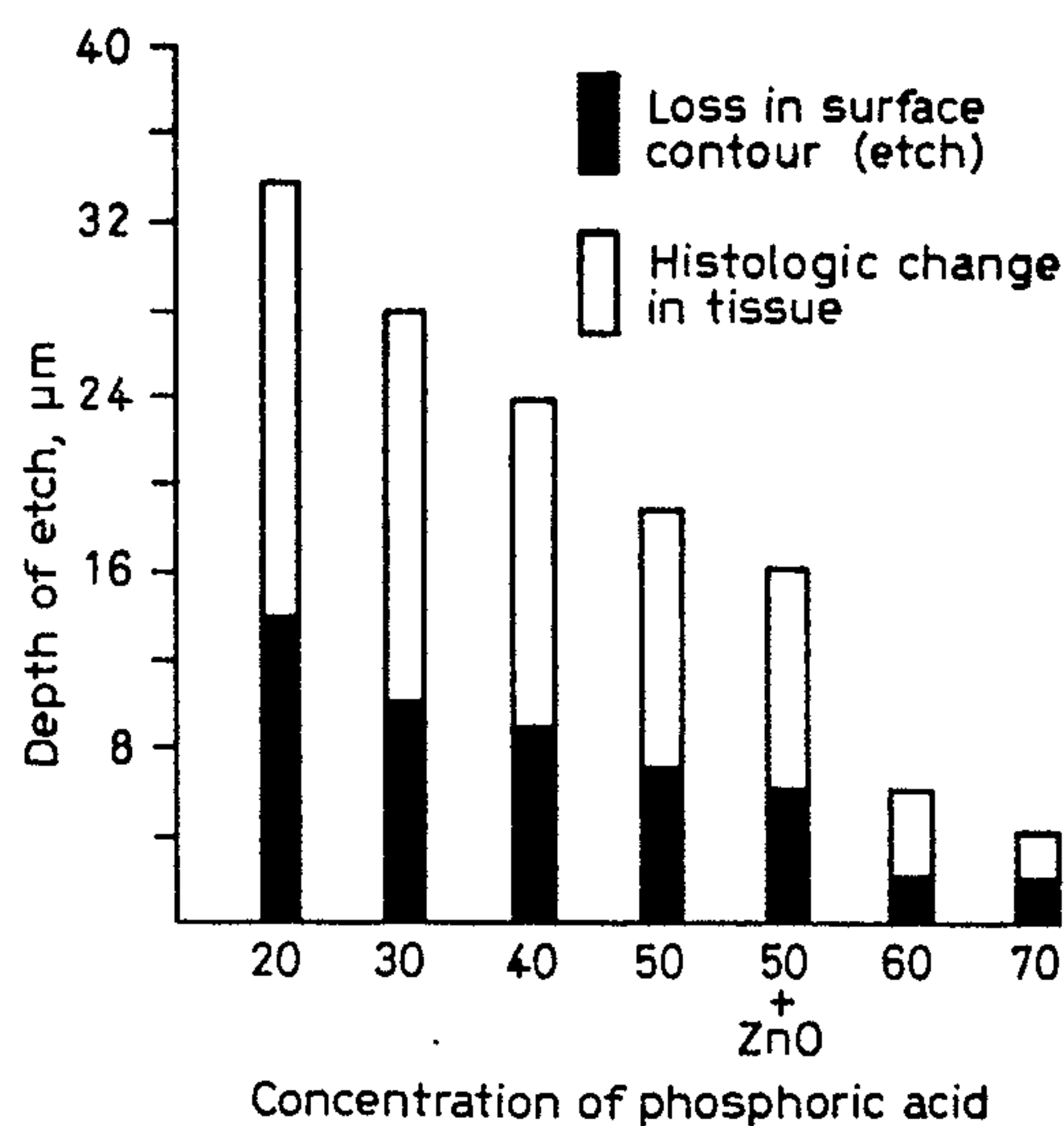
### **5.4 DEPTH OF ETCH AND ENAMEL POROSITY**

It is necessary to examine the literature as it relates to enamel loss and histologic change after etching with various concentrations of phosphoric acid, and to some extent, minor variations in etching times. This is in order to provide a baseline to later look at reduced etching times, per se, and the subsequent effects that have been reported.

Smith, Spinelli & Tartakow (1976) showed that phosphoric acid solutions used for etching can penetrate to a depth of 50  $\mu$ m into enamel after acid application for a period of two to five minutes. Etching of enamel surfaces with phosphoric acid results in a total loss of

Depth of etch and histological change in enamel (to nearest  $\mu\text{m}$ ) following a 1-min exposure to various concentrations of phosphoric acid

| 1-min exposure                 | Concentration of phosphoric acid, % |    |    |    |           |    |    |
|--------------------------------|-------------------------------------|----|----|----|-----------|----|----|
|                                | 20                                  | 30 | 40 | 50 | 50<br>ZnO | 60 | 70 |
| Depth of etch                  | 14                                  | 10 | 9  | 7  | 6         | 2  | 2  |
| Depth of histologic change     | 20                                  | 20 | 15 | 12 | 10        | 4  | 2  |
| Total depth of enamel affected | 34                                  | 30 | 24 | 19 | 16        | 6  | 4  |



Depth of enamel affected by 1-min exposures to various concentrations of phosphoric acid. This consists of both the loss in depth due to etching and the region showing histological change.

**From Silverstone (1974), Fissure sealants: laboratory studies.  
Caries Res. 8:2-26**

superficial enamel and preferential dissolution of the underlying enamel (Gottlieb, Retief & Jamison, 1982).

The depth of etch (ie amount of superficial enamel removed) is dependent on the type of acid used, the acid concentration, the duration of acid application and the enamel fluoride concentration (Nasir, Retief & Holbrook, 1981).

Gwinnett (1971b) reported on the depth of etch with various acids, noting that 10% and 50% phosphoric acid solutions etched enamel to a depth of between 5 and 25  $\mu\text{m}$  for a 2 minute application.

The degree of damage to the enamel surface, in terms of both depth of etch and extent of porosity of the tissue, was found to be inversely proportional to the concentration of phosphoric acid employed (Silverstone, 1974; Silverstone, Saxton, Dogon & Fejerskov, 1975). Silverstone (1975) is in general agreement with this. However, he investigated phosphoric acid concentrations over a wider range (ie 5 to 80% cf 20 to 70%). The lower acid concentrations (5, 10 & 15%) created surface defects similar to those produced by 70 and 80% solutions applied for one minute.

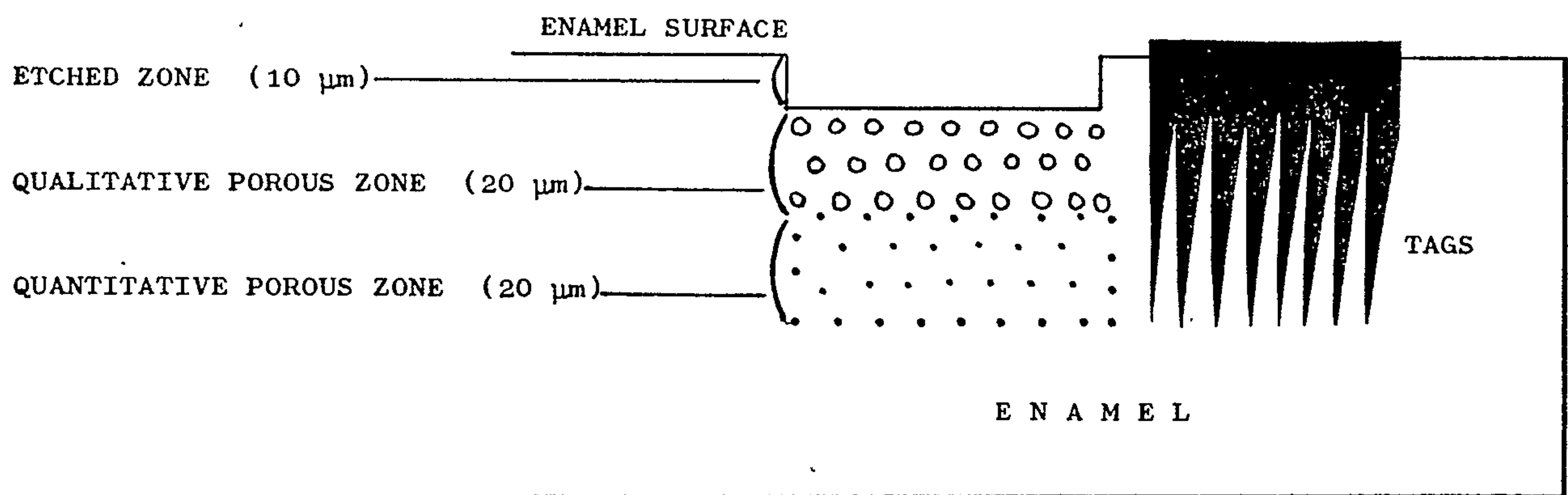
The use of an unbuffered 30% phosphoric acid solution for 1 minute caused the loss of approximately 10  $\mu\text{m}$  of enamel and a further histological change (ie porosity) of 20  $\mu\text{m}$  (Silverstone, 1974).

Silverstone (1975) found that the etch created by a 30% solution for 1 minute was evenly distributed over the entire surface, as was that of a 40% solution. However, the etching patterns created by solutions below 30% or above 40% were both less evenly distributed, and less porous, than those in the range 30-40%. The most consistent results

| Acid (2 min application<br>of 0.3 cm <sup>3</sup> ) | Depth of etch     |            |                       |
|-----------------------------------------------------|-------------------|------------|-----------------------|
|                                                     | Less than<br>5 µm | 5-25<br>µm | Greater than<br>25 µm |
| 50% Citric                                          | *                 |            |                       |
| 10% Formic                                          | *                 |            |                       |
| Zinc phosphate liquid <sup>§</sup>                  | *                 |            |                       |
| 85% Phosphoric                                      | *                 |            |                       |
| 50% Phosphoric                                      |                   | *          |                       |
| 10% Phosphoric                                      |                   | *          |                       |
| 0.1 N Hydrochloric                                  |                   |            | *                     |
| 0.5 N Hydrochloric                                  |                   |            | *                     |

<sup>§</sup>LD Caulk Tenacin®

From Gwinnett, AJ (1971b) p 733, Table 1  
 Histologic changes in human enamel following treatment with acidic  
 adhesive conditioning agents  
 Archs Oral Biol 16:731-738



Diagrammatic representation of an etched and porous enamel surface showing the three distinct zones, and the subsequent penetration of tags of resin.

**From Silverstone (1975), The acid etch technique: in vitro studies with special reference to the enamel surface and the enamel-resin interface.**

**In: Proceedings of an International Symposium on the Acid Etch Technique (Eds: Silverstone & Dogon) pp 213-229  
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were obtained using an unbuffered solution of 30% phosphoric acid, irrespective of the specific pattern produced. The depth of enamel loss appeared to be at a maximum using 20% phosphoric acid. However, the underlying porosity was considerably variable. Silverstone (1974) reported a 14  $\mu\text{m}$  loss for a 20% solution, the highest of any solution tested (range 20-70%).

Silverstone (1975) compared the birefringence of a section of etched enamel with a control. The outer 10  $\mu\text{m}$  depth is lost completely (Etched zone). The remaining, underlying surface enamel is approximately 20  $\mu\text{m}$  in depth and is observed to be porous histologically with the polarising microscope (Qualitative zone). Beneath this is another 20  $\mu\text{m}$  layer that is not histologically porous, but does in fact exhibit a lower observed birefringence than sound enamel (Quantitative zone), into which resin tags penetrate.

Retief (1975c) calculated the depth of etch from enamel biopsy samples and concluded that a 50% solution of phosphoric acid produced a reduced depth of etch (enamel loss) compared to other concentrations which produced similar bond strengths to epoxy resin. He found that surface roughness, a measure of peak-to-valley height, was greater than that for more dilute acid concentrations. He states that it is not the overall depth of etch which is important, but the surface roughness, in relation to the retention of the resin.

An evenly distributed etching pattern with marked surface roughness, but little actual loss of enamel, is most desirable to achieve a good mechanical interlock between adhesive and enamel (Gorelick, Masunaga, Thomas & Zachrisson, 1978b).

Fitzpatrick & Way (1977) calculated a mean loss of tooth enamel

(which is not replaced), due to etching enamel with a 30% solution for 90 seconds, of 9.9  $\mu\text{m}$  (corresponding to the Etched zone of Silverstone, 1975). Brown & Way (1978) in a similar study reported a median loss of 3.0  $\mu\text{m}$  (80% range 0.2-11.7 $\mu$ ) for a 37% solution applied for 90 seconds. Pus & Way (1980) similarly, using a 37% solution or a 43% gel applied for 90 seconds, found a mean enamel loss of 6.9  $\mu\text{m}$ . The preceding figures (Fitzpatrick & Way, 1977; Brown & Way, 1978; Pus & Way, 1980) are for enamel loss due to etching only, and do not include those which were presented for pumicing, debonding or polishing procedures. Diedrich (1981) measured an irreversible loss of enamel of 5  $\mu\text{m}$  for a two-minute etch. Lehman & Davidson (1981) found a mean loss of 0.7  $\mu\text{m}$  for fluoridated enamel and a mean loss for unfluoridated enamel of 6.7  $\mu\text{m}$  when etched for 60 seconds using a 50% phosphoric acid solution.

Shey & Brandt (1982) quantified the enamel loss and computed the depth of etch for three commercial etchants applied for 60 or 120 seconds. There was more enamel lost in the teeth that were etched for 120 seconds. The mean depth of etch was less for the 60 second group (22.5  $\mu\text{m}$ ) than for the 120 second group (39.8  $\mu\text{m}$ ). Monolok™ etchant was the least damaging in terms of enamel loss or depth of etch produced.

Manson-Rahemtulla, Retief & Jamison (1984) examined the effects of phosphoric acid etchants in the concentration range 10-70%. They found that the total amount of calcium dissolved ( $\mu\text{g}/\text{mm}^2$ ) from the enamel increased as the concentration of the acid solution increased, until a maximum was reached at 40%, thereafter the calcium loss decreased as acid concentration rose. The measured depth of etch ( $\mu\text{m}$ ), that is, irreversible loss of superficial enamel (Silverstone's etched zone), followed a similar pattern. This measured depth of etched enamel

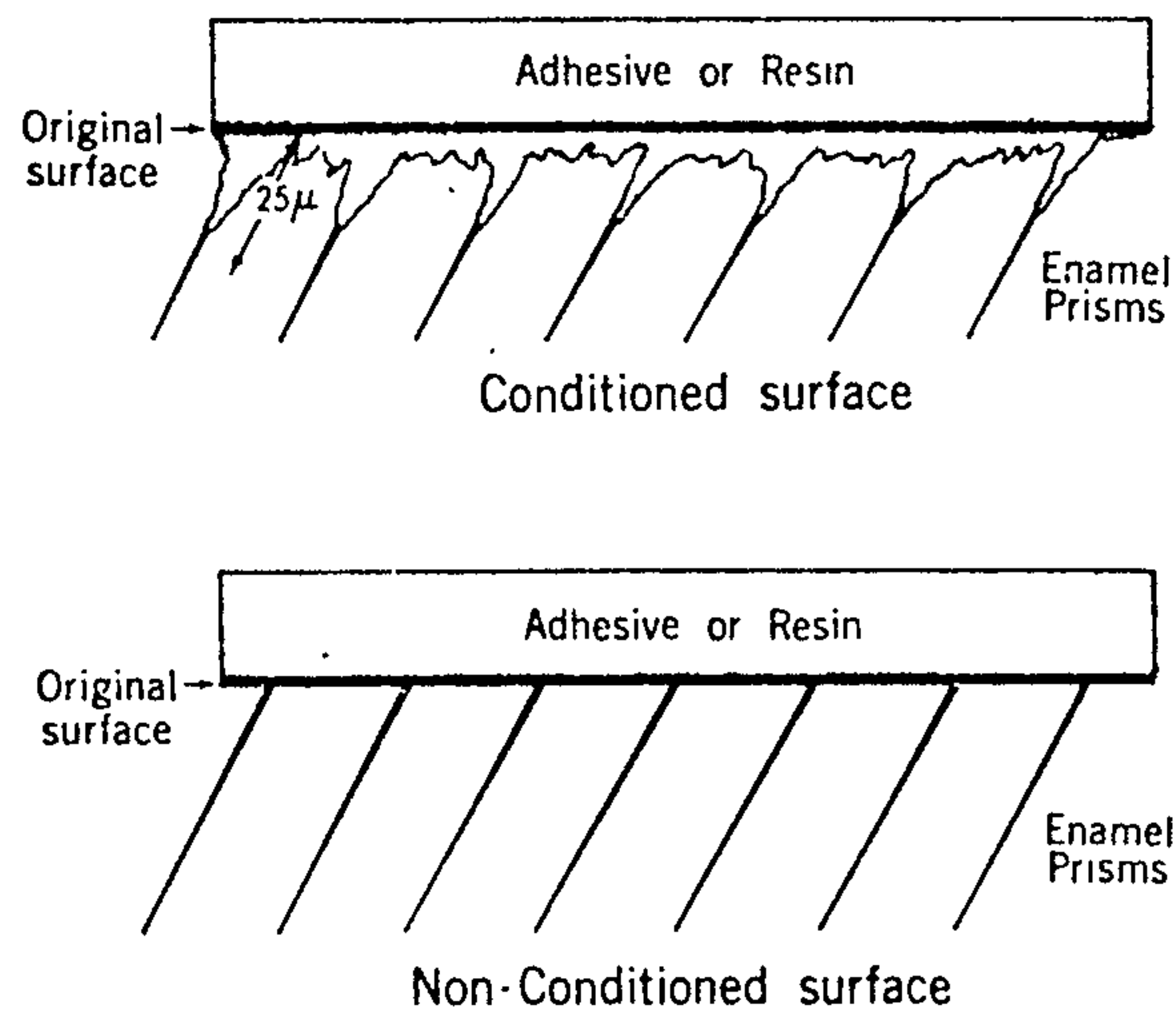


Diagram representing a section through a row of enamel prisms with penetration of resin into spaces produced on an acid conditioned enamel surface. The resin, in combination with protein matter, forms the prism-like tags. Penetration is represented as greater at the interprismatic areas in accordance with the results of our polarized light examination of thin cross sections of etched surfaces.

**From Buonocore, Matsui & Gwinnett (1968), Penetration of resin dental materials into enamel surfaces with reference to bonding. Archs Oral Biol. 13:61-70**

lost was not significantly different for 30, 40 or 50% phosphoric acid, which produced a zone of the order of 18-22  $\mu\text{m}$  in depth. However, the amounts of subsurface enamel removed ( $\mu\text{g}/\text{mm}^2$ ), that is, the porous zones created, were not significantly different for acids in the range 10-60%.

Zidan & Hill (1986) investigated surface loss of enamel using 0.5-80% solutions of phosphoric acid. The maximum depth of etch (surface layer loss) occurred within the range 20-50% concentration (peaking at 52  $\mu\text{m}$  for a 35% solution). The depth of etch progressively decreased as the two extremes of acid concentration were approached. No differences were found for bond strengths for 2, 5 or 35% solutions. They feel that it is preferable to use low concentrations causing minimal loss of enamel while securing an adequate bond.

### 5.5 LENGTH OF TAGS

It is axiomatic that tag formation mirrors the etched enamel surface, provided adequate wetting is allowed to take place. Arakawa, Takahashi & Sebata (1979) demonstrated the presence of exceptionally short tags formed by resin bonded to etched enamel from the cervical third of premolars. This reflects the poorly etched (pitted) surface on this area of the buccal surface. The formation of tags represents an intimate relationship between adhesive and enamel components (Silverstone, 1974). He reported average tag lengths of 50  $\mu\text{m}$ , some up to 100  $\mu\text{m}$  long.

Buonocore, Matsui & Gwinnett (1968) reported resin tags of up to 25  $\mu\text{m}$  for a number of resins. They considered that these observed tags were a combination of resin and entrapped organic matter of the enamel. Gwinnett & Ripa (1973) measured the penetration of two fissure sealant

materials into enamel that was etched and sealed *in vivo*. Tags averaged approximately 25  $\mu\text{m}$  in length.

Retief (1973) demonstrated tags up to 50  $\mu\text{m}$  projecting from the resin, replicating the etched enamel surface. Silverstone (1975) examined the lengths of Nuva Seal tags after demineralising the subjacent etched enamel surface, and reported lengths of up to 50  $\mu\text{m}$ . In addition, he reported tags in partially demineralised enamel (thus having the support of the enamel) as being 50–60  $\mu\text{m}$  in length, before disappearing into the remaining supporting enamel, which had not been removed by the subsequent intentional demineralisation. Tags may be fractured, and therefore appear shorter, once support from the enamel is lost.

Mean tag lengths of 8 to 9  $\mu\text{m}$  were reported by Jørgensen (1975b) for three adhesives. Maximum lengths were of the order of 22 to 26  $\mu\text{m}$ . Brandt, Servoss & Wolfson (1975) measured tags of 5–10  $\mu\text{m}$  in length, with a density of 10 000–20 000 tags per square centimetre.

Tag lengths produced on enamel cut transversely (ie perpendicular to prism axis) and then etched, were 5–10  $\mu\text{m}$  in length, and those from longitudinally cut enamel, 3–5  $\mu\text{m}$  (Voss & Charbeneau, 1979). Enamel etched on surfaces parallel to the prism direction (longitudinal sections) showed troughs and ridges (Poole & Johnson, 1967). Voss & Charbeneau (1979) reported that the tags formed on etched, longitudinally cut enamel were in the form of wavy rows of troughs and ridges.

SEM micrographs showed that resin tags generally reached a depth of 80  $\mu\text{m}$ , but sometimes fine extensions as far as 100–170  $\mu\text{m}$  into the enamel were seen. This, he says, concurs with Silverstone's (1975)

observations about the fragility of the extremities of the tags (Diedrich, 1981).

## 5.6 FLUORIDE AND SURFACE ENAMEL

The concentration of fluoride in surface enamel and its effects on etching and bonding, along with the use of fluoride prior, and subsequent, to etching is controversial. The topical application of fluorides subsequent to the bonding procedure itself, is encouraged, in order to ensure enhance remineralisation of etched, but uncovered, enamel (Buonocore, 1981). In the interests of standardising this research project as much as possible, only patients who demonstrated at least complete post-natal residence in an area of water fluoridation were accepted. No consideration was given to professionally provided topical fluorides or to that in dentifrice use. The purpose of this study was to attempt to limit the variables to etching time, not to examine the various reported fluoride effects.

It is important to consider fluoride in relation to enamel and etching for a number of reasons. Firstly, because enamel surface fluoride concentrations may have an effect on etching and subsequent bond strengths. Secondly, because the clinician must be aware of the possible dangers of over-etching and the concern that the enamel may be rendered more caries-susceptible. Thirdly, that the (controversial) application of fluorides before, during or subsequent to etching may affect wetting, adhesion and micromechanical locking and hence bond strengths. Below, therefore, is some consideration of the variability of opinion in the literature.

### 5.6.1 Topical fluoride treatment

Lehman & Davidson (1981) state that topically fluoridated enamel has a highly acid-resistant layer 2-4  $\mu\text{m}$  in thickness. A series of three one-minute etches (50% phosphoric acid) showed a significantly lower enamel solubility for fluoridated teeth compared to controls. At least 2 minutes of etching was needed to produce a surface with a similar solubility to unfluoridated enamel. The total enamel loss after 3 successive minutes of etching was 7.6  $\mu\text{m}$  and 18.4  $\mu\text{m}$  for the fluoridated and unfluoridated enamel, respectively. Sampson, Wookey & Rouse (1987) examined teeth that had been exposed to fluoridated water and had not recently received topical fluoride treatments. They report the existence of a fluorine gradient that diminishes appreciably within the surface 2-4  $\mu\text{m}$ , although noticeable inter- and intra-tooth variation occurs. Etching for 120 seconds will remove this higher fluorine-containing 2-4  $\mu\text{m}$  layer, so that the lower (0.1-0.2%) fluorine-containing subsurface becomes exposed.

Kochavi, Gedalia & Anaise (1975) found that pretreatment of enamel with either 2% NaF or 2% SnF<sub>2</sub> reduced the subsequent etching action of a 50% solution of phosphoric acid, probably because the formation of microporosities was inhibited by a decrease in enamel crystal solubility. Surface changes in enamel not so treated (with a fluoride solution) were considerably greater. This suggests an inverse relationship between fluoride content and bond strength.

In contrast, Brännström, Nordenvall & Malmgren (1978) and Brännström, Malmgren & Nordenvall (1982) reported no significant differences in the SEM appearance of etched enamel surfaces of teeth pretreated with a fluoride varnish (Duraphat) compared to control teeth.

The former study applied a 37% solution of phosphoric acid for one minute and the latter study a 50% phosphoric acid gel for 15 seconds.

### **5.6.2 Application to etched enamel**

Teeth treated with a number of topical fluorides subsequent to etching show a greater mean fluoride mass than do control teeth at corresponding depths within the surface enamel. The existence of a fluoride gradient was noted, and the use of 4% acidified NaF on etched enamel results in a greater uptake of fluoride than does the use of neutral NaF (Kajander, Uhland, Ophaug & Sather, 1987).

The application of a variety of topical fluorides to etched enamel surfaces produces reaction products or precipitates which interfere with resin penetration and tag formation due to both their physical presence and the reduction of wettability of the enamel surface (Sheykholeslam, Buonocore & Gwinnett, 1972).

Wei (1975) examined etched enamel samples that were subsequently treated with a variety of topical fluoride applications. He found that calcium fluoride and an amorphous layer formed on the enamel surface.

### **5.6.3 Fluoride and bond strength**

Fluoride solutions should not be applied to etched enamel as the formation of reaction products can be shown to adversely affect bond strength (Sheykholeslam, Buonocore & Gwinnett, 1972; Gwinnett, Buonocore & Sheykholeslam, 1972).

Low, von Fraunhofer & Winter (1975) found that treatment of enamel with acidulated phosphate fluoride (APF) solution prior to

etching significantly reduced the tensile bond strength of pit and fissure sealants. Likewise, treatment of etched enamel with APF prior to resin application led to decreased bond strengths. In contrast, the treatment of etched enamel with 8% stannous fluoride produced an increased tensile bond strength, the reverse of what Sheykholeslam et al (1972) reported. However, Gwinnett et al (1972) noted no reaction products formed (on the enamel surface) when stannous fluoride was used after etching.

Hirce, Sather & Chao (1980) tested shear bond strengths of orthodontic brackets after various fluorides were applied subsequent to etching. Two percent NaF in 50% phosphoric acid significantly decreased bond strength, whereas basic (alkaline) phosphate fluoride and 8%  $\text{SnF}_2$  did not significantly alter it, compared to a control (no fluoride applied).

Bryant, Retief, Bradley & Denys (1985) report that the results of their *in vitro* study suggest that the application of topical fluorides to enamel surfaces 7 days before orthodontic bonding will not have an effect on bond strengths. They found no significant correlation between acquired enamel fluoride concentration and tensile bond strength and no significant differences between bond strengths for 4 topical fluorides and a control.

Thornton, Retief, Bradley & Denys (1986), found that the addition of fluoride (NaF) to a 50% phosphoric acid solution did not impede the etching effect on enamel, nor the subsequent tensile bond strengths.

## 5.7 RECOMMENDED TIMES FOR ACID-ETCHING

Before considering reduced etching times it is necessary, and indeed of some historical interest, to explore the literature for reported etching times. It would seem that much of what has evolved in terms of etching times is rather arbitrary. Russell, Beech & Brown (1985) report the most commonly used etch time amongst Australian orthodontists to be 60 seconds.

Buonocore used a 30 second etch in his seminal 1955 study. Rock (1974) used the recommended 30 second etch for Epoxylite 9075 in a study of fissure sealants. A 45 second etch was used in a laboratory study by Laswell, Welk & Regenos (1971).

For permanent tooth enamel, an etching time of 60 seconds has often been accepted and used or recommended (Newman, 1965; Buonocore, Matsui & Gwinnett, 1968; Buonocore, 1971; Sheykholeslam & Buonocore, 1972; Gwinnett, Buonocore & Sheykholeslam, 1972; Gwinnett, 1973a; Retief, 1973; Retief, 1974a; Rock, 1974; Silverstone, 1974; Adipranoto, Beech & Hardwick, 1975; Silverstone 1975; Jörgensen, 1975a; Jörgensen & Shimokobe, 1975; Pahlavan, Dennison & Charbeneau, 1976; Galil & Wright, 1979; Beech & Jalaly, 1980; Barkmeier, Gwinnett & Shaffer, 1985).

Occasionally longer times have been used, such as 90 seconds (Galil & Wright, 1979) or 120 seconds (Gwinnett & Matsui, 1967; Gwinnett, 1971a,b; Brandt, Servoss & Wolfson, 1975; Pahlavan, Dennison & Charbeneau, 1976; Voss & Charbeneau, 1979).

It was found that an etching time of 120 seconds was required to produce a characteristic etching pattern for deciduous teeth, 60 or 90

seconds not being sufficient (Silverstone, 1975).

### **5.8 PROPHYLAXIS**

Gwinnett (1981) states that acid etching cannot remove all enamel contaminants. The acquired pellicle is relatively resistant to mechanical removal (Meckel, 1965; Hay, 1967; Armstrong, 1971). Miura, Kakagawa, & Ishizaki (1973) showed that maximum bond strengths were only obtained when an oral prophylaxis was done before etching.

Dissolution of the subjacent enamel can remove surface films from the enamel, particularly the SSC (Meckel, 1965; Armstrong, 1971). Acid etching can completely remove the acquired pellicle from the tooth surface according to Main, Thomson, Cummings, Field, Stephen & Gillespie (1983), although this does seem to conflict with the findings reported by various authors in the preceding paragraph.

It was felt that pumicing was a necessary adjunct prior to etching, and was carried out accordingly. In this study the labial surfaces of the teeth were carefully cleaned with a slurry of water and flour of pumice in a rubber cup, as this has been shown to remove the least surface enamel (Pus & Way, 1980).

### **5.9 WASHING**

Smith, Spinelli & Tartakow (1976) showed that phosphoric acid solutions used for etching can penetrate to a depth of 50  $\mu\text{m}$  into enamel, and that after washing the enamel surface, a significant residue of the etchant may be left behind (ie 20-40% of the applied etchant).

Soetopo, Beech & Hardwick (1978) report increased bond strength of resins bonded to etched enamel that had been washed for 60 seconds

compared to 15 seconds.

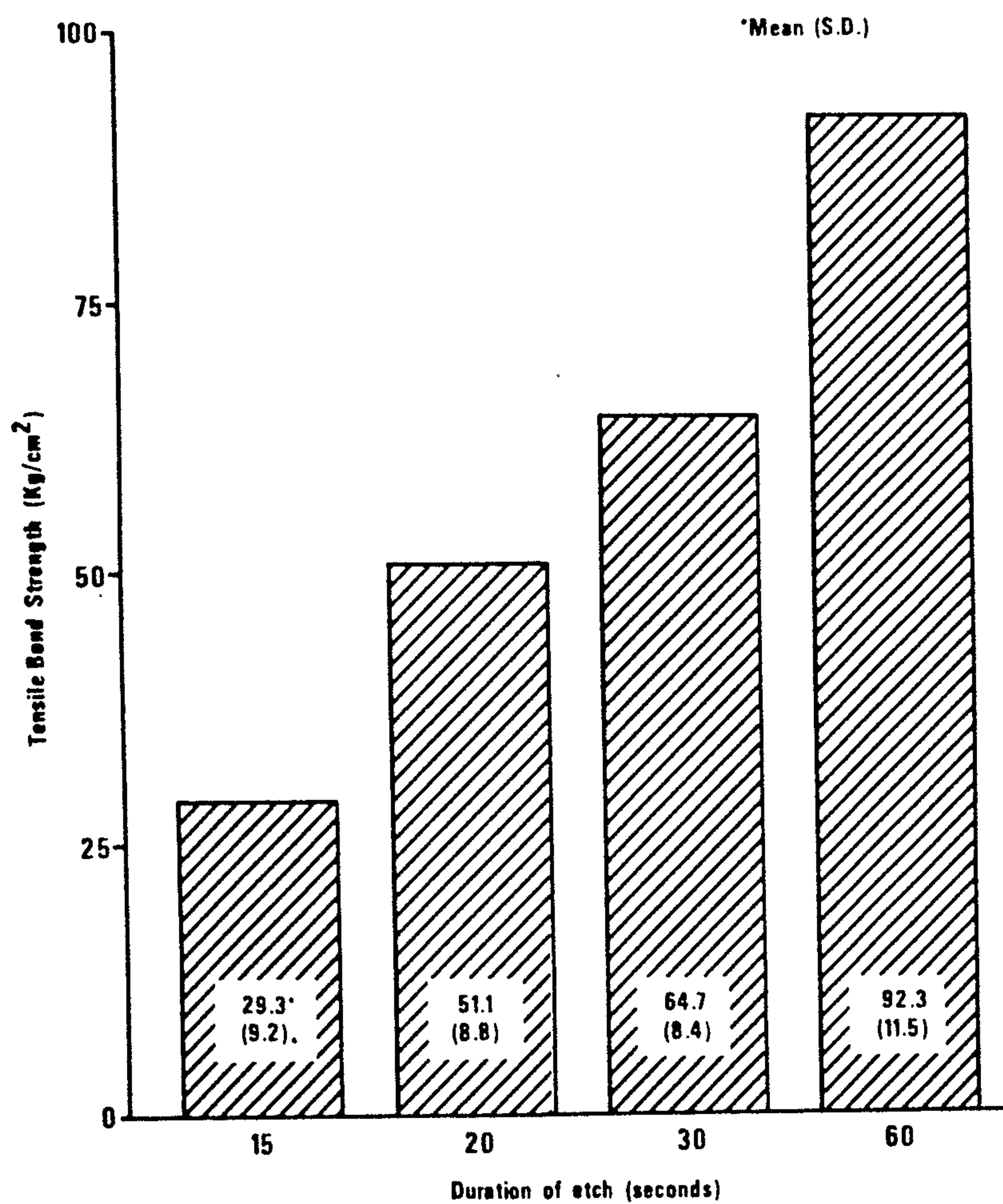
Beech & Jalaly (1980) found a minimum washing time below which the mean shear bond strengths of resins bonded to the etched enamel decreased. They attribute this to the removal of surface contaminants, which were demonstrated as lamellar crystals obscuring the fine prism details under SEM. This precipitated layer of calcium phosphate(s) has a markedly deleterious effect on bond strengths of self curing resins and composites.

Diedrich (1981) found that a minimum of 5 seconds of washing would remove acid residues or crystal precipitates from the etched surface even when using a gel etchant.

Gwinnett (1981) recommends that at this time it would appear preferable to wash each tooth thoroughly for 10-15 seconds (and for up to one minute when a gel is used). There is, he says, no substitute for washing; attempts to remove the acid and its reaction products by other means simply precipitates crystalline reaction products on the surface.

The most frequently given washing time for orthodontists in Australia is 10 seconds (Russell, Beech & Brown, 1985).

Proper drying is also necessary after washing. Compressed air gives a more rapid and thorough drying than chemical drying agents, and resulted in higher bond strengths of fissure sealants (Main, Thomson, Cummings, Field, Stephen & Gillespie, 1983).



Effect of etching time on tensile strength of direct bonded orthodontic attachments.

From Mardaga & Shannon (1982), Decreasing the depth of etch for direct bonding in orthodontics.  
J Clin Orthod 16:130-132

### 5.10 REDUCED ETCHING TIMES

Brännström & Nordenvall (1977) report no apparent difference in the appearance of enamel etched for 15 seconds or two minutes, using a 37% phosphoric acid solution. They suggest that shorter etching times than those hitherto used, can now be accepted.

Beech & Jalaly (1980) feel that it is desirable to dissolve only the minimum amount of enamel from the tooth surface, and therefore only the minimum etching time consistent with obtaining optimal bonding should be used. Longer etching times result in increased tissue destruction. In testing the mean shear bond strengths of resins bonded to enamel, they found that a 5 or 15 second etch was sufficient to produce a bond not sufficiently different to a 60 second etch for unfluoridated teeth. The need for a one minute etch for unfluoridated enamel is therefore questionable.

Nordenvall, Brännström & Malmgren, (1980) demonstrated that a 15 second etch of young permanent teeth (central areas of buccal and lingual surfaces) with a 37% phosphoric acid solution created more retentive conditions than etching for 60 seconds. The reverse was true for old permanent teeth.

Brännström, Malmgren & Nordenvall (1982) using a 50% phosphoric acid gel, found superior etching on young permanent teeth for a 15 second etch, compared to a 60 second etch.

Mardaga & Shannon (1982), found a reduction in tensile bond strengths when reduced etching times with 37% phosphoric acid were used on extracted molars. Compared to the 60 second etch (92.3 kg/cm<sup>2</sup>), the 30 second etch produced a tensile strength decrease of

29.9% (64.7 kg/cm<sup>2</sup>). The decrease for the 20 second etch group was 44.6% (51.1 kg/cm<sup>2</sup>) and that for the 15 second etch, 68.3% (29.3 kg/cm<sup>2</sup>). What is of interest here is not the absolute values for bond strength, but rather, the effect of reducing the etching time on bond strength.

Reynolds (1975) has stated that a maximum mean tensile strength for direct bonded brackets of between 60 to 80 kg/cm<sup>2</sup> would seem reasonable, although clinically successful bonding has been recorded with *in vitro* tensile bond strengths as low as 50 kg/cm<sup>2</sup>. Mardaga & Shannon (1982) feel confident in recommending an etching time of no less than 30 seconds based on these data. However, it should be noted that the 20 second etch group (51.1 kg/cm<sup>2</sup>) modestly exceeded Reynold's (1975) lower figure of 50 kg/cm<sup>2</sup>, and it is tempting to suggest that a twenty-second etch may produce a clinically acceptable bond strength.

Stephen, Kirkwood, Main, Gillespie & Campbell (1982), in a split-mouth trial (20 second versus 60 second etch) of fissure sealants, found retention rates superior to any in the literature for an autopolymerising resin. The teeth etched for 20 seconds had 100% retention at two years; the difference between 20 and 60 second etching being not statistically significant.

Main, Thomson, Cummings, Field, Stephen & Gillespie (1983) assessed the effect of reducing the etch time from 60 to 10 seconds (50% buffered phosphoric) in fissure sealant application. The result was a finer etching pattern which led to a reduced (by 21%), but still high, bond strength, albeit with slightly poorer long-term adhesion (as shown by thermal cycling).

Eidelman, Shapira & Houpt (1984) reported that fissure sealant retention subsequent to a 20 second etch was comparable to the result obtained after a 60 second etch (two year retention rate of 99%).

It is perhaps arguable whether to apply the results of studies on fissure retention to orthodontic bonding directly, and that is not the intention of this review. Instead it is intended to show the effects of reduced etching times in a related technique, as both procedures involve the application of a resin to etched enamel in the expectation of, if not adhesion, then at least durable micromechanical locking. These fissure sealant studies, therefore, provide some circumstantial optimism for the orthodontic application of reduced etching times.

Barkmeier, Gwinnett & Shaffer (1985) evaluated the retentive characteristics of etched enamel surfaces using a 50% phosphoric acid solution for 15 or 60 seconds. The only difference between the two etching times was that the 60 second series showed greater loss of enamel (depth of etch). Examination of the two surfaces showed no morphologic difference in etch patterns produced. Shear bond strengths (of orthodontic brackets) were not statistically different for 15 vs 60 second etching.

Barkmeier, Shaffer & Gwinnett (1986) found no differences in the pattern or character of etched enamel rods following conditioning with 37% phosphoric acid gel for either 15 or 60 seconds. In addition, no difference was found for shear strength testing of composite resin cylinders bonded to these etched surfaces.

Carstensen (1986a) published results of shorter etch times and edgewise bracket retention. After a mean treatment time of some 15 months, he experienced an overall failure rate of 1.23% for 1134

brackets (using a 30-35 second etch). In the second part of his study, 2 brackets were lost in 9 months, both in the 15-20 second etch group; none failing in the 30-35 second etch group. The overall failure rate in the second part was 1.11% (180 brackets).

Sampson, Wookey & Rouse (1987) found that enamel surface loss was significantly increased with etching for 120 seconds compared to 60 seconds. There was little difference in enamel surface loss between 15, 30 and 60 seconds of etching. Enamel surface fluorine levels are lost by 60 seconds of etching. Because of this, and the increased loss of surface enamel beyond 60 seconds, they suggest that optimal etching times probably lie in the 30-60 second range.

## 6 BONDING OF ORTHODONTIC ATTACHMENTS

The bonding of orthodontic attachments is possible because Buonocore (1955) showed the potential of acid etching. Newman (1965) provided evidence of the direction this would take in orthodontics.

Bonding for the orthodontist is less of a problem than for those concerned with preventive and restorative dentistry. This is so for a variety of reasons, especially as the bonding is to enamel only, and the need for bracket retention of only about two years (Reynolds, 1975; Shey & Brandt, 1982).

### 6.1 ACID-ETCH TECHNIQUE

Gwinnett & Matsui (1967) showed that acid pretreatment of enamel surfaces opens up pores or spaces in the enamel into which adhesive resin flows.

Retief (1973) showed that contact between epoxy adhesive and unconditioned enamel was poor, whilst there was intimate interfacial contact between the adhesive and the etched enamel surface. This latter interface did not exhibit leakage. Contact angle measurements obtained with the uncured epoxy resin show a reduction from  $28.3^\circ$  to  $14.3^\circ$  (comparing unconditioned enamel to etched enamel), thus indicating increased wettability of the surface.

Successful bonding is characterised by resin tags within the tissue surface, responsible for mechanical interlocking (Smith, 1975).

If the material is strong intrinsically (ie cohesive strength), the strength of the mechanical bond would increase with increased penetration of the material into the enamel. Assuming the material has

good wetting properties for the enamel, the extent of penetration will depend very largely upon the number of spaces in the enamel into which the material can flow. The depth of penetration, ie tag length, will also depend on the speed of polymerisation of the material. If rapid setting, the free monomer present in the early stages of polymerisation may have little time to penetrate (Gwinnett & Matsui, 1967).

## **6.2 ADVANTAGES AND INCIDENCE**

There are many reported advantages, for both operator and patient, to bonding over banding, including, inter alia, aesthetics, oral hygiene, ease of use and time savings, patient comfort and for unbandable teeth (Zachrisson, 1985; Russell, Beech & Brown, 1985).

Silverman, Cohen, Gianelly & Dietz (1972) presented their method of bonding metal and plastic brackets for approximately 100 patients over a six-month period. These bonded appliances withstood all manner of applied forces successfully, including headgear and substantial root torquing movements.

In a survey of 2 000 American orthodontists, Gorelick (1979) reported that 93% of orthodontists surveyed bonded some teeth. Those teeth bonded most often were the upper anteriors (90% of respondents) and the lower anteriors (77%). Bicuspids were bonded by 40-60% and molars by approximately 10% of respondents.

Russell, Beech & Brown (1985), in an Australian survey, elicited an even higher figure of respondents bonding (98%). Ninety-two percent of those surveyed bonded anterior teeth routinely, and 41% routinely bond anteriors and premolars. Only 8.5% extend this to molars as well.

### 6.3 BONDING RESINS

According to Reynolds (1975) and Read (1984), there are two main types of direct bonding resins, acrylic resins or diacrylate resins. The most common diacrylates are based on bisphenol A glycidyl dimethacrylate (Bis GMA), or Bowen's resin.

Russell, Beech & Brown (1985) report a clear preference for (Bis-GMA) composite resin adhesives over acrylics in orthodontic bonding, in their national survey of Australian orthodontists.

A basic difference between the two resins is that the acrylics form linear polymers only, whereas the diacrylates form cross-linked, three-dimensional networks. Both types of resin can occur in filled or unfilled forms (Gorelick, Masunaga, Thomas & Zachrisson, 1978a).

The polymerised (Bis GMA) resin is extremely rigid, is stronger, and has lower water absorption and less polymerisation shrinkage than the acrylic resins (Read, 1984).

To overcome some of the difficulties with acrylic resins, materials based on Bis-GMA have been developed. Owing to their high viscosity, diluent monomers are added (Maijer, 1982).

To the base resin of Bis-GMA is added the diluent monomer; usually methacrylate, triethylene glycol dimethacrylate or ethylene glycol dimethacrylate; the initiator is peroxide amine or benzoyl peroxide (Altuna & Freeman, 1987).

Monolok™ is a no-mix adhesive designed for orthodontic bracket bonding. The primer contains Bis-GMA resin with an alkylated aromatic amine accelerator. The paste is Bis-GMA resin with silica filler (65% by

weight), and benzoyl peroxide initiator. The manufacturer is Rocky Mountain Orthodontics of Denver, Colorado (Wright & Powers, 1985).

No comparison has been made in this study regarding the different properties, for example cohesive strengths, for two reasons. Firstly a comparison of adhesives was considered to be beyond the scope of this thesis, and secondly, in order to make this study a realistic one with regard to everyday clinical applicability, it was felt reasonable to use one adhesive throughout. Monolok™ is widely commercially available and popular. Therefore it was assumed that it must be performing to an acceptable level of efficiency for those clinicians using it in the orthodontic market-place.

#### **6.4 ADHESIVE RESIN VISCOSITY**

Monolok™, being a filled paste sandwiched between the unfilled primer which is placed both on the tooth and bracket pad, would appear to be covering all contingencies, in relation to the question of whether an unfilled, intermediate resin is required for optimal tag penetration.

Jørgensen & Shmoke (1975), in comparing an unfilled resin to a number of filled resins, concluded that viscosity did not affect resin penetration. That is, tag formation, length and shape were independent of the materials examined; and they suggest that there is no need for the use of an intermediate low viscosity resin as there is sufficient liquid phase in the composite resins to form adequately long tags (which were observed to contain no fillers).

Jassem, Retief & Jamison (1981) found no significant differences in bond strength with and without the use of a sealing resin (ie intermediate low viscosity resin).

Tags from the composite resin adhesives do not contain filler particles, as the resin penetration does not depend on the viscosity of the composite material but on, among other factors, the viscosity of the resin component and whether this component is available in sufficient amounts at the interface (Jørgensen, 1975b). Monolok™ tags are, of course, unlikely to contain filler particles anyway, as the system employs an unfilled primer or sealant which is applied directly to the etched enamel surface.

Pahlavan, Dennison & Charbeneau (1976) found no significant differences between four resin materials with different viscosities in terms of tag lengths formed. These tags penetrated to 5–10  $\mu\text{m}$  (mean 7  $\mu\text{m}$ ) after etching with 50% phosphoric acid for 60 seconds. One filled resin exhibited filler particles near the interface, but a fairly clear zone of resin was seen immediately adjacent to the enamel.

Diedrich (1981) also found no differences for three adhesives in terms of penetration (tag length) despite their different viscosities and composition.

## 7 CLINICAL FAILURE

The following review of bond strengths and occlusal forces is not included in order to relate the two with any precision but to provide some background information, and for completeness. One must also question the relevance of tensile strength testing, as under *in vivo* conditions, dental adhesive materials would more likely be subjected to shear and compressive forces (Retief, 1974b). Occlusal force values are included so that the reader is aware that the forces of occlusion are far and away greater than applied orthodontic forces (Reynolds, 1975). One would assume that failure rates of bonded attachments might vary as occlusal forces are less incisally than at the first molars, although a number of other factors also relate to failure, as will be seen below.

### 7.1 BOND STRENGTHS

Phillips (1980) states that the minimum suitable bond strength is an unanswered question. Whilst it is beyond the scope of this study to review bond strength testing, it is interesting to note that Jassem, Retief & Jamison (1981) found no significant differences between bond strengths in shear or tension for bonded orthodontic attachments. Retief (1974b) states that tensile loading tests are the preferred method of testing, ratified by the Dental Materials Group of the IADR.

Reynolds (1975) states that although maximum orthodontic forces are unlikely to exceed 1.5 kg, the bonded attachment must withstand occlusal loading, the major intra-oral force. He recommends a maximum mean *in vitro* tensile bond strength between 60 to 80 kg/cm<sup>2</sup>, although clinically successful bonding has been recorded with tensile bond strengths as low as 50 kg/cm<sup>2</sup>.

Faust, Grego, Fan & Powers (1978) reported tensile bond strengths of 19-53 kg/cm<sup>2</sup> for brackets bonded to teeth with a variety of adhesives. And Zidan & Hill (1986) recorded bond strengths of 201 to 207 kg/cm<sup>2</sup> for composite resin bonded to etched enamel for phosphoric acid concentrations of 2, 5 & 35%.

## **7.2 BOND STRENGTH AND TAG LENGTH**

Retief, (1978) states that tag penetration into the artificially created microspaces allows for mechanical bonding between enamel and resin, and that this is a major factor in bond strength.

If the material is strong intrinsically, the strength of the mechanical bond would increase with increased penetration of the material into the enamel (Gwinnett & Matsui, 1967).

However, Adipranoto, Beech & Hardwick (1975) state that the length of resin tags penetrating into the enamel showed no correlation with observed bond strengths. Low acid concentrations produced strong bonds with little tag formation. They conclude that adhesion arises from the formation of secondary bonds rather than from mechanical interlocking. Zidan & Hill (1986) likewise concluded that tag length is of little, if any, consequence for adequate bonding.

A direct relationship between tag length and bond strength of dental resins to etched enamel has not yet been demonstrated (Gwinnett, 1979; Manson-Rahemtulla, Retief & Jamison, 1984).

### 7.3 BOND STRENGTH AND TOOTH TYPE

Knoll, Gwinnett & Wolff (1986) showed a marked difference between shear strengths for incisors and molars, in laboratory shear strength testing. The incisor bonding failures occurred on average at a point 42% higher than for the molars. They postulate that the lower values for the molar teeth may relate to the adaptation of the bracket and non-uniform resin thickness. Gorelick, Masunaga, Thomas & Zachrisson (1978a) relate bonding failure to excessive thickness of the adhesive. Good adaptation of the bracket and a thin layer of adhesive should be aimed for.

### 7.4 OCCLUSAL FORCES

Jenkins (1966) reports maximum loads of approximately 45 kg on molar teeth during normal mastication; and that average masticatory loads are usually approximately one-third of the maximum. Carlsson (1974) feels likewise, that chewing forces are generally much lower than the maximal bite force.

Mizrahi & Smith (1971) cite Mizrahi (1968 MSc thesis, Manchester) who states that a load of 12 kg may be produced during mastication. Further, they believe the forces of mastication are more likely to produce bond failure than the forces from the orthodontic appliance.

Garner & Kotwal (1973) measured incisive biting forces of males and females from the age of ten years to the early or mid-twenties. They report mean values of approximately 18 and 14 kg for males and females, respectively.

Reynolds (1975) reports a mean maximum biting force of 70 kg (range 10-100 kg) and a maximum force produced by an orthodontic

headgear of only 1.5 kg. Thus the maximum occlusal force may be 47 times higher than the maximum orthodontic force.

Proffit, Fields & Nixon (1983) reported a mean maximum biting force of 31 kg and 35.6 kg (for 2.5 and 6 mm opening, respectively) in mesofacial subjects. The values for chewing were less than half these values and for swallowing, around one-tenth of these values.

### **7.5 INCIDENCE OF FAILURE**

Sheykholeslam & Brandt (1977) suggest that if bonding failures are to be minimised, there should be a proper etching in strict accordance with the manufacturer's instructions, preceded by a thorough prophylaxis, and that strict moisture control should be maintained throughout the entire bonding procedure.

The chief causes of bond failure are: inadequate removal of the organic pellicle; improper etching; poor choice of bonding system; moisture contamination; disturbing the bracket during setting of the adhesive; failure to comply with manufacturer's instructions; premature archwire engagement; thick adhesive layer due to poor adaptation of bracket base to tooth surface; and, premature placement of bracket in occlusion (Gorelick, Masunaga, Thomas & Zachrisson, 1978c).

Other factors influence the bonding and hence bond strengths of metal brackets to enamel. These relate to the nature of the base such as the design of its retentive components and manufacturing effects, for example, weld spots (Maijer & Smith, 1981).

In Gorelick's 1979 survey, 56% of respondents felt that bonded brackets held as well as bands, whilst 35% did not. 64% said that improved technique, such as isolation methods, improved their bonding

success rates.

Zachrisson (1977) recorded data for bracket failure ( $n=705$ ) in 46 children over a mean treatment time of 17 months. Incisors, canines and first premolars in both arches failed at the rate of 4-10%. Second premolars and molars had higher failure rates. To reduce failure rates Zachrisson urges the improvement of the clinical procedure rather than increasing the strength of the adhesive.

Gorelick (1977) reported a failure rate of 5.8% for 549 brackets over a 12-month period. Upper anterior brackets had a failure rate of 4%; lower anteriors, 6.5%; upper premolars, 6.2%; and 7% for lower premolars. An additional study of 800 brackets observed for a 6-month period showed very similar failure rates. Subsequent studies using various adhesive combinations resulted in overall failure rates over a 6-month period of 3-5.2% for upper and lower anteriors and bicuspid.

Zachrisson & Brobakken (1978), in a 6-month observation period, noted a failure rate of 2.5% for 243 direct bonded brackets.

Mizrahi (1982) found that the lowest rates of "adhesion" failure can be achieved by placing bands on all posterior teeth and mesh-backed brackets on incisors and canines. He states that the failure rates for lower incisors is similar whether banded or bonded; and that bonded brackets are more successfully retained than bands placed on upper anterior teeth. His overall adhesion failure rate in this study was 4.7%, compared to a failure rate of 7% for a fully banded study, both using the Begg LWT.

Silverman (1979) assessed his overall failure rate for bonding, including molars, as being 2% or less using a heavily filled composite

resin adhesive.

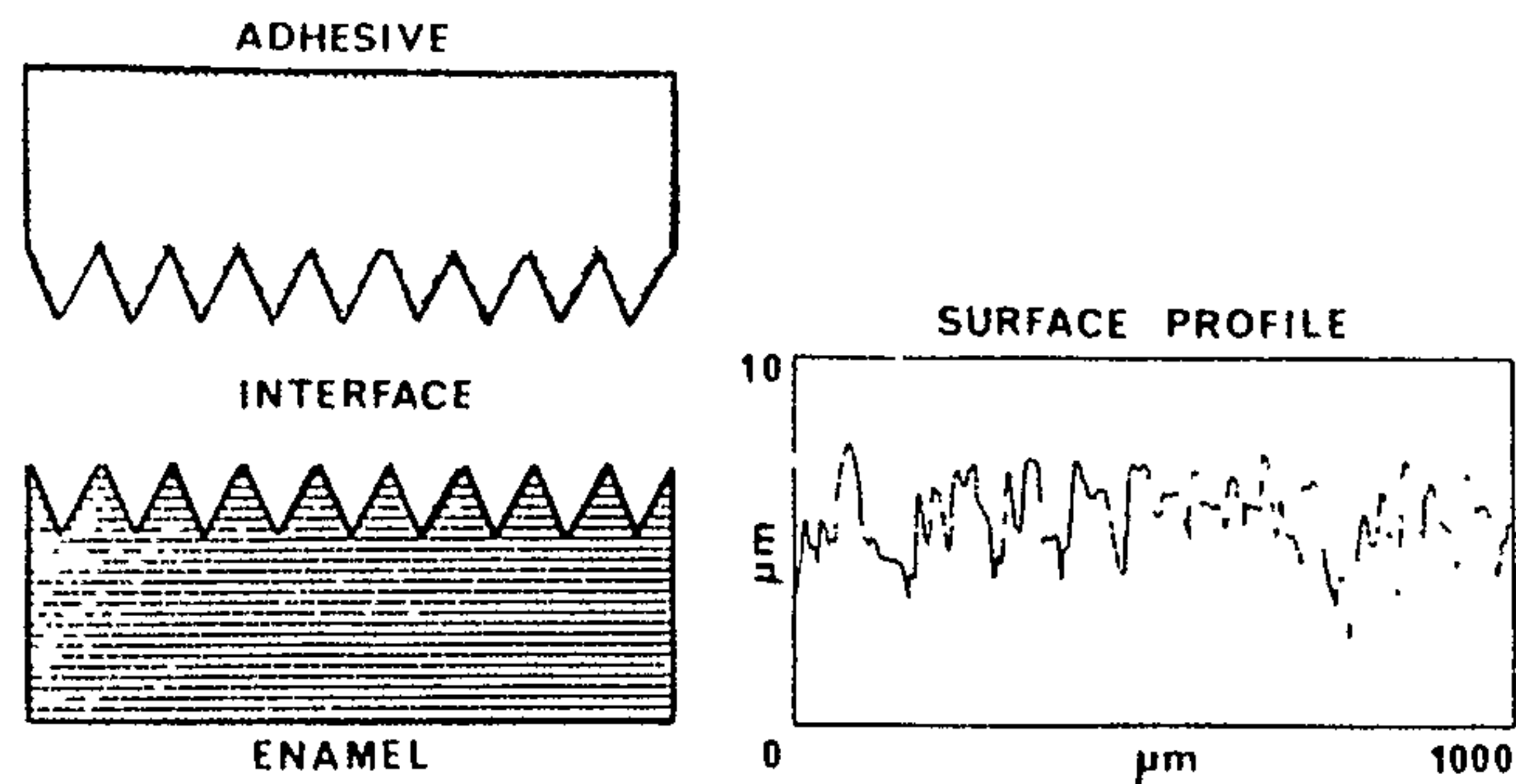
Aguirre, King & Waldron (1982) experienced an overall failure rate of 5.3% for direct-bonded brackets in a three-month period. Of this 5.3%, the majority of failures were anteriors - three upper and one lower. One lower 1st bicuspid failed.

Geiger, Gorelick & Gwinnett (1983) reported that early failure of a bond occurs more frequently than late failure; the majority failing within the first three months. Sheykholeslam & Brandt (1977) feel that if a bond fails shortly after being secured, it is probably due to faulty technique.

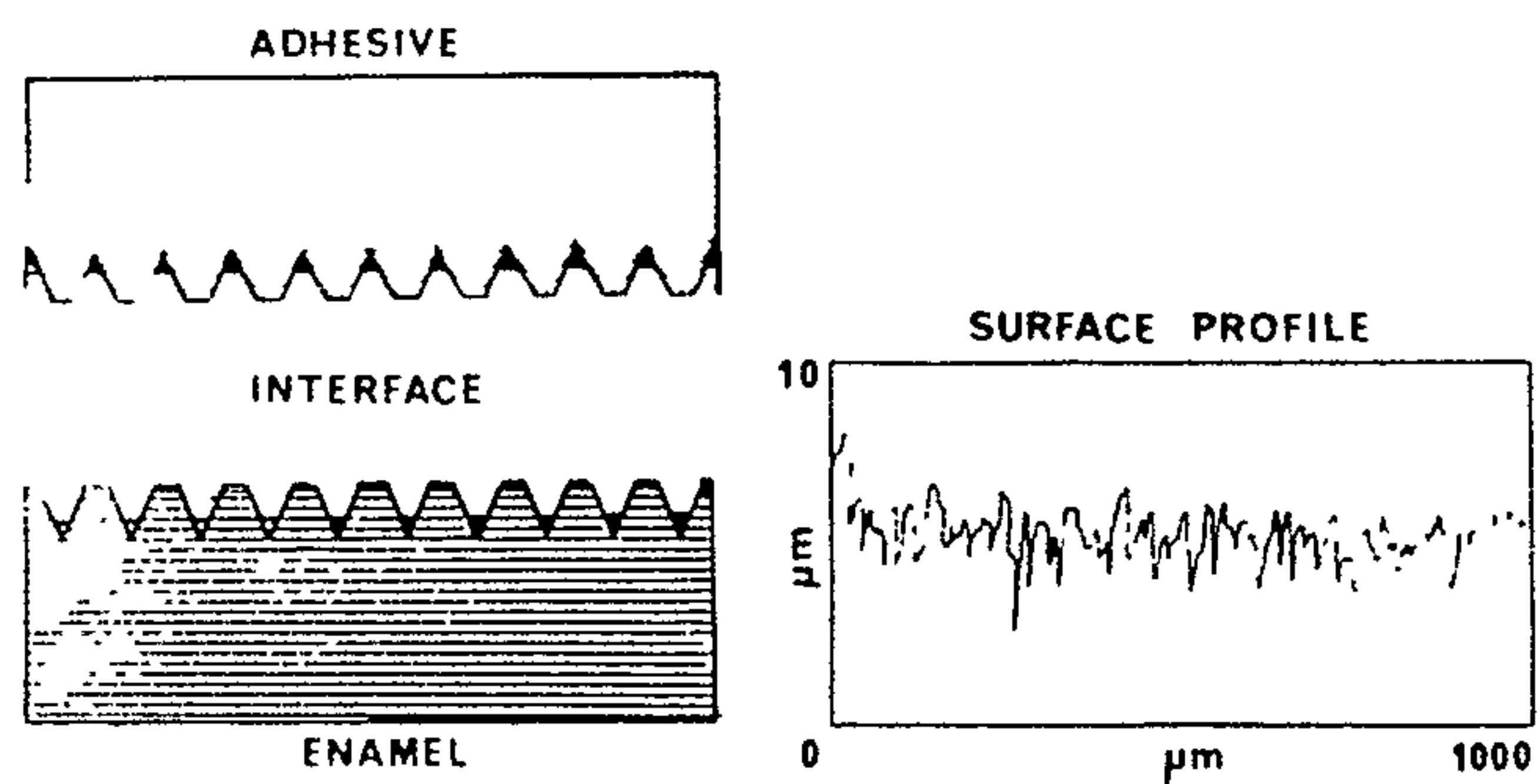
Geiger, Gorelick & Gwinnett (1983) note that brackets bonded to upper molars failed most often (21.1%), followed by lower incisors (8.8%), then upper bicuspids (5.1%) and, least often, upper cuspids (3.5%) at 5 months.

Artun & Bergland (1984), in a study comparing crystal-growth conditioning to a 60 second etch with a 37% phosphoric acid solution (control), found overall failure rates for the control brackets in the range 0-8.5%, in 4 treatment groups, over a six-month period. Incisor brackets failed not at all; other teeth experienced some failures.

Russell, Beech & Brown (1985) recorded the reported rates of Australian orthodontists' bracket failures as a mean of 5.6%. The respondents attributed these failures to (in descending order of frequency): faulty technique; patient abuse, accidents; occlusal force (poor tooth selection); applying too much force too soon; adhesive or bracket faults; or adverse enamel properties (eg enamel hypoplasia).



Diagrammatic presentation of clean interfacial break and surface profile of enamel surface etched with 50%  $H_3PO_4$  for 1 minute.



Diagrammatic presentation of model to explain interfacial failure and surface profile of the enamel aspect of a fractured bond on etched enamel.

**From Retief (1975d), Pulpal responses to phosphoric acid and failure at the dental resin-etched enamel interface.  
In: Proceedings of an International Symposium on the Acid Etch Technique (Eds: Silverstone & Dogon) pp 213-229  
North Central Pub. Co., St Paul, Minnesota**

## 7.6 FAILURE SITES

Failure can be classified as cohesive, adhesive or a combination of the two. Cohesive failure is not restricted to the adhesive, as, for example, plastic brackets may fracture under various loadings. The following authors report observations on failure sites which indicate that the problem of adhesion to the tooth is less of a problem than, say, attachment of the bracket base to the adhesive resin. It is beyond the scope of this study to analyse the ætiology of failure as it relates to the different characteristics and properties of the adhesive resins and brackets used. However, it was felt pertinent to include some information about where such failures might occur.

Sites of failure cannot be reliably classified visually by the unaided eye or even under low magnification (Retief, 1974b, 1975d). He demonstrated that so-called interfacial failures involved both fracture of enamel and resin tags, that is, failure partly within the enamel and partly within the resin.

Keizer, Ten Cate & Arends (1976) state that the attachment of the bracket to the adhesive is the bottleneck of the direct bonding procedure, at least where plastic brackets are concerned. They found a mean maximum bond strength to enamel of 121 kg/cm<sup>2</sup> compared to the mean maximum bond strength to the bracket of 53 kg/cm<sup>2</sup> using a variety of Bis-GMA resins and plastic brackets that had, or had not, received surface treatment prior to bonding.

Low & von Fraunhofer (1976) state that the weakest link in their study (of *in vitro* bracket bonding) was not at the tooth-adhesive interface but at the mesh-adhesive junction (90% failed at the latter site). They also found no correlation between the load at failure and the

surface area of attachment.

Bond failure was found (by SEM) to occur at the bracket-adhesive interface approximately 50% of the time; a combination of fracture at the bracket, within the adhesive and at the adhesive-enamel interface in an additional 50% of cases; and, in a very small percentage, at the adhesive-enamel interface itself (Perry, 1980).

Alexandre, Young & Bowman (1981) reported bond failure for one adhesive at the enamel-adhesive interface in half the brackets tested, and at the bracket-adhesive interface for another one-third of the brackets tested. Two other adhesives showed predominant failure at the bracket-adhesive interface (69% and 83%).

Farquhar (1986) found that the predominant fracture site for shear strength testing of edgewise brackets occurred at the bracket-resin interface, although fractures involving more than one interface were often seen.

Zidan & Hill (1986) found that the majority of fractures occurred within the composite resin, either a bulk failure or an apparent interfacial failure which was later shown (by SEM) to be within the resin.

## 8 MATERIALS AND METHODS

### 8.1 STANDARDISATION

More times than not, the cause of bond failures can be linked to poor operating procedures (Brandt, Servoss & Wolfson, 1975; Sheykholeslam & Brandt, 1977). For this reason each bonding was standardised, and the etching, timed.

According to Nasir, Retief & Holbrook (1981), acid etching (depth) depends on the type of acid used, the acid concentration, the duration of etching and the enamel fluoride concentration. In this study the etch time was the variable tested, as all reasonable attempts were made to hold the other three possible variables constant.

Permanent teeth only were used as these are more commonly bonded to and because a number of authors have reported on prismless enamel (Ripa et al, 1966; Gwinnett, 1966, 1967) and its resistance to etching (Sheykholeslam & Buonocore, 1972; Gwinnett, 1973b; Silverstone, 1975).

The one type of Begg bracket was used. This was the Begg type light wire bracket (A-1086) manufactured by Rocky Mountain Orthodontics, Denver, Colorado. The attached mini-bases were contoured for incisors (D-2520) or for cuspids and bicuspids (D-2521), and were of the mesh type. Mesh-backed brackets give stronger bonds than those with perforated pads (Zachrisson & Brobakken, 1978). Fine mesh bracket bases of the woven mesh type gives the best resin penetration and highest bond strengths (Maijer & Smith, 1981). Russell, Beech & Brown (1985) report that 82% of Australian orthodontists who currently bond use metal brackets, and that of these, 97.5% use mesh-based brackets.

TABLE 1

|           | 20 seconds | 60 seconds | Totals |
|-----------|------------|------------|--------|
| Incisors  | 56         | 56         | 112    |
| Canines   | 28         | 28         | 56     |
| Premolars | 33         | 33         | 66     |
| Totals    | 117        | 117        | 234    |

**Numbers of brackets: tooth type and etch times**

Also, the overwhelming majority use mini or regular (or the two in combination) sized bracket bases.

The direct bonding system used throughout was the commercially-available Monolok™ from Rocky Mountain. The supplied etchant was a 37% solution. Russell, Beech & Brown (1985) report that 75% of Australian orthodontists etch some, or all, of the time with an etching solution. Monolok™ is the second most popular bonding adhesive in this country (Russell, Beech & Brown, 1985).

One operator (the author) performed the bonding procedure on all patients. A split-mouth technique was carried out to compare the two etch times. It is known that contralateral pairs of teeth display very similar etching patterns (Jørgensen, 1975a,b). In an attempt to eliminate left-right bias, alternate patients were etched in reverse (ie, left replaced right). Aguirre, King & Waldron (1982) noted that there was a trend in their study for the left side bonds to be more accurately placed in the upper, and the right side bonds to be more accurately placed in the lower.

In an attempt to simulate routine treatment, the seventeen patients were not selected for malocclusion type. 234 brackets were bonded to anteriors and premolars as required. A total of 112 incisors, 56 canines and 66 premolars were bracketed directly. Any occlusal interferences were accepted as being part of routine treatment, although some were relieved where possible, if this did not adversely affect the bracket's integrity. It was anticipated that the occlusal interferences would be eliminated as tooth movement progressed, especially on lower incisors in deep bite cases.

Patients all had, as far as can be ascertained, a 100% positive

fluoride history. Sydney was the population drawn upon, with a population of 2.87 million, fluoridated since 1968 (Fluoridation of Water in Australia, 1984). Carstensen (1986b, personal communication) did not consider the fluoride histories for his patients in his analysis of bracket failure. Other aspects of fluoride treatment or the use of fluoridated dentifrices, were not considered for the patients in the present study. The practice area of 83% of Australian orthodontists is fluoridated (Russell, Beech & Brown, 1985).

## **8.2 TECHNIQUE**

### **8.2.1 Pumicing of enamel**

The acquired pellicle is relatively resistant to mechanical removal (Meckel, 1965; Hay, 1967; Armstrong, 1971). Miura, Kakagawa & Ishizaki (1973) showed that maximum bond strengths were only obtained when an oral prophylaxis was done before etching.

Gwinnett (1981) states that acid etching cannot remove all enamel contaminants. Dissolution of the subjacent enamel can remove surface films from the enamel, particularly the SSC (Meckel, 1965; Armstrong, 1971). However, not all workers in this area agree. Acid-etching may completely remove the acquired pellicle from the tooth surface according to Main, Thomson, Cummings, Field, Stephen & Gillespie (1983).

It was felt safer to proceed with this project on the assumption that a prophylaxis with a slurry of water and flour of pumice would remove surface contaminants. The labial surfaces of the teeth were carefully cleaned with pumice in a rubber cup, as this has been shown to remove the least surface enamel (Pus & Way, 1980).

### **8.2.2 Etching**

After pumicing, the teeth were rinsed until visibly free of pumice. Lip and cheek retractors with a saliva ejector attached to a bite-block, were immediately inserted. The teeth were air dried and then etched for either 20 or 60 seconds. Carstensen (1986a) feels that it takes 5 seconds to etch 3 teeth, in other words, the third tooth receives 5 seconds less in etching. In this study no more than 3 teeth were etched at any one time, sometimes only one or two. It was felt that the etchant could be applied more quickly than Carstensen suggests, resulting in a typical etch time for 3 teeth of, say, 17-20 seconds. The etchant placed on the teeth for 60 seconds was agitated gently twice after placement with the mini-sponge supplied.

### **8.2.3 Washing**

After etching, the teeth were washed thoroughly with a vigorous water spray for 15-20 seconds, starting with the first tooth etched, and working around. The importance of washing has already been reported. The etched surfaces were carefully air-dried until the characteristic mat appearance was obtained.

### **8.2.4 Contamination of the etched surface**

Diedrich (1981) stresses that great care should be taken of the etched surface. He showed that detrimental effects result from saliva or mucosal contact, oil contamination and hand contact. Artun & Bergland (1984) reported a difference in failure rates of orthodontic brackets when using conventional (8.5%) versus high speed (3.8%) evacuation.

### **8.2.5 Bonding**

Monolok™ primer was applied in a thin film to the tooth surface. A dental assistant applied primer and then the Monolok™ paste to the bracket base, as would occur in routine application of the procedure. The bracket was as described above with a small mesh bonding base. The brackets were placed in routine positions on the teeth and pressed into place to minimise adhesive thickness. Following setting, gross adhesive flash was removed, where required, from the tooth surface and an APF gel applied for 4 minutes. Retractors and saliva ejector were then removed and the patient dismissed.

### **8.2.6 Recall**

The patients undergoing routine Begg light wire treatment were referred back to their respective operators and any bond failures were recorded by them. The author collated data on failure directly from the patients' files at the end of the observation period.

## **8.3 RESULTS**

Zachrisson (1977) recorded failure rates of 4-10% for brackets bonded to incisors, canines and first premolars. Gorelick (1977) reported failure rates of 5.8%. Upper anterior brackets had a failure rate of 4%; lower anteriors, 6.5%; upper premolars, 6.2%; and 7% for lower premolars. Zachrisson & Brobakken (1978) report an overall failure rate of 2.5% for 243 direct-bonded brackets. Additionally, they present figures for maxillary versus mandibular bracket failures and tooth by tooth failures. A total of 3.9% failed in the maxilla, whilst 1.4% failed in the mandible. Teeth that failed were the upper central incisor (n=2), upper first premolar and upper 1st molar. In the mandible, one first

TABLE 2

|           | 20 seconds | 60 seconds |
|-----------|------------|------------|
| Incisors  | 3          |            |
| Canines   |            | 1          |
| Premolars |            | 1          |
| Total     | 3          | 2          |

**Number of bracket failures: tooth type and etch times**

TABLE 3

|                  | 20 seconds | 60 seconds |
|------------------|------------|------------|
| Brackets         | 117        | 117        |
| Bracket failures | 3          | 2          |
| Percent failure  | 2.56%      | 1.71%      |

**Percentage of bracket failures and etch times**

premolar and one lateral incisor failed. Silverman (1979) assessed his overall failure rate for bonding, including molars, as being 2% or less using a heavily filled composite resin adhesive.

Mizrahi (1982) found that the lowest rates of "adhesion" failure can be achieved by placing bands on all posterior teeth and mesh-backed brackets on incisors and canines. His overall adhesion failure rate in this study was 4.7%, compared to a failure rate of 7% for a fully banded study, both using the Begg Light Wire Technique.

Aguirre, King & Waldron (1982) experienced an overall failure rate of 5.3% for direct-bonded brackets in a three-month period. The teeth that failed were: UR3; UR1,2; LR3; and LR4.

Russell, Beech & Brown (1985) recorded the reported rates of Australian orthodontists' bracket failures as a mean of 5.6%.

### **8.3.1 Incidence of failure**

Five brackets failed out of a total of 234. The observation period was 6 to 8 months since bracket bonding. The reported causes of failure are seemingly not consistent in terms of both time of failure or circumstance; the fortuitous debondings that occurred may not be truly representative of bracket failures in general. Three may fall into the category of patient abuse, and two may possibly constitute abuse by the operator.

The first bracket failed at 1 day. This was probably due to direct occlusal loading by an opposing tooth. The tooth bonded was the lower left lateral incisor (20 second etch).

The second tooth to fail was at 61 days due to a blow to the mouth

from a surf-ski. The tooth was the upper left central incisor (20 second etch).

The third to fail was at 84 days due to a shearing force imparted during normal bypass clamp removal. Tooth was upper left first premolar (60 second etch).

The fourth failed at 91 days, reportedly due to a normal attempt to pin in the arch wire (0.016"). This tooth was the upper right lateral incisor (20 second etch).

The fifth to fail was at (calculated median time between appointments) 137 days. This may be partly due to direct occlusal loading by an opposing tooth, as this upper left canine was in lingual crossbite initially (the etch time was 60 seconds).

### **8.3.2 Failure rates**

Three brackets out of the 117 20-second-etch group failed (2.56%). Two brackets out of the 117 60-second-etch group failed (1.71%). The overall failure rate was 5 out of 234 brackets or 2.14%.

Time at failure was determined by the patient's report in four cases, and by calculating the median time between the last two appointments for one patient who could not accurately report the time of failure. The mean time at failure was 74.8 days. This is within a 2 to 3 month period. One bracket failed within one month (at one day); three failed between 2 and 3 months (61, 84 and 91 days); and one failed before 5 months (137 days).

Mean time of failure for the 20-second-etch group was 51.0 days. For the 60-second-etch group, the mean was 110.5 days.

### **8.3.3 Tooth type**

Three incisor brackets failed. One canine bracket failed. One first premolar bracket failed.

Upper tooth brackets failed more often than lowers (ratio of 4:1). More left-side teeth failed than right-side teeth (ratio 4:1).

### **8.3.4 Statistical significance**

The details of the statistical tests used in this thesis are contained in Appendix 2.

There was no significant difference in failure rates of Begg brackets bonded to enamel that had been etched for either 20 or 60 seconds (5% level of confidence). Two statistical tests were applied to determine this. They were a chi-squared analysis comparing the dependency of expected versus observed failures, and a chi-squared analysis of the homogeneity of (proportion of) failure rates between the two groups.

Neither were there any significant differences for bracket failures between upper and lower teeth or between left and right sides when tested for proportion of failures and subjected to a chi-squared analysis (5% level of confidence).

A two-sample, unpaired t-test comparing the mean times at failure for 20 and 60 seconds found no significant difference for the two etch times (5% level of confidence).

A chi-squared analysis of the testing of dependency between the two etch times found that there was a significant difference for tooth type (10% level of confidence). In other words, failure is dependent on

tooth type as the proportions of failure in each tooth type sample are not homogeneous.

#### **8.4 DISCUSSION**

The results of this study compare favourably to reports in the literature for orthodontic bond failures. It was important to establish whether the results of this study differed from the published results of others. If they did not, and indeed this was the case, we can have some confidence in the validity of the results of the reduced etching time group.

The overall failure rate in this clinical study was 2.14%. The ratio of upper to lower bracket failures was similar to Zachrisson & Brobakken (1978), viz, 4:1. Aguirre et al (1982) recorded a ratio of upper to lower bracket failures of 3:2. All brackets that failed in their study were bonded to teeth on the right side. In this study the ratio of failures was reversed; that is, more left side brackets failed.

Aguirre et al (1982) reported a finding of predominantly anterior bracket failure which matches that found in this study, that is, 80% or a ratio of four to one (anterior to posterior). As reported above, tooth type only was statistically significant in relation to bracket failure. The circumstances surrounding the failure of the 5 individual brackets are discussed above.

Greater gross loss of enamel has been shown to occur with a 60 second etch compared to a 20 second etch (Barkmeier, Gwinnett & Shaffer, 1985). Shey & Brandt (1982) and Sampson, Wookey & Rouse (1987), amongst others, note that enamel loss is proportional to etch time. Further studies need to be carried out, along the lines of those

reported by Fitzpatrick & Way (1977), Brown & Way (1978) and Pus & Way (1980), to determine if less enamel loss occurs on debonding an attachment which is bonded to enamel that has been etched with a reduced etching time. This may or may not be true, as bond strengths to enamel which has been etched for a shorter time, may or may not be reduced (although Mardaga & Shannon, 1982, found this to be so for orthodontic buttons bonded *in vitro*). It is suggested that further studies be carried out to ascertain bond strengths of orthodontic attachments to enamel etched for a shorter time, under conditions which may more nearly approximate those found in the oral environment.

## 9 SUMMARY AND CONCLUSIONS

### 9.1 SUMMARY

The underlying hypothesis is that reduced etching times, specifically a twenty second etch time, would result in clinically acceptable retention rates in the bonding of Begg orthodontic brackets for the duration of what was felt to be a reasonable observation period during routine fixed appliance therapy. Allied with this is the presumption that a reduced etching time will cause less damage to tooth enamel: indeed, that a reduced depth of etch should result and the underlying histologic change should be similar to that produced by a 60 second etch.

A clinical study was undertaken on a group of Sydney school-children, who were accepted for routine fixed appliance therapy using the Begg light wire technique at the United Dental Hospital of Sydney. Begg brackets with mesh bases were bonded directly to the teeth of seventeen patients who met the selection criterion of a total, positive fluoride history. In all, 234 teeth were bracketed, including incisors, canines and some premolars. The bonding procedure was carried out by the author and was standardised for each patient. A split-mouth technique was utilised. The etch time was the test variable: 20 seconds being employed on one side of the mouth, and 60 seconds on the other. Routine bonding of brackets was carried out using Monolok™ from Rocky Mountain Orthodontics, Inc.

These patients were from then on treated by colleagues either in the United Dental Hospital Orthodontic Department, or by fellow Master of Dental Science students in the University (of Sydney) Orthodontic Department. As far as can be determined, these patients were treated as

routinely as any other fixed appliance patients undergoing orthodontic treatment at the United Dental Hospital. Bracket failures were recorded in the patients' files and collated by the author after a period of 6 to 8 months from the bonding appointment.

A total of 5 bracket failures was reported. Three incisors, one canine and one premolar failed. Three were from teeth etched for 20 seconds and two from teeth etched for 60 seconds. Statistically, no differences were found between failure rates for the two etch times. Further, there were no significant differences between bracket failures for either left versus right side or upper versus lower teeth bracketed. Differences in times of bracket failure for the two etch times were also found to be not significant.

However, when tooth type was considered, there was a significant relationship between tooth type and etch time; that is, the failure rates differed significantly from what would be expected from chance alone. In other words, tooth type affects bracket failure rates within the two groups (20 second versus 60 second etch times).

The bonding of brackets for routine Begg light wire treatment using a reduced etching time gave comparable results to etching for 60 seconds. Moreover, the failure rate of brackets in this study compares favourably to other studies reported in the literature both for conventional and reduced etch times.

## 9.2 CONCLUSIONS

From the review of the literature, it would seem that several things are achieved by reducing the etching time of phosphoric acid agents applied to human dental enamel. Firstly, the depth of etch is reduced, that is, the absolute loss of surface enamel. Secondly, because of this, the surface enamel remaining (which would have been removed by deeper etching) may have a higher fluoride content. Thirdly, the reported histologic changes produced are as good as, and, according to some authors, better, than those obtained by "normal" etching. Fourthly, bond strengths of resins and orthodontic attachments appear to range from at least adequate to values similar to those achieved with the longer, conventional etching times. Fifthly, retention rates of fissure sealants and bonded brackets using shorter etching times compare favourably to the longer etching times.

In this clinical study there was no statistically significant difference between the two etching times in terms of number of failures, or for mean times at failure.

One can only conclude that reduced etching times produce favourable success rates in orthodontic Begg bracket retention, at least in the short- to medium-term. There remains the assessment of failure rates over a full course of orthodontic treatment; and perhaps using different types of brackets. Likewise, optimal etching times need to be determined: the lower limit of etching times is yet to be established.

It was beyond the scope of this thesis to investigate the depth of etched enamel using a reduced etch time. Perhaps a complementary *in vitro* study could be undertaken to assess the depth of etch (SEM examination) on enamel from extracted premolars from school-children

from a similar sample. Further, debonding and cleanup procedures and their effects on enamel loss and surface damage could be examined for both a reduced and a normal etching time group. An assessment of staining potential of residual resin tags for both etch times may indicate if there is likely to be any difference in stain uptake subsequent to treatment. These suggested studies may yield clinically useful information that would allow the orthodontist to minimise damage to the enamel surface during all stages of bonding and debonding (and perhaps rebonding).

One might suggest, on evidence from both this study and the literature, that orthodontists might cautiously embrace the use of reduced etching times, with the concomitant realisation that success or failure depends on a fairly strict adherence to certain principles of the acid-etch technique. It is hoped that a reduced etching time for orthodontic bonding will become a part of an already well accepted technique; and that further studies will clarify the unanswered questions discussed above, or those that are posed by practitioners at large.

The clinical study undertaken for this thesis has not produced evidence to refute the hypothesis that a reduced etching time should lead to favourable retention rates of Begg brackets during fixed appliance therapy.

## APPENDIX 1

RESULTS

| Tooth type |    | n   | Etch time |        |
|------------|----|-----|-----------|--------|
|            |    |     | 20 sec    | 60 sec |
| Incisors   | 11 | 14  | 7         | 7      |
|            | 21 | 13  | 6*        | 7      |
|            | 31 | 14  | 6         | 8      |
|            | 41 | 14  | 8         | 6      |
|            | 12 | 15  | 8*        | 7      |
|            | 22 | 14  | 7         | 7      |
|            | 32 | 14  | 6*        | 8      |
|            | 42 | 14  | 8         | 6      |
| Canines    | 13 | 14  | 8         | 6      |
|            | 23 | 14  | 6         | 8*     |
|            | 33 | 14  | 6         | 8      |
|            | 43 | 14  | 8         | 6      |
| Premolars  | 14 | 9   | 5         | 4      |
|            | 24 | 9   | 4         | 5*     |
|            | 34 | 11  | 5         | 6      |
|            | 44 | 11  | 6         | 5      |
|            | 15 | 8   | 4         | 4      |
|            | 25 | 8   | 4         | 4      |
|            | 35 | 5   | 3         | 2      |
|            | 45 | 5   | 2         | 3      |
| TOTALS     |    | 234 | 117       | 117    |

\*Denotes one bracket failure in each group so marked.

## APPENDIX 2

### STATISTICS

#### Etching time and failure

(a) Testing of independency of 2 factors, time (20 seconds and 60 seconds) and failure (failed, not failed).

$H_0$ : factors are independent

$r$  = number of row

$H_1$ : factors are dependent

$c$  = number of column

|          |            | <u>Observed frequency</u>         |                                       |                  |
|----------|------------|-----------------------------------|---------------------------------------|------------------|
|          |            | Failed                            | Not failed                            | Totals           |
| Times    | 20 seconds | 3                                 | 114                                   | 117              |
|          | 60 seconds | 2                                 | 115                                   | 117              |
| Totals   |            | 5                                 | 229                                   | 234              |
|          |            | <u>Expected frequency</u>         |                                       |                  |
|          |            | Failed                            | Not failed                            | $n_{i.}$         |
| Times    | 20 seconds | $\frac{5 \cdot (117)}{234} = 2.5$ | $\frac{129 \cdot (117)}{234} = 114.5$ | 117 ( $n_{1.}$ ) |
|          | 60 seconds | $\frac{5 \cdot (117)}{234} = 2.5$ | $\frac{129 \cdot (117)}{234} = 114.5$ | 117 ( $n_{2.}$ ) |
| $n_{.j}$ |            | ( $n_{.1}$ ) 5                    | ( $n_{.2}$ ) 229                      | 234 ( $n$ )      |

Expected:  $\frac{n_{i.} n_{.j}}{n}$

$$\chi^2 = \sum_{i=1}^k \frac{(\text{obs}_i - \text{exp}_i)^2}{\text{exp}_i} \approx \chi^2_{(r-1)(c-1)}$$

$$= \frac{(3-2.5)^2}{2.5} + \frac{(114-114.5)^2}{114.5} + \frac{(2-2.5)^2}{2.5} + \frac{(115-114.5)^2}{114.5}$$

$$= 0.2044$$

$$\chi^2_{1(0.05)} = 3.841$$

Do not reject  $H_0$ , so accept independent model at 5% level of significance.

**(b)** Comparing a 2 binomial population: 20 and 60 seconds.

$$H_0: p_1 = p_2$$

$$H_1: p_1 \neq p_2 \quad (\text{testing the homogeneity of proportion})$$

Effect of 2 times on failure rates:

| Times   | failed<br>$x_i$ | not failed<br>$n_i - x_i = y_i$ | $n_i$ |
|---------|-----------------|---------------------------------|-------|
| 20 secs | 3               | 114                             | 117   |
| 60 secs | 2               | 115                             | 117   |
|         | 5               | 229                             | 234   |

$$p = \frac{\sum_{i=1}^k x_i}{\sum_{i=1}^k n_i} = \frac{5}{234} = 0.02137$$

$k$  = number of independent sets of binomial trials.

Expected frequency table:  $n_{ip}$

| Times   | failed<br>$n_{ip}$ | not failed<br>$n_i - n_{ip}$ |
|---------|--------------------|------------------------------|
| 20 secs | 2.5                | 114.5                        |
| 60 secs | 2.5                | 114.5                        |

$$n_{ip} = 117.(0.02137)$$

$$n_i - n_{ip} = n_i(1-p) = 117.(1-0.02137)$$

$$\chi^2 = \sum_{i=1}^k \left[ \frac{(x_i - n_i p)^2}{n_i p} + \frac{(y_i - n_i (1-p))^2}{n_i - n_i p} \right]$$

$$= \frac{(3-2.5)^2}{2.5} + \frac{(114-114.5)^2}{114.5} + \frac{(2-2.5)^2}{2.5} + \frac{(115-114.5)^2}{114.5}$$

$$= 0.2044$$

$$\chi^2_{(k-1)(0.05)} = \chi^2_{1(0.05)} = 3.841$$

Therefore accept  $H_0$ , that the failure proportions of 20 and 60 seconds are homogeneous.

### **Tooth type and failure**

(a)  $H_0$ : tooth type and time are independent

$H_1$ : tooth type and time are dependent

#### Observed frequency of failure

| Time       | Tooth type |        |          | Totals |
|------------|------------|--------|----------|--------|
|            | Incisor    | Canine | Premolar |        |
| 20 seconds | 3          | 0      | 0        | 3      |
| 60 seconds | 0          | 1      | 1        | 2      |
| Totals     | 3          | 1      | 1        | 5      |

#### Expected frequency

| Time       | Tooth type |        |          | Totals |
|------------|------------|--------|----------|--------|
|            | Incisor    | Canine | Premolar |        |
| 20 seconds | 1.8        | 0.6    | 0.6      | 3      |
| 60 seconds | 1.2        | 0.4    | 0.4      | 2      |
| Totals     | 3          | 1      | 1        | 5      |

$$\chi^2 = \sum \frac{(\text{observed} - \text{expected})^2}{\text{observed}}$$

$$\begin{aligned} \chi^2 &= \frac{(3-1.8)^2}{1.8} + \frac{(0-0.6)^2}{0.6} + \frac{(0-0.6)^2}{0.6} + \frac{(0-1.2)^2}{1.2} + \frac{(1-0.4)^2}{0.4} + \frac{(1-0.4)^2}{0.4} \\ &= 5 \end{aligned}$$

However,

$$\chi^2_{(3-1)(2-1)(0.10)} = \chi^2_{2(0.10)} = 4.605$$

Therefore, the hypothesis  $H_0$  is rejected; the dependency between time and tooth type affects failure.

**(b)** The statistical analysis chi squared can also be used to test the homogeneity of proportion:

$$H_0: p_1 = p_2 = p_3$$

$$H_1: p_1 \neq p_2 \neq p_3 \quad p_1 = 3/5 \quad p_2 = 1/5 \quad p_3 = 1/5$$

The alternative hypothesis is accepted (proportions of failure according to tooth type are not homogeneous).

### Upper versus lower teeth and failure

$$H_0: p_1 = p_2 \text{ (proportion of failure of upper and lower teeth are equal)}$$

$$H_1: p_1 \neq p_2 \text{ (proportion of failure of upper and lower teeth are not equal)}$$

| Time       | <u>Observed failures</u> |       | Totals |
|------------|--------------------------|-------|--------|
|            | Upper                    | Lower |        |
| 20 seconds | 2                        | 1     | 3      |
| 60 seconds | 2                        | 0     | 2      |
| Totals     | 4                        | 1     | 5      |

| Time       | <u>Expected failures</u> |       | Totals |
|------------|--------------------------|-------|--------|
|            | Upper                    | Lower |        |
| 20 seconds | 2.4                      | 0.6   | 3      |
| 60 seconds | 1.6                      | 0.4   | 2      |
| Totals     | 4                        | 1     | 5      |

$$\chi^2 = \frac{(2-2.4)^2}{2.4} + \frac{(1-0.6)^2}{0.6} + \frac{(2-1.6)^2}{1.6} + \frac{(0-0.4)^2}{0.4}$$

$$= 0.8333$$

$$\chi^2_{1(0.05)} = 3.841$$

Therefore, accept  $H_0$ , that proportion of upper and lower tooth failures are equal.

### Right versus left teeth and failure

$H_0: p_1 = p_2$  (proportion of failure of right and left teeth are equal)

$H_1: p_1 \neq p_2$  (proportion of failure of right and left teeth are not equal)

| Time       | <u>Observed failures</u> |       | Totals |
|------------|--------------------------|-------|--------|
|            | Left                     | Right |        |
| 20 seconds | 2                        | 1     | 3      |
| 60 seconds | 2                        | 0     | 2      |
| Totals     | 4                        | 1     | 5      |

| Time       | <u>Expected failures</u> |       | Totals |
|------------|--------------------------|-------|--------|
|            | Left                     | Right |        |
| 20 seconds | 2.4                      | 0.6   | 3      |
| 60 seconds | 1.6                      | 0.4   | 2      |
| Totals     | 4                        | 1     | 5      |

$$\chi^2 = \frac{(2-2.4)^2}{2.4} + \frac{(1-0.6)^2}{0.6} + \frac{(2-1.6)^2}{1.6} + \frac{(0-0.4)^2}{0.4}$$

$$= 0.8333$$

$$\chi^2_{1(0.05)} = 3.841$$

Therefore, accept  $H_0$ , proportion is not significant, left and right side teeth are homogeneous.

### Etching time and mean time of failure

Analysis using a 2 sample unpaired t-test to compare mean times of failure for 20 versus 60 seconds.

$$H_0: \sigma_1^2 = \sigma_2^2$$

$$H_1: \sigma_1^2 \neq \sigma_2^2$$

| n                         | 1 = 20 seconds | 2 = 60 seconds |
|---------------------------|----------------|----------------|
| 1                         | 1              | 84             |
| 2                         | 61             | 137            |
| 3                         | 91             |                |
| $\bar{X}$                 | 51             | 110.5          |
| $S^2$                     | 2100           | 1404.5         |
| $F = \frac{2100}{1404.5}$ | = 1.495        |                |

$$F_{\text{critical}} = F_{2,1} (0.05) = 200$$

Accept  $H_0$ , variances ( $\sigma^2$ ) are equal, so they can be pooled:

$$\begin{aligned} S_p^2 &= \frac{(n_1-1)S_1^2 + (n_2-1)S_2^2}{(n_1-1) + (n_2-1)} \\ &= \frac{(3-1)2100 + (2-1)1404.5}{(3-1) + (2-1)} \\ &= 1868.1667 \end{aligned}$$

$H_0: \mu_1 = \mu_2$  (mean times of failure for 20 and 60 seconds are equal)

$H_1: \mu_1 < \mu_2$  (mean times of failure for 20 seconds less than 60 seconds)

$$\begin{aligned} t &= \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{S_p^2(1/n_1 + 1/n_2)}} \\ &= \frac{51 - 110.5}{\sqrt{1868.1667(1/3 + 1/2)}} \\ &= -1.508 \end{aligned}$$

$$t_{(n_1-1)+(n_2-1)}(0.05) = t_3(0.05) = 2.353$$

$H_0$  accepted as  $-1.508 > -2.353$ . therefore, mean times of failure for 20 and 60 seconds are equal.

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