TITLE

A LABORATORY EVALUATION OF CONVENTIONAL CHEMICALLY ACTIVATED AND MICROFILLED LIGHT ACTIVATED COMPOSITE RESTORATIVE RESINS.

BY

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OBJECTIVES.

To evaluate,in vitro,the tensile bond strengths of some conventional and light cured resins, to study the effects of etchants on polished and ground enamel using the scanning electron microscope, and to examine the resin/etched enamel interface by scanning electron microscopy.

DEDICATION.

To my wife Shirley and the boys Dale, Robert and Michael for their love and understanding throughout this study.

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INTRODUCTION.

The universal acceptance of the composite resins and their clinical success have resulted in a major emphasis of materials research in this area.

The composite resins are currently the most common restorative material utilized in anterior restorations. They are esthetic in appearance and may be made to adhere to the enamel of the teeth. The technique of acid-etching the enamel prior to placement of the composite resin has been found to greatly increase the retentive bond strength of the resin restoration which bonds mechanically with enamel and which improves the marginal seal.

The interface between a restorative material and enamel is of crucial importance in the preservation of a complete seal of the margin of the restoration to prevent microleakage. Thus an ideal restoration should have a bonding capacity to enamel and be able to prevent penetration of fluids and bacteria at the restoration/tooth interface.

Bonding agents, low viscosity resins, are utilized by some clinicians in the belief that a lower viscosity material will more readily penetrate the pores created by acid-etching the enamel margins of a cavity preperation, resulting in improved bonding. This is controversial and research studies report conflicting results.

The objective of this study is to evaluate, in vitro, the effects of etchants on polished and ground enamel using scanning electron microscopy (S.E.M.) and to examine the resin/etched enamel interface by S.E.M., and to study the tensile bond strengths of some conventional and light cured, marofilled and microfilled composite resins.

REVIEW OF THE LITERATURE

In the review of the literature it is apparent that the following topics are of importance to this study:

1. Background of composite resins
2. Acid etch studies
3. Tensile bond strength studies
4. Scanning electron microscope studies
5. Microleakage studies.

BACKGROUND of COMPOSITE RESINS.

Some of the newest and most rapidly evolving classes of dental materials are the restorative resins. Since their introduction at the close of World War II, these materials have become the most commonly used tooth coloured restorative material, apart from dental porcelain. Included in this class of materials are the unfilled resins, the composite or filled resins, and pit and fissure sealants.

Two important events in the development of the resins have had a major effect on restorative dentistry. Bounocore (1955) developed and showed that the acid etch technique removes surface debris, etches and roughens the enamel, enlarges the contact area and increases the wettability of the enamel surface. Bowen (1962) developed the aromatic dimethacrylates (unfilled resins) which produced harder and

less toxic resins, and which showed less exothermic heat production and lower polymerization shrinkage than the methyl methacrylate resins that were currently in use. The unfilled resins were the fore-runners of composite resins. The main difference between the unfilled and composite resins is the addition of inorganic filler particles to the resin. The addition of filler particles to the resin results in a lower coefficient of thermal expansion, higher compressive and tensile strengths, greater hardness, lower water sorption and increased resistance to abrasion (Bounocore, 1968).

The filled resins consist of an organic matrix, commonly the BIS-GMA resin system, reinforced with a fine dispersion of an inorganic filler such as quartz, glass or lithium-aluminium silicate. The filler may be coupled chemically to the organic matrix by a silane coating on the filler particles.

There are two main types of composite resins based upon the method of polymerization : chemically cured and light cured composites. The setting reaction, involving polymerization of the methacrylate or acrylate groups of the resin, may be activated in one of two ways. For two component materials activation of the polymerization process is accomplished when the peroxide initiater present in one component reacts with a chemical activator, normally a tertiary amine, present in the other component. This process is often referred to as chemical activation of the polymerization process.

For single paste materials, activation of a light sensitive initiator system (camphoroquinone) is accomplished by exposure to high intensity radiation of the correct wavelength. This process is called light activation or photocuring of the composite resin.

Other variables are the type, amount and particle size of the filler. In the development of the resins the macrofilled type was the first to be used. These early composite resins consisted of resins which had filler particles in the 15 um size range. Some examples of these early macrofilled resins are Adaptic (Johnson and Johnson, East Windsor, NJ.) and Concise (3M Company, St Paul, MN.). These resins could not be polished to a high lustre and tended to become rough with resultant discolouration and plaque accumulation. These resins were also chemically cured and thus allowed the incorporation of air and porosities when mixed as a two paste system. The amine accelerator in the material also tended to discolour the body of the restoration after a few years.

Microfilled resins were later developed to improve the earlier composite resins. They contain silicon dioxide filler particles which are in the submicron range and because of this their polishability is improved. However they are more brittle than the macrofilled resins due to their increased filler content. Many of the microfilled resins are light cured (e.g. Silux, 3M Company, St. Paul, MN.)and this is a distinct advantage over the chemically cured microfilled resins (e.g. Silar, 3M Company, StPaul, MN.). The problems of porosity which may occur during the

mixing of these resins is lessened when a light cured material is used as a one paste system of dispensing (Joseph and Cohen 1986). Amine accelerators are not present in light cured resins and subsequent discolouration of the body of the restoration is less likely.

There is a trend towards the use of blends of fillers having different particle size in order to maximise the filler content (McCabe 1984). Hybrid resins were developed to attempt to bridge the gap between the microfilled and macrofilled resins. These resins have a filler particle size ranging between 1 and 7 um and because of this can be relatively well polished. They are intended to be used in situations of high stress. Pillar, Smith and Maric (1984) showed that microfilled materials had lower wear rates when compared to conventional resins in the class 1 and class 2 situations but that they are more brittle and are prone to fracture when compared to the macrofilled composite resins.

ACID-ETCH STUDIES.

To obtain increased adhesion of resin to enamel Buonocore (1955) etched the enamel with 85% phosphoric acid. This improved the bonding to enamel of the acrylic restorative materials that he was using and paved the way for the mechanical retention of composite resins as we know them today. Lack of adhesion between dental restorative materials and tooth structure results in percolation and microleakage with the possibility of pulpal irritation and recurrent caries. A bond between the restoration and the enamel would decrease the percolation microleakage which may occure.

Initially the acid was presented for use as a clear liquid in small bottles. Application of the acid to enamel was achieved by using a small sponge or brush to transfer the acid to the tooth. Later the acid was marketed as a coloured gel, in squeeze bottles and applied with a brush or non-metallic instrument. Acid gel dispensed in syringes is now available to make the application more precise than before.

A fundamental requirement for good bonding between a dental resin and enamel surface is the intimate interfacial contact between the resin and the enamel. For this to occur the resin must flow over the etched surface and penetrate into the microspaces produced in the surface by

the etchant (Retief 1978). The extent to which a liquid will wet a surface depends upon the viscosity of the liquid, the topography of the surface, the free energy of the surface and the contact angle formed between the surface of the liquid drop and the surface of the adherend upon which it is resting (Zisman 1963). Etching increases the wettability of the enamel surface (Retief 1973).

By combining the acid etch technique with conventional cavity preparation, the common microleakage problems can be greatly reduced (Torney, Denehy and Teixeira 1977). With acid etching, the resin can flow more easily into the retentive pits of the enamel prior to polymerization due to the decrease in the contact angle and greater wetting action. The penetration results in the encapsulation by the resin of the crystallite component of the enamel, thus providing a mechanical bond.

The condition of the surface to be etched is important. Brannstrom and Nordenvall (1977) showed that the ground enamel surface resulted in a more irregular surface compared to the enamel surface which has not been ground. Similar observations of uncoated (no gold splutter coating) human enamel have been made by Risnes and Stolen (1981).

Various acids have been used in the past to produce etched enamel as shown by Retief, Bischoff and van der Merwe (1976). They stated that the use of 10% pyruvic acid applied for 90 seconds to enamel surfaces did not adversely affect the tensile bond strength of Concise Brand Composite with Concise Enamel Bond System when compared to enamel surfaces etched with 37% phosphoric acid for a similar period of time.

The effect of the concentrations of phosphoric acid were investigated by Mansson-Rahemtulla, Retief & Jamison (1984) and they concluded that the total amount of calcium dissolved increased with greater concentration of phosphoric acid and reached a maximum with 40% phosphoric acid. A further increase in acid concentration resulted in a decrease in the total calcium dissolved.

Gwinnett (1981) showed that 37% - 50% phosphoric acid seems to be the optimum concentration. He also showed that rubbing the acid on the enamel instead of dabbing it on decreased the surface available for bonding by burnishing and fracturing friable rods of enamel with their resultant loss of availability for encapsulation by the resin.

Different concentrations of phosphoric acid produced similar etch patterns as shown by Denys and Retief (1982). They also felt that it was not possible to define the optimal concentration of phosphoric acid as an etchant by scanning electron microscopy alone.

A desirable property of any low viscosity bonding resin is to be able to "wet" the surface of enamel entirely and easily. It is necessary for an adhesive to "wet" the surface of the enamel to establish good adhesion. If a liquid exhibits a small contact angle, it is said to have better wetting than one exhibiting a larger contact angle. After phosphoric acid is applied to the enamel surface the wettability is increased.

Retief (1974) studied the effect on enamel of three etchants: 50% phosphoric acid for 1 minute, 50% phosphoric

acid plus 7% zinc-oxide for 1 minute and 50% citric acid for 2 minutes. All three solutions reduced the contact angle, but the 50% phosphoric acid plus zinc-oxide reduced it the most. He found that the epoxy resin used in this study had the highest tensile bond strength when using this solution for the etching. He therefore concluded that the high bond strength may be caused by the small contact angle of the resin on that surface. The wettability of the conditioned surface was found to determine the extent of penetration into the pores. In this study Retief found that attenuated phosphoric acid produced the greatest reduction in contact angles of resins and the most marked increase in bond strengths.

One of the very first clinical applications of acid etched bonding was in sealing pits and fissures for the prevention of caries. Even though the penetration coefficient into the etched prisms of 3 resins varied, tensile bond strengths did not differ significantly (Retief and Mallory 1981).

Another major application of the acid etch technique is in the attachment of orthodontic appliances. In an in vitro study Retief and Dreyer (1967) attached stainless steel orthodontic attachments to extracted teeth using epoxy resin. The teeth had previously been etched with 50% phosphoric acid for 1 minute. The force necessary to break the bond was greater than the force which would be exerted on the attachment during orthodontic treatment.

From the above it is obvious that etching enamel surfaces ensures a better bond of resins to enamel and thus the acid etching of enamel of a cavity preparation before placement of a composite retoration has become a common proceedure in restorative dentistry.

MICROLEAKAGE STUDIES

Microleakage is dependant upon the bond of the resin to enamel and the subsequent contraction and expansion of the resin. The bond to enamel may be influenced by various factors: whether the enamel is polished or ground, whether the enamel is correctly etched and whether the enamel is rendered wettable and if good tag formation occurs. Polymerization contraction occurs when a composite resin cures and this may encourage microleakage if of sufficient magnitude. After a period of time water sorbsion occurs and the resin expands. To create a bond between a restoration and tooth structure is an ideal property of a restorative material. A material must be found that will bond to both enamel and dentine and/or cementum and so prevent microleakage.

However, this is not yet the case and "microleakage" is still commonly found at the enamel margins, and even more severely at the dentine interface, or where the cavity margin is on dentine.

In a study by Retief, Rutland and Jamison (1982) they found that microleakage was greater gingivally than occlusally in class V cavities. They also found that no significant difference in microleakage existed between conventional and microfilled resin materials.

In another study on microleakage, Retief, Woods and Jamison (1982) showed again that the microleakage at the cervical margins of class V restorations was more severe

than at the incisal margins. They also could not support the use of low viscosity bonding resin applied to the walls of the preparation prior to the placement of a resin, based upon the findings of this study.

A later study by Retief and Jamison (1982) showed that class V restorations frequently showed microleakage at the gingival margin and questioned if this could be due to the prismless enamel (giving a class 3 etch pattern). They showed that bevelling the cavosurface at this margin had some effect in reducing the microleakage.

However, Retief, Woods, Jamison and Denys (1982) suggest that the variations in prism orientation in parazones and diazones of Hunter-Schreger bands is the main reason why no significant difference in microleakage patterns were observed in restorations placed in butt and bevelled preparations.

This may be the reason why Hembree (1984) reported that changes in cavity design did not alter the leakage pattern. Hembree compared the microleakage of two microfilled composite resins and a conventional composite resin using three different cavosurface angles. It was thought that by increasing the surface area available for etching, the microleakage of the microfilled composite resin might be reduced. It is possible therefore to postulate that the Hunter-Schriger bands, being wave like in form, are responsible for the uniformity in leakage patterns.

In a study by Arends, van Groeninger and Schutthoff (1984) it was shown that microleakage of all the resins they

tested decreased with time and became negligible after 1 month. This suggests that water uptake and subsequent expansion of the composite resin accounts for the decrease in leakage.

Asmussen (1985) also noted that if the contraction gap was small enough and hygroscopic expansion large enough the marginal gap will, as a consequence, close in two days. This is conditional on the gap not being filled by polishing debris. Delayed finishing of the resin allows for the expansion of the resin by water sorbsion to counteract the stresses caused by polymerization shrinkage. The higher the filler content of the resin the less the hygroscopics expansion will be. Hygroscopic expansion of a certain magnitude is a desirable property in a composite resin. Microfilled resins contain less filler by weight (30-60wt%) than macrofilled resins (75-80wt%), and therefore have more hygroscopic expansion (McCabe 1984). Asmussen (1985) claims that the greater percentage of BIS GMA in conventional filled resins the smaller the contraction gaps and the less the microleakage.

Welsh and Hembree (1985) found that the bond to dentin and cementum (using dentine bonding agents) still shows microleakage even though material manufacturers claim otherwise. A glass ionomer control by way of contrast showed no significant leakage at any time. However, Hembree (1984) suggested that the conventionally filled resins show less microleakage than microfilled resins. Retief, Rutland and Jamison (1982) were not in agreement with this study. In a previous study they concluded that microleakage of

preparations restored with a conventional composite resin and a microfilled composite resin was not significantly different.

Gross, Retief and Bradley (1985) showed that in vitro, posterior composite resins exihibited microleakage. They evaluated the microleakage of MOD preparations in extracted human mandibular molar teeth restored with a chemically activated posterior composite resin (P10) and a visible light cured composite resin (P30) used in conjuction with a low viscosity bonding resin (Enamel Bond) and a dentine bonding agent (Scotchbond) respectively. The restored teeth were subjected to 100 complete temperature cycles in an aqueous calcium chloride solution between 5 and 55 degrees centigrade and the microleakage evaluated from autoradiographs. They demonstrated that P10, and P30, posterior composite resins (3M Dental Company),both showed microleakage but was less when Scotchbond (dentine bonding agent) was used. The penetration of various dental materials into etched enamel surfaces has been observed by several workers. Gwinnett and Buonocore (1965) were among the first to describe this phenomenon. They found that tags of methyl-2-cyanoacrylate of approximately 10 um in length penetrated into enamel surfaces etched with a phosphoric acid solution.

Jorgensen and Shimokobe (1975) reported maximum tag lengths of Adaptic, Concise & Nuva Seal to be between 22-26 um. These tags did not contain filler particles and they

concluded that the penetration of the material is dependant on the viscosity of the resin component of the composite. They did not believe that an intermediate film of low viscosity bonding resin was necessary for good bonding.

Pahlavan, Dennison & Charbeneau (1976) showed by means of scanning electron and polorizing light microscopy, that the depth of penetration of all materials tested (Nuva Seal, Concise Enamel Bond and Restodent) into etched human enamel to be the same, at +-7 um. They found that the viscosity of the resin did not change the penetration depths nor whether the enamel was previously ground or not. This observation showed that low viscosity resin was not necessary to achieve adequate tag lengths and the possibility of a good bond forming with a highly filled resin.

Other researchers have claimed that the viscosity of the resin applied to the etched surface is related to the penetration of the resin into the tags. It was shown in a study by Dogon (1976) that the frequency as well as the lengths of the tags increased as the viscosity of the resin decreased. Dogon also showed that the greatest penetration of tags into the enamel was achieved with the intermediary low viscosity bonding resins.

Later Prevast, Fuller and Peterson (1984) also showed that there was no difference in tag length or regularity for Concise, Adaptic, Concise Enamel Bond, Adaptic Bonding agent, Silar and Isopast. It appears therefore that the successful adaption of composite resins is not dependant on the use of an intermediary unfilled resin as a bonding agent (Low, Lee and Von Fraunhofer 1978).

SCANNING ELECTRON MICROSCOPE STUDIES

The scanning electron microscope has been utilized in this study because it allows for the examination of the entire labial surface of a tooth with both low and very high magnifications, which cannot be examined with light or transmission electron microscopes. The scanning electron microscope also allows for an increased depth of focus by 300 times when compared to conventional light instruments.

The morphology of the interface between "adhesive" resins and the etched enamel shows the formation of tags when examined under scanning electron microscopy. These tags vary in length from 5 to 50 um. Macrofilled resins tend to exhibit longer tags and the microfilled resins shorter tags. The diameter of the tags can be as wide as 5 um, which then narrows as the tags lengthen.

Silverman, Saxton, Dogon and Fejerskov (1975) reported that variations in the pattern of acid etched human dental enamel examined by S.E.M. were noticeable. They claimed that there is no one specific etch pattern in human enamel and highlighted the variations that can occur in enamel not only from tooth to tooth or surface to surface but also from site to site on a single tooth. They showed that extensive loss of the prism core material was the most common feature. This is called a Type I etch pattern. Less frequently the core material remained but the periphery was lost. This is a Type II etch pattern. A Type III etch pattern occurs on enamel surfaces where the enamel prisms do not reach the

surface and results in an amorphous indefinate etch pattern. Crystallite orientation was suggested to be significant in causing the various etch patterns.

These different etch patterns were thought to be attributed to intrinsic differences in enamel, possibly explained on the basis of enamel solubility. The demineralization was limited to the outer few microns of enamel. Poole and Johnson (1972) used the scanning electron microscope to examine the etch pattern of surfaces that were not parallel to the prisms. A troughlike surface was produced. They believed that this confirmed a differential dissolution of enamel which was associated with different structures. Silverman et.al. (1975) were not in agreement with the Poole and Johnson (1972) findings.

Lees, Trombly, Skobe, Gapiety and Trull (1979) noted that local variations of enamel can account for the observation by Silverman et.al. (1975) that more than one type of etch pattern can be obtained on a single enamel specimen with a single reagent - thus the concept was proposed that the etching pattern is controlled by the residual organic content of dental enamel.

Similar observations of the variability of enamel topography had been observed by Tyler (1976) as shown by the coexistance of prism cleft formation, the loss of prism cores and the change of structure associated with surface enamel which may contribute to etch pattern irregularity.

Gwinnett (1981) showed that etching had a preferential effect on the enamel prisms resulting in cores of resin being formed, while the preference of dissolving prism peripheries produced craters of resin. The exact boundaries of the resin tags were difficult to distinguish except where they enveloped prism cores. Etching resulted in an increased surface area which leads to a greater mechanical retention of the resin. Voss and Charbeneau (1979) in a scanning electron microscope study demonstrated tag projections of resin into etched enamel surface when cut both transversely and longitudinally. Adaptic with bonding agent, Simulate with primer and Simulate without primer were applied to etched enamel surfaces. The resin projections into the etched enamel rod ends at the enamel surface were 5mu to 10mu in length. No observable differences in the lengths and pattern of tags were noticed amongst the three bonded materials.

Pahlavan, Dennison and Charbeneau (1976) showed that under scanning electron and polorizing light microscopy, the penetration of the three materials representing unfilled and filled resins into etched enamel to be the same. Differences in viscosity did not appear to change the penetration depths of the resin.

The findings of the above study is supported by Prevost, Fuller and Peterson (1984) who showed no difference in tag lengths and regularity using 6 different composite resins representing conventional composites, intermediate

resins and microfilled resins. They concluded that the need for an intermediate resin might depend on the composite resin's ability to wet enamel.

TENSILE BOND SRENGTH STUDIES.

Bounocore (1955) was the first to introduce the concept of mechanically bonding to enamel and reported that bonding of acrylic restorative materials to enamel could be substantially increased by etching the enamel surface with 85% orthophosphoric acid.

Retief and Dreyer (1967) showed the strength of this bond when used for orthodontic therapy to be adequate to maintain the orthodontic brackets which were etched and bonded to the enamel of the teeth.

Retief (1978) stated that mechanical bonding is the major factor responsible for the increase bond strength of dental resins to etched enamel surfaces and for decreased microleakage at the tooth resin interface.

Lambrechts (1983) reported that values for tensile strength of conventional composite resins are greater than microfilled resins. Tensile strengths of conventional composites are 35-55 MPa ; microfilled resins are 30-40MPa; amalgam is 54MPa; enamel is 10MPa; and dentine is 51MPa (megapascal = N.mm2).

Munechika, Suzuki, Nishiyama, Ohashi and Horie (1984) showed that the tensile bond strengths of resin bonded to enamel were increased in transversly cut sections of enamel as opposed to longitudinal sections which exhibited parallel

rods. Tensile bond strength of 18-19MPa in transverse section as opposed to 10-11MPa in longitudinal section were obtained. They concluded that the tensile bond strength was effected by the nature of the crystal planes in the enamel.

Retief (1974) in his study with epoxy resin examined the failure of the dental adhesive-etched enamel interface and stated that epoxy tags exceeding 50 um in length were observed extending into the enamel. These tags produced a saw tooth appearance when torn apart, with some of the resin still in the enamel and some enamel embedded in the resin. No interfacial fracture occured strictly in either material, but occures in both the enamel and the composite resin. However the bond strength of the resin at the interfacial zone was usually sufficient to withstand the forces applied to it, and if a fracture did occure, it usually appeared in the body of the composite resin close to the interfacial zone.

Zidan, Asmussen and Jorgensen (1982) stated that except for rare cases of enamel facture, the fracture always occured in the restorative resin when the tensile bond strength tests were being applied. The fracture propagates either through the bulk of the material, or through the material very close to the interface. The residual film on enamel is so thin that only the penetrating tags can be discerned by the microscope study. This study reported no correlation between the bond strength and type of failure. This contrasts with work by Retief (1974) and Mitchem and Turner (1974) who found a progressive decrease in bond strength as the area of apparent interfacial fracture

increased.

Higher bond strengths have been reported following a 0.1mm reduction of enamel surface as shown by Schneider, Messler and Douglas (1981). Mechanical surface reduction was associated with increased residual composite at the bond site after debonding.

In another study by Aker, Aker and Sorensen (1979) claims were made that the surfaces prepared with some reduction of enamel with a diamond bur gave higher retention values than unprepared enamel or carbide bur prepared surfaces.

Kemper and Kilian (1976) described a testing system for bond strength determination which is now widely used. The commonly used apparatus for testing bond strengths is the Instron machine which was reported on, amoungst others, by Low, Davies and von Fraunhofer (1975).

The tensile bond strength of macrofilled and microfilled resins show some variation according to Zidan, Asmussen and Jorgensen (1980). They showed that of the 5 composites they tested (two microfilled and three macrofilled resins) the microfilled showed a lower bond strenth. This probably reflects the brittle nature of the microfilled resin compared to the macrofilled resin which is more "plastic".

This concept is supported by a study by Short, Hembree and McKnight (1976) who tested the bond strengths of resins to etched enamel and found that the highest values were with the macrofilled resins (Concise and Adaptic)

Researchers Soetopo, Beech and Hardwick (1978) claimed

that the tensile bond strength to etched enamel is dependant upon the concentration of phosphoric acid, used as an etchant. This is contradicted by Gottlieb, Retief and Jamison (1982) who showed that the tensile bond strengths of a resin to enamel surface etched with 10% to 60% phosphoric acid were not significantly different. Neither the depth of etch nor the amount of superficial enamel removed during the etching proceedure is dependent on the acid concentration or the duration of etch. The depth of etch ranged from 10 to 12 um with 10 to 45w/w% phosphoric acid, dropped to 7 um with 50w/w% phosphoric acid and decreased to 3.5 um with 60w/w% phosphoric acid. All these etching procedures were over a 1 minute time period. Etching with 70% phosphoric acid produced a significant decrease in the bond strengths.

Bates, Retief, Jamison and Denys (1982) also showed that moderate variations of the method of application, etch times and wash times did little to effect the bond strength to enamel. The conclusions were that in an in-vitro study, the method of acid application by dabbing, rubbing or no agitation had no significant effect on the tensile bond strength of a composite resin to etched enamel. The tensile bond strength to enamel surfaces etched for 30, 60 and 120 seconds were not signicantly different. A 5 second wash after acid etching was adequate to maintain bond strength.

Bowen, Nemoto and Rapson (1983) noted that the strength of the adhesive bond should develop more rapidily than the contraction tensile force in the material during its

polymerization. These tensile polymerization stresses may be large enough to open up lamellae, cracks or cause surface crazing. They concluded that beveled or chamfered margins which are acid etched are recommended to distribute the shrinkage forces over a large area of substrate enamel.

Davidson, de Gee and Feilzer (1984) showed that in a single dimensional study, the adhesion of the composite/dentin bond survived the contraction forces, but in a three dimensional cavity the contraction values can exceed the bond strengths. This is particularly important when the bulk of material to be cured is large. Polymerization contraction stresses in large restorations can produce considerable internal stresses upon the tooth to which it is bonded.

Improved adhesion of acrylic restorative materials to enamel by precoating with monomer containing vinyl benzyl phosphoric acid has been demonstrated by Farley, Jones and Anbar (1977). This is attributed to the interacton between calcium ions in the enamel surface and phosphanate groups in the vinyl benzyl phosphoric acid.

Maijer and Smith (1977) also showed that tensile bond strengths to enamel when using polyacrylic acid as an etchant were equal or greater than bond strengths obtained using 37% phosphoric acid as the etchant. Crystals which form at the interface can be used as mechanisms for attachment.

The use of low viscosity bonding resin has been in question for some time. It was shown by Raadal (1978) that adherance of undiluted composite to enamel surfaces was not

improved by a layer of intermediate low viscosity resin. This has also been reported by Retief and Woods (1982).

However, Prevost, Fuller and Peterson (1982) concluded that it was better to use a low viscosity bonding resin due to the risk of not obtaining adequate wetting when using composite resin only.

Ortiz, Phillips, Swartz and Osborne (1979) evaluated the efficacy of the bonding agents supplied with 3 different resins to human teeth. They found that little or no difference existed in the tensile bond strengths and microleakage when the restorations were placed with or without use of the respective bonding agents.

Jassem, Retief and Jamison (1981) reported on the tensile and shear strengths of bonded and rebonded orthodontic attachments. They stated that the use of low viscosity bonding resin had no significant different effect on the tensile and shear bond and rebond strengths. Retief and Woods (1982) investigated the need for a low viscosity bonding resin and found that it did not increase the tensile bond strength. They questioned the continued use of the low viscosity resin in clinical dentistry.

Pretreatment of enamel with various chemicals may effect the bond strengths by altering the effects of the etchants. Several investigators have shown that the etching effect of phosphoric acid on the enamel surfaces pretreated with topical flouride agents was impeded, causing reduced bond strengths of the resins. However Bryant, Retief, Bradley and Denys (1985) showed that the application of

topical flouride agents to enamel. 7 days prior to the bonding of orthodontic attachments did not have an adverse effect on the bond strengths. It may be contraindicated to apply flouride agents to the enamel of teeth immediately prior to acid etching and bonding.

METHODS OF EVALUATION.

TENSILE BOND TESTS.

REASON FOR THIS TYPE OF EVALUATION:

1) To demonstrate the differences, if any, between the microfilled and macrofilled composite resins tested.

2) To record the strength of the bond to enamel.

3) To ascertain the site of fracture of the materials.

4) To correlate the tensile bond strengths of the materials with their filler component.

SCANNING ELECTRON EXAMINATION.

REASON FOR THIS TYPE OF EVALUATION:

1) To demonstrate the result of acid etching on enamel and the production of etch patterns.

2) To demonstrate differences, if any, in the resultant etch pattern produced by the different etchants supplied.

3) To demonstrate and measure the tag lengths produced when low viscosity bonding resins are used in conjuction with the composite materials.

METHODS AND MATERIALS.

PART ONE ---TENSILE BOND STRENGTH STUDIES.

MATERIALS:

1) 60 extracted human permanent central incisor or canine teeth.

2) 3 M products which consisted of :

a) Concise --- 3M Dental Products, St Paul, MN55144 U.S.A. Batch No. 1925S.

b) Silux --- 3M Dental Products, St Paul, MN55144 U.S.A. Batch No. 5501S.

3) Johnson and Johnson Products which consisted of :

a) Adaptic --- J&J Dental Products Company, East Windsor, NJ 08520 U.S.A. Batch No. OHO14 for catalyst paste and OFO14 for the universal paste. Components of the kit consisted of a base and a catalyst paste and a mixing spatula.

The J&J Bonding agent, which consisted of a liquid etchant and a two part low viscosity bonding resin, a brush handle and disposible brushes, was used with Adaptic.

b) Certain --- J&J Dental Products Company, East Windsor, NJ 0852O, U.S.A. Batch numbers for the light curing resins presented in light syringes were 401106, 1H1991 and 4F1416. On the recommendation of the manufacturer the light curing low viscosity bonding resin Aurafil (made by J&J) was used in conjuction with Certain. This was presented as a single part light curing resin,

liquid etching agent, brush handle and disposable brushes. Batch No. was 302099

4) 60 Brass cups. (Ref. P31)

5) Fast curing acrylic resin ("Fastcure"). Manufacturer was Kerr Sybron, Romulus, Michigan U.S.A. Batch no. 1358.

6) 60 Perspex tooth stubs, machined at the University of the Western Cape, South Africa.

7) Instron Testing machine. (Ref. P31-32).

8) Aligning blocks. (Ref. P33).

9) Grinding and Polishing machine (" Masterserv") with 200 grit and 600 grit paper and its aligning block. Manufactured by Metallurgical Services, Surrey, England. (Ref. P32).

10) Incubator. Manufactured by Laboratory Scientific Equipment, Johannesburg, South Africa.

11) White light scource. Model "Luxor" 4000. Manufactured by Imperial Chemical Industries (I.C.I.), Pharmaceutical Division, Macclesfield, Cheshire, England. (Ref. P34).

FIGURE NO:1 Brass cup with perspex stub and prepared tooth.

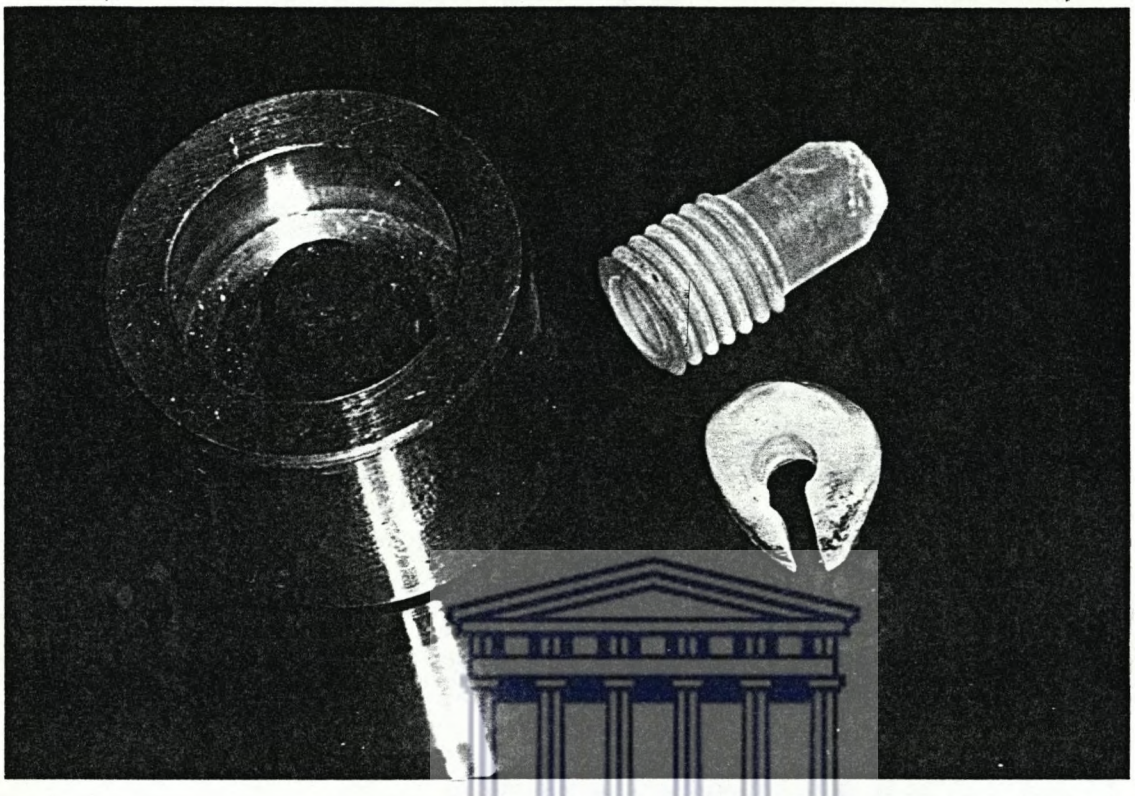


FIGURE NO:2

Instron Machine.

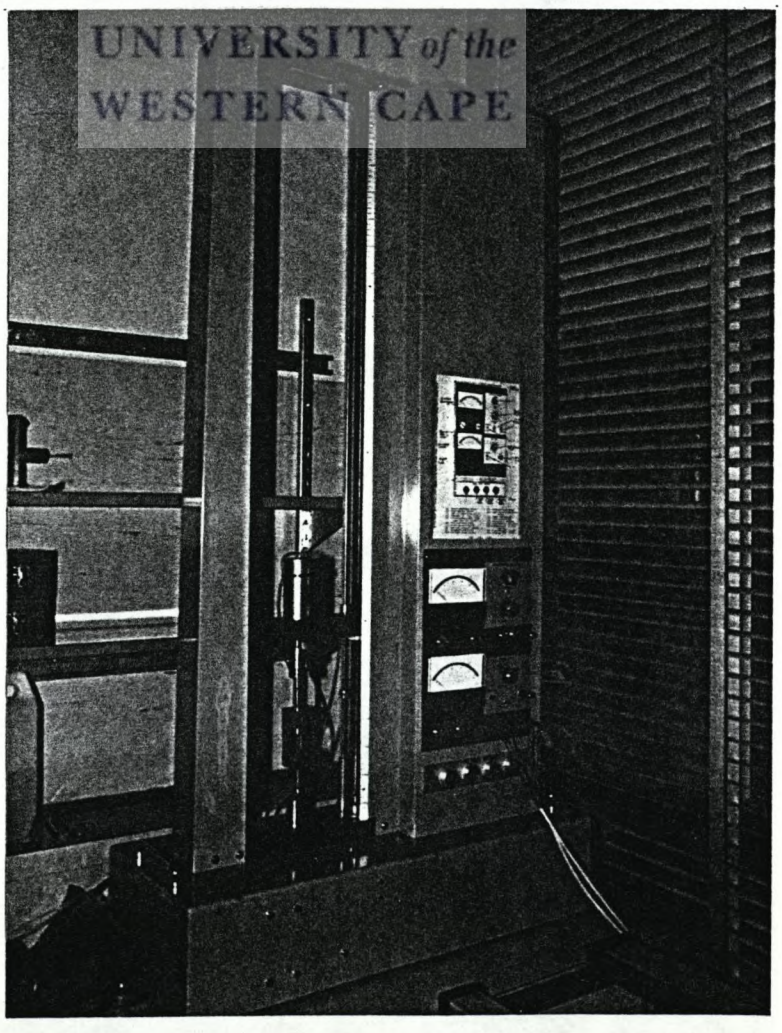


FIGURE NO.3:Instron graph plotter.

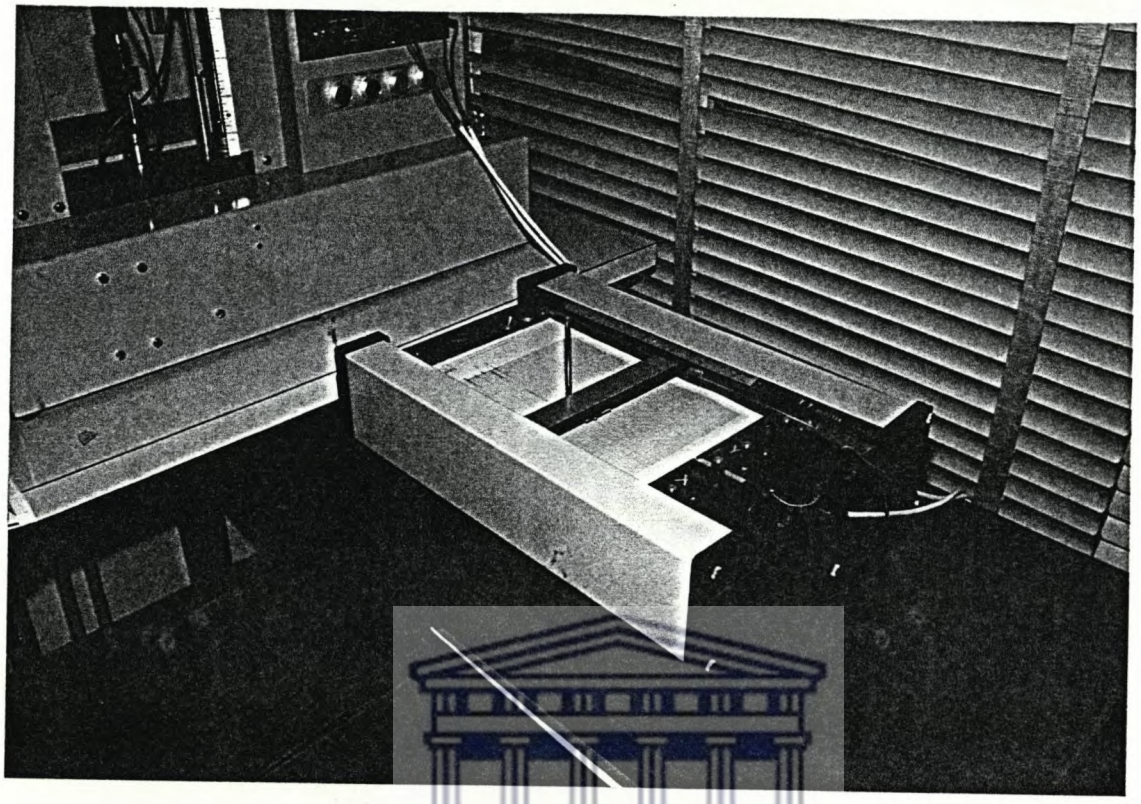


FIGURE NO.4: Grinding machine.

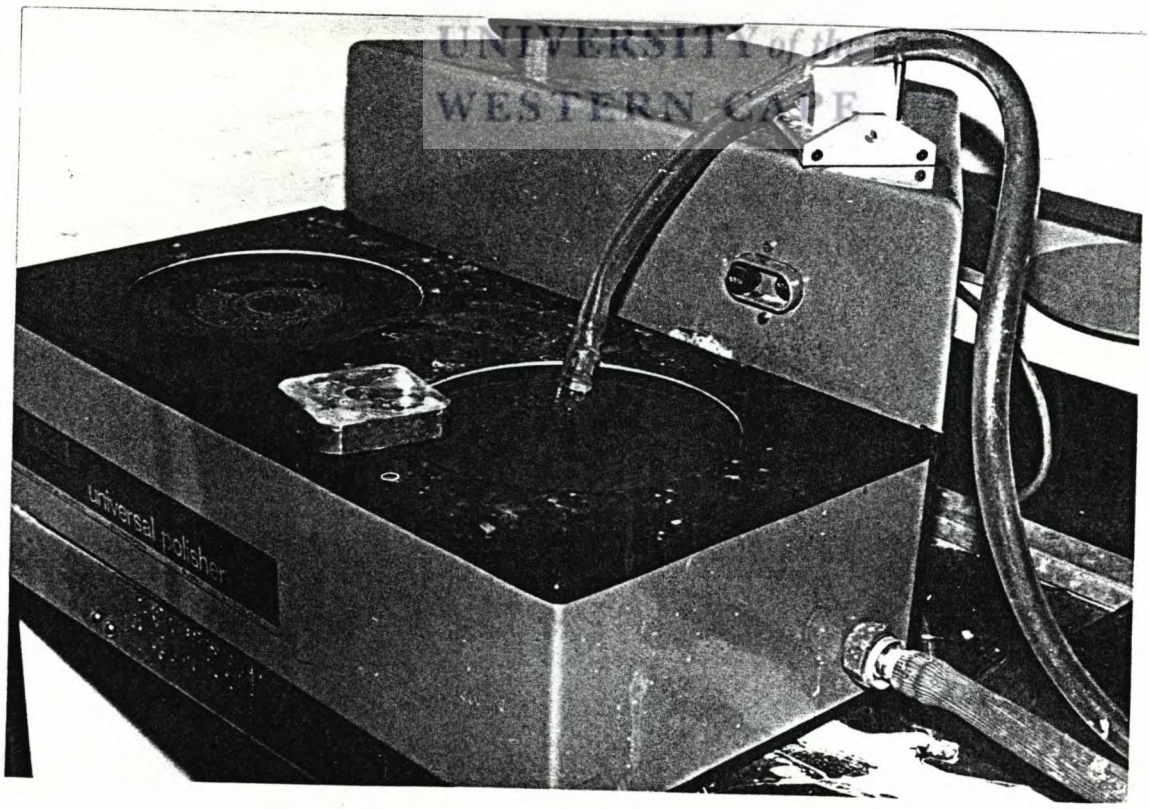


FIGURE NO.5: Bonded specimen.

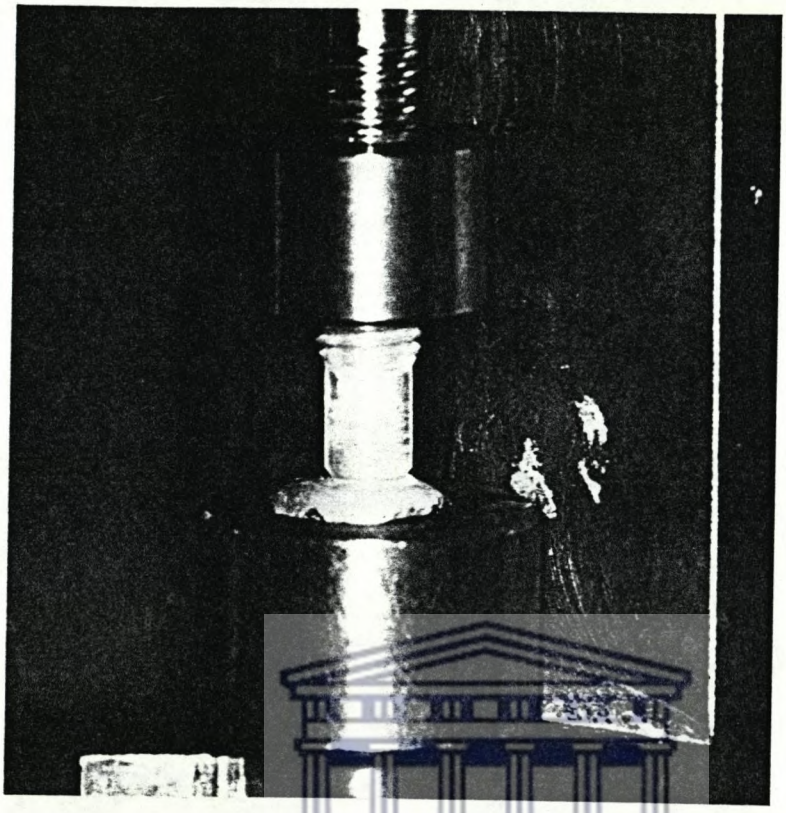


FIGURE NO.6: Aligning blocks with weighted specimen.

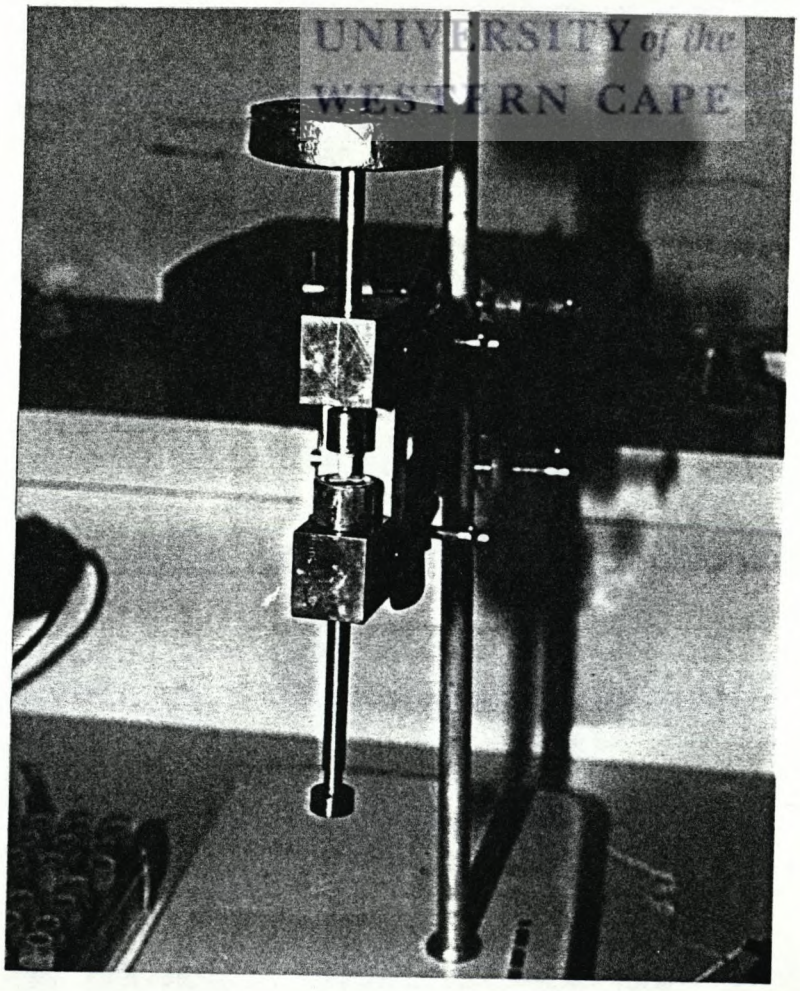


FIGURE NO:7. Specimen after fracture showing stub and tooth.

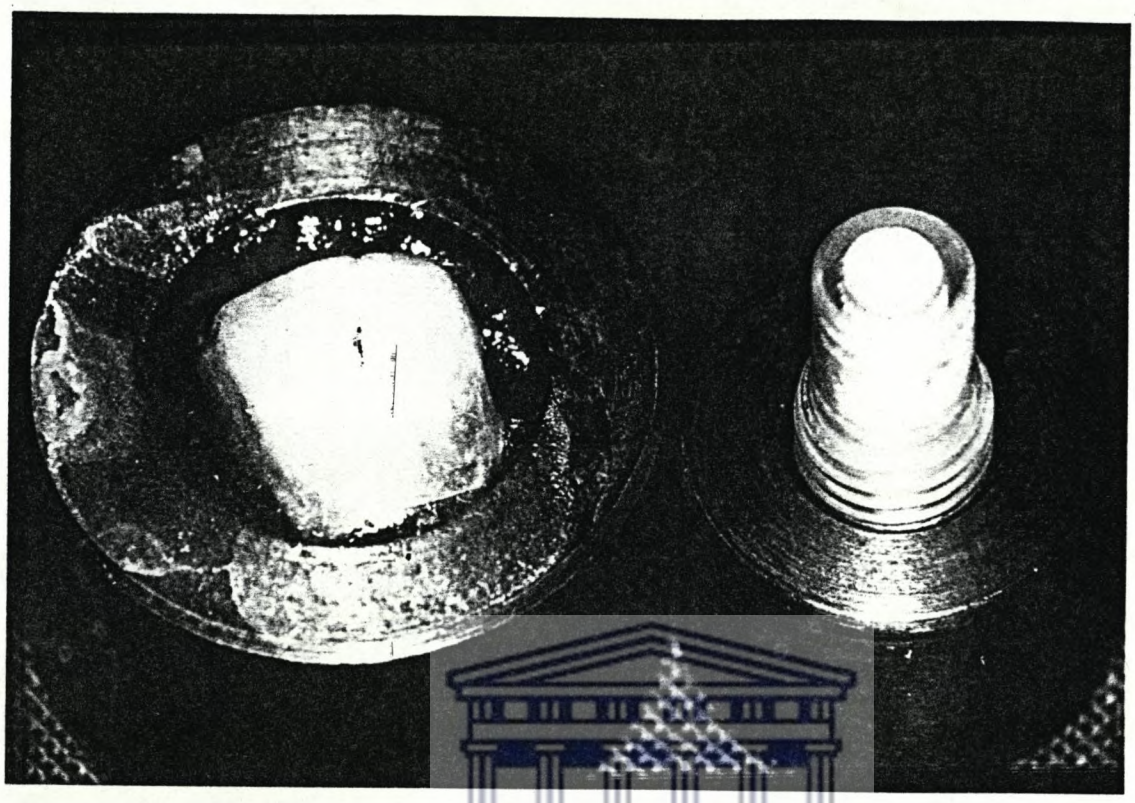
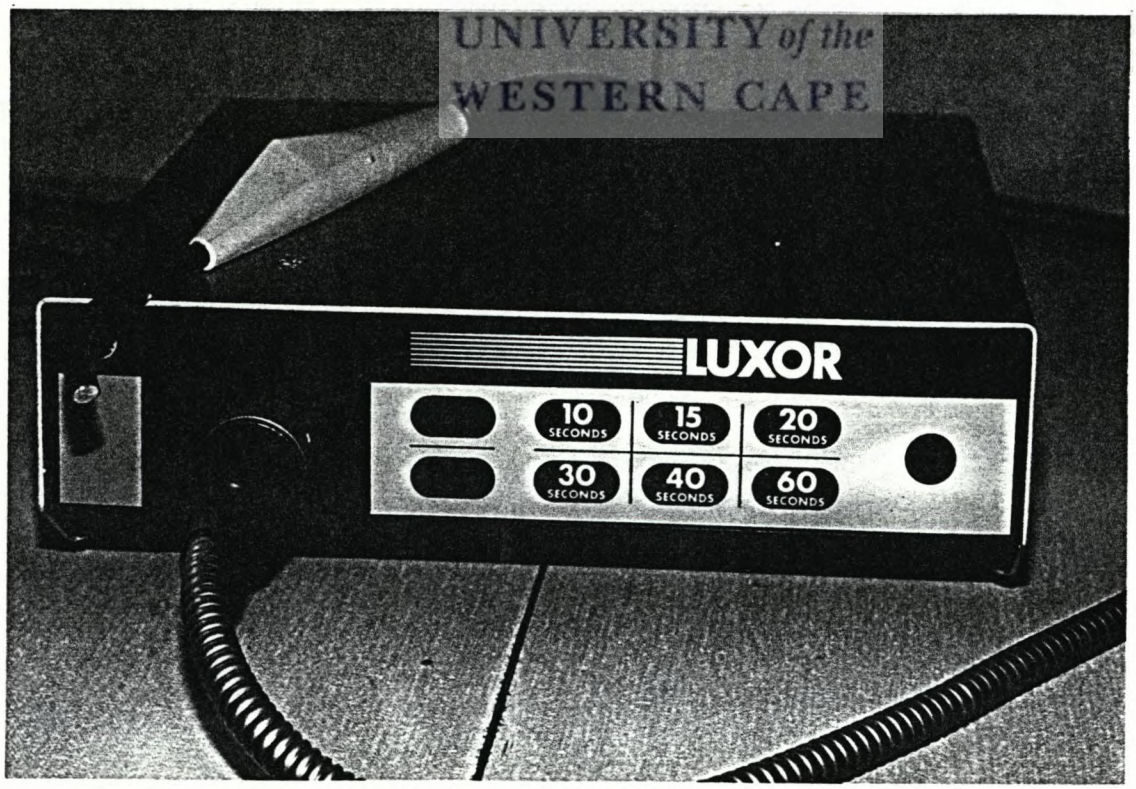


Figure No:8. Light unit: Luxor 4000.



METHODOLOGY FOR THE TENSILE BOND STRENGTH STUDIES.

Sixty non-carious freshly extracted human maxillary incisor or canine teeth were stored in 70% ethanol after having been cleaned of any soft tissue debris.

The crowns of all the teeth were sectioned from the roots at the cemento-enamel junction with a diamond disc. These crowns were then prepared by cutting a wedge shaped channel through the palatal side from incisal edge to cervical neck, removing the pulp chamber and contents at the same time. This was performed using a water cooled diamond bur in a high speed handpiece. This provided the mechanical retention needed to hold the crowns in the embedding medium.

These teeth were secured in brass cups by means of a self curing acrylic resin (Kerr's "Fastcure"). Care was taken to allow the buccal face of the crowns to extrude 1mm above the lip of the brass cup so that the facial aspect was approximately perpendicular to the vertical. The acrylic resin was allowed to cure for 24 hours at room temperature. They were then stored wet in an incubator at 37 degrees centigrade for at least 24 hours.

Fifteen of these prepared teeth were used for each material. The cups were all numbered and a computer generated random sequence order was used to determine which material was to be used in the determined sequence.(ref to table 4 on page 48). Immediately prior to the preparation of the bond test specimen, the crown surface was polished

lightly by placing the tooth cup in a polishing block on a 600 grit silicon carbide disc in the polishing machine, using water as a coolant and lubricant. This polishing block assured that the surface of the crown was perpendicular to the stub to eliminate the risk of shear forces being introduced at the time of tensile testing. The ground area on the enamel allowed the stubs to seat correctly when aligned in the blocks. At no time was dentine exposed.

This surface of the tooth was then etched with 37% phosphoric acid for one minute using the the material sequence order described earlier. In all cases the manufacturers instructions were adhered to. The resulting etched surface was then washed in running tap water for 15 seconds to remove the reaction products and dried by means of a hand held chip syringe.

Perspex stubs were machined at this university using two studies to assertain the strength and dimensional stability of the perspex required to withstand the tensile forces generated in this study. These perspex stubs were necessary for this study as two of the materials were light cured.

Where indicated the low viscosity bonding resins were mixed according to the manufacturers instructions and placed on the teeth. The composite resins were bulk packed into the stubs against a back filler. If they were of the light curing variety a bulk packing method was applied directly from the syringe, or mixed by hand and then manually packed into the stubs if they were of the chemically cured

variety. The order in which the materials were to be used in the numbered cups was determined by a computer generated random table as mentioned previously (Ref. P48). After placement of the paste in the stubs, the stubs were placed in the bonding aligning block. The low viscosity bonding resins were applied according to the manufacterers instructions to the etched enamel surfaces. The composite filled stubs were then lowered onto the teeth in the block, until the stub's knife edge made contact with the enamel surface. The excess was removed with an explorer and "superfloss" and allowed to cure under a load of 500 grams for 15 minutes in the case of a chemically cured resin. The light cured resins were exposed to a beam of white light from a white light unit (I.C.I's Luxor 4000 unit). Using a three side exposure proceedure and a total of 60 seconds exposure a complete cure of the materials was achieved. A load of 500 grams was also applied to the light curing specimens at the curing stage. These stubs were then removed and stored wet in an incubator at 37 degrees for 48 hours until ready for the tensile bond strength tests.

To determine the tensile bond strengths, the specimens were mounted in the measurement alignment block, which was then suspended in the upper jaw of the Instron testing machine (Instron Corporation, Canton, Mass. U.S.A.). A tensile load was then applied at a speed of .02 in. per minute and the force required to break the experimental bond was recorded.

This recording was in the form of a bar chart on calibrated graph paper. Using the information known it was then possible to calculate the bond strengths in Mega Newtons/metre squared (MN/m2) or Newtons /millimetre squared (N/mm2). The fractured specimens were examined under a dissecting microscope and the site of the fracture was noted.

Statistical comparisons of the data were made by an Analysis of Variance and by means of Duncans Multiple Range Test at the 0.05 level of signifance.

PART TWO -- SCANNING ELECTRON MICROSCOPE STUDY.

MATERIALS:

1) 24 freshly extracted Human permanent maxillary incisors.

2) Cambridge Sterio-Scan 180 Scanning Electron Microscope (Ref. P 40).

3) Gold splutter machine ("Splutter Device") by Blazers Union, Lichtenstein.

4) 3M Products:

a)Concise --- 3M Dental Products, St Paul, MN55144 U.S.A. Batch no. 1925S.

b)Silux --- 3M Dental Products, St.Paul, MN55144 U.S.A. Batch no.5501S.

5) Johnson and Johnson Products.

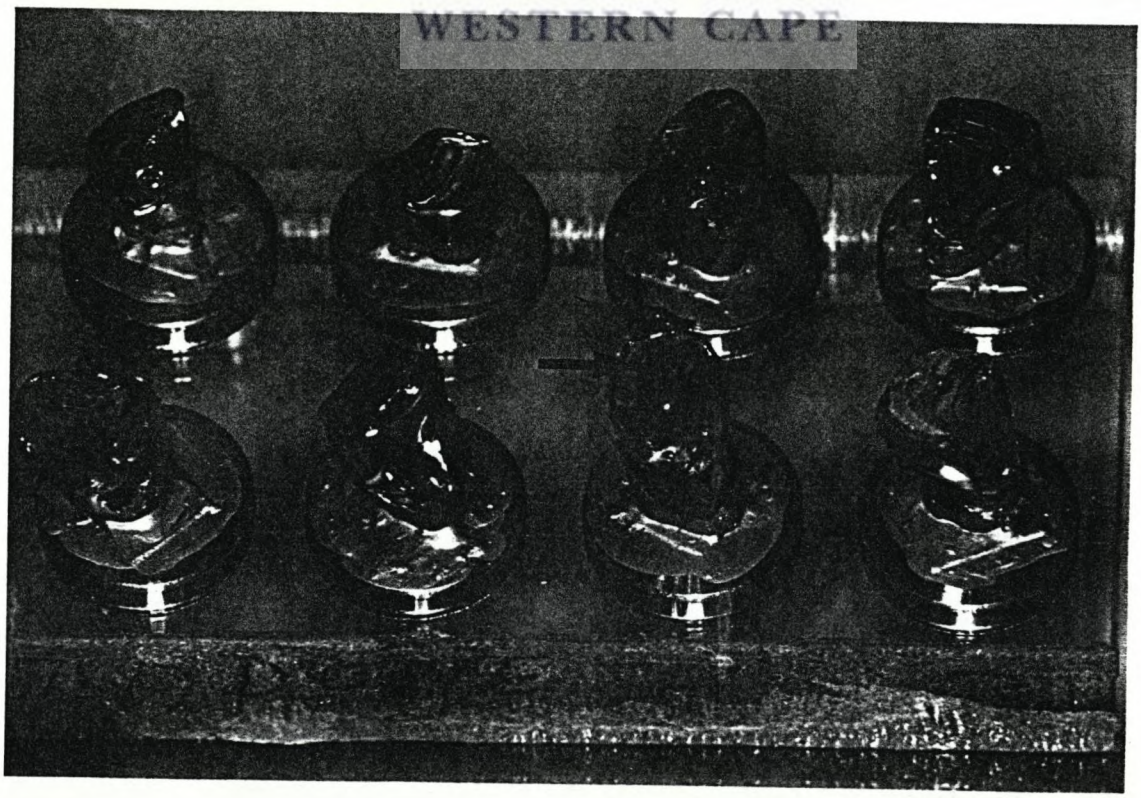
a)Adaptic --- Jonhson and Johnson Dental Products Company, East Windsor, NJ.08520 U.S.A. Batch No. 302099.

b)Certain --- Johnson and Johnson Dental Products Company, East Windsor, NJ.08520 U.S.A. Batch No.302099.

6) White light unit --- I.C.I. Luxor. Model 4000.

FIGURE NO.9:Stereoscan electron microscope.

FIGURE NO.10: Gold spluttered specimens, showing the interfacial zone.



METHOLOGY OF THE SCANNING ELECTRON MICROSCOPE STUDY.

The twenty four teeth to be examined were divided into two major groups, one for use with 3M products and the other for the use with J&J products.

The six teeth used for polished enamel and acid etch studies were polished with fine pumice and rubber cups and then etched according to the manufacturers'instructions. The enamel surfaces were etched for 60 seconds, using a dabbing motion to apply and circulate the acid, washed under running tap water for 15 seconds and dried using a hand operated chip syringe. These teeth were then dried in a desiccator overnight and prepared for scanning electron microscopy by gold splutter coating of the samples.

The six teeth used for ground enamel and acid etch studies were all subjected to 600 grit grinding of their labial surfaces, washed for 15 seconds under running tap water, and then etched according to the manufacturers instructions. After washing for a futher 15 seconds under running tap water they were also desiccated and prepared for scanning electron microscopy.

The 12 interfacial zone specimen teeth were all prepared by first polishing the labial surfaces with pumice and a rubber cup. One of the four composite resins was then bonded onto this surface producing a small island of material in the middle of the labial surface. Bonding was preceeded by acid etching the tooth as recommended

by the individual manufacturer. They were tnen stored wet for 24 hours at 37 degrees centigrade in an incubator.

The interfacial zones were examined by sectioning the teeth through the enamel/resin interface with a diamond disc. All surfaces except this cut section of interface were then covered with sticky wax. This interfacial surface was then decalcified by exposing it to 5% hydrochloric acid for 30 seconds. The specimen was then thoroughly washed in running tap water and dried overnight in a desiccator. They were then prepared for scanning electron examination as described previously.

Thus each major group consisted of 12 test teeth and they were examined in the following ways:

1) Enamel etching;

a) 3 teeth allocated for a polished enamel and then etched surface examination.

b) 3 teeth allocated for a ground enamel and then etched surface examination.

2) Interfacial zones;

a) 3 teeth were allocated for the conventional macrofilled resins.

b) 3 teeth were allocated for the microfilled resins.

Schematically this could be represented as follows:

TABLE 1: Methodology table for S.E.M. study:

	GROUP A	GROUP B
ETCHED ENAMEL:	3M	J&J
Polished	3 teeth	3 teeth
Ground	3 teeth	3 teeth
INTERFACIAL ZONE:		
Conventional resin	3 teeth	3 teeth
Microfilled resin	3 teeth	3 teeth
Total	12 teeth	12 teeth

This part of the study was a descriptive and qualitative evaluation only.

RESULTS.

Part One --- Tensile bond strengths.

Instron machine readings:

The Instron Testing Machine was used to produce a graph of the forces applied to the various samples. The pen movements on the graph were measured in millemetres.

The machine was used with the load cell sensitivity set at 0.1. With this setting and a load cell rating of 5000N (5k) a stress of 500 N would give a pen movement of 250mm on the graph paper (with standard calibration).

Recalling that the diameter of the bonded areas were 3mm, the radius is therefore 1.5mm. The surface area of the bond was thus;

PI R2

=3.14 * (1.5)2

=7.065mm2

All the recordings were measured in mm and the stress in Newtons was calculated by applying the formula:

$$\frac{\text{movement of pen (mm)} * 500}{250} = \text{X Newtons}$$

The tensile bond strength was then calculated by dividing this stress by the surface area:

$$\frac{\text{"X"}}{7.065} = \text{"Y"N/mm2}$$

By applying these calculations and subjecting the various samples to the testing proceedure and recording the results, the following table of results was obtained.

Below is a total of 12 specimen bond strength measurements in each group in N/MM2.

TABLE 2: Tensile bond strengths of 4 composite resins.

GROUP: 1	2	3	4
ADAPTIC	CERTAIN	CONCISE	SILUX
26.89	16.98	30.00	25.19
31.42	25.76	22.64	23.21
22.92	25.19	32.83	30.29
23.21	27.17	26.61	22.64
30.57	22.36	30.00	22.64
27.45	19.53	28.02	22.36
31.41	10.19	22.36	31.70
29.44	16.98	16.41	26.04
26.89	18.11	27.17	24.34
22.08	18.11	28.59	24.34
23.77	22.36	35.10	20.94
32.27	31.42	26.61	19.53
n: 12	12	12	12
Mean: 27.36	21.18	27.19	24.43
SD: 3.69	5.70	4.98	3.55
CV% 13.5%	26.9%	18.3%	14.5%

(where Critical Value=SD/Mean*100/1)

STATISTICAL ANALYSIS.

Analysis of variance.

This may be defined as a technique whereby the total variation present in a set of data is partitioned into several components. Associated with each of these components is a specific scource of variation, so that in the analysis it is possible to ascertain the magnitude of the contributions of each of these scources to the total variation.

Analysis of variance finds its widest application in the analysis of data derived from experiments. It is used to: (1)estimate and test hypotheses about population variances and (2)estimate and test hypotheses about population means.

Duncans Multiple Range Test.

Duncan has contributed a considerable amount of research to the subject of multiple comparisons with the result that at the present time a widely used proceedure is Duncan's new multiple range test.

Analysis of Variance test was performed to test the hypothesis that there was no significant difference between the four groups. The Analysis showed an F value of 4.84 which gave a p value of 0.0054. This indicated that there was a significant difference in the four groups.

Where these differences lay was tested for by the Duncans Multiple Range Test:

TABLE 3: Duncans multiple range test.

GROUP:	1	2	3	4
	Adaptic	Certain	Concise	Silux
MEAN	27.360	27.195	24.435	21.180
BOND STRENGTH	----------	----------	----------	----------

The bond strengths are in N/MM2.

This shows that the groups 1,2 & 3 are not significantly different amongst themselves. It also shows that groups 3 & 4 are not significantly different. However groups 1 and 2 are significantly different to group 4.

Random table

Random sequence order table:

This table was generated by computer for the purpose of ordering the sequence of tests. Adaptic was given the symbol "A", Certain was "B", Concise was "C" and Silux was "D"

TABLE 4: Computer generated random sequence order.

1=A, 2=D, 3=B, 4=C, 5=B, 6=D, 7=C, 8=A, 9=A, 10=B,

11=D, 12=C, 13=D, 14=A, 15=B, 16=C, 17=B, 18=C, 19=D, 20=A,

21=C, 22=B, 23=D, 24=A, 25=B, 26=D, 27=C, 28=A, 29=C, 30=B,

31=D, 32=A, 33=D, 34=C, 35=B, 36=A, 37=B, 38=C, 39=D, 40=A,

41=A, 42=D, 43=B, 44=C, 45=D, 46=C, 47=A, 48=B, 49=C, 50=B,

51=A, 52=D, 53=B, 54=A, 55=C, 56=D, 57=C, 58=D, 59=B, 60=A.

FRACTURE SITES - part 1

Once the specimens had been stressed to fracture in the Instron machine, the teeth were examined under a light microscope for the site of fracture.

The following table represents the differences:

TABLE 5: Fracture sites of specimens.

SITE OF FRACTURE	NUMBER OF TEETH
1) In the composite	33
2) At the interfacial zone	21
3) In enamel	1
4) Combination of 1) and 2)	5
TOTAL:	60 TEETH

FRACTURE SITES. - part 2

The sites of fracture can futher be divided to include the materials as is shown below:

TABLE 6: Fracture sites of specimens.

	Composite	Interfacial zone	Enamel	Combination
Adaptic	1	10	0	4
Certain	15	0	0	0
Concise	4	9	1	1
Silux	14	1	0	0

This table shows a predominance of fractures in the body of the composite resins associated with the microfilled resins tested. The macrofilled resins tested showed a predominant tendency to fracture at the interfacial zone or in a combination of composite and interfacial zone. Only one specimen fractured in the body of enamel.

The following photographs show the different fracture sites.

FIGURE NO:11. Fracture in the body of the composite resin.

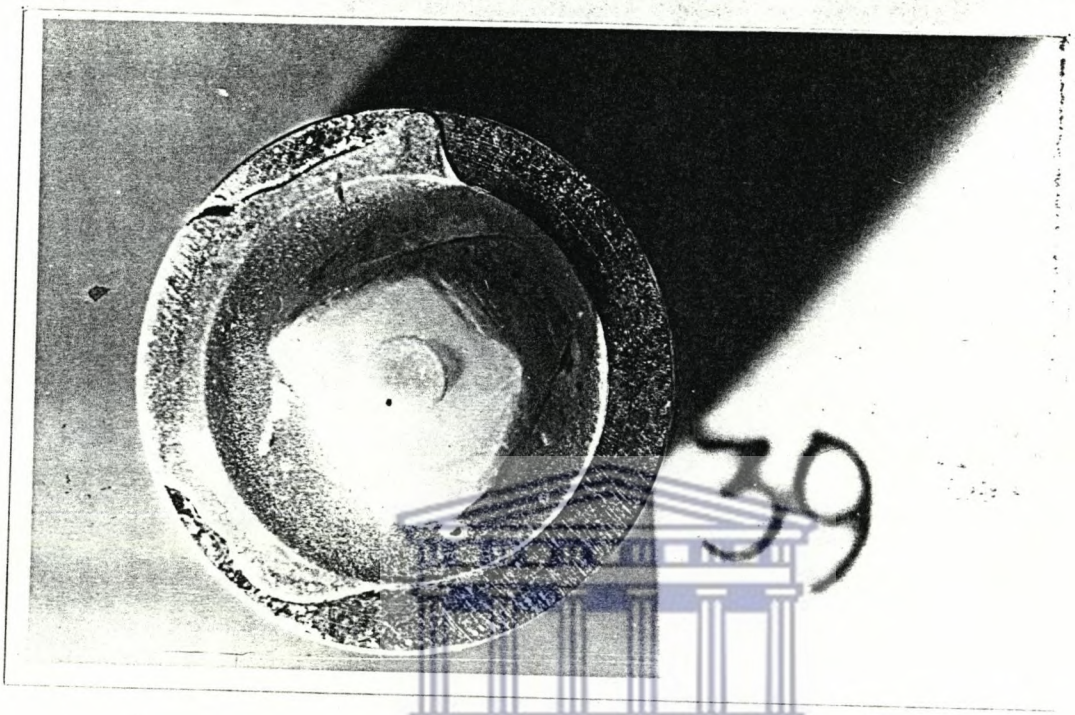


FIGURE NO:12. Fracture, partly within the resin and at the interfacial zone.

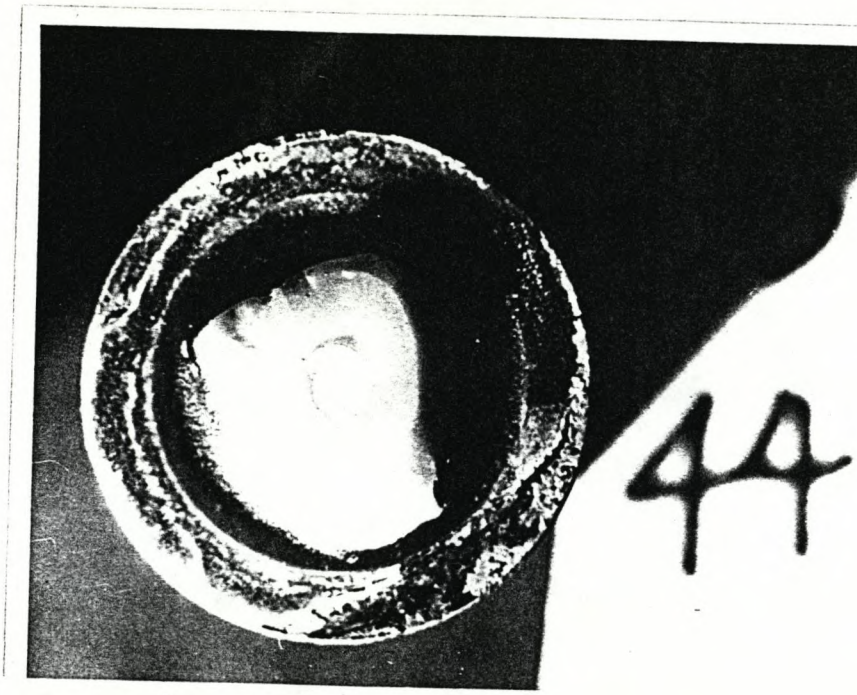
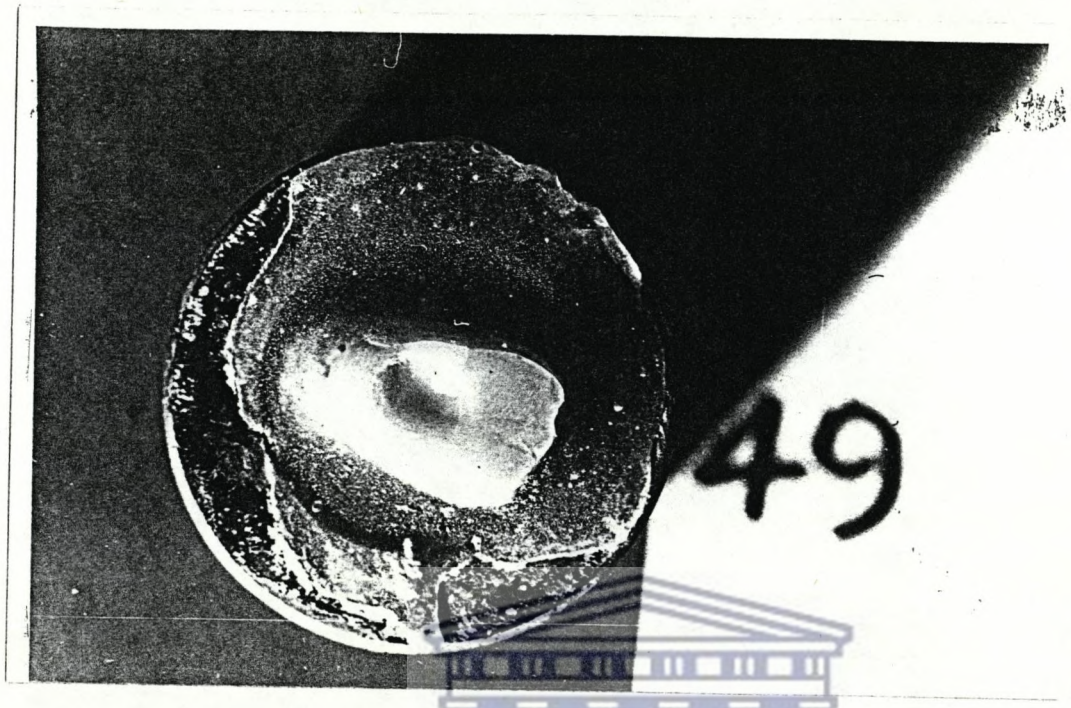


FIGURE NO:13. Fracture in enamel.



Part Two- Scanning Electron Microscope Study

This part of the study is descriptive and qualitative only and will be dealt with in the discussion section.

University of Cape Town S.E.M.Course.

In order to operate the scanning electron microscope I completed a computer generated self instructional course at the University of Cape Town. The graphics for the SEM screen are done in machine language and operated from an Applesoft basic environment. The hardware consisted of a 16 channel A/D converter interfacing the buttons and knobs to the Apple. The Apple was the 48K + language card version having a Videx 80 column card, three disc drives and an Apple silentype printer. On completion of this course I was allowed to operate the unit at the Medical Research Council in Cape Town.

Medical Research Unit.

The Stereoscan 180 scanning electron microscope incorporates the following features:

1 to 60kV electron optical system.

Magnification fully compensated for all gun accelerating voltage and working distance changes.

Dual magnification imaging.

Clean, high performance vacuum system, with auto control and fail safe protection.

DISCUSSION.

Tensile Bond strength study.

Perspex stubs:

In this part of the study a unique problem had to be overcome before any bond strengths could be assessed on the light cured materials.

Previously all stubs were made of brass, which would not allow the curing of the resin by a light scource. A perspex stub of similar dimension to the brass stubs was constructed and two pilot studies were completed on the design of this stub. Some modifications to the prototype stub had to be made before they were ready for this study.

The stubs were fabricated from a solid 7mm, cylindrical sample of perspex, with half the length of the external surface being threaded. The apex of the sample had an external bevel placed to ensure that the sample would be accurately placed without margin excess as well as to ensure a uniform bond area. These stubs were 12mm in length with a 3mm internal diameter at the apex flaring to a 4 mm diameter at the opposite end.

The internal diameter of the stubs were measured after the tensile tests to determine whether change in shape had occured. The diameter of the bonded area was also measured to ensure that a constant surface area was always obtained for the test.

The pilot study revealed that the perpex stubs could be used only once, and that the composite bonded to the tooth had obtained a total cure and was of the correct dimension. Once the composite sample was removed from the stub some damage invariably occured to the stub in the form of a fracture or break which precluded their re-use. Ref. to Figs.14 and 15 on page 56.

Figure No:14. Photograph of newly fabricated perspex stubs.

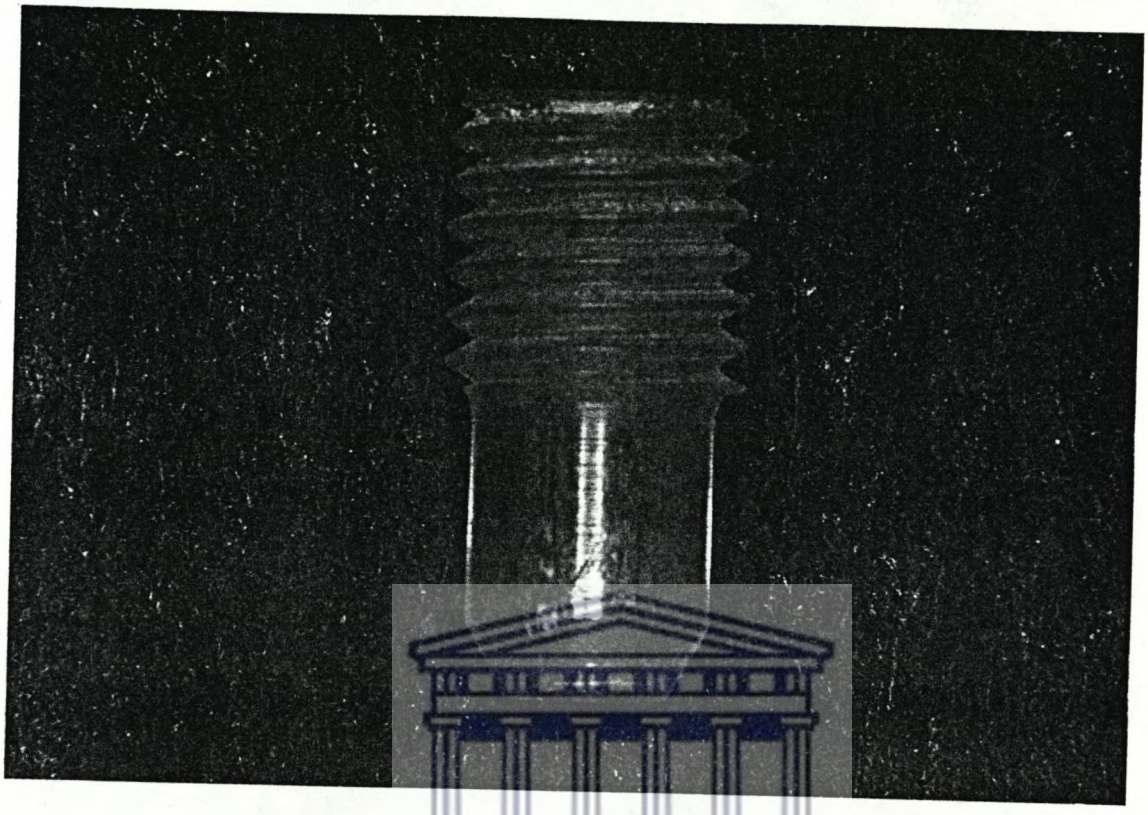
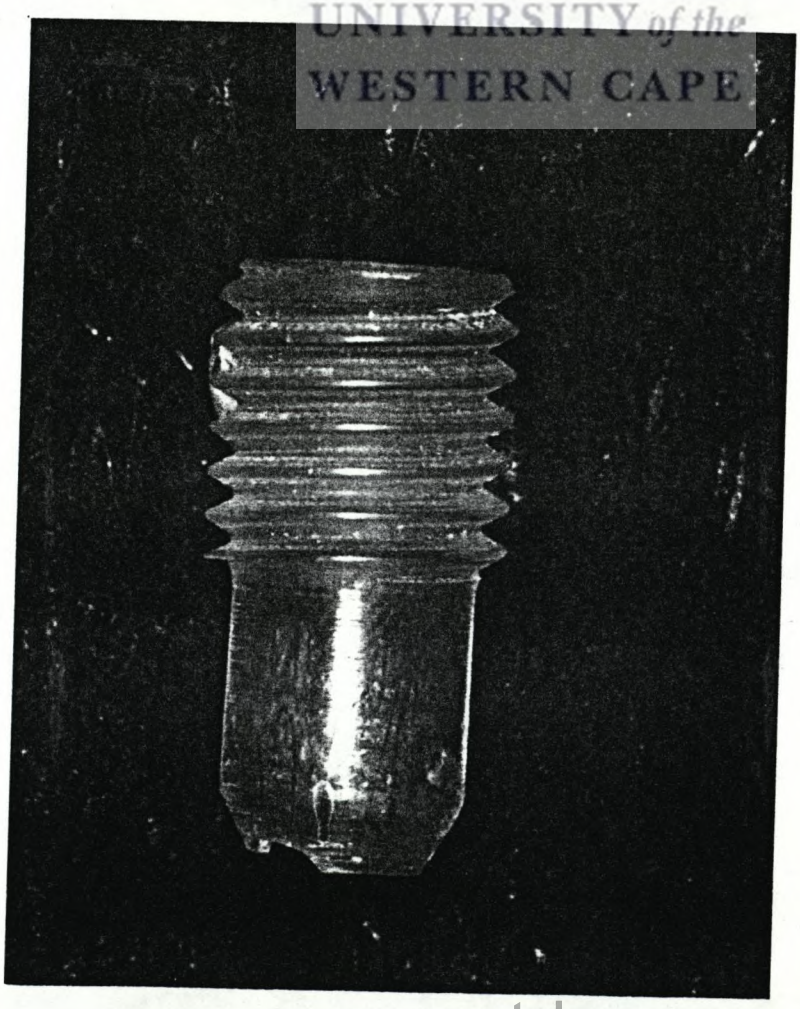


Figure No:15. Photograph of fractured stub.



Discussion of Johnson and Johnson Products.

The results indicate that there was no significant difference in the tensile bond strengths between the J&J products, Adaptic and Certain. Refer to page 51 for the Duncans Multiple Range Test.

Adaptic, being a macrofilled resin is expected to behave in a more "plastic" manner than the more brittle microfilled Certain. However the results did not show a signicant difference in bond strengths when Adaptic was compared to Certain (microfilled resin). Adaptic did not show a significant difference to Concise (macrofilled resin by 3M). Adaptic did however show a significant difference in bond strengths when compared to Silux (microfilled resin by 3M). Refer to Duncans Multiple Range Test on page 47.

Adaptic showed a tendency to fracture at the involved interfacial zone when stressed to failure. Certain showed a tendency to fracture only in the body of the composite resin which is expected since Certain is a microfilled resin. Refer to table 6 on page 50.

Discussion of 3M Products.

The difference between the the 3M product Concise (macrofilled) and Silux (microfilled) was greater than in the J&J sample but was not statistically significant.

It is not surprising that the microfilled Silux has a tensile bond strength which is signifacantly less than the macrofilled counterpart (Adaptic). The smaller particle resins are heavily filled and are more brittle than the larger particle composite resins. What is surprising is that no signifacant difference could be shown between the macrofilled "Adaptic" and the microfilled " Certain". Refer to the Duncans Multiple range test on page 47.

Concise showed a tendency to fracture at the interfacial zone when stressed to failure. Silux however fractured almost exclusively in the body of the composite resin. Refer to table 6 on page 50.

In general the following observation were made;

1) The bond strengths reported on page 45 compare favourably to published bond strength data (Asmussen 1985), albeit on the high side.

2) The difference in bond strengths between the samples (macrofilled and microfilled) may be due to the nature of the composite resin. The macrofilled resins are said to be plastic in nature whereas the microfilled resins are more brittle (refer to table 2 on page 45). The microfilled resins are, by weight, more heavily filled than the macrofilled resins and this varies the reported physical properties.

3) Differences in the observed etch patterns may be due to the type of pretreatment of the enamel. Polished enamel exhibited an indistinct etched pattern due to the presence of the prismless layer of enamel. Ground enamel

sections exhibited more classifiable etch patterns due to the exposure of the enamel prisms. Thus these different etch patterns are thought to be attributed to intrinsic differences in enamel, possibly explained on the basis of enamel solubility (Poole and Johnson 1972).

4) Tags were formed irrespective of the etch pattern. There was a difference between the tag lengths of the macrofilled and the microfilled resins. The depth of tag penetration for macrofilled resins was longer than the microfilled resins due to a lesser interference of the large filler particles with the wetting and penetration of the resin into the prisms of the enamel. The depth of tag penetration of the microfilled resins was about half that of the macrofilled resins (5-10 microns). This is likely to be due to the submicron particles interfering with the penetration of the resin into the enamel.

Retief and Mallory (1981) showed that even though the penetration coefficient of the resins they tested varied, the tensile bond strength did not vary significantly. The materials usually fractured in the body of the composite resin.

SCANNING ELECTRON STUDY.

1) Acid etching of the polished and ground enamel.

2) Interfacial Zones.

1.11 Polished Surfaces of the enamel using 3 M products.

The acid etch patterns of the polished enamel of three labial surfaces of the anterior teeth show a variable etch pattern ; some areas show an etching of the central core of the prisms (class one etch pattern) and in other areas an amorphous pattern is discernable. (Fig 16, 17, 18 at 1K magnification).

Even at higher magnification of 3 K the pattern is still indistinct (Fig 19). Some etching has occured but the pattern is mixed.

1.12 Ground Surface of the enamel using 3 M products.

The ground surfaces of three teeth show a definate etch pattern of either class 1, class 2 or class 3 types . This can be seen under high magnification of 3 K showing a class 1 pattern in Fig 20, class 2 in Fig 21 and a mixed pattern in Fig 22.

It would appear from the results that polished enamel surfaces do not produce the best etching pattern, and that the removal of a surface layer of enamel enhances the etched pattern. This observation is in agreement with the work done by Brannstrom and Nordenvall (1977). There is also no way of determining which etch pattern will result. (Silverman et.al.1975).

Etch patterns vary with the position of the tooth and because of this the incisal and cervical areas of enamel were examined. Using a polished enamel surface with 3 M products, the etch pattern showed that incisally a class 1 pattern resulted (Fig 23), whereas cervically a mixed pattern resulted (Fig 24).

When measuring the tags, the measurement accuracy may be influenced in various ways: during sectioning of the sample, fracture or degradation of resin during specimen preparation and throughout the examination of the sample. Angulation, tilt and height of the pedestal may also influence the viewing by altering the perspective of the specimen.

FIGURE NO :16. Etched surface of polished enamel at 1 K.

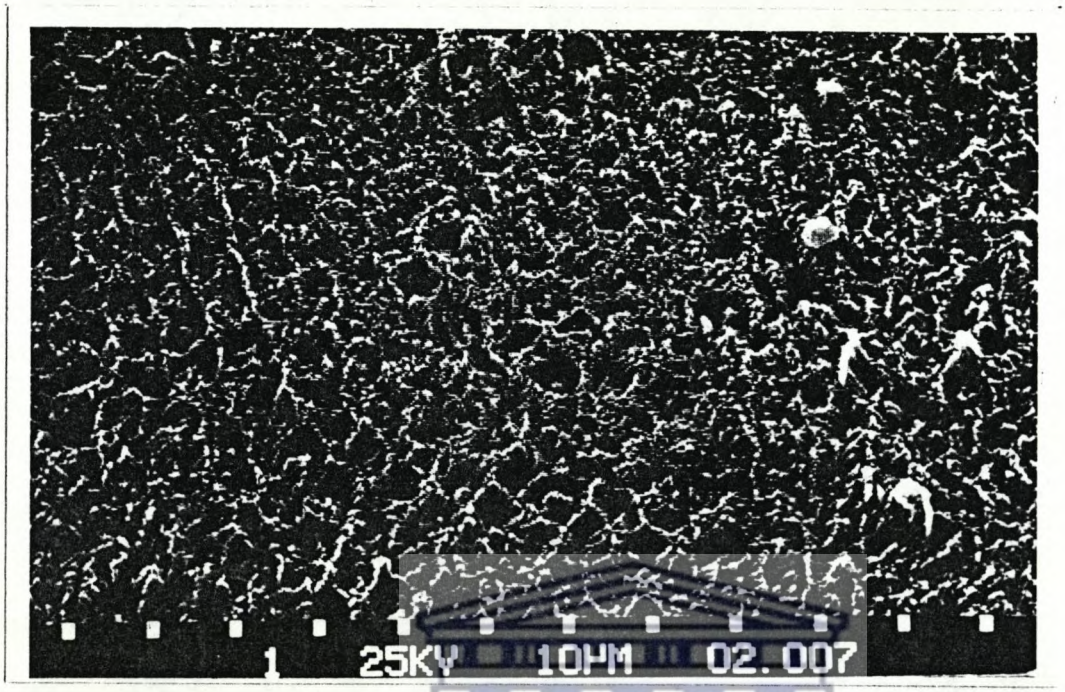


FIGURE NO:17. Etched surface of polished enamel at 1 K.

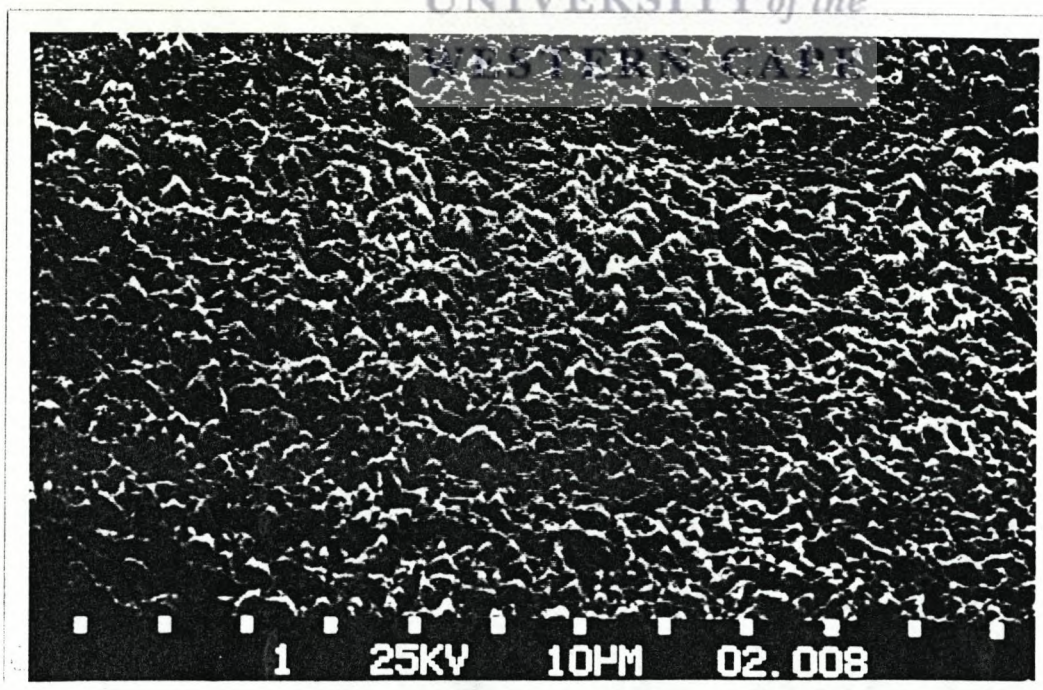


FIGURE NO:18. Etched surface of polished enamel at 1 K.

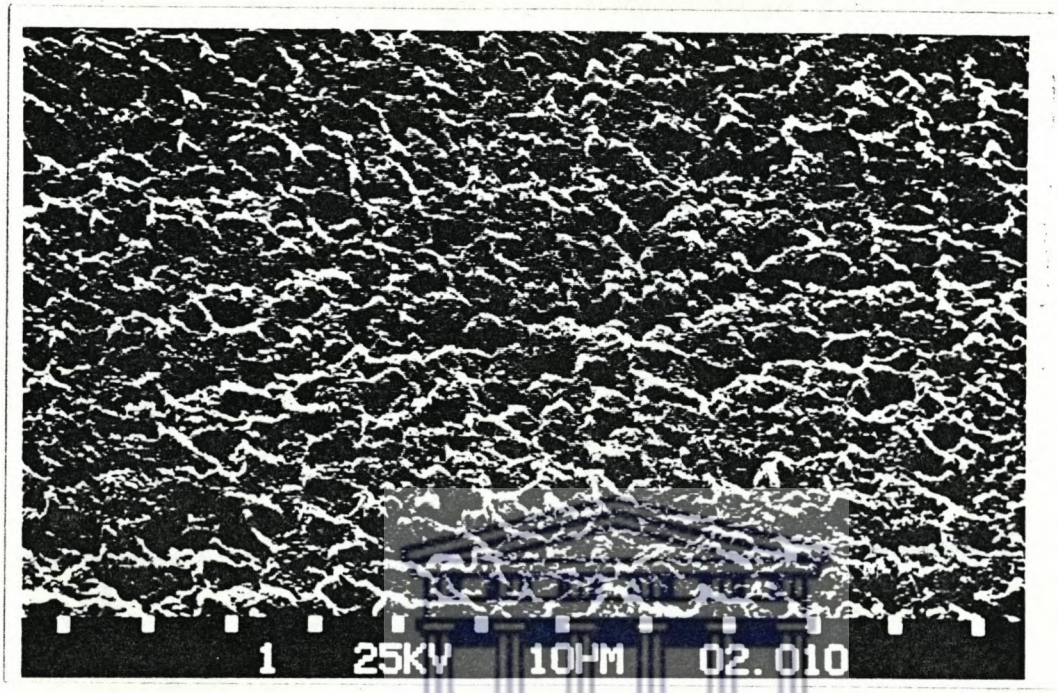


FIGURE NO:19. Etched surface of polished enamel at 3 K.

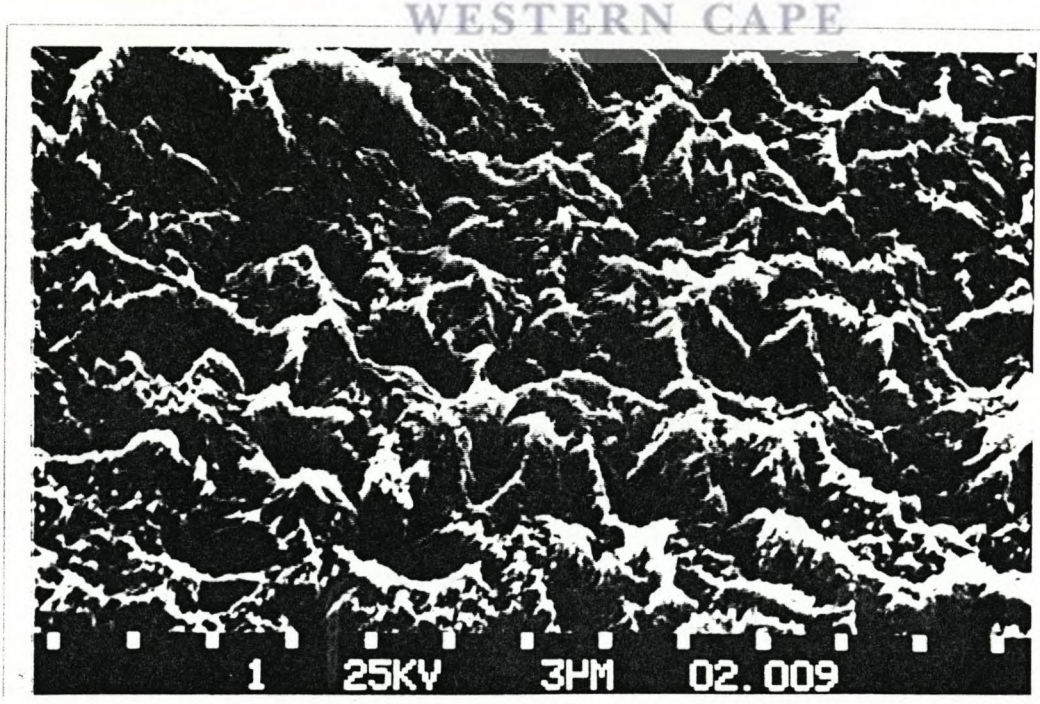


FIGURE NO:20. Class 1 etch pattern of ground enamel at 3 K.

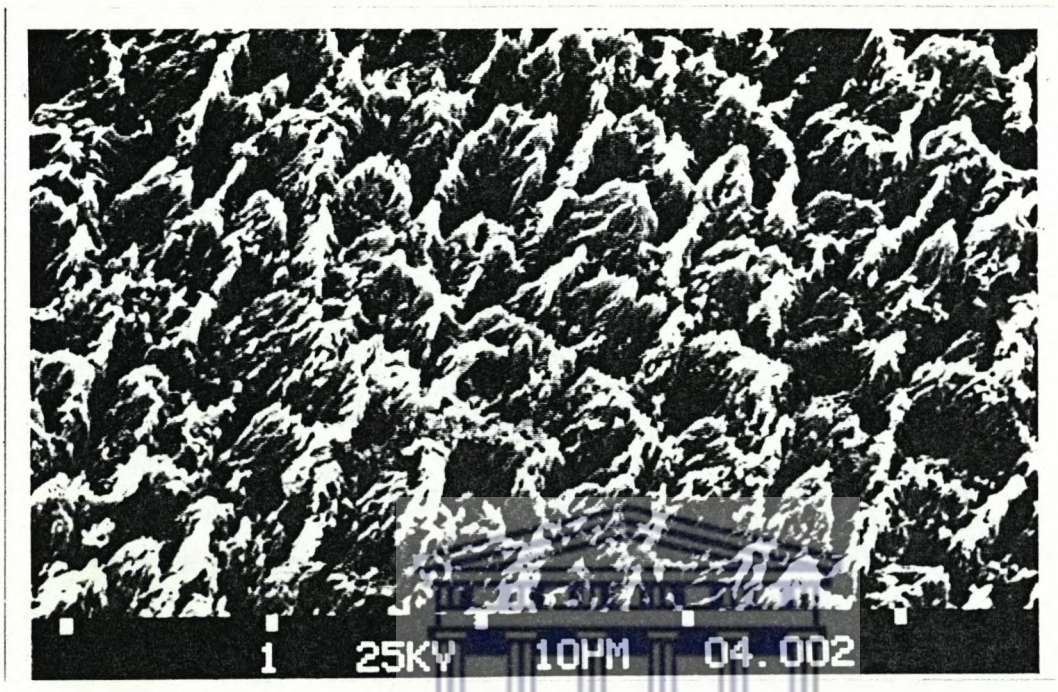


FIGURE NO:21. Class 2 etch pattern of ground enamel at 3 K.

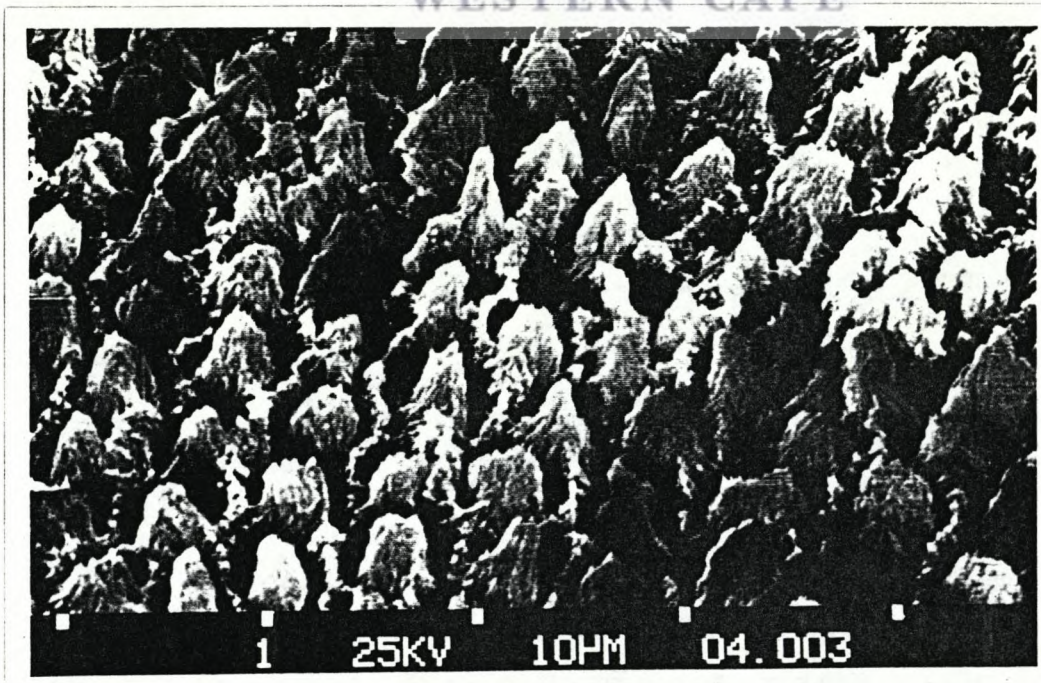


FIGURE NO:22. Mixed etch pattern of ground enamel at 3 K.

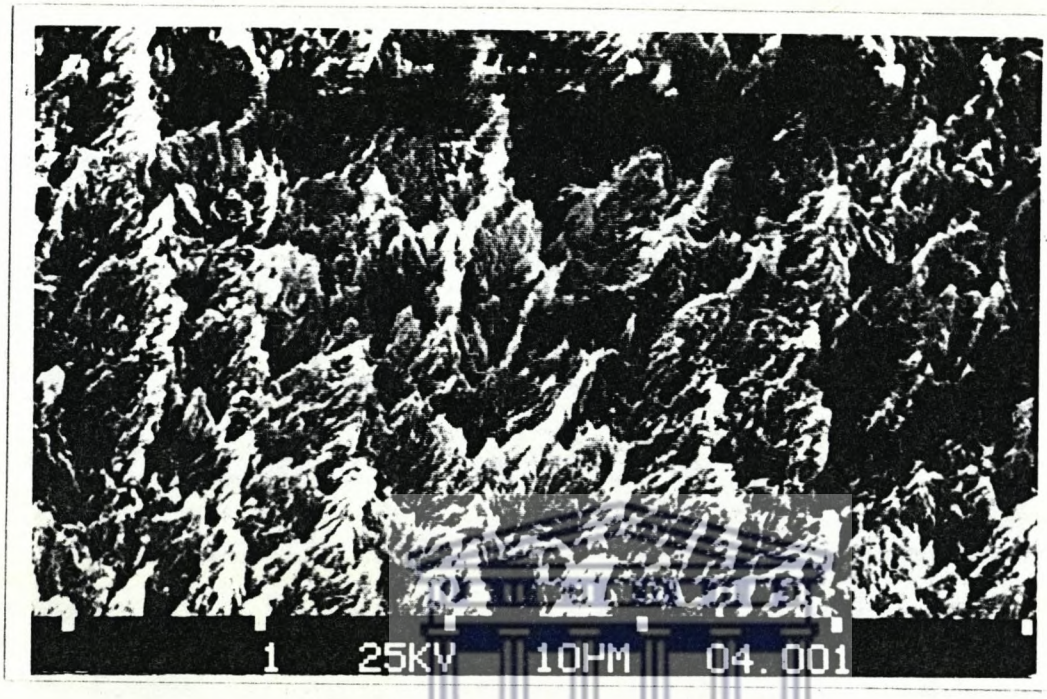


FIGURE NO:23. Class 1 etch pattern of polished incisal enamel.

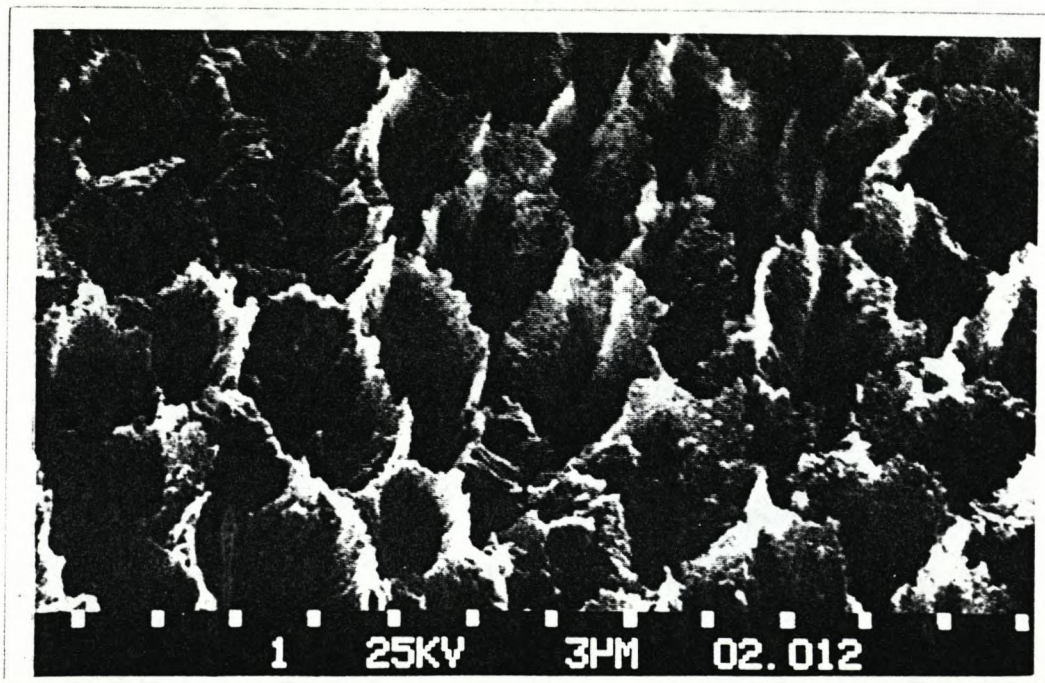
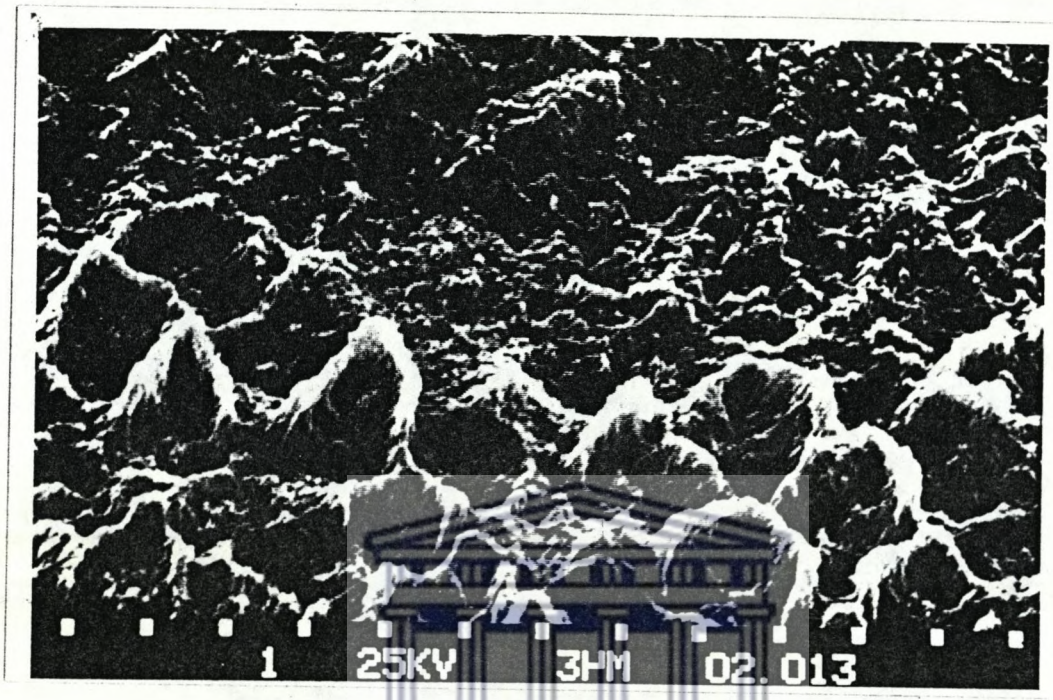


FIGURE NO:24. Mixed etch pattern of polished cervical enamel (upper section).



1.21 Polished surface of enamel using J&J products.

The electron microscopy examination showed the irregular patterns of etched enamel, some areas exhibiting a class 1 pattern (Fig 25), others a weak class 1 and class 2 pattern (Fig 26) and then again some flat irregular areas pitted by some class 1 patterns (Fig 27). The magnification of all these figures was at 1K.

1.22 Ground surface of enamel using J&J products.

These sections showed a more well defined etch pattern throughout the field. This was predominantly a class 1 etch pattern as was shown in Fig 28 and a class 2 etch pattern in Fig 29. A class 1 etch pattern is shown in Fig 30.

Once again this is in agreement with Brannstrom and Nordenvall (1977) and Silverman et. al. (1975).

FIGURE NO:25. Class 1 etch pattern of polished enamel at 1 K.

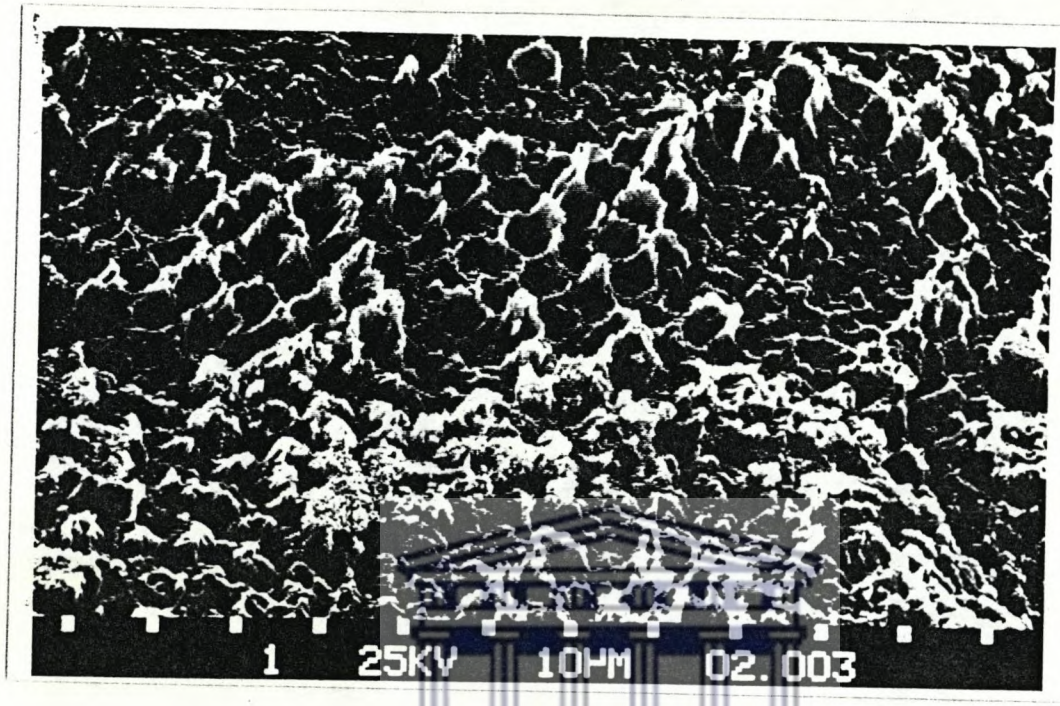


FIGURE NO:26. Class 1 and 2 weak etch pattern of polished enamel at 1 K.

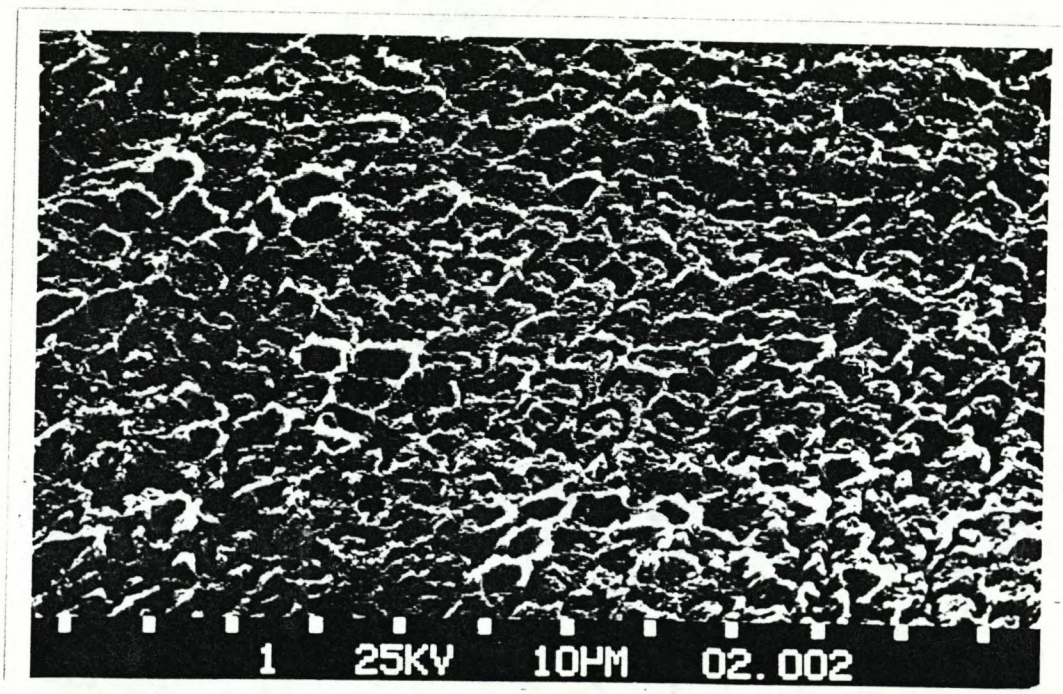


FIGURE NO:29. Class 2 etch pattern of ground enamel at 1 K.

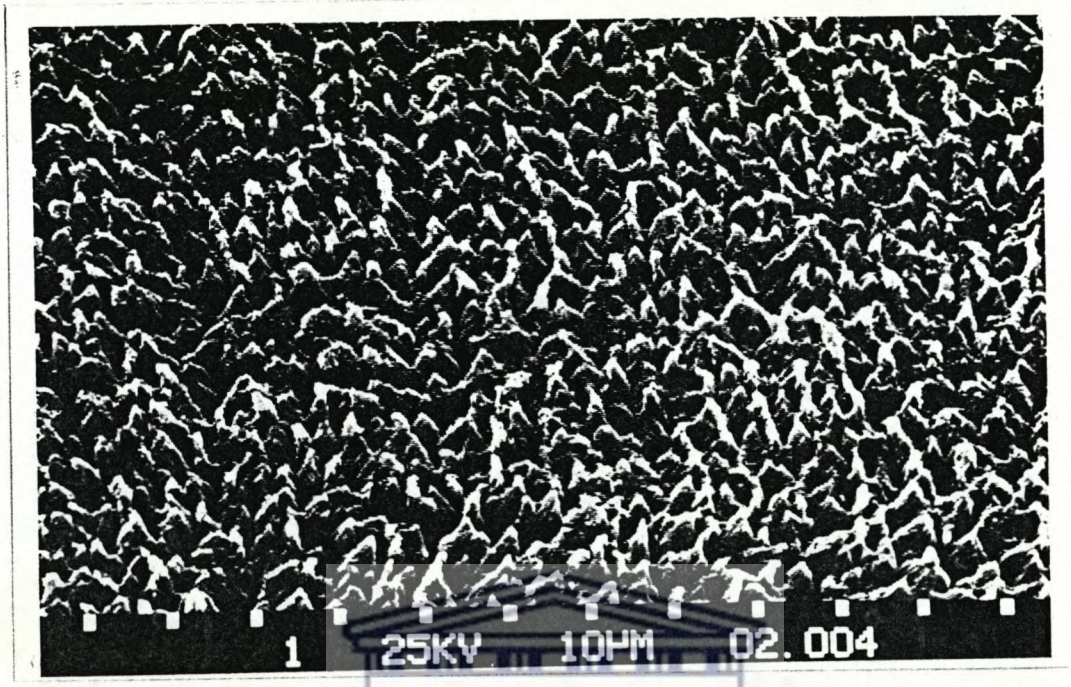
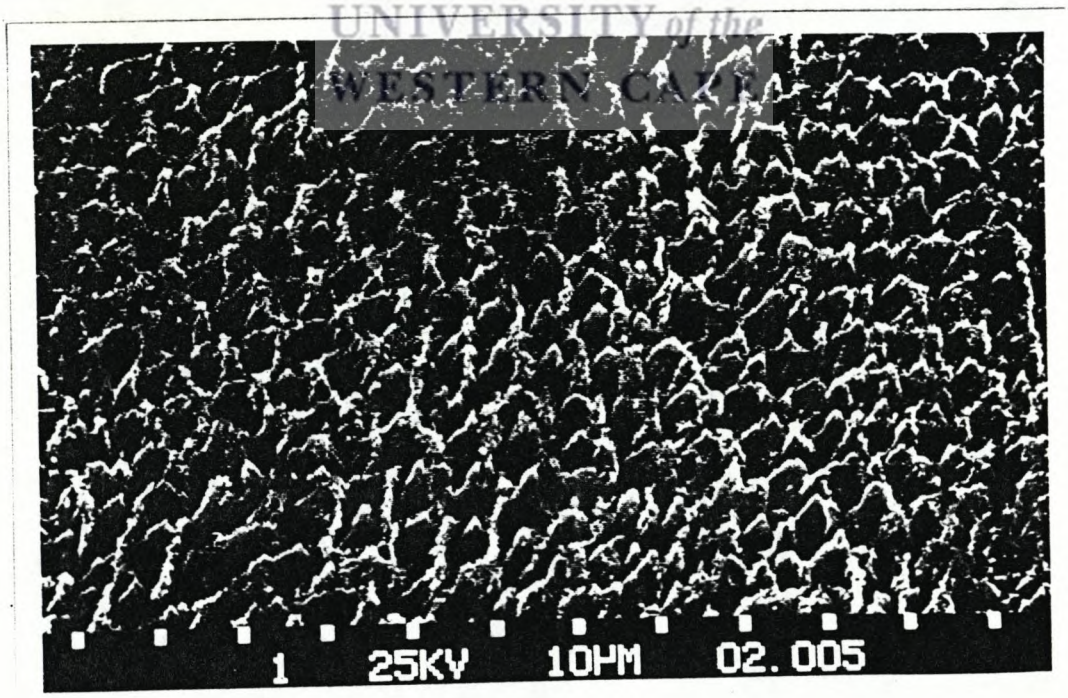


FIGURE NO:30. Class 1 etch pattern of ground enamel at 1 K.



2) Interfacial zones.

The testing proceedure involving the two materials was divided into 2.1 : J&J products of Certain and Adaptic.

2.2 : 3M products of Concise and Silux.

2.11 Adaptic Interfacial Zones.

The interfacial zones of the three specimens all showed good tag formation as can be seen in a low powered magnification photograph of the zone at 300 magnification (Fig.31). The appearance of a void or air bubble in the lower left hand part of the photograph has not interupted the tag formation suggesting that the void existed in the composite resin material and not in the low viscosity bonding resin.

Higher powered magnification photographs in Fig 32 at 1 K show this tag formation clearly. This can again be seen in Fig 33 at 1K, and this time demonstrating the inclusion of a void near the interfacial zone. This photograph also shows the size of some of the filler particles to be so large, that they apparently do not interfere with the formation of the delicate tags by obstructing the passage of the resin into the etched enamel. It is argued that this is the reason why the tag lengths of of macrofilled resins are often longer than those of microfilled resins where the smaller filler particles may obstruct the passage of the resin. These tags appear to be in the range of 10-20 microns in length and compare morphologically to the tags reported on by Voss and Charbeneau (1979).

FIGURE NO:31. Photograph of the Adaptic/enamel interfacial zone (*30).

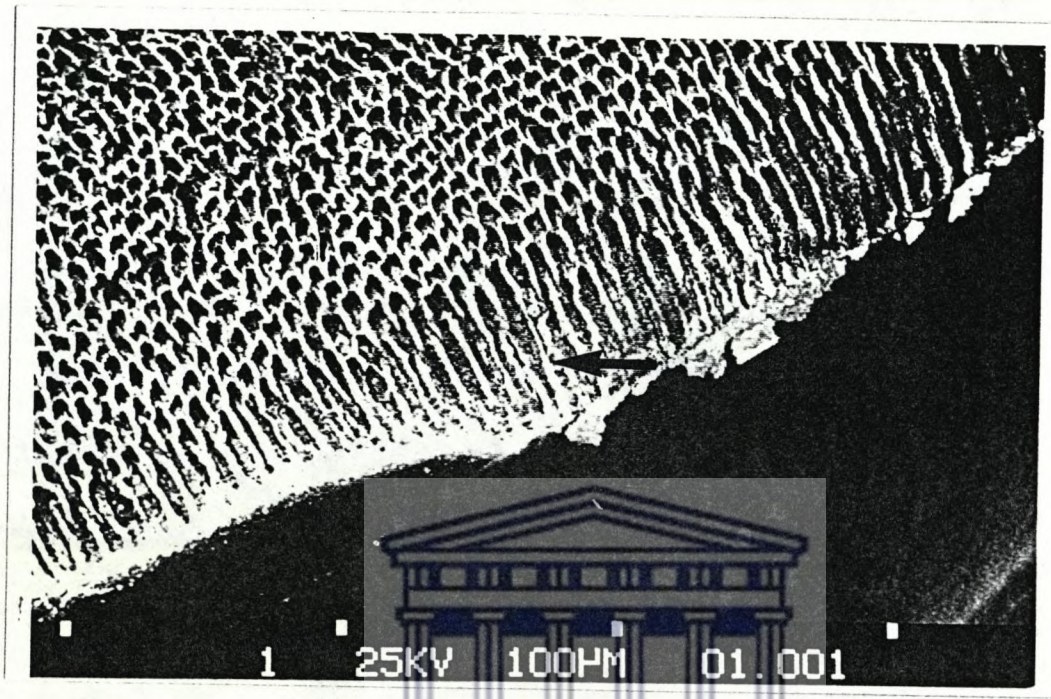


FIGURE NO:32. Photograph of the Adaptic/enamel interfacial zone showing tag formation at 1 K (arrowed).

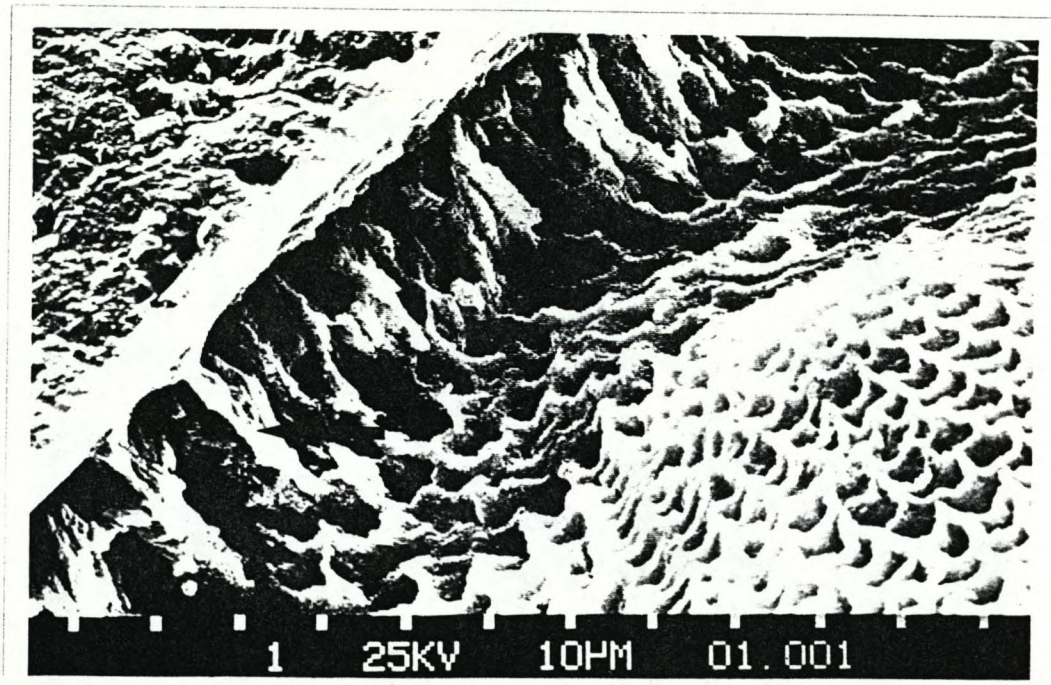
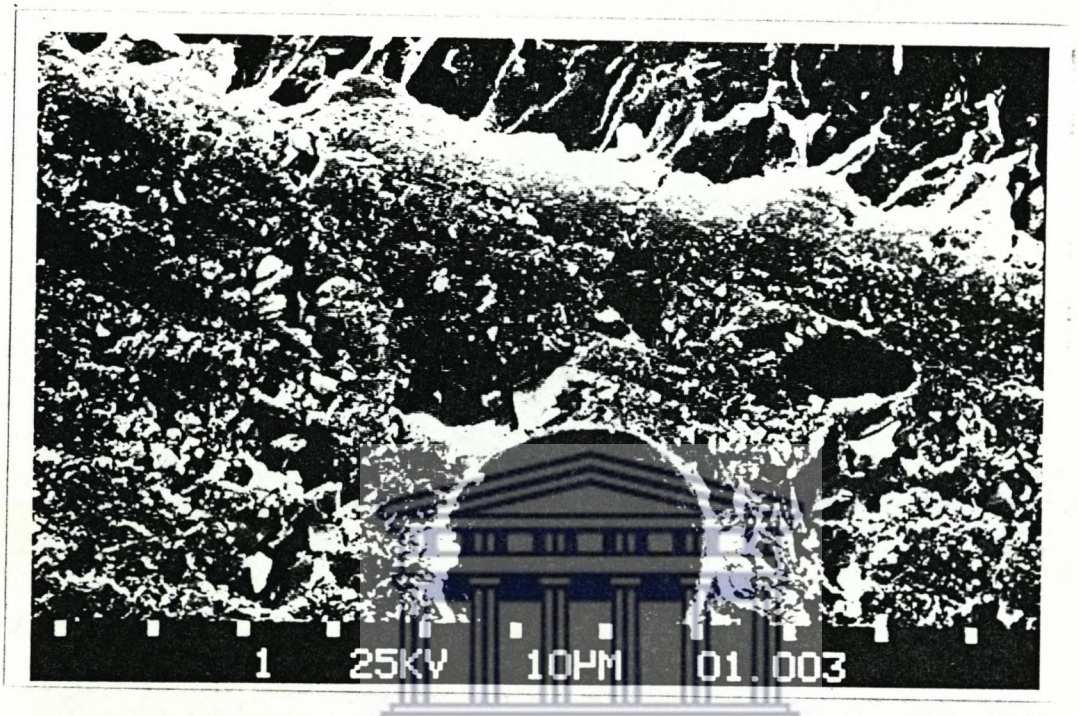


FIGURE NO:33. Inclusion of a void near the interfacial zone at 1 K.



2.12 Certain Interfacial Zones.

The interfacial zones of Certain, a microfilled resin, were examined and it was seen to exhibit tags of irregular dimension and length. The resin tag lengths averaged 5-10 microns in length. (Fig 34, 35, & 36). Fig 36 shows a class 2 etch pattern but the length of the tags is noticibly shorter than with Adaptic.

FIGURE NO:34. Resin tag formation under low power magnification (*30).

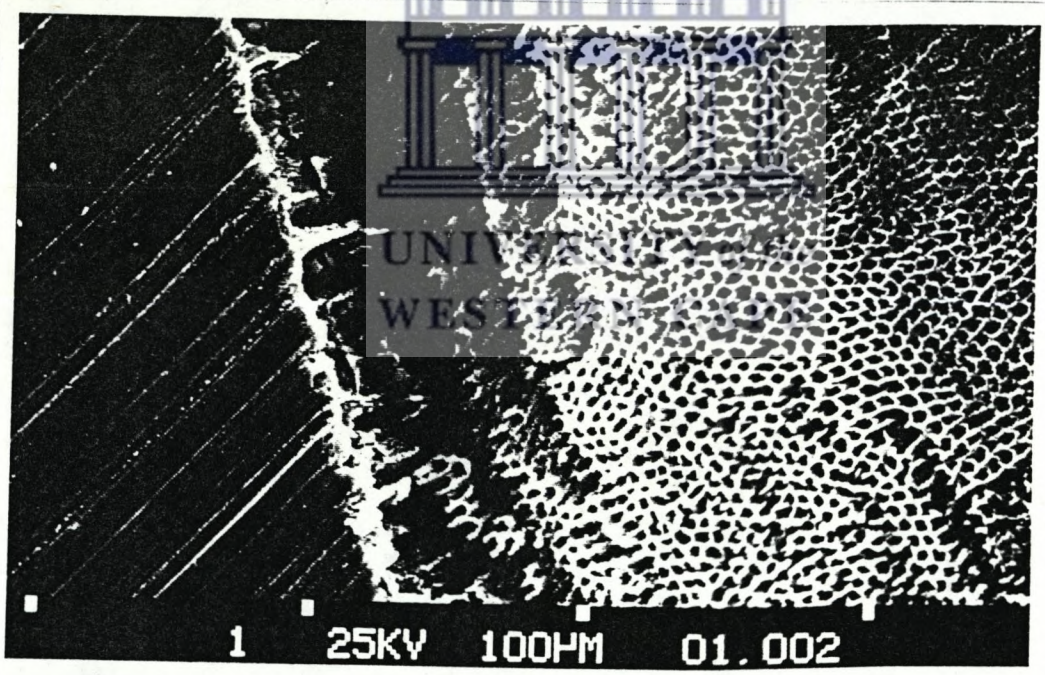


FIGURE NO:35. Resin tag formation under low power (*30).

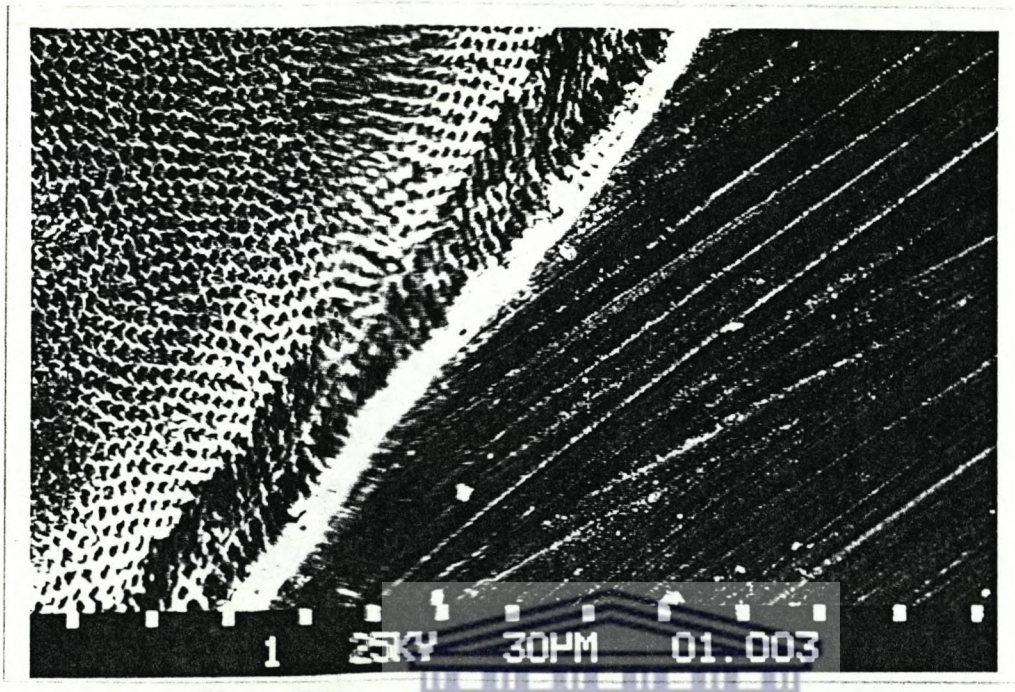
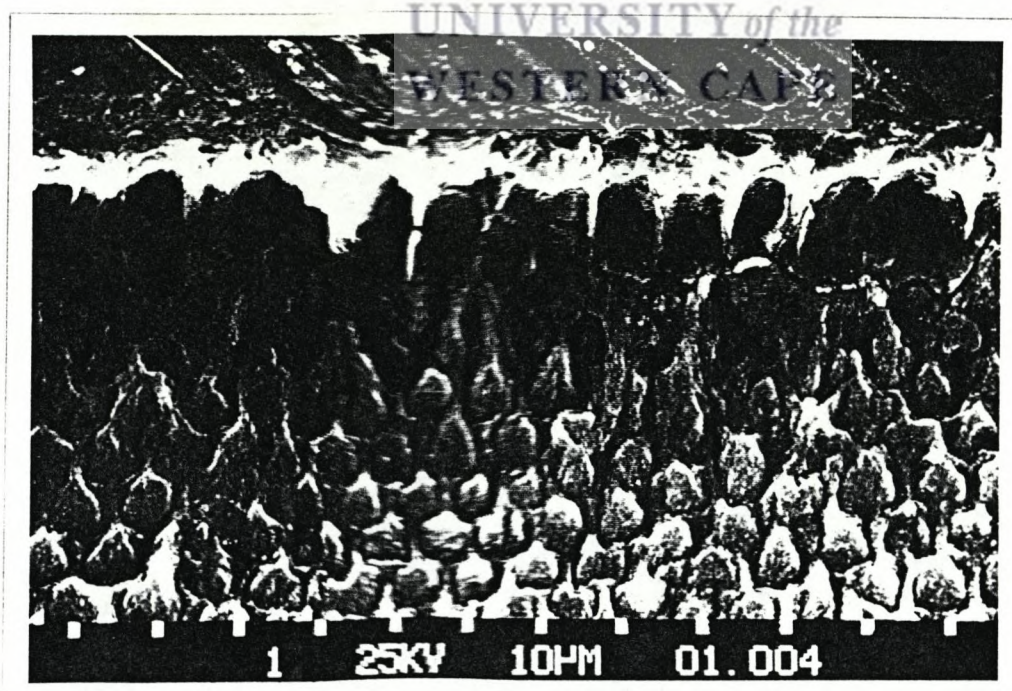


FIGURE NO:36. Resin tag formation at 1 K. (arrowed).



2.21 Concise Interfacial Zone.

Fig 37 shows good tag formation at 300 magnification and in an enlargement (by zooming the 300 magnification) in Fig 38. Macroscopic particles of filler were again evident at this magnification.

Higher magnification (Fig 39) shows the tags penetrating into the enamel. Fig 40 at 1 K magnification shows the extent of the tags (approx.20 mu) with what appears to be a resin rich layer at the interfacial zone free of filler.

FIGURE NO:37. Tag formation under low power magnification. (*30).

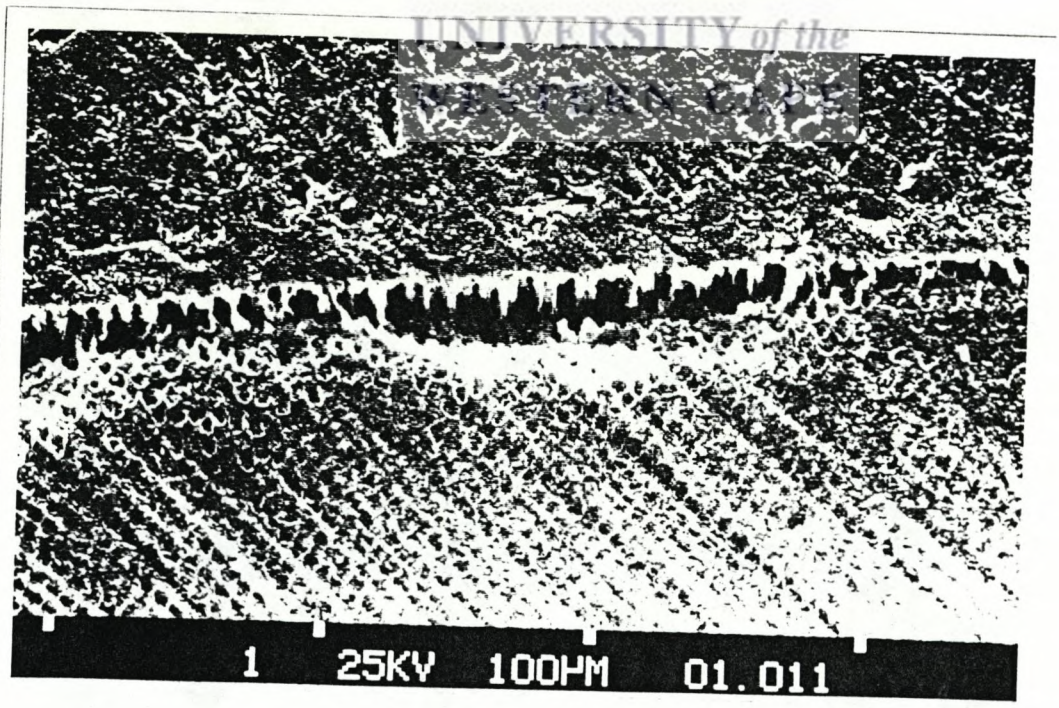


FIGURE NO:38. Tag formation under higher power at 3 K. magnification.

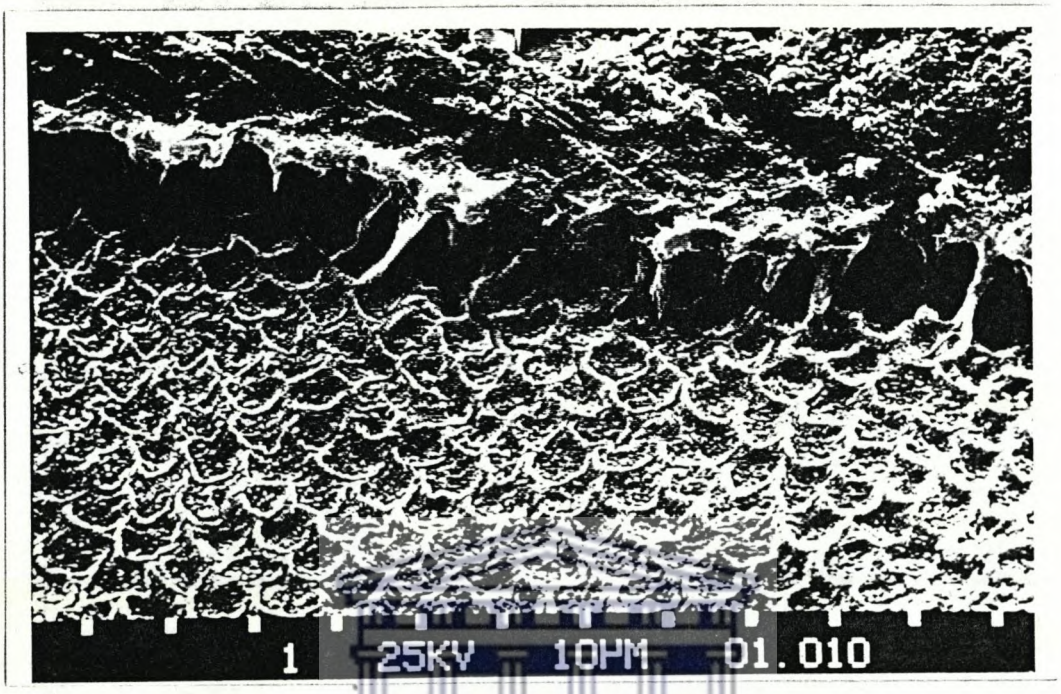


FIGURE NO:39. Tag formation at 3 K. magnification.

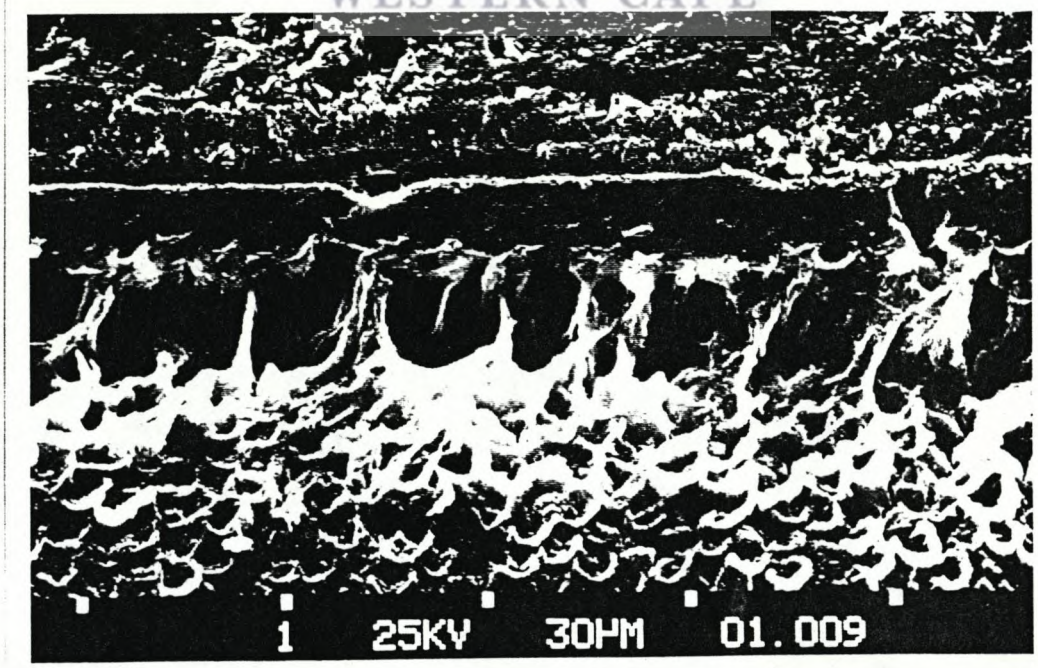
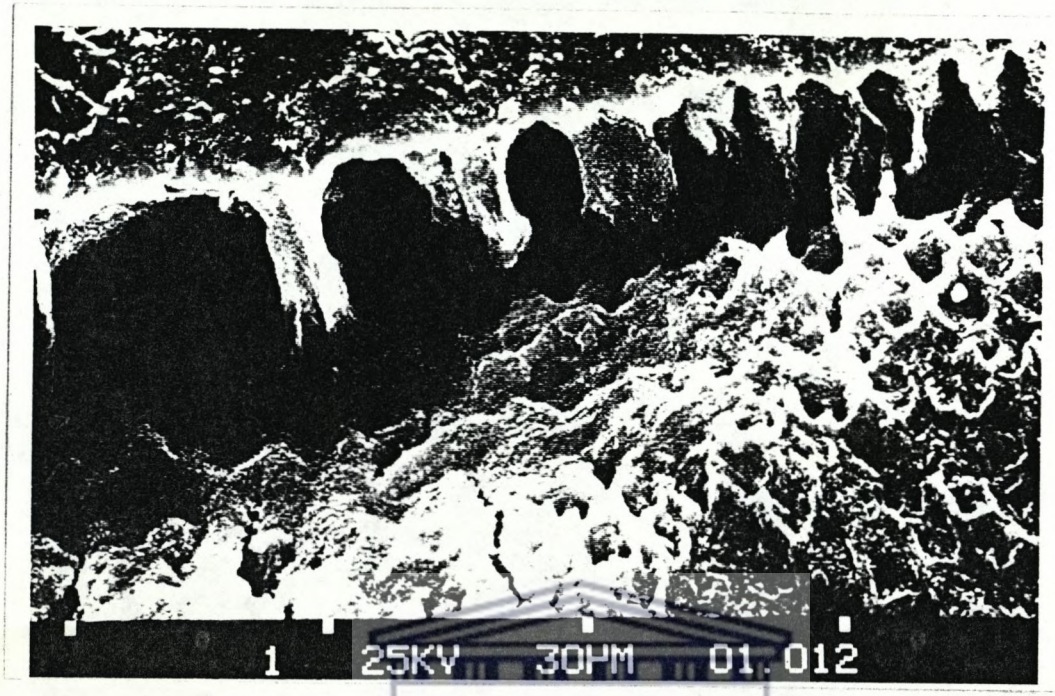


FIGURE NO:40. Tag formation at 1 K magnification.



2.22 Silux Interfacial Zone.

At a magnification of 300 in Fig 41 some well formed but short tag formation can be seen.

At higher magnification in Fig 42 and 43 at 1K the tags can clearly be seen to be shorter than those for the macrofilled resin of Concise.

FIGURE NO:41. Interfacial zone at low power (*30).

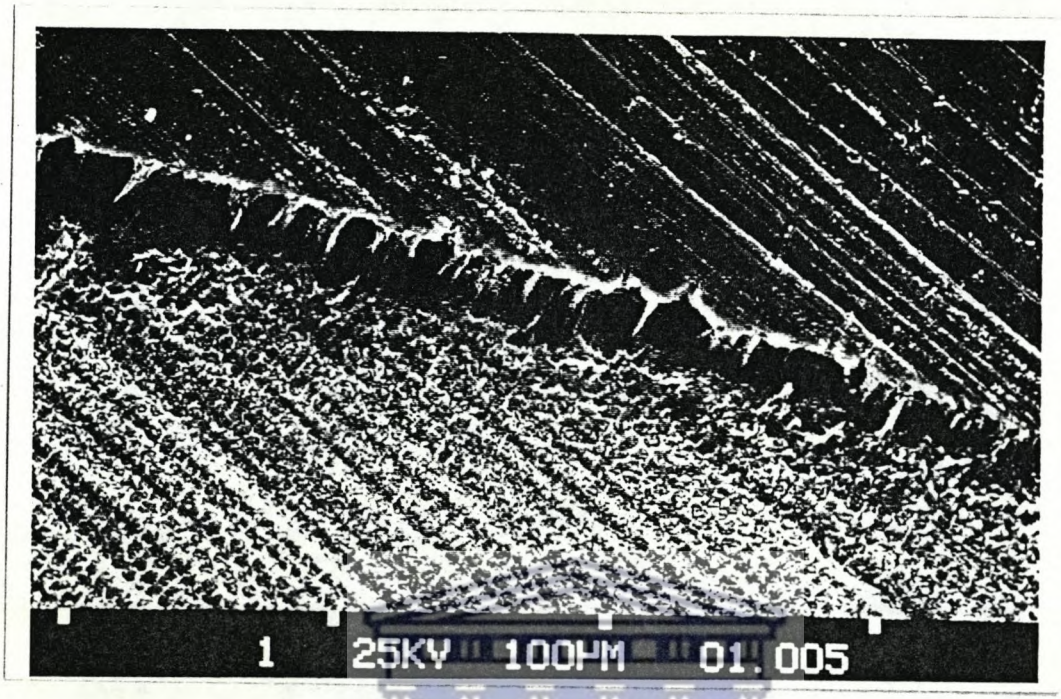


FIGURE NO:42. Interfacial zone at 1 K. magnification.

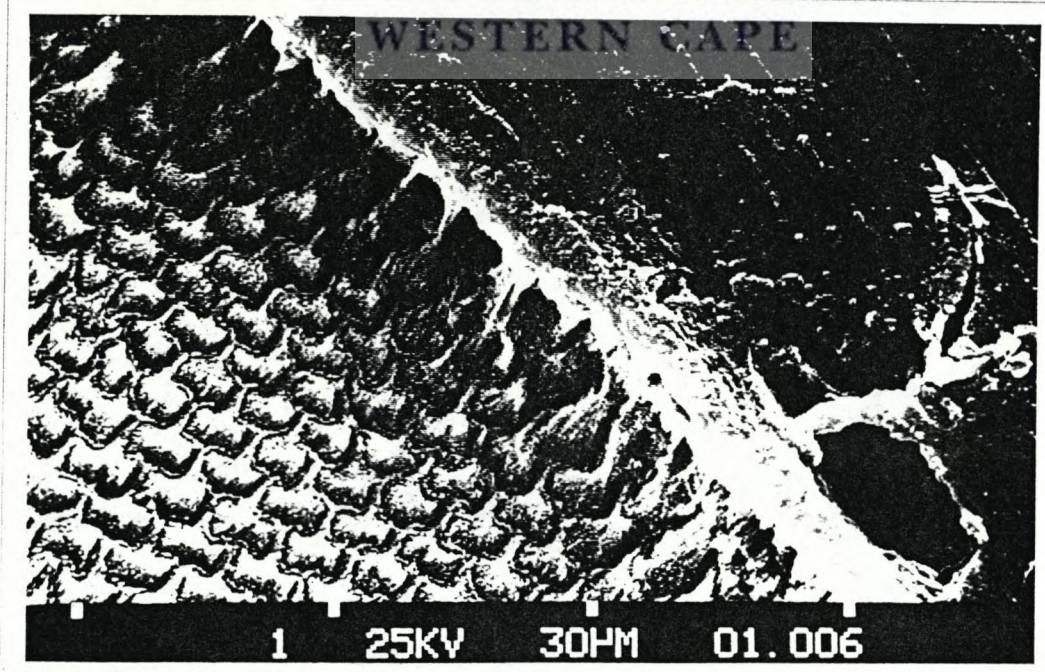
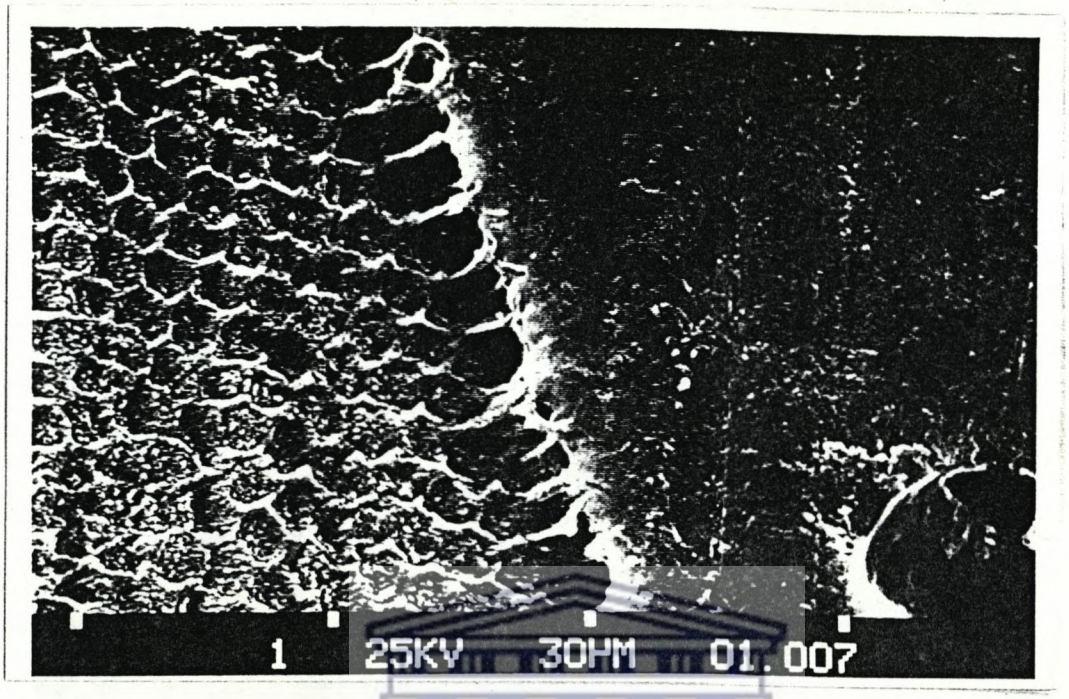


FIGURE NO:43. Interfacial zone at 3 K. magnification.



Summary.

The purpose of this study was to evaluate in vitro, the tensile bond strengths of some conventional and light cured resins, to study the effects of etchants on polished and ground enamel using the scanning electron microscope, and to examine the resin/etched enamel interface by scanning electron microscopy.

This study was divided into: 1) laboratory tensile bond strength study of four composite resin materials. 2) a scanning electron microscopic study of the effect of four etchants on enamel. 3) the S.E.M. examination of the interfacial zone after the application of a demineralizing agent.

The tensile bond strength study consisted of testing the bond strengths of two macrofilled and two microfilled resins in common use. These resins were applied directly to the etched enamel surfaces of extracted human teeth by utilizing newly developed perpex stubs which allowed a uniform surface area and for light curing of the resin.

In this laboratory study the extracted teeth and the composite filled stubs were stressed to failure of the material bond to tooth in an Instron Machine. Bond strengths were recorded and tabulated in Newtons/mm2.

The application of statistical analysis by Analysis of Variance and Duncans Multiple Range Test showed that there was a significant difference in the bond strengths of some of the four materials.

The scanning electron microscope study consisted of an examination of etched enamel, either polished or ground using the etchants supplied by the two manufacturers i.e. 3 M Dental Company and J&J Dental Company.

The interfacial zones of the resin/tooth interface systems were examined, using the two macrofilled and the two microfilled resins in the study. Differences were found to exist at this zone.

Conclusions.

Tensile Bond Strength Study.

1) There were significant differences between the tensile bond strengths of the macrofilled and the microfilled resins.

2) The area of failure in most cases occured within the body of the resin, showing that the interfacial bonding was stronger than the cohesive strength of the material itself.

3) Statistics showed a significant difference between the bond strengths of the some four materials.

4) The difference in the fracture sites of the macrofilled resins and the microfilled resins indicates how the increased filler loading affects the elastic properties of the material.

5) In areas of high stress therefore a material which is of the microfilled type is contra-indicated.

Scanning Electron microscope study.

Enamel:

1) The etched pattern of ground enamel appeared more defined than that of polished enamel.

2) Different types of etch patterns were observed (class 1, 2 & 3) but they were not predictable.

Tags:

3) Cup and cone like patterns of resin tags were formed on tranversely cut etched enamel, as seen on the interfacial zones of specimens studied.

4) The average tag length of the macrofilled resins was approximately twice as long as the tags of the microfilled resins. The macrofilled resins exhibited tags of 10-20 um while the microfilled resins showed tags in the order of 5-10 um.

Interfacial zone:

5) This zone exhibited that adequate mechanical bonds were obtained irrespective of the etch pattern, that are necessary for practical applications of the material in dentistry.

BIBLIOGRAPHY.

Aker,A.A., Aker,J.R. and Sorensen,S.E. (1979) Effects of methods of tooth enamel preparation on the retentive strength of acid etch composite resins. Journal of the American Dental Association.99:185-189.

Arends,J., v.d.Veen,H., v.Groeningen,G. and Schuthof,J. (1984) Quantified marginal leakage of composites in vitro. Journal of Oral Rehabilitation.12:229-234.

Asmussen,E. (1985) Clinical relevance of physical, chemical and bonding properties of composite resins. Operative Dentistry.10:61-73.

Bates,D., Retief,D.H., Jamison,H.C. and Denys,F.R. (1982) Effects of acid etch parameters on enamel topography and composite resin-enamel bond strength. Pediatric Dentistry.4(2):106-110.

Bounocore,M.G. (1955) A simple method of increasing the adhesion of acrylic filling material to enamel surfaces. Journal of Dental Research.34:849-853.

Bounocore,M.G. (1965) New anterior restorative materials. International Dental Journal.18:406-420.

Bounocore,M. (1981) Retrospections on bonding; Symposium on composite resins in dentistry. Dental Clinics of North America.25(2):241-255.

Bowen,R.L. (1962) Dental filling material comprising vinyl silane treated fused silica and a binder consisting of the reaction product of bisphenol and glycidyl acrylate. U.S. Patent No.3,066,112.

Bowen,R.L., Nemoto,K. and Rapson,J. (1983) Adhesive bonding of various materials to hard tooth tissues: forces developed in composite materials during hardening. Journal of the American Dental Association.106:475-477.

Brannstrom,M. and Nordenvall,K.J. (1977) The effects of acid etching on enamel, dentin and the inner surface of the resin restoration: A scanning electron microscope investigation. Journal of Dental Research.56(8):917-923.

Bryant,S., Retief,D.H., Bradley,E.L. and Denys,F.R. (1985) The effect of topical flouride treatment on enamel flouride uptake and the tensile bond strength of orthodontic bonding resin. American Journal of Orthodontics.87(4):294-302.

Daniel,W.W. (1983) Biostatistics: A foundation for analysis in the health sciences. Publishers:John Wiley and sons.Third edition:206-224.

Davidson,C.L., de Gee,A.J. and Feilzer,A. (1984) The competition between the composite - dentin bond strength and the polymerization contraction stress. Journal of Dental Research.63(12):1396-1399.

Denys,F.R. and Retief,D.H. (1982) Variations in enamel etching patterns produced by different concentrations of phosphoric acid. Journal of the American Dental Association.37:185-189.

Dogon,I.L. (1976) The influence of viscosity on the penetration of resin into acid etched enamel. Abstract No. 307 (Delivered at) The annual meeting of the American Association for Dental Research.

Farley,E.P., Jones,R.L. and Anbar,M. (1977) Improved adhesion of acrylic restorative materials to dental enamel by precoating with monomer containing phosphonate groups. Journal of Dental Research.50(8):943-952.

Gross,J.D., Retief,D.H. and Bradley,E.L. (1985) Microleakage of posterior composite restorations. Dental Materials.1:7-10.

Gottlieb,E.W., Retief,D.H. and Jamison,H.C. (1982) An optimal concentration of phosphoric acid as an etching agent. Part 1: Tensile bond strength studies. Journal of Prosthetic Dentistry.48(1):48-51.

Gwinnett,A.J. (1981) Acid etching for composite resins. Dental Clinics of North America.25(2):271.

Gwinnett,A.J. and Buonocore,M.G. (1965) Adhesives and caries prevention. A preliminary report. British Dental Journal. 119:77-80.

Hembree,J.H. (1984) Microleakage of microfilled composite resin restorations with different cavosurface designs. Journal of Prosthetic Dentistry.52(5):635-656.

Jassem,H.A., Retief,D.H. and Jamison,H.C. (1981) Tensile and shear strengths of bonded and rebonded orthodontic attachments. American Journal of Orthodontics.79:661-668.

Jorgensen,K.,and Shimokobe,H. (1975) Adaption of resinous restorative materials to acid etched enamel surfaces. Scandanavian Journal of Dental Research.83:31-36.

Joseph,V.P. and Cohen,J. (1986) Porosities of chemically cured and light cured composite resins. I.A.D.R. Abstract No.17. (Delivered at) The annual meeting of the I.A.D.R.

Kemper,R.N. and Kilian,R.J. (1976) New system for tensile bond strength testing. Journal of Dental Research. 55B:B138(abstract 308).

Lambrechts,P. (1983) Basic properties of dental composites and their impact on clinical performance. Thesis submitted at Leuven.(Belgium).

Lees,S., Trombly,P.L., Skobe,Z., Gapiepy,E.E. and Trull,A.E. (1979) Modifications of etching patterns in bovine dental enamel. Journal of Dental Research.58(8):1840-1847.

Low,T., Davies,E.H. and Von Fraunhofer,J.A. (1975) A method of determining the tensile bond strength of fissure sealants. Journal of Oral Rehabilitation.2:341-347.

Low,T., Lee,K.W. and Von Fraunhofer,J.A. (1978) The adaption of composite resin materials to etched enamel surfaces. Journal of Oral Rehabilitation.5:349-355.

Manson-Rahemtulla,B., Retief,D.H. and Jamison,H.C. (1984) Effect of concentrations of phosphoric acid on enamel dissolution. Journal of Prosthetic Dentistry. 51(4):495-498.

Maijer,R. and Smith,D.C. (1977) A new surface treatment for bonding. I.A.D.R. Abstracts. 56:A152.

McCabe J.F. (1984) Developments in composite resins. British Dental Journal. 157:440.

Mitchem,J.C. and Turner,L.R. (1974) The retentive strengths of acid etched retained resins. Journal of the American Dental Association. 89:1107-1110.

Munechika,T., Suzuki,K., Nishiyama,M., Ohashi,M. and Horie,K. (1984) A comparason of tensile bond strengths of composite resins to longitudinal and transverse sections of enamel prisms in human teeth. Journal of Dental Research. 63(8):1079-1082.

Ortiz,R.F., Phillips,R.W., Swartz,M.L. and Osborne,J.W. (1979) Effect of composite resin bond agents on microleakage and tensile bond strength. Journal of Prosthetic Dentistry. 41(1):51-57.

Pahlavan,A., Dennison,J.B. and Charbeneau,G.T. (1976) Penetration of restorative resin into acid etched human enamel. Journal of Dental Association of America. 93:1170-1176.

Pilliar,R.M., Smith,D.C. and Maric,B. (1984) Oscillatory wear tests of dental composites. Journal of Dental Research. 63(9):1166-1172.

Poole,D.F. and Johnson,N.W. (1972) The effects of different demineralizing agents on human enamel surfaces studied by scanning electron microscopy. Archives of Oral Biology. 12:1621-1634.

Prevost,A.D., Fuller,J.L. and Peterson,L.C. (1982) The use of an intermediate resin in the acid etch proceedure: retentive strengths, microleakage and failure mode analysis. Journal of Dental Research.61(2):412-418.

Prevost,A.P., Fuller,J.L. and Peterson,L.C. (1984) Composite and intermediate resin tag formation in the acid etched enamel: A scanning electron microscope evaluation. Journal of Prosthetic Dentistry. 52(2):204-207.

Raadal,M. (1978) Bond strength of composites applied to acid etched enamel. Scandanavian Journal of Dental Research. 86:157-162.

Risnes,S., and Stolen,S.O. (1981) Uncoated specimen of human enamel observed in the scanning electron micrscope. Scandanavian Journal of Dental Research.89:205-212.

Retief,D.H. (1973) Effect of conditioning the enamel surface with phosphoric acid. Journal of Dental Research.52:333-341.

Retief,D.H. (1974) A comparative study of three etching solutions. Journal of Oral Rehabilitation. 1:381-390.

Retief,D.H. (1974) Failure at the dental adhesive -etched enamel interface. Journal of Oral Rehabilitation. 1:265-284.

Retief,D.H. (1978) The Mechanical bond. International Dental Journal.28(1):18-27.

Retief,D.H., Bishoff,J. and van der Merwe,E.H.M. (1976) Pyruvic acid as an etching agent. Journal of Oral Rehabilitation. 3:245-265.

Retief,D.H. and Dreyer,C.J. (1967) Epoxy resin for bonding orthodontic attachments to teeth. Journal of the Dental Association of South Africa. 22(11):338-346.

Retief,D.H. and Jamison,H.C. (1982) Effect of cavo-surface treatment on microleakage in class five composite resin restorations. Journal of Prosthetic Dentistry. 47(5):496-501.

Retief,D.H. and Mallory,W.P. (1981) Evaluation of two pit and fissure sealants:an in vitro study. Paediatric Dentistry.3:12-16.

Retief,D.H., Rutland,J.R. and Jamison,H.C. (1982) Microleakage of conventional and microfilled restorative resins.Journal of Biomedical Materials Research. 16:901-910.

Retief,D.H. and Woods,E. (1982) Is a low viscosity bonding resin necessary?. Journal of Oral Rehabilitation.8:255-266.

Retief,D.H.,Woods,E. and Jamison,H.C. (1982) Microleakage of selected composite restorative resins. Journal of Dentistry.10(4):292-299.

Retief,D.H., Woods,E., Jamison,H.C. and Denys,F.R. (1982) Surface topography of the enamel margins of butt and bevelled class five cavity preperations. Journal of Prosthetic Dentistry.48(2):166-170.

Schneider,P.M., Messer,L.B. and Douglas,W.H. (1981) The effect of enamel surface reduction in vitro on the bonding of composite resin to permanent human enamel. Journal of Dental Research 60(5):895-900.

Short,G.M. Hembree,J.H. and McKnight,J.P. (1976) The bond strengths of resin systems to etched enamel. Journal of Prosthetic Dentistry. 36(5):538-543.

Silverman,L.M., Saxton,C.A., Dogon,I.L. and Fejerskov,O. (1975) Variation in the pattern of human dental enamel examined by scanning electron microscopy. Caries Research.9:373-387.

Soetopo, Beech,D.R. and Hardwick,J.L. (1978) Mechanisms of adhesion of polymers to acid-etched enamel. Journal of Oral Rehabilitation.5:69-80.

Torney,D.L., Denehy,G.E. and Teixeira,C.D. (1977) The acid etch class three composite resin restoration. Journal of Prosthetic Dentistry. 38(6):623-626.

Tyler,J.E. (1976) A scanning electron microcsope study of factors influencing etch patterns of human enamel. Archives of Oral Biology. 21:765-769.

Voss,J.E. and Charbeneau,G.T. (1979) A scanning electron microscope comparason of three methods of bonding resin to enamel rod ends and longitudinally cut enamel. Journal of the American Dental Association. 98:384-389.

Van Groeningen,G., Jongebloed,W. and Arends,J. (1985) Composite resins after one and a half years of in vivo abrasion:an scanning electron microscope investigation. Quintessence International. 4:253-258.

Welsh,E.L. and Hembree,J.H. (1985) Microleakage at the gingival wall with class four anterior restorative materials. Journal of Prosthetic Dentistry.53(3):370-372.

Zidan,O., Asmussen,E. and Jorgensen,K.D. (1982) Microscopic analysis of fractured restorative resin/etched enamel bonds. Scandanavian Journal of Dental Research.90:286-291.

Zisman,W.A. (1963) Influence of constitution on adhesion. Ind. Eng. Chem. 55:19-38.