PHYSICAL STUDIES

OF

DENTAL PIT AND FISSURE SEALANTS

A Thesis submitted to the University of Glasgow for the degree of Doctor of Philosophy

By

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SUMMARY

The application of sealants to pits and fissures on occlusal surfaces of teeth as a caries preventive measure has been evaluated in numerous clinical studies. However, the duration of sealant cover and the caries reductions found in these different trials were extremely variable, even where the same materials, and ostensibly the same techniques, had been used. The primary objective of this study was to investigate physical factors involved in applying sealants and to devise changes in materials and application procedures to ensure long term sealant retention and caries reduction. A second objective was to explain the very poor retention found by Stephen, Sutherland and Trainer (1976) for the TP2206 (Alphaseal) fissure sealant system.

The effects of various clinical factors, including moisture contamination and surface treatments, on the adhesion between sealant and enamel were assessed using a new tensile testing technique. These tests also indicated the importance of applying sufficient u.v. radiation to set the resins. A subsequent investigation of the output and distribution of radiation from u.v. sources supplied for clinical use, showed a wide range in performance for different units of the same type. In particular, one of the sources used by Stephen et al. (1976) had a very low output.

Studies with microhardness measurements showed that Nuva-seal set more rapidly and to a greater final hardness when higher u.v. intensities were used. Alphaseal set more slowly than Nuva-seal, and did not set readily in depth. U.V. absorption measurements indicated that the transmission through Nuva-seal was sufficient to allow setting to the depths normally expected of fissure sealants. Alphaseal showed much stronger absorption, which was partly due to a fluorescent dye incorporated to aid long term sealant detection, and partly due to the

high concentration of catalyst used (5% w/w).

The setting behaviours of three u.v. activated sealants (Nuva-seal, Alphaseal and Nuva-cote) were compared using a new method, which relied on the passage of acoustic vibrations to determine the set. The nature of the dependence of the rate of set on the u.v. intensity was similar for all three materials. However at any given intensity Nuva-seal set faster than Nuva-cote and Alphaseal by factors of about 4 and 13, respectively. The slow setting of Alphaseal was largely due to the strong absorption of the u.v. radiation in the surface layers. Removing the fluorescent dye and reducing the catalyst concentration to 1% w/w, resulted in an increase in the rate of set of Alphaseal by a factor of 10.

Thus it was concluded that the poor retention found by Stephen et al. (1976), was probably due to the low output of the u.v. sources used, and the inadequate setting of the TP2206 resin, due to the strong absorption of the polymerising radiation in the surface layers.

A replication technique for use with the Scanning Electron Microscope was developed to study in vivo changes in sealant morphology on selected teeth. In an eight month study of the retention of Nuva-seal, material was observed to be lost by abrasion over the entire sealant surface, and by occasional brittle fracture which resulted in the loss of relatively large fragments. Retention of thick layers of sealant appeared to be superior to thinner applications, and premolar surfaces retained the sealant better than molars.

Based on all these studies, an improved protocol for sealant application was developed and its effectiveness confirmed by a clinical trial using the Nuva System. Here, a dental auxiliary achieved sealant retention, over one year, which was superior to that found in all previous trials except that reported by Helle (1975). Blind

examinations after 6 and 12 months revealed 98% and 93% complete retention in first permanent molars, which have been shown to be the most difficult, yet most desirable, to seal.

CHAPTER ONE

LITERATURE REVIEW AND AIMS OF PRESENT INVESTIGATION

1.1 Introduction

Increasingly, preventive techniques are sought as an alternative approach to the problem of dental caries. Various methods of fluoride treatment have demonstrated considerable success in preventing caries. However, a number of studies have shown that such fluoride treatments are least effective in preventing the development of pit and fissure caries in molars and premolars. (Ast et al., 1956; Backer-Dirks, Houwink and Kwant, 1961; Blayney and Hill, 1967). However, pit and fissure lesions represent a significant portion of observed carious sites, particularly in the first permanent molars of six to nine-year-old children. Thus the extremely rapid development of fissure caries in first permanent molars following their eruption in six-year-olds, has been recognised as a major problem in the dental care of young children (Hargreaves, 1964).

The complex morphology of pits and fissures prevents an adequate self-cleansing process and cleaning with a toothbrush is quite ineffective. Hence these sites form havens for the accumulation of debris and micro-organisms. Fed with nutrients from our high sucrose diets, some intra-oral bacteria produce acids which cause the carious destruction of adjacent enamel. It has long been appreciated that the sealing of pits and fissures from the oral environment might prevent occlusal caries. Not only would the accumulation of bacteria be prevented, but any organisms accidentally sealed into the fissures would be isolated from their supply of dietary nutrients.

1.2 Caries Susceptibility of Occlusal Pits and Fissures

In a review of the dental literature Hyatt (1930) noted that pits and fissures had been associated with a high incidence of occlusal caries as early as the 19th century. Since then the high incidence and rapid development of occlusal caries has been well documented (Day and Sedwick, 1935; Grainger and Reid, 1954; Parfitt, 1955; Barr, Diodatti and Stephens, 1957; Backer Dirks, 1961; Hargreaves, 1964; Berman and Slack, 1973).

Parfitt (1955) examined the caries distribution in children aged from 2 to 15 years. The greatest proportion of caries in the permanent dentition was found on occlusal surfaces of first and second molars. The two most commonly found carious sites for children of 8 years and over were the occlusal surfaces of upper and lower first permanent molars, which represented at least 30% of the caries present in any of the age groups.

Day and Sedwick (1935) stated that occlusal decay in 13 year old children represented 45% of the caries in the permanent dentition. Hargreaves (1964) found that up to 12 years, the first molar was the main tooth affected by caries in the permanent dentition, with the initial attack in the pits and fissures, approximal lesions occurring later.

The incidence of caries in different tooth sites was studied by Backer Dirks (1961) in a longitudinal study of a group of children from 9 to 15 years of age. The distribution of caries at age nine is shown in Fig. 1.1. Here, pit and fissure lesions had developed in 85% of the upper and 70% of the lower first permanent molars.

Berman and Slack (1973) concluded that the occlusal surfaces of all teeth, with the possible exception of the lower first premolar, showed a high degree of susceptibility and rapidly developed caries in advance of approximal and smooth surfaces.

Thus the dental needs of primary school children over the age of

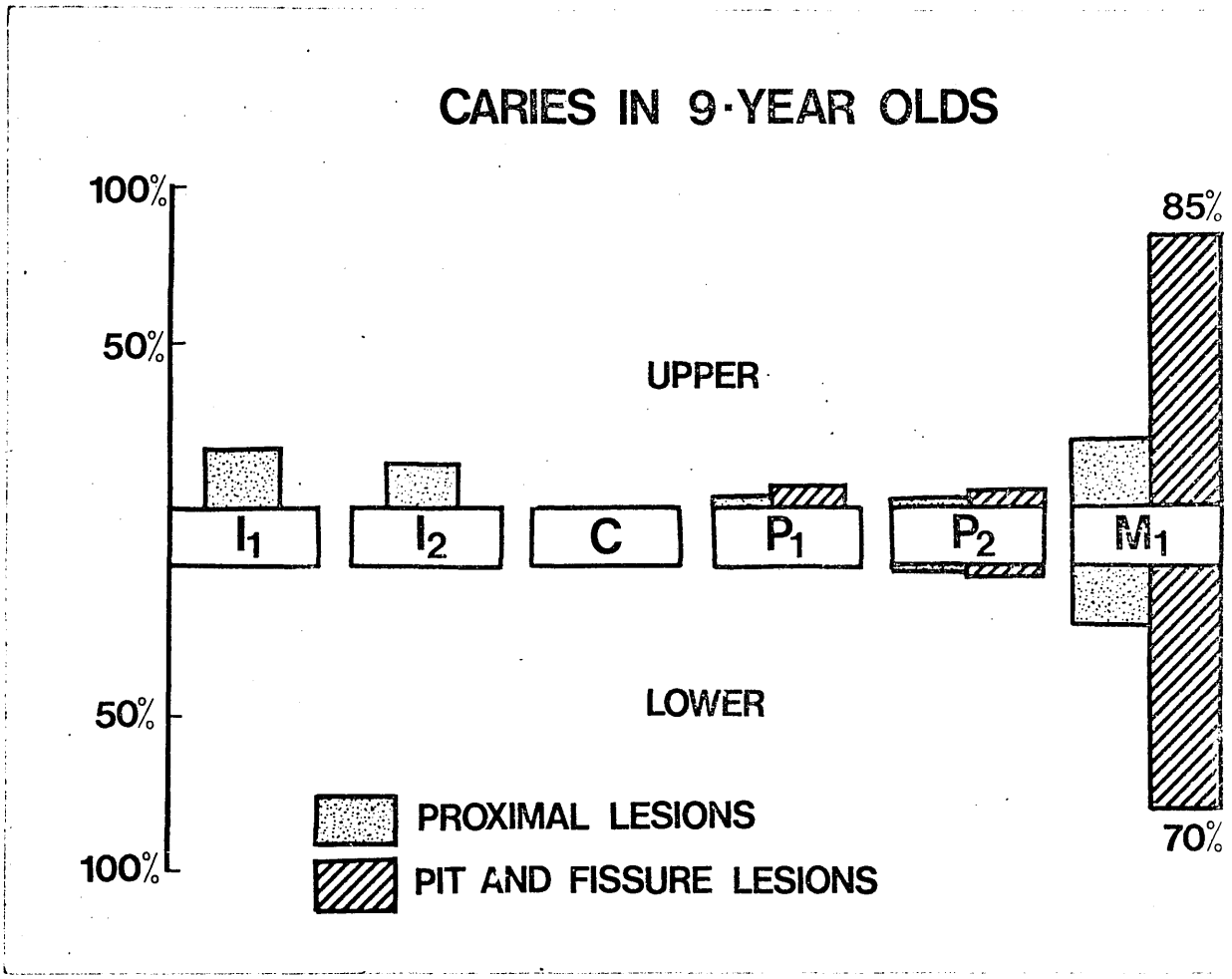


Fig. 1.1 The incidence of proximal and fissure carious lesions in a group of nine-year-old children is shown for upper and lower incisors (I_1,I_2), canines (C) premolars (P_1,P_2) and first permanent molars (M_1) (Backer Dirks, 1961).

six and dominated by the rapid development of fissure caries in first permanent molars. Lewis and Hargreaves (1975) emphasised that fissure sealants should be applied to first permanent molars as soon as possible after eruption.

1.3 The Effect of Fluoride on Pit and Fissure Caries

Fluoride is now widely used in a variety of forms for the prevention of caries, and its effectiveness has been well established. Therefore one may ask whether there is a role for fissure sealants to play in preventive dentistry. However, the most successful trials have indicated that sealants have the potential to completely prevent occlusal caries - albeit with regular re-applications of sealant. Such a potential has never been claimed for fluoride treatments, where one might expect a reduction of about 50% in the overall caries levels. fluoride has also been shown to be less effective in preventing caries of pits and fissures than on smooth surfaces and interproximal areas. Ripa (1975) noted that this effect has been demonstrated where the fluoride was present in toothpaste (Muhler et al., 1954), in table salt (Marthaler and Schenardi, 1962), in tablet form (Marthaler, 1967 and 1969), in a topical gel (Englander et al., 1969) in prophylactic paste (Peterson et al., 1969) or in the community water supply (Ast et al., 1956; Backer Dirks et al., 1961; Blayney and Hill, 1971). Therefore many authors have stressed that sealants should be used in conjunction with other preventive systems such as fluoride treatments, since there would be little benefit in preserving the occlusal surface while allowing the others to decay (Buonocore, 1975; Ripa, 1976).

1.4 The Acid Etch Technique

The use of an acid conditioning treatment to improve adhesion to enamel was first advocated by Buonocore (1955). An 85% concentration of phosphoric acid was applied to enamel for 30 seconds prior to being

washed off. The increased adhesion following such an acid treatment has been attributed to a number of factors:

(a) a tremendous increase in the surface area due to etching;

(b) exposure of the organic material of enamel to which the resin can adhere;

(c) removal of old, fully reacted and inert enamel surface to expose a more reactive surface;

(d) adsorption of highly polar phosphate groups from the acid;

(e) increase in the wettability of the surface (Gwinnett and Buonocore, 1965).

It was about ten years after the pioneering work of Buonocore (1955) that materials were developed to take advantage of the "acid etch technique". These are now used in a wide range of adhesive dental procedures including composite restorative materials, direct orthodontic bonding systems and pit and fissure sealants. Current systems employ a variety of acid etch procedures, the Nuva-System (L.D. Caulk Co.) using a one minute application of 50% w/w phosphoric acid buffered by 7% by weight zinc oxide. A similar time with 30% w/w phosphoric acid is recommended for the Alphaseal system (Amalgamated Dental Co.) whereas Epoxylite 9075 (Lee Pharmaceuticals) requires the enamel to be exposed to a 50% w/w phosphoric acid solution for only 30 seconds, and Concise Enamel Bond (3M Co.) uses a 37% w/w concentration of phosphoric acid applied for one minute. Such a wide range in acid etching techniques suggests that there is little agreement on which is the best method.

Prism-like tags of sealant penetrating into acid conditioned enamel surfaces have been observed in sections of sealed teeth by many research workers (Gwinnett and Matsui, 1967; Buonocore, Matsui and Gwinnett, 1968; Sharp and Grenoble, 1971; Sheykholeslam and Buonocore, 1972; Gwinnett and Ripa, 1973; Retief, 1973; Silverstone, 1974; Jorgenson

and Shimokobe, 1975; Soetopo, 1975). Reports on the lengths of these tags have varied from about 9 μm (Jorgenson and Shimokobe, 1975) up to 60 μm (Silverstone, 1974). A wide range of enamel adhesives have been found to penetrate acid-conditioned enamel, although Dogon (1976) showed that the length of tags formed depended on the viscosity of the resin. Sealant penetration has also been shown to vary from one tooth to another and from one site to another on the same surface (Gwinnett and Matsui, 1967). The duration of exposure, concentration and type of acid used to condition the enamel also affects the sealant penetration (Silverstone, 1974; Soetopo, 1975).

The extent of sealant penetration into partially decalcified enamel was demonstrated at the microscopic level with a Transmission Electron Microscope (T.E.M.) by Simmelink, Nygaard and Scott (1974), who concluded that the network of resin not only surrounded the crystals of hydroxyapatite but penetrated and polymerised in their dissolved cores.

The Scanning Electron Microscope (S.E.M.) has been used to examine the appearance of enamel following acid conditioning, and the undersurface of sealant after demineralisation of the enamel (Gwinnett, 1971a; Gwinnett and Buonocore, 1972; Hoffman, 1972; Gwinnett and Ripa, 1973; Myers, Rossi and Cartz, 1974; Silverstone et al., 1975; Marshall, Olsen and Lee, 1975; Jorgenson, 1975; Jorgenson and Shimokobe, 1975; Kochavi, Gedalia and Anaise, 1975). In particular this technique has been used to compare the etching of different concentrations of phosphoric acid (Ohsawa et al. 1972; Silverstone, 1974; Soetopo, 1975). At low concentrations the degree of etching was found to increase with increasing concentrations of phosphoric acid up to 20 - 40% w/w. Thereafter, further increases resulted in a decreased etching effect.

This phenomenon has been explained by Chow and Brown (1973) as

being due to the deposition of salts on the enamel surface when higher concentrations of phosphoric acid are employed. Marshall et al. (1975) used a scanning electron microscope to show variations in the etching pattern on the occlusal surfaces of individual teeth and between molars and premolars. Enamel was not found to be etched in the region of the pits and fissures, and premolars were more readily etched than molars.

Many authors have presumed that it is possible to deduce the degree of adhesion which an adhesive attains on the basis of the degree of penetration into the enamel. Therefore the presence and length of penetrating tags of sealant has been taken as a measure of the extent of the adhesion attained. In addition, some authors have tried to relate the different degrees of porosity present in surface enamel to the resultant adhesion achieved in vivo. Since there have been very few direct studies to prove these assumptions, the results of tests of sealant adhesion to enamel, using various acid etch procedures, are presented in Chapter Two.

1.5 Remineralisation After Acid Etching

The use of acids to promote adhesion to enamel has caused concern that etched surfaces might be more susceptible to caries. When sealants are applied not all of the etched enamel will be covered with sealant. It has been suggested, however, that these etched surfaces are able to "remineralise" using constituents in the saliva. In a review of the literature, Wei (1967) concluded that although there was much evidence for the existence of "remineralisation" the nature of the process was not fully understood. The recovery of enamel following the use of an acid etch to promote adhesion has been studied by Retief, Dreyer and Gavron (1970), Albert and Grenoble (1971), Arana (1974) and Meurman and Asikainen (1976). Retief et al. (1970) reported that enamel treated with 50% phosphoric acid showed a normal appearance after one week. Albert and

Grenoble (1971) reported that four teeth etched with 50% phosphoric acid for one minute regained their normal appearance after 96 hours, when examined on the Scanning Electron Microscope. Although Meurman and Asikainen (1976) agreed that etched enamel regained its normal appearance within the mouth, they found that it was only rehardened up to 84% of its original value. Closer examination with the Scanning Electron Microscope showed that the enamel surface did not fully regain its intact, non-porous nature since micropores and defects remained in the surface topography during the three week study period. They concluded that rehardening was due to the calcification of salivary deposits in the microspaces left after etching, but pointed out that this renewed enamel surface was not necessarily less resistant to bacterial attacks since it may have an enhanced fluoride content and therefore be more resistant to further decalcification.

It should also be noted that examination of the occlusal surfaces of teeth after acid conditioning has shown that the etch is confined to the cuspal slopes and does not penetrate into the pits and fissures, where occlusal caries usually develops (Marshall et al., 1975). Further evidence that acid etching does not promote occlusal caries is provided by clinical trials with sealants, which show no increase in caries even after the loss of the sealant (Horowitz, Heifetz and Poulsen, 1976).

1.6 Microleakage Studies on Fissure Sealants

Sealants may be regarded as a barrier between occlusal pits and fissures and the oral environment, preventing the accumulation of bacteria in the fissures and eliminating the supply of nutrients to any bacteria already present. If sealants allow significant microleakage from the oral environment their caries preventive effect may be lost. Therefore a variety of techniques, which have previously

been used to assess the marginal leakage of restorative dental materials, have been used to determine the microleakage allowed by fissure sealants. Although the significance of the microleakage levels observed has been difficult to assess, it has been assumed that the lower it is the better, and different materials compared on that basis.

In a review of the literature on the microleakage permitted by restorative materials, Kidd (1976) listed a wide variety of techniques, including the use of dyes, radioactive isotopes, air pressure, bacteria, neutron activation analysis, artificial caries and scanning electron microscopy. Many of these methods have now been adapted for the study of fissure sealants. The first study of fissure sealant microleakage was by Khowassah and Sahs (1966), when methyl-2-cyanoacrylate adhesive was shown to have superior sealing ability than three dental cements using an in vitro dye penetration technique.

The sealing effectiveness of cyanoacrylate adhesives was confirmed by Buonocore, Matsui and Gwinnett (1968) using a dye and radioactive sulphate to compare the sealing effectiveness of a cyanoacrylate adhesive, a self-curing methyl methacrylate resin, and silicate and zinc phosphate cements. After storage for 6 and 12 months neither of the two cements remained bonded to enamel specimens, but both the cyanoacrylate and methyl methacrylate materials prevented the penetration of the radio-isotope and the dye along the adhesive/enamel interface where an acid conditioning treatment had been used.

Lee and Swartz (1971) investigated the sealing effectiveness of a polyurethane resin by examining radioautographs of 30 sectioned teeth which had been stored at 37°C for 7 days after sealing and immersed in a solution of Calcium 45. Although the isotope was detected beneath the fissures in seven specimens, this was attributed to improper technique.

Woody, Moffa and McCune (1972) used a similar method to show that

microleakage was prevented by a u.v. activated dimethacrylate (presumably Nuva-seal), but not by the polyurethane, cyanoacrylate and polycarboxylate materials which they tested. Rudolph, Phillips and Swartz (1974) demonstrated sealing effectiveness of two commercial sealants (Nuva-seal and Epoxylite 9075) and one experimental diacrylate sealant. Sixty teeth sealed with each material exhibited little or no leakage of isotope, even when stored for three months and thermally cycled. Martinez, Cooley and Greener (1974) compared the sealing effectiveness of three sealants by a scanning electron microscope examination and a resistance test, on teeth sealed in the laboratory. They found that Nuva-seal and Epoxylite 9075 failed in 25 out of 100 cases immediately after placement, with subsequent temperature cycling and toothbrushing producing no further failures. Dennison et al. (1974) tested three sealant materials for their ability to reduce the uptake of dysprosium, detected by neutron activation analysis. The authors did not state that leakage was prohibited, but asserted that the fluid exchange detected was minimal for all the materials (a bis-GMA resin, a- cyanoacrylate and polyurethane).

Williams, Fraunhofer and Winter (1975a) used dye penetration and zero resistance current measurements to study the microleakage of five fissure sealants. They found that the polymeric materials (Nuva-seal, Epoxylite 9075 and Espe 717) resisted microleakage through the body of the sealant, and when leakage did occur it was along the sealant/enamel interface. However, Aspa and Poly F cement-like sealants showed microleakage through the body of the material as well as at the sealant/enamel interface.

Thus the studies carried out with dyes and isotopes found that diacrylate fissure sealants did not generally allow microleakage, while other adhesives showed poorer sealing ability. Only Martinez et al. (1974) observed leakage with the diacrylates, which they attributed to

incomplete polymerisation of Nuva-seal and shrinkage of Epoxylite 9075. Although microleakage experiments have been able to compare the sealing effectiveness of different materials in the laboratory, the difficulties involved in realistically simulating an oral environment limit any deductions about clinical performance.

1.7 Bacteriological Studies on Fissure Sealants

Sealing is normally applied to caries free occlusal surfaces. However, because of the difficulty in detecting early caries development it is likely that many incipient lesions are inadvertently sealed. Bacteria may also enter the fissures by penetrating between sealant and enamel. Therefore it is important to determine whether bacteria can survive under fissure sealants, and whether they retain the capacity to promote caries.

Pink (1972) demonstrated bacterial penetration around fissure sealants using an in vitro technique. Handelman (1976) has suggested that the leakage observed by Pink may have been due to cracks in the enamel produced at the time of extraction. However, Mednick, Loesche and Corpron (1974) found bacterial leakage around sealants using an in vivo technique. Sterile paper points which were sealed into cavities with Nuva-seal were found to be contaminated in 10 of the 19 teeth which were sealed for periods of 21 to 116 days. Although no precise estimate was made, Mednick and his co-workers concluded that the magnitude of bacterial leakage around sealants was rather small.

Since bacterial leakage seems to occur, and bacteria may well be sealed into fissures, the viability of any retained organisms is important. Handelman, Buonocore and Heseck (1972) made a quantitative estimate of the fate of bacteria under sealants. Six teeth with fissure caries were sealed with Nuva-seal and the lesions bacteriologically sampled one month later. The count of viable

bacteria in the carious dentine of sealed teeth was reduced fifty-fold below that of nine unsealed control teeth. These preliminary findings were confirmed in later studies of teeth which were sealed for periods up to two years. After an initially rapid fall in the first few weeks the organism count fell gradually until after two years, a thousand-fold reduction was noted (Handelman, Buonocore and Schoute, 1973; Handelman, Washburn and Wopperer, 1975; Handelman, 1976).

Mednick et al. (1974) also evaluated the viability of organisms under sealants by placing paper points contaminated with bacteria into cavities in primary molars and covering with Nuva-seal. An examination of 18 teeth which were extracted after having been sealed for 30 to 73 days, revealed that half the paper points had increased their bacterial content and half had decreased, although details of the numbers involved were not specified. Nevertheless, Mednick and his co-workers concluded that the viability of bacteria under sealants was seriously impaired by the inadequacy of the nutrient supply.

Jeronimus, Till and Sveen (1975) assessed the viability of micro-organisms in carious lesions of teeth which were sealed with three commercial pit and fissure sealants. The lesions were classified as being incipient, moderate or deep and bacteriologically sampled within 10 minutes of sealing and after two, three and four weeks. The retention of the three sealants employed varied considerably, with Nuva-seal being retained in all of the teeth treated, while considerable losses were observed with Epoxylite 9075 and 3M Caries Preventive Treatment. The majority of incipient lesions contained cultivable micro-organisms 10 minutes after the placement of the sealant, although in three instances negative cultures were obtained suggesting that the acid treatments used may have affected the bacteria. Later examinations after two, three and four weeks resulted in negative cultures from the dentine of these incipient lesions whenever the sealant was well

retained. Where sealant was lost the cultures were generally positive. Micro-organisms were found in almost all cases where moderately deep and deep lesions were sealed. However, there was no assessment of any reduction in the number of organisms present, as had been demonstrated by Handelman et al. (1972, 1973, 1975 and 1976).

Thus, current knowledge indicates that fissure sealing will greatly impair the viability of micro-organisms, particularly in superficial lesions. Where deep or moderately deep lesions are sealed the survival of bacteria remains debatable. However, by closing the lesions from the oral environment one expects the supply of fermentable substrates to be greatly reduced, thus perhaps slowing the progress of caries. The arresting of caries lesions deep in the dentine by closure from the oral environment has already been demonstrated by Besic (1943) for amalgam cavities. He concluded that: "It appears as though (a) the carious process in dentine definitely stops or gradually ceases as soon as the lesion is closed from the oral environment even when the organisms remain alive; (b) the bacteria have a tendency to die out; but (c) in 30% of the cases studied positive cultures of streptococci persisted after being sealed for more than a year."

Jeronimus et al. (1975) and Handelman (1976) observed that dentinal caries sealed from the oral environment became dry, dark in colour and of leathery appearance suggesting that this was consistent with the arrestment of the lesion. Micik (1972) demonstrated the arrestment of caries-like lesions with fissure sealants using an in vitro method. Further evidence that caries does not progress under sealants is provided by the numerous clinical trials in which caries development under transparent sealant, such as Nuva-seal, has not been observed.

1.8 Clinical Trials of Sealants

1.8.1 Early attempts at fissure sealing

Although the desirability of sealing fissures has long been appreciated, early attempts to achieve this were unsuccessful. Miller (1905) was the first to put the concept into practice, by applying silver nitrate to close off pits and fissures. Later Hyatt (1923) recommended the preventive application of dental cement immediately following eruption, with later application of amalgam restorations. However, this process of "prophylactic odontotomy" met with protest due to the removal of healthy tissue. Bodecker (1924) proposed an initial application of oxyphosphate cement, and subsequent eradication of the fissures without placement of an amalgam filling. However the dental profession remained unconvinced and declined such radical procedures. Prime (1937) returned to the use of silver nitrate and claimed some success in reducing occlusal caries, but Klein and Knutsen (1942) failed to reproduce this. The use of nitrocellulose to penetrate naturally decalcified areas as proposed by Gore (1939), was not successful. Gottlieb (1948) employed zinc chloride treatments to impregnate occlusal surfaces soon after eruption with some success, but not enough to gain widespread popularity. Less successful was the application of copper cement by Miller (1950).

Thus between 1905 and 1950, numerous materials were tried but none were really successful. The fundamental concept of sealing was valid but the right materials not yet available. However, since 1950 a vast array of synthetic resins and adhesives has been developed in industry, and it seemed that eventually such advances might solve the problem of adhesion to enamel.

Buonocore (1955) improved these prospects when he showed that a phosphoric acid etch enhanced the bonding of methyl methacrylate to enamel. Nonetheless it was twelve years before Cueto and Buonocore

(1967) reported the results of a pit and fissure sealant trial using such an acid etch technique. This trial marked the start of a revival of attempts to seal occlusal pits and fissures in the United States, although the first adhesive sealants to be tested clinically were the cyanoacrylate resins, already under investigation in Japan.

1.8.2 Cyanoacrylate sealant systems

Takeuchi (1964) first reported the use of a cyanoacrylate resin to prevent occlusal caries. He described a method whereby ethyl-2-cyanoacrylate was applied to the tooth and powdered polymethyl methacrylate added to the liquid, and the mixture pushed into fissures. By re-applying the sealant every six months a 90·5% reduction in caries was established over a five year period (Takeuchi et al., 1971). However, Lee and Orlowski (1975) reported that while Ninomiya et al. (1968) also found a considerable reduction in occlusal caries, Nakagaki et al. (1971) failed to confirm Takeuchi's success.

Gwinnett and Buonocore (1965) reported the development of a sealant system in which methyl-2-cyanoacrylate was blended with polymethyl methacrylate powder before being applied to tooth surfaces which had received a treatment with phosphoric acid. Cueto and Buonocore (1967) used this technique to produce an 86·3% reduction in occlusal caries after one year, and Ripa and Cole (1970) confirmed its effectiveness. However, Parkhouse and Winter (1971) failed to reproduce the early success of the American clinical trials. A lively debate ensued in the pages of the British Dental Journal with both Ripa (1971) and Gwinnett (1971b) suggesting that the changes which Parkhouse and Winter had made in the application technique, explained their poor results.

Although these studies with cyanoacrylate resins confirmed the potential of pit and fissure sealants for preventing occlusal caries,

their success was limited by deterioration on exposure to moisture in the mouth and these materials have been superceded by diacrylate sealants.

1.8.3 Diacrylate fissure sealants

Bowen (1963) described the preparation of a monomer comprising the adjunct of bisphenol-A and glycidyl methacrylate (bis-GMA). This monomer has played a major part in the development of new adhesives in dentistry, and is now a major constituent in most of the currently available fissure sealants and composite restorative materials. Roydhouse (1968) reported the first use of a diacrylate fissure sealant, consisting of the bis-GMA monomer diluted 20% by methyl methacrylate (MMA) and cured with benzoyl peroxide. The sealant was applied in a thin layer to the occlusal surfaces, without using acid etching to promote adhesion. A 29% caries reduction was observed 3 years after the initial application, although little bulk retention was apparent.

Buonocore (1970) modified the diacrylate system used by Roydhouse to allow rapid polymerisation on exposure to ultra-violet radiation. Here, three parts by weight bis-GMA, diluted one part by weight MMA was initiated by the addition of 2% benzoin methyl ether. The resin was applied with a brush to enamel surfaces pre-conditioned with a 50% phosphoric acid etch for 60s. After one year 99% of the sealant applications remained intact and occlusal caries was completely prevented. Later, Buonocore (1971) reported that 87% of the permanent teeth and 50% of the deciduous teeth remained covered after 2 years, and 99% and 87% caries reductions were observed, respectively. These successful results represented a considerable advance over all previous trials, both in terms of sealant retention and caries reduction. Using the same system, Rock (1972) could not fully reproduce Buonocore's success with only 54% of treated teeth covered after one year, and a

65% reduction in occlusal caries. However, the initial studies were sufficiently encouraging that the system was marketed as the pit and fissure sealant Nuva-seal (L.D. Caulk Co., Milford, Delaware, U.S.A.). A compact Nuva-lite u.v. source replaced the bulky Spectoline lamp which Buonocore had adopted for his first clinical trial.

The Nuva-seal system has now been evaluated in over 28 independent clinical trials, and is by far the best tested and most popular system in use today. More recently developed materials have yet to exceed it in either popularity or in successful application in clinical trials. The results of these Nuva-seal trials are examined in detail in the next section of this chapter.

Another diacrylate fissure sealant "Epoxylite 9075" (Lee Pharmaceuticals Co. Ltd.) is now available. This system is similar to the formulation used by Roydhouse (1968), with the material being applied in two layers. The first layer contains a peroxide catalyst and the second, an accelerator. The subsequent diffusion of the two layers results in polymerisation and any excess monomer is wiped off. Robb and Garcia (1972) reported complete prevention of occlusal caries one year following the application of the material, with 97% of unfluoridated teeth remaining completely covered. However, Rock (1973) found only 59% retention after six months and 53% after one year. By two years, Rock (1974a) found that 52% of these teeth were still totally covered with an 84% reduction in occlusal caries. Williams and Winter (1976) reported that 49% of pits and fissures treated with Epoxylite 9075 were still covered after one year, and 44% at two years. Both Rock (1974a) and Williams and Winter (1976) found poorer retention than that claimed by Robb and Garcia (1972). In addition to the published trials of Epoxylite 9075, Lee and Orlowski (1975) summarised the findings of a number of research reports by the manufacturers indicating successful

retention of sealant and claiming significant caries reduction. In general, Epoxylite 9075 has not achieved the same degree of success as Nuva-seal and has therefore not been the subject of as many clinical and laboratory studies.

Alphaseal is an u.v. polymerised fissure sealant recently developed by the Amalgamated Dental Company, London. This material was originally studied as the experimental product TP2206 in vitro by Silverstone (1974) and in vivo by Rock (1974a) and Stephen, Sutherland and Trainer (1976). The main resin component is the reaction product of an aliphatic chain di-isocyanate and a hydroxyalkyl methacrylate, diluted with a cross-linking monomer, the dimethacrylate of 1, 3, butanediol and a fluorescent dye. After the addition of the catalyst (Trigonal 14, Akzo Chemie Ltd., Wandsworth, London) polymerisation is activated by exposure to u.v. radiation in the 365 nm region, from a quartz fibre-optic u.v. source, described by Rock (1974b) and Stephen et al. (1976) as the Quartzlite, prior to marketing as the Alphalite. The fluorescence of the sealant on exposure to the radiation is designed to facilitate detection after long periods in the mouth. Using this system Rock (1974a) found 82% of the treated teeth completely covered after one year, and noted an 85% reduction in caries. The only other published trial found as little as 2·3% of TP2206 applications intact the following year, although a 43% reduction in occlusal caries was noted (Stephen et al., 1976).

Williams, Casson and Winter (1974) studied another u.v. polymerised fissure sealant Espe 717 (Espe GmbH, Germany). They reported disappointingly poor sealant retention with a correspondingly low caries reduction. A modified version of the material, Espe 71729, failed to improve on these results, with poorer retention and caries reduction than the original formulation (Williams and Winter, 1976). As yet no other trials of this material have been reported in the dental

literature.

The Concise Enamel Bond System (3M Co., St. Paul, Minnesota, U.S.A.) is used by mixing equal parts of resin and catalyst. The tooth surface is preconditioned for 60s with a 37% concentration of phosphoric acid. Helle (1975) compared the performance of this system with Nuva-seal and found poorer retention of Concise Enamel Bond than with Nuva-seal, but the difference was not claimed to be significant. Ulvestad (1976) evaluated a diluted form of the Concise composite material and found sealant retained on 95% of all treated surfaces of first permanent molars, and on 100% of all premolar surfaces, after a two year period.

Delton pit and fissure sealant is marketed in the United States by Johnson & Johnson Ltd. Bojanini et al. (1976) carried out a clinical trial on a number of formulations of this material and reported that the best of these (SF-119) was completely retained after one year on 91·6% of treated first permanent molars, and the incidence of occlusal caries was reduced by 90%. Brooks et al. (1976) compared Delton with Nuva-seal. After one year, complete sealant retention was found in 95% of the first permanent molar teeth treated with Delton and 84% of the teeth treated with Nuva-seal. Thus these two recent studies suggest that Delton may give better caries reduction than Nuva-seal because of its improved retention. However, Delton pit and fissure sealant requires further evaluation before the merits of the two systems can be properly compared.

Kerr pit and fissure sealant is a filled bis-GMA resin which is available, as yet, only in the United States. Dennison, Charbeneau and Ryge (1975) and (1976) used this sealant to treat first permanent molars in five to nine year old children. After six months, sealant retention was 91% with an effective caries reduction of 83%. After 18 months complete retention was found in 74% of the teeth with a

corresponding caries reduction of 76%. As with Delton, the Kerr system has demonstrated some results which compare favourably with those of Nuva-seal but further evaluation by other researchers will be required before any firm conclusions can be reached.

1.8.4 Other sealants

Epoxylite 9070 (Lee Pharmaceuticals, California, U.S.A.) was a polyurethane sealant which relied on the release of fluoride for caries prevention. McCune and Cvar (1971) reported a caries reduction of 59% at six months but found poor sealant retention. Rock (1972) confirmed the early loss of this sealant with only 1·4% of fissures fully sealed by Epoxylite 9070 after six months, and even these were uncovered at the one year examination. However, Rock did report a 43% reduction in occlusal caries, although the number of teeth tested was rather small. Despite this limited degree of success, Epoxylite 9070 is no longer available, since it has been superceded by the more successful diacrylate formulation, Epoxylite 9075.

Aspa is a glass-ionomer cement marketed by the Amalgamated Dental Co., London, and developed by the Laboratory of the Government Chemist for sealing dental pit and fissures. McLean and Wilson (1974) reported encouraging results with 84% of fissures treated with Aspa cement, still fully covered after one year, and 78% fully covered after two years. Caries was observed to develop solely where the sealant was lost. The only other trial with Aspa found poor results (Williams and Winter, 1976). However, Williams and Winter neglected to use the cavity varnish which the originators of the technique (McLean and Wilson) had stated was essential.

The effect of a methyl methacrylate and tri-n-butylboron (MMA-TBB) sealant in preventing occlusal caries was studied by Ohmori et al. (1976). Sealant retention was not quoted but a caries reduction

of 57% was observed in first permanent molars after two years.

The use of silico-phosphate cement (Petralit, Dental Filling Ltd., London) to fill fissures was tested by Wallis (1973). Only 41% of the 78 treated fissures remained covered after six months, and although a caries reduction of 60% was observed, it was not found to be statistically significant.

1.9 Clinical Trials with Nuva-Seal

In view of the many clinical trials which have been carried out with Nuva-seal, a comprehensive review of fissure sealing with this system is now possible. In these trials, sealant has generally been assessed as being wholly present, or partially or completely absent at regular intervals after application, while the caries incidence in control and treated groups of teeth have allowed the caries reduction for the treated group to be calculated. Comparisons of the results found in the different trials are complicated by the different age-groups treated, different types of teeth involved, and inevitable variations in the methods of assessment used. However, a number of trends can be confirmed from an overall view of these studies.

1.9.1 Caries susceptibility according to retention status of sealant

Nearly all of the trials with Nuva-seal have found that caries does not develop where sealant is wholly retained. Horowitz, Heifetz and Poulsen (1976) studied the caries incidence in paired sites according to the retention status of the sealant after four years. Only 1 of the 465 sites in which sealant was fully retained became carious, while 87% of the 465 paired, untreated sites had developed caries. Where sealant was only partially retained, 6 out of 149 sites were carious, while 60 of the 149 paired sites had developed caries. Where sealant was lost entirely, 57% of sites were carious compared to

60% in the paired unsealed sites. Thus one can assert that there is no increase in caries where sealant is wholly or partially lost. Indeed, caries incidence appears to be reduced where sealant is partially retained. However, these results do not confirm suggestions that caries prevention may continue after sealant is lost in bulk, due to the retention of microscopic tags of resin.

1.9.2 Variation in sealant retention from one trial to another

Even after allowing for differences in the types of teeth treated, large variations in sealant retention sill emerge between the various trials. Fig. 1.2 shows the retention found in first permanent molars for a number of different studies. Thus while Helle (1975) was able to claim complete retention in almost all the teeth which he sealed, other studies showed much poorer retention. This suggests that the results obtained depended on some unknown factors in the application technique.

1.9.3 Retention by tooth type

It has generally been found that premolars retain the sealant better than molars (Rock, 1972; Rock, 1974a; Graves, Bagramian and Bhat, 1975; Horowitz et al., 1976; Meurman and Helminen, 1976). To a lesser extent first premolars show better retention than second premolars (Rock, 1973; Rock, 1974a; Horowitz et al., 1976) and first molars show better retention than second molars (Rock 1972; Rock, 1974a; Graves et al., 1975; Burt et al., 1975; Horowitz et al., 1974; Bagramian and Graves, 1976).

These trends seem stronger than any differences between maxillary and mandibular arches, for although individual trials have sometimes shown distinct differences no consistent trend has emerged. Thus, while Risager and Poulsen (1974) found better retention in lower than upper first permanent molars, Horowitz et al. (1976) observed no difference.

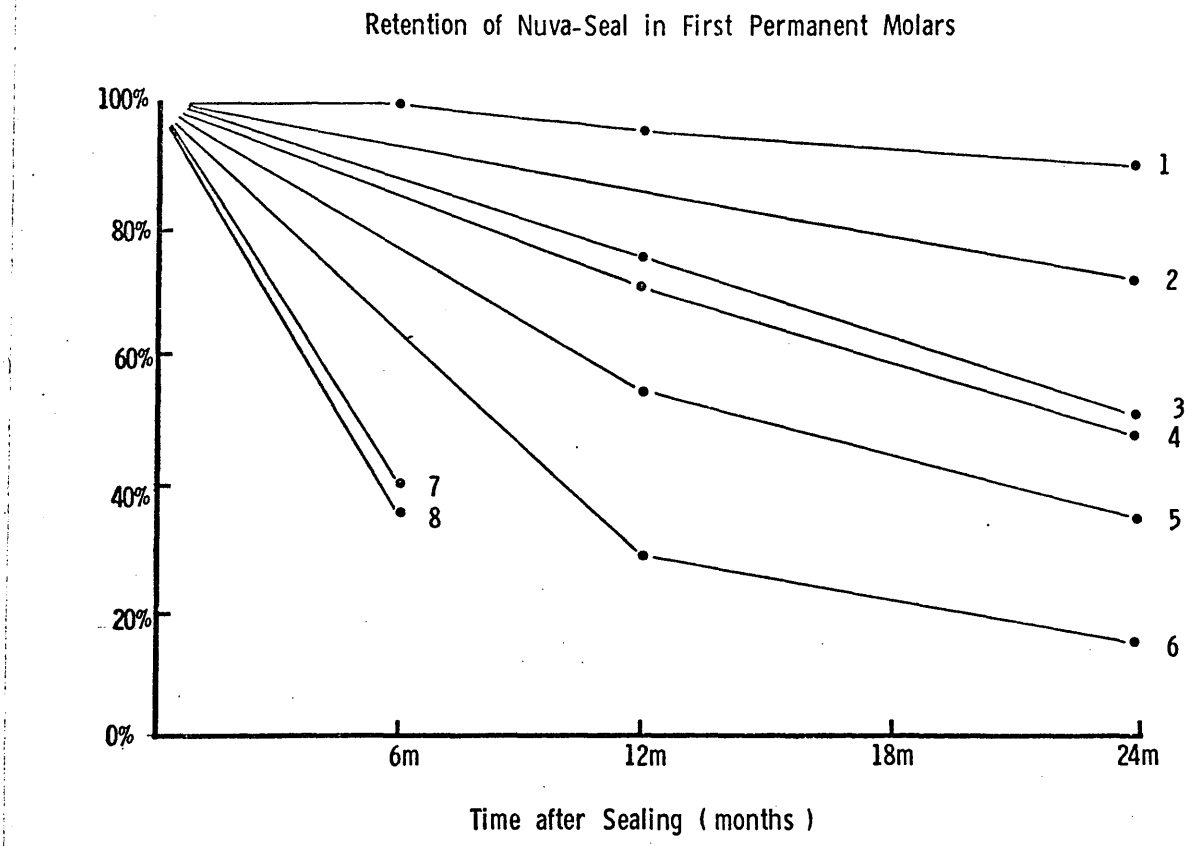


Fig. 1.2 Percentage of first permanent molars completely covered by Nuva-seal at various times following application in eight clinical trials.

1. Helle (1975)
2. Rock (1974a)
3. Cons, Pollard and Leske (1976)
4. Horowitz, Heifetz and McCune (1974)
5. Bagramian and Graves (1976) - 13 year olds
6. Bagramian and Graves (1976) - 7 year olds
7. Rock (1972)
8. Burt et al. (1975)

Cons, Pollard and Leske (1976) treated a very large number of first permanent molars in about 1800 children and assessed retention in four sites in these teeth; the mesial fossa and disto-palatal groove of the upper molar and the occlusal surface and buccal pit of the lower molars. Retention was markedly worse in the buccal pit.

1.9.4 Duration of sealant applications

The pattern of sealant loss after sealing is an important factor in determining when sealant should be re-applied. Where sealant has generally been well retained (e.g. in excess of 70% after one year) losses tended to occur steadily during the trial period. Thus if 20% of the seals are no longer complete at one year 40% will be "lost" or "partial" after 2 years, and it may therefore be useful to express sealant losses in terms of a rate of loss per year. Thus Helle found a loss rate of about 4%/annum; Going et al. (1976) about 22%/annum and others found as much as 50%/annum. When sealant was less well retained, there was sometimes a very rapid loss of sealant initially, followed by a more gradual loss (e.g. Rock, 1972 and Burt et al., 1975). It may be that the incorrect application to some teeth results in very early loss of sealant, while successfully applied layers are lost only in the long term due to other factors.

1.9.5 Retention by age of subject

Many authors have pointed to the difficulties in sealing teeth in very young children, in particular the first permanent molar in six-year-olds (e.g. Burt et al., 1975 and Stephen et al., 1976). However, few studies have made direct comparisons of the retention achieved in the same tooth type in children of different ages. However, Graves et al. (1975) and Bagramian and Graves (1976) found 55% complete retention in first permanent molars of 13-year-olds, but only 29% complete retention

in the same teeth of 7-year-olds after one year. Such a result may be explained by the difficulty of access to this tooth in the younger children.

1.9.6 Retention in permanent and deciduous teeth

Some clinical trials in which Nuva-seal has been evaluated on both deciduous and permanent teeth have shown poorer sealant retention on the former. (Buonocore, 1970 and 1971). It has been suggested that this may be due to the occurrence of a layer of prismless enamel on deciduous teeth, which does not allow the usual penetration of sealant into the enamel (Gwinnett, 1973). Consequently, Gourley (1974, 1975) used an etching period increased from 60 to 90s for deciduous teeth, and found better retention in deciduous than permanent teeth. However, Graves et al. (1975) also found better retention in primary molars than in the first permanent molar, although they did not specify whether an increased etching period had been used or not. The effect on sealant retention of possible differences in adhesion to permanent and deciduous enamel may have been masked in all these trials by morphological effects and differences in the ages of subjects.

1.9.7 Retention in fluoridated and non-fluoridated areas

Retention of sealant in fluoridated areas might be expected to be poorer than in non-fluoridated areas due to the increased resistance of teeth to acid etching. Laboratory studies have demonstrated that adhesion of sealant can be affected by the presence of fluoride in the enamel (Lee et al., 1972; Low, Fraunhofer and Winter, 1975 and 1976). However, there is little clinical evidence to indicate the effect of fluoride in the water-supply on sealant retention. Although Cons et al. (1976) and Bagramian and Graves (1976) carried out sealing in fluoridated communities, it is not clear whether their results would have

been better in a non-fluoridated community. Stephen et al. (1976) found the surprising result that TP2206 retention was better in fluoridated than non-fluoridated areas. However since the retention in both areas was very low, this difference may have been caused by other factors. Clearly further clinical studies will be required before this issue can be clarified.

1.10 Aims of Present Investigation

Numerous studies on fissure sealants have shown that effective caries prevention depends on successful retention of the sealant. Provided the sealant cover is maintained occlusal caries can be completely prevented. However, a major problem with sealants has been the variable retention found from one trial to another. Before such materials can be adopted in a community programme of preventive dentistry the reasons for failures in sealant retention need to be understood so that corrective measures can be taken. Therefore, factors affecting the retention of the most widely used material, Nuva-seal, and the more recently developed sealant, Alphaseal, have been studied. In particular, the role of u.v. radiation in polymerising these materials, has been examined. The objective of these studies was the development of a detailed clinical protocol to ensure optimum sealant retention with the available materials and equipment. The effectiveness of such a protocol has been evaluated in a clinical trial with the Nuva System. An explanation is also sought for the very poor retention found in the trial of Alphaseal (TP2206) carried out in Galloway by Stephen et al. (1976), so that modifications to the materials and equipment can be made to ensure more successful results in the future.

CHAPTER TWO

IN VITRO STUDIES OF PHYSICAL FACTORS AFFECTING ADHESION OF FISSURE SEALANT TO ENAMEL

2.1 Introduction

In the early stages of the development of dental adhesives, test methods were employed which simply determined whether or not a bond was formed with enamel. However with the gradual improvement of these materials more quantitative tests became necessary. The two basic types now in common use are the tensile and shear tests. In the tensile test a uniform tensile stress is applied perpendicularly to a flat adhesive/enamel interface, and the maximum sustained stress recorded. In a shear test the stress is applied parallel to the adhesive/enamel interface and is not generally uniformly distributed. More elaborate testing methods have also been used with dental adhesives, such as the pin-hole test (Williams, De Vries and Despain, 1973) and punch shear test (Patrick and Kaplan, 1965). In addition special set-ups have been designed to simulate the oral loading experienced by directly bonded orthodontic brackets (Mizrahi and Smith, 1969a and 1969b; Cohl, Green and Eick, 1972; Bishara, Khowassah and Oesterle, 1975; Rich, Leinfelder and Hershey, 1975).

A number of tensile test methods found in the dental literature are compared in Table 1. A wide variety of dental adhesives have been tested including methyl methacrylate based direct filling resins, composites, cyanoacrylates, and diacrylate fissure sealants. The substrate used was generally either bovine or human enamel or dentine. Bovine incisors have the advantage of being more readily available than human teeth, and provide larger areas of flat enamel. The tensile strengths quoted in Table 1 are typical of the results found in each

TABLE 1 : A comparison of tensile bond tests on dental adhesives

Reference	Adhesive	Substrate	Tensile Strength (kgf/cm^2)	Coeff. of Variation
Bowen (1965)	Methylmethacrylate	Human enamel and dentine	80	17%
Mulholland and Deshazer (1968)	Addent restorative resin	Human enamel	60	50%
Lee, Swartz and Culp (1969)	Epoxylite 8985	Bovine enamel and dentine	93	50%
Eden, Craig and Peyton (1970)	Various commercial restorative resins	Bovine enamel and dentine	90	20% - 40%
Phillips, Swartz and Rhodes (1970)	Polycarboxylate and zinc phosphate cement	Bovine enamel and dentine	21	25% - 45%
Khowassah and Shippy (1971)	Cyanoacrylates and zinc phosphate	Human enamel	42	15%
Laswell, Welk and Regenos (1971)	Sevriton restorative resin	Bovine enamel	140	30%
Lee, Phillips and Swartz (1971)	Methyl methacrylate	Bovine enamel	75	25%
Beech (1972)	Cyanoacrylates	Human enamel	70	20%
Brauer and Termini (1972)	Kadon restorative resin	Bovine enamel	54	20% - 50%
Otsuki et al. (1973)	Cyanoacrylate	Bovine enamel	140	20%, 38%
Wright and Beck (1973)	Nuva-seal	Human enamel	36	61%
Jenkins (1974)	Commercial composite resins	Human enamel	135-175	27% - 50%
Rock (1974c)	Nuva-seal and Epoxylite 9075	Human enamel	30-45	13% - 25%
Williams, Fraunhofer and Winter (1974)	Nuva-seal Epoxylite 9075	Human enamel	30 42	43% 55%
Breakspeare, Tranter and Weldon (1975)	Nuva-seal	Human enamel	9	--

case, and have reached a maximum of about 150 kgf/cm^2. The coefficient of variation provides a measure of the reproducibility of each test method, and was generally rather large.

Test procedures which have been used previously with dental adhesives are generally unsuitable for the sealants studied here, since they do not allow for the application of u.v. radiation. Although Wright and Beck (1973) described a simple test for measuring the bond strength between u.v. polymerised sealants and enamel, reproducibility was poor, with a coefficient of variation of about 60%. Williams, Fraunhofer and Winter (1974) obtained better results, but their method did not permit a uniform application of u.v. radiation. A new procedure was therefore developed to provide reproducible results and permit uniform irradiation of the resin.

2.2 Materials and Methods

Enamel specimens were taken from extracted human or bovine teeth which were mounted horizontally in small plastic boxes using self-curing acrylic resin. Each box was fitted securely to the working table of a vertical drill, as shown in Fig. 2.1. For each tooth a fresh disc of 600 grit, self-adhesive silicon carbide paper was pressed firmly to an attachment set in the drill chuck. The rotating abrasive was then applied to the embedded tooth, until a sufficiently large area of flat enamel was prepared. Cylinders of enamel, 2·8 mm in diameter, were cut perpendicular to this flat surface using diamond-tipped core drills fitted in the chuck, as shown in Fig. 2.2.

The enamel surface was etched by totally immersing each specimen in a dappen's glass of the recommended acid for one minute. To wash the acid thoroughly from the enamel, the specimen was transferred to a beaker of distilled water which was stirred for at least 1 min. The enamel specimen was dried for 5 s in a jet of dry, hospital air

maintained at a flow of 5 l/min. The tooth cylinder was placed in a "non-stick" PTFE mould as shown in Fig. 2.3, care being taken to ensure that the cylinder formed a tight fit to prevent leakage of sealant down the side of the specimen. Nuva-seal was then brushed slowly over the etched enamel to avoid trapping air bubbles. The quartz u.v. guide of the Nuva-lite was fitted directly over the sealant, and maintained at a fixed distance of 2 mm by a brass washer. In early experiments, an exposure time of 45 s was found sufficient to completely polymerise Nuva-seal, but later a thin layer of Nuva-seal was polymerised before applying the bulk of the sealant.

The shape of the polymerised sealant in the completed specimen allowed for a direct pull without the need for a second adherend. Thus no attachments were necessary, which could have blocked the u.v. radiation thereby impeding the polymerisation process and reducing the strength of the bond.

Completed specimens were stored in distilled water at 37°C for about 24 hours prior to testing. A universal tester (Instron Ltd., High Wycombe, Bucks., U.K.) was used to pull the sealant from the enamel at a constant rate of 0·02 cm/min and the tensile load at which the bond failed was registered on a paper chart recorder.

The loading arrangement shown in Fig. 2.4 was designed to apply a uniform force perpendicular to the bond surface. The bonded specimen was fitted into a plastic collet, and the protruding enamel cylinder firmly gripped in a three jaw chuck fixed to the Instron crosshead. A wax lining between the attached sealant and the plastic collet ensured a good fit. When a load was applied to the rig, the wax helped to distribute the stress more uniformly over the load bearing surface of the sealant.

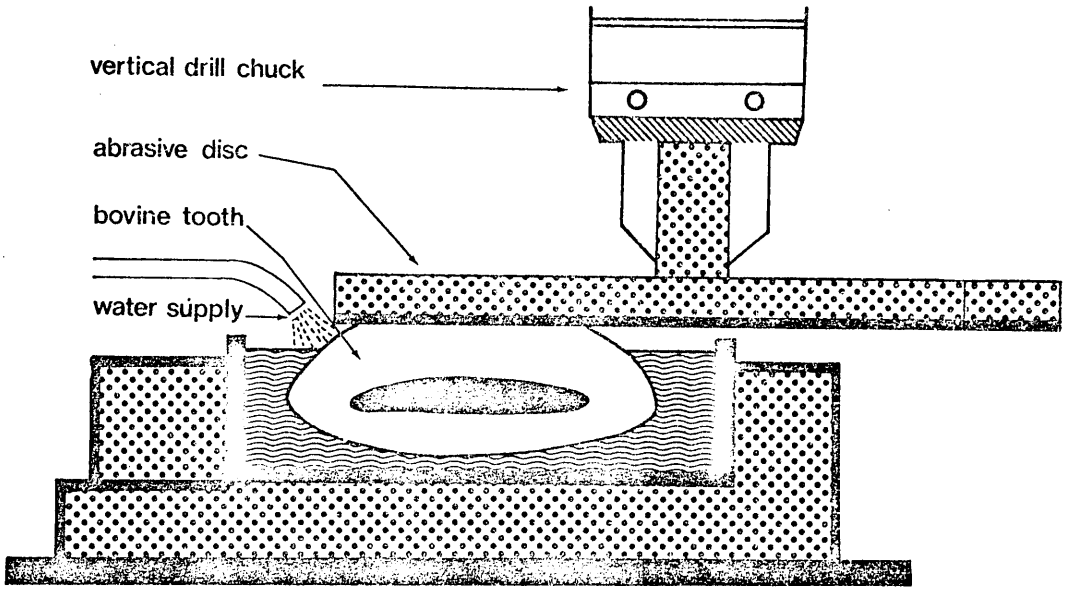


Fig. 2.1 Arrangement for preparing a flat enamel surface on a tooth mounted in a plastic box, with self-curing acrylic resin.

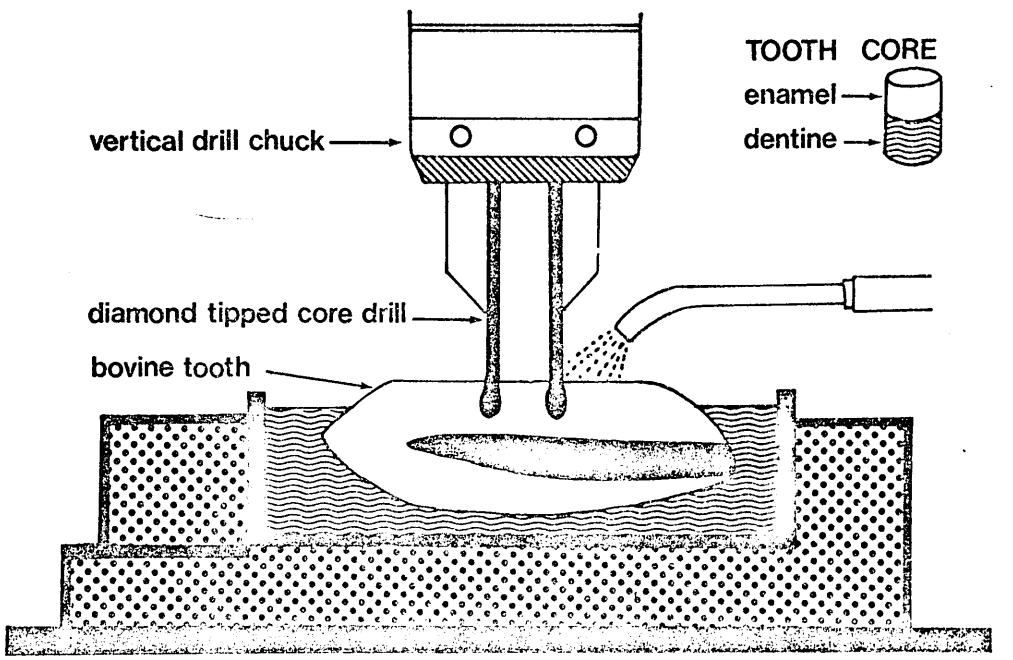


Fig. 2.2 Arrangement for cutting a tooth cylinder with a diamond tipped core drill.

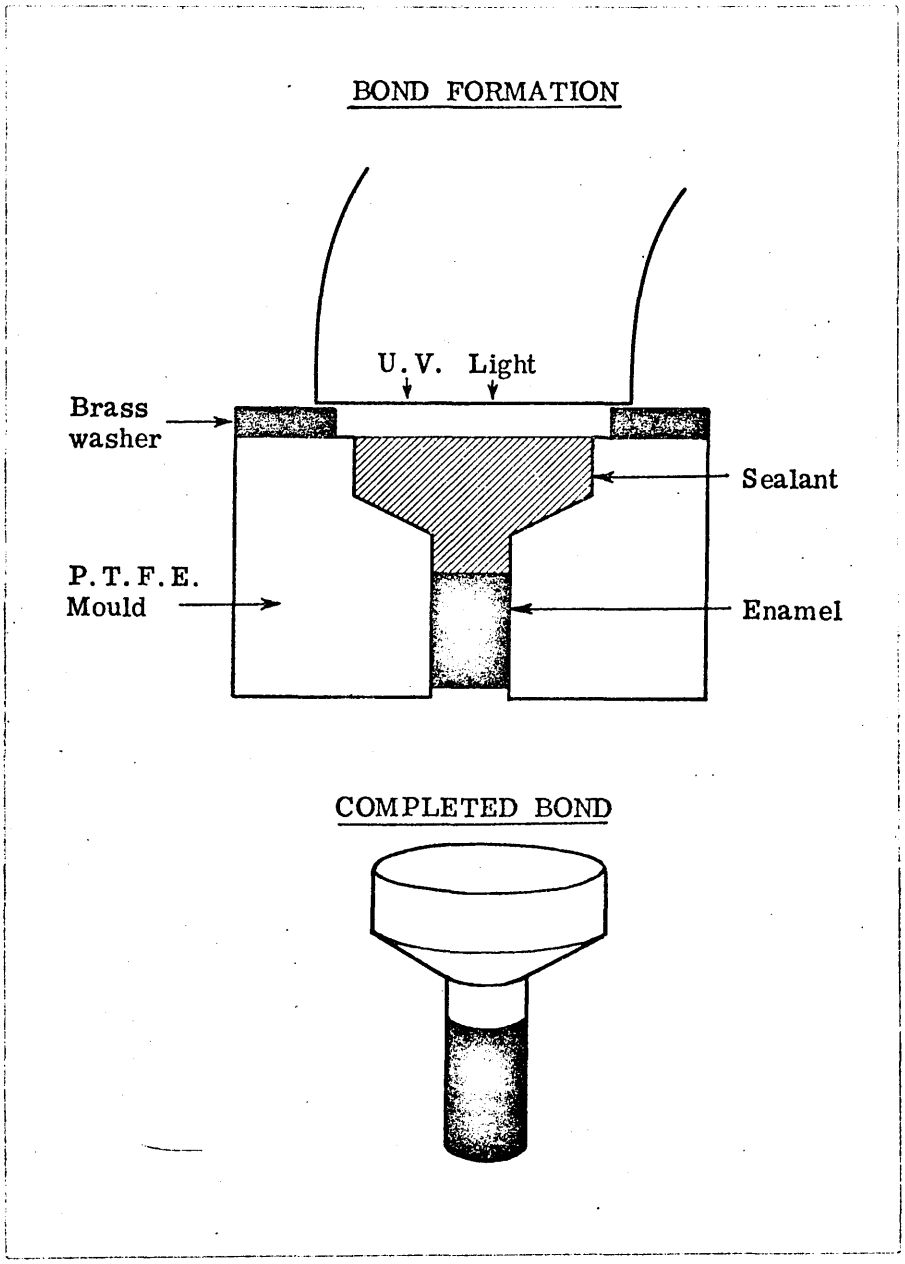


Fig. 2.3 The formation of a bond between an enamel cylinder and sealant polymerised by u.v. radiation.

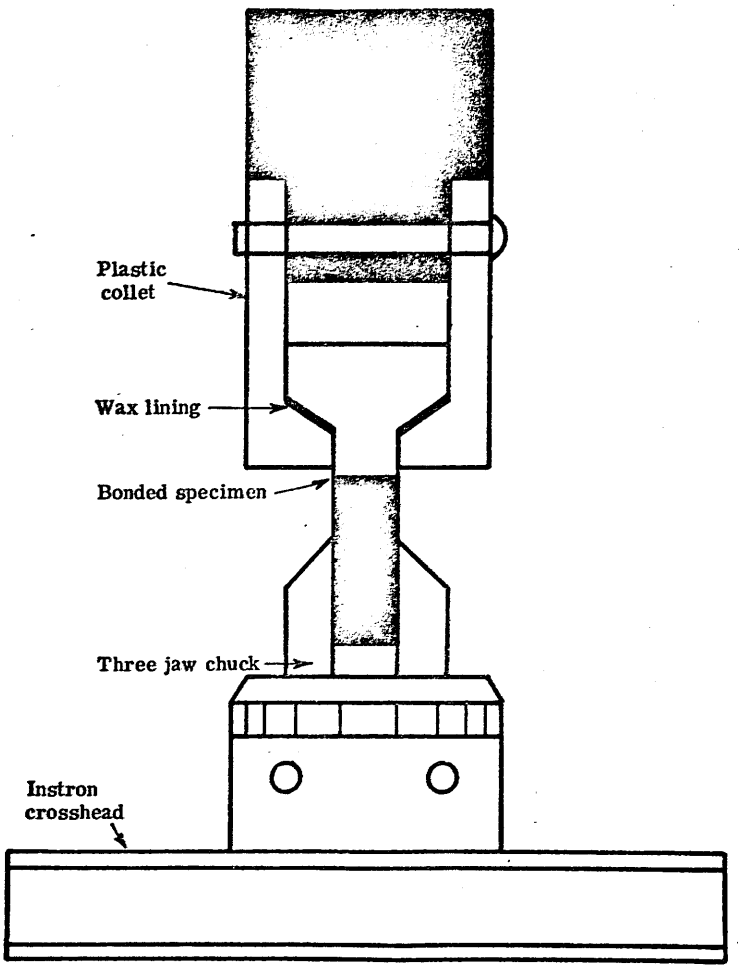


Fig. 2.4 The loading arrangement used for testing the tensile strengths of bonded specimens.

2.3 Investigation of Experimental Factors

A number of experimental factors which might influence the measured values of tensile bond strength between sealant and enamel were investigated using the above bond testing technique with the Nuva-seal system.

2.3.1 Effect of the wax lining

To assess the effect of using a wax lining in the testing rig to distribute the load more evenly, 18 bonded specimens were prepared. The bond strengths found when 9 specimens were tested with the wax lining, and 9 without the wax lining revealed two immediate benefits (Fig. 2.5). Firstly, it reduced the spread in the measured values, as evidenced by the reduction in the coefficient of variation from 51% to 16% (Coeff. of Var. = Standard Dev./Mean) and secondly, it produced an increase in the mean measured bond strength from 84 to 124 kgf/cm^2.

2.3.2 Type of enamel

Of the 16 tensile bond testing arrangements listed in Table 1, 9 were performed on human enamel and 7 on bovine enamel. Bovine enamel is not, however, the same as human enamel, as shown in the scanning electron microscope study of Yamamoto et al. (1971) but it is available in virtually unlimited quantities and a large area of flat enamel on the labial surface of bovine incisors is an advantage. Therefore, tests were performed on both types of enamel to see whether the differences between them would affect the tensile bond strength. For this, 22 human enamel and 56 bovine enamel specimens were prepared.

The mean tensile bond strength attained for the human samples was $94{\cdot}7 \pm 5{\cdot}9$ kgf/cm^2, and for the bovine samples $96{\cdot}4 \pm 4{\cdot}2$ kgf/cm^2. Both types of enamel demonstrated high bond strengths close to 100 kgf/cm^2, representing about 25% of the claimed tensile strength of

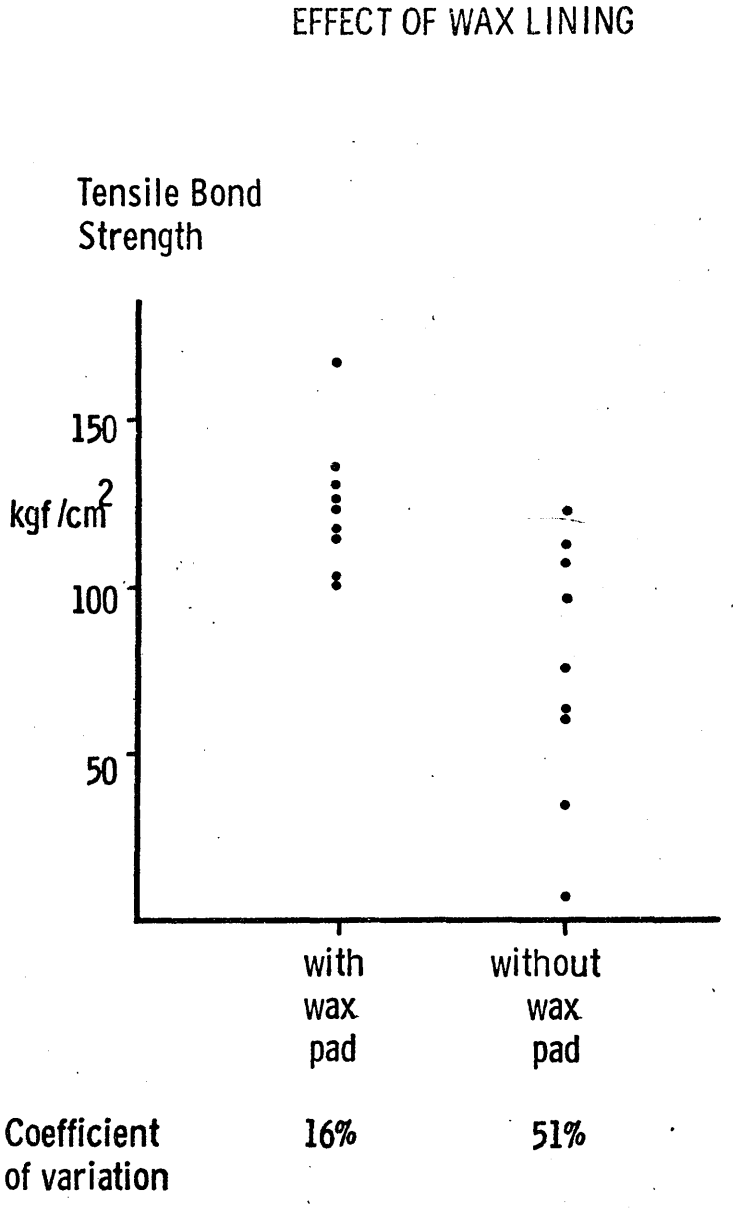


Fig. 2.5 The effect on measured bond strengths of incorporating a wax lining into the loading arrangement shown in Fig. 2.4.

the Nuva-seal resin itself (Caulk, 1974). Since the two mean bond strengths were not found to be significantly different using Student's t-test ($P > 0.4$) all subsequent tests used the more convenient bovine specimens.

2.3.3 Effect of variation between different teeth

Bond tests with dental adhesives have invariably shown poor reproducibility. While the best systems achieved coefficients of variation of about 15%, very high values ranging up to 60% were often found. This large variability has generally been attributed to anatomical variations between teeth used to provide experimental specimens. Therefore, an investigation was carried out to evaluate differences in measured bond strength from one tooth to another.

Three specimen cylinders were cut from each of eight bovine incisors and the resulting bond strengths are displayed in Fig. 2.6. The coefficient of variation of the three bond strengths appropriate to each tooth was generally much smaller than the coefficient of variation in the values of bond strengths found when specimens were taken from a number of different teeth. Therefore, steps were taken in all subsequent experiments to allow for variations between individual teeth, by ensuring that any comparative tests were carried out between paired enamel specimens from the same tooth. Having evaluated these three basic criteria for the reliability of the tensile bond testing technique, attention was next focussed on a number of physical factors of clinical relevance in order to assess their effects on measured bond strengths.

2.4 Physical Factors of Clinical Importance

2.4.1 Moisture contamination

To investigate the effect of moisture at the bond surface three

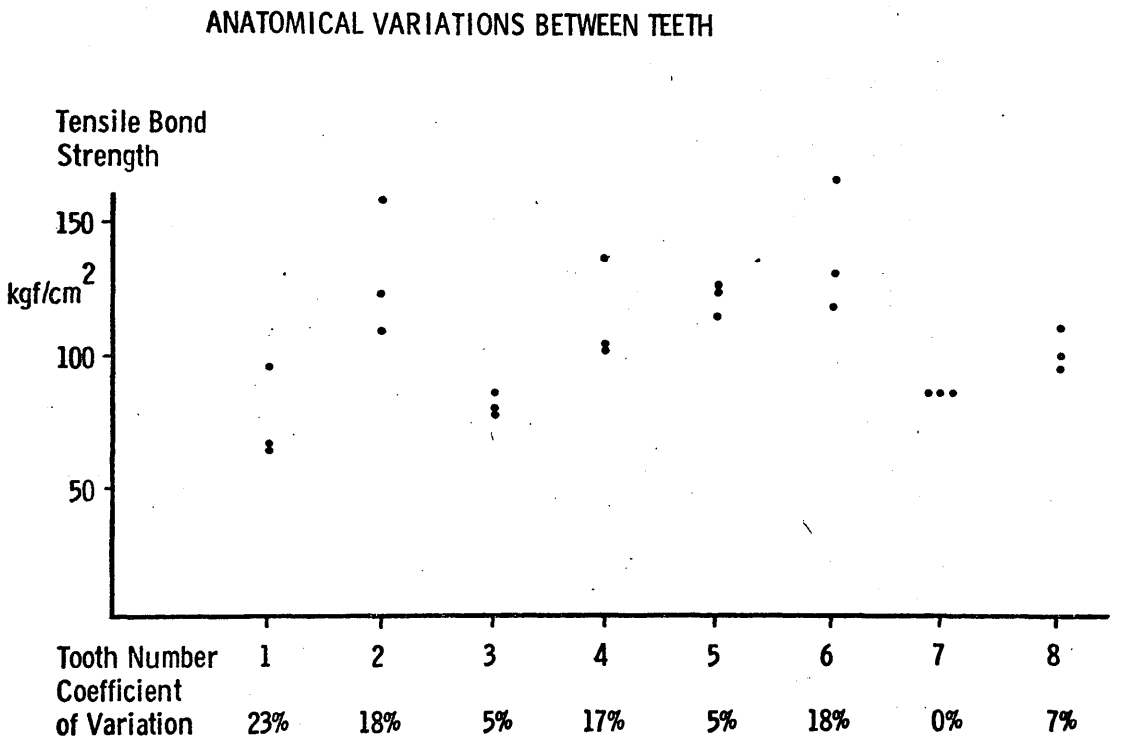


Fig. 2.6 Tensile bond strengths for the adhesion of Nuva-seal to three enamel cylinders cut from each of eight bovine incisors from different animals.

moisture conditions were established:

(a) Dry: The normal "dry" condition used a 5 s drying period with compressed hospital air after a preliminary wipe with a dry tissue; the air flow being maintained at a constant 5 l/min.

(b) Extra Dry: The "extra dry" condition had a drying period of 60 s.

(c) Wet: The "wet" condition was established by placing 0·5 µl of distilled water directly on to a "dry" enamel surface. This amount of water formed a small spot in the centre of the enamel. Once sealant was applied to this "wet" surface, the sealant resin was brushed thoroughly into the enamel.

To allow for possible variations between teeth, pairs of bonds were formed with specimens from each tooth; one bond formed under the control "dry" condition and the other at either "extra dry" or "wet". Fig. 2.7 compares the bond strengths found under these three moisture conditions. Under the "wet" condition the bond strength was drastically reduced ($p < 0{\cdot}001$), but between 14 pairs of "dry" and "extra dry" bonds there was no significant difference ($p > 0{\cdot}4$), using Student's t-test.

2.4.2 Intensity of u.v. radiation

In the course of laboratory experiments using a Nuva-lite u.v. source, tensile bond strengths were found to be much lower than had previously been observed and the variability in the measured values much greater than before. In was eventually determined that a reduction in the output of the u.v. source was the cause. Considerable visible radiation was still being emitted but insufficient u.v. radiation was present to provide adequate polymerisation. A new bulb was obtained and a dramatic improvement resulted. Tensile bond tests, prepared using an old and new bulb, showed that the greater intensity of u.v. radiation from the new bulb produced higher and more consistent bond

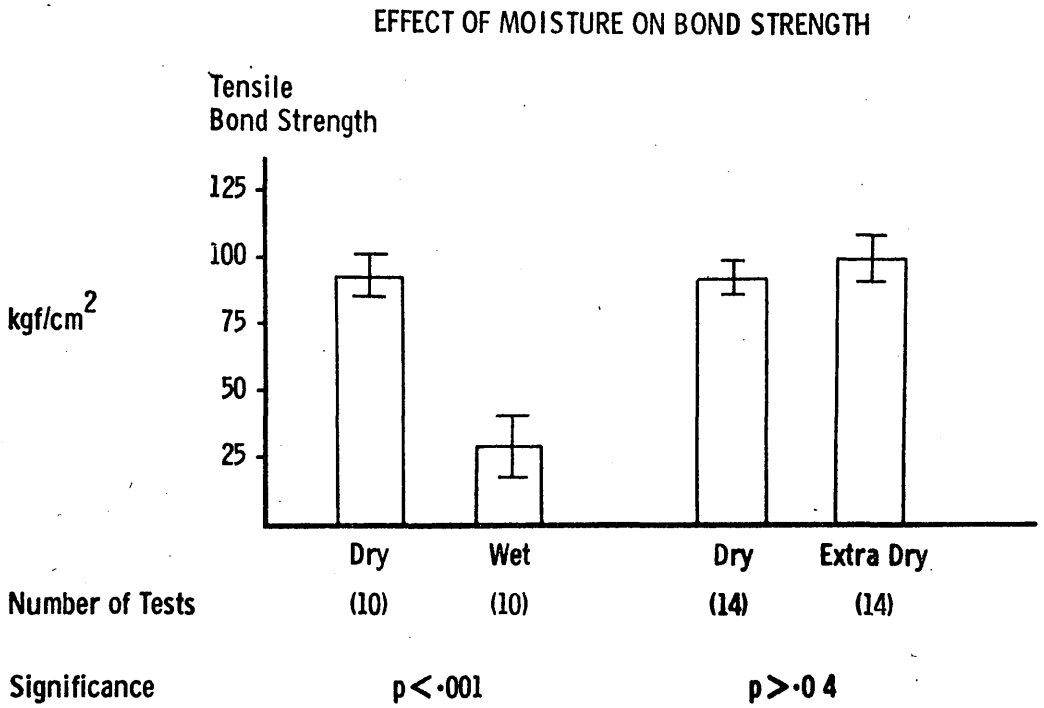


Fig. 2.7 Tensile bond strengths of Nuva-seal bonded to pairs of enamel cylinders, with one specimen's surface "dry" and the other's either "wet" or extra "dry".

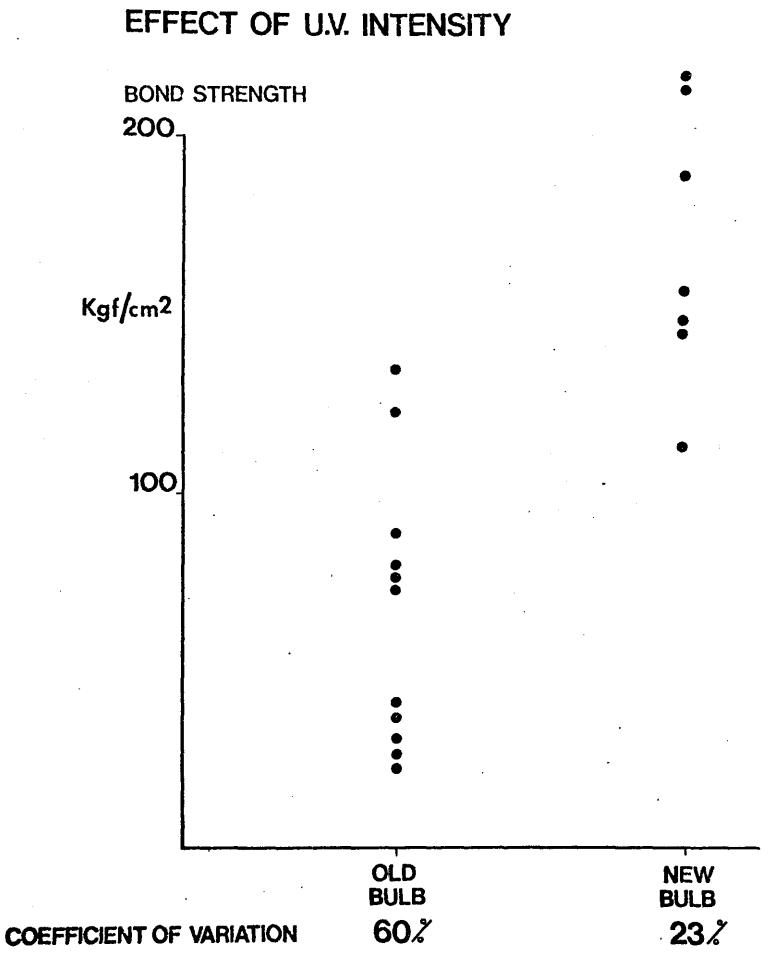


Fig. 2.8 Tensile bond strengths measured between enamel and Nuva-seal, exposed to radiation from a Nuva-lite, fitted with its old u.v. bulb and with a new replacement bulb.

strengths, as shown in Fig. 2.8. A visual examination of the two bulbs used in the experiment revealed that the older bulb had developed an opaque deposition on the inside of its quartz window, which may explain its reduced output. These findings prompted an investigation of the emission of u.v. radiation from the two commercial sources currently in clinical use; namely the Nuva-lite and Alphalite sources (Amalgamated Dental Co. Ltd., London, U.K.). The results of these studies are presented in the next chapter.

2.4.3 Concentration of etching acid

Silverstone (1974) has investigated the effect of different concentrations of phosphoric acid (H_3PO_4) on the depth of etched surface enamel. He showed that between concentrations of 20% and 70%, the depth of surface etch and depth of histological change decreased steadily with increasing acid concentration. Some of his results are shown in Fig. 2.9. The greatest depth of surface etch was caused by the 20% and 30% H_3PO_4 solutions, while at higher concentrations the etch produced in the remaining surface layer was very much reduced.

Scanning electron micrographs of four neighbouring areas of a 4 mm x 4 mm surface of bovine enamel are shown in Fig. 2.10. Each area was etched for 60 s with one of the phosphoric acid concentrations, 10, 30, 50 and 70% w/w. The 30 and 50% solutions produced the greatest change in the enamel surface while the 70% etchant appears to have had only a limited effect. Four corresponding sealant bond surfaces were exposed by dissolving the enamel in 10% hydrochloric acid for 24 hours. Scanning electron micrographs of these surfaces are shown in Fig. 2.11. The degree of sealant penetration appears to correspond to the degree of etching produced by the different etchants. Thus the 10, 30 and 50% solutions resulted in a "honeycomb" pattern of sealant penetration over large areas of the enamel surface, while the enamel treated with

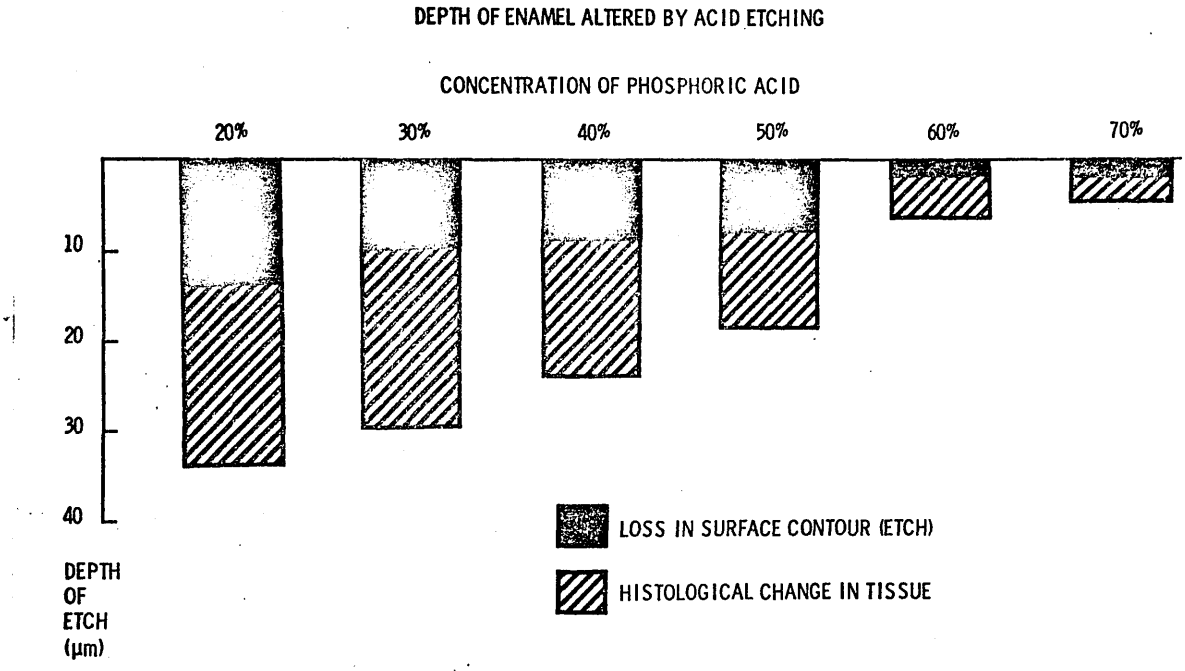


Fig. 2.9 Depth of etch and histological change in enamel following a one minute exposure to various concentrations of phosphoric acid (Silverstone, 1974).

the 70% solution, showed no evidence of such penetration by the resin.

The appearance of the enamel at areas of interfacial failure following tensile destruction of the four types of bond are shown in Fig. 2.12. Any penetrating tags of sealant have been torn off near the interface, so that the deeply etched patterns shown on the enamel surfaces in Fig. 2.10 are no longer apparent. The appearance of sealant surfaces at areas of interfacial failure for the four types of bond, are shown in Fig. 2.13. The original "honeycombs" of penetrating sealant have been pulled off the bulk of the resin leaving a smoother surface than observed in Fig. 2.11. This difference was particularly marked for the bonds formed using the 30 and 50% solutions, where sealant penetration was greatest.

Tensile strengths of bonds formed using the same four concentrations of phosphoric acid etchant are presented in Fig. 2.14. While there was a tendency towards higher bond strengths for the more severely etched surfaces, the 30 and 50% H_3PO_4 solutions did not produce significantly different results. This may be surprising since Silverstone (1974) showed that the lower of these two concentrations permitted greater sealant penetration. Although the 70% solution achieved lower values, the recorded bond strengths were quite high, at 125 kgf/cm^2. Assuming a mechanical theory of retention, relying on "tags" of penetrating sealant for adhesion, one would expect the 70% solution to result in much lower bond strengths than the 30% etchant, since the depth of etch found by Silverstone (1974) and the surface appearance and sealant penetration shown here, were all dramatically different for the two etchants.

The "tags" of penetrating sealant which have frequently been referred to in the dental literature (cf. Chapter One), are normally those which occupy the spaces left by dissolution of enamel prism cores. Consequently they have diameters of about 4 μm. However it has been

Fig. 2.10 Scanning electron micrographs of four neighbouring areas of a surface of bovine enamel etched with 10, 30, 50 and 70% w/w H_3PO_4 for 60s.

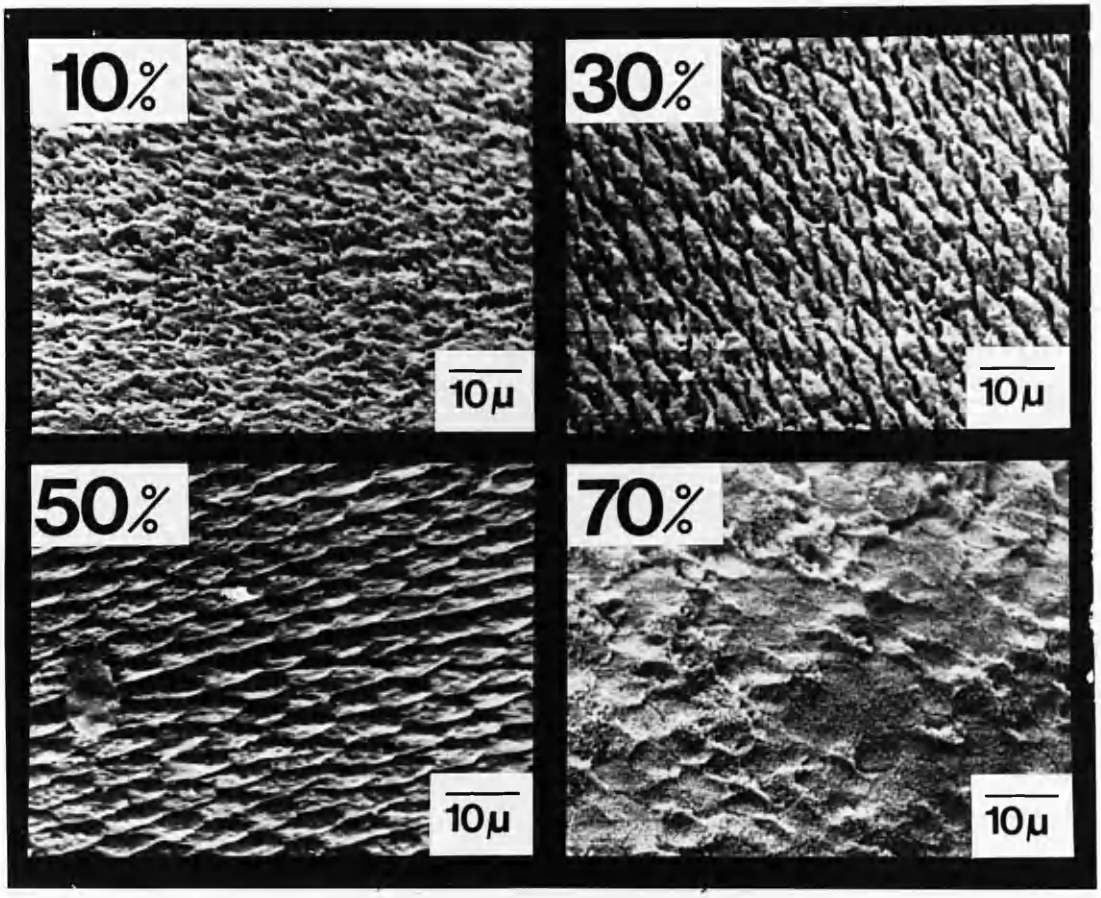

10%
10μ
30%
10μ
50%
10μ
70%
10μ

Fig. 2.11 Scanning electron micrographs of sealant surfaces penetrating bovine enamel etched for 60s with 10, 30, 50 and 70% w/w H_3PO_4, exposed by dissolving the enamel in 10% w/w HCl for 24 hours.

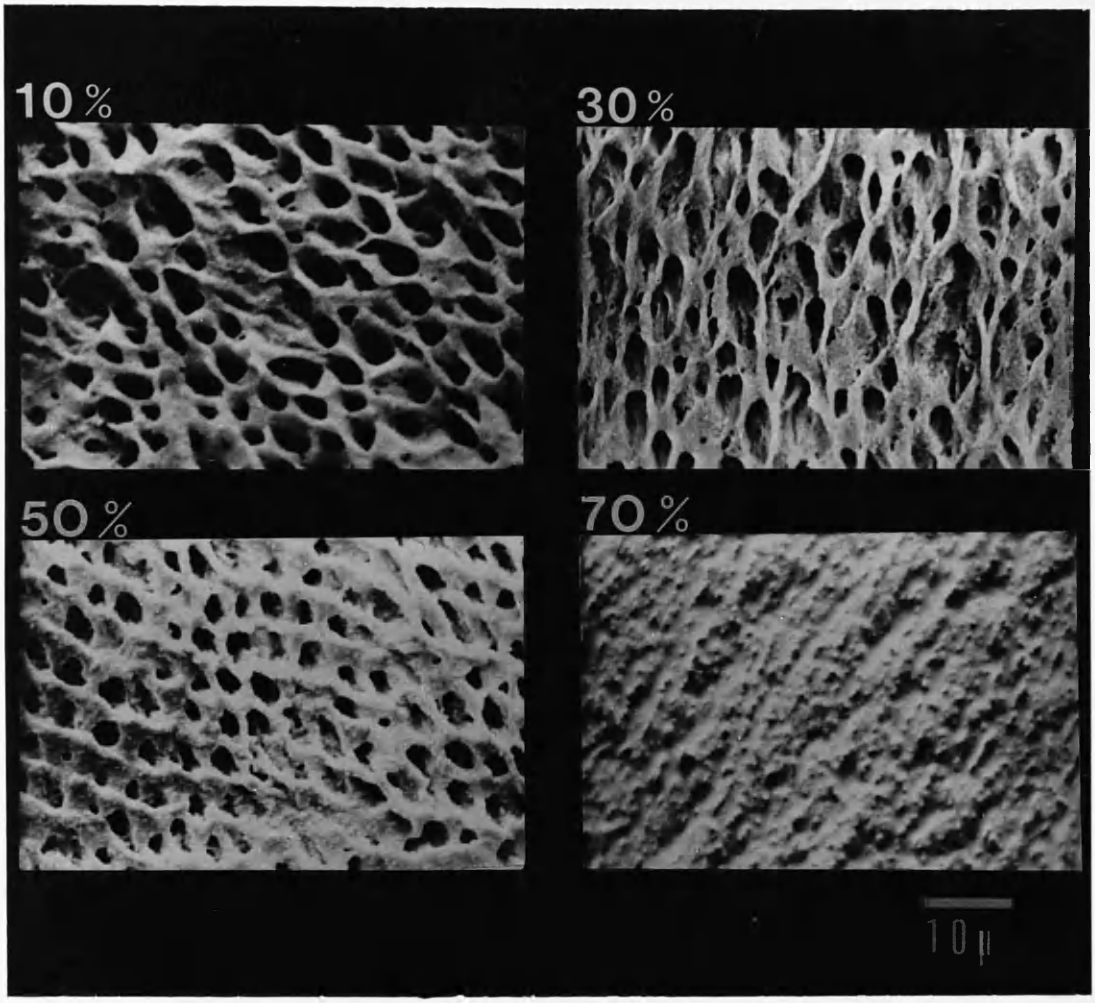

10 %
30 %
50 %
70 %
10 μ

Fig. 2.12 Scanning electron micrographs of enamel surfaces at areas of interfacial failure, where 10, 30, 50 and 70% w/w H_3PO_4 had been used to condition the enamel for 60s, prior to the application of Nuva-seal.

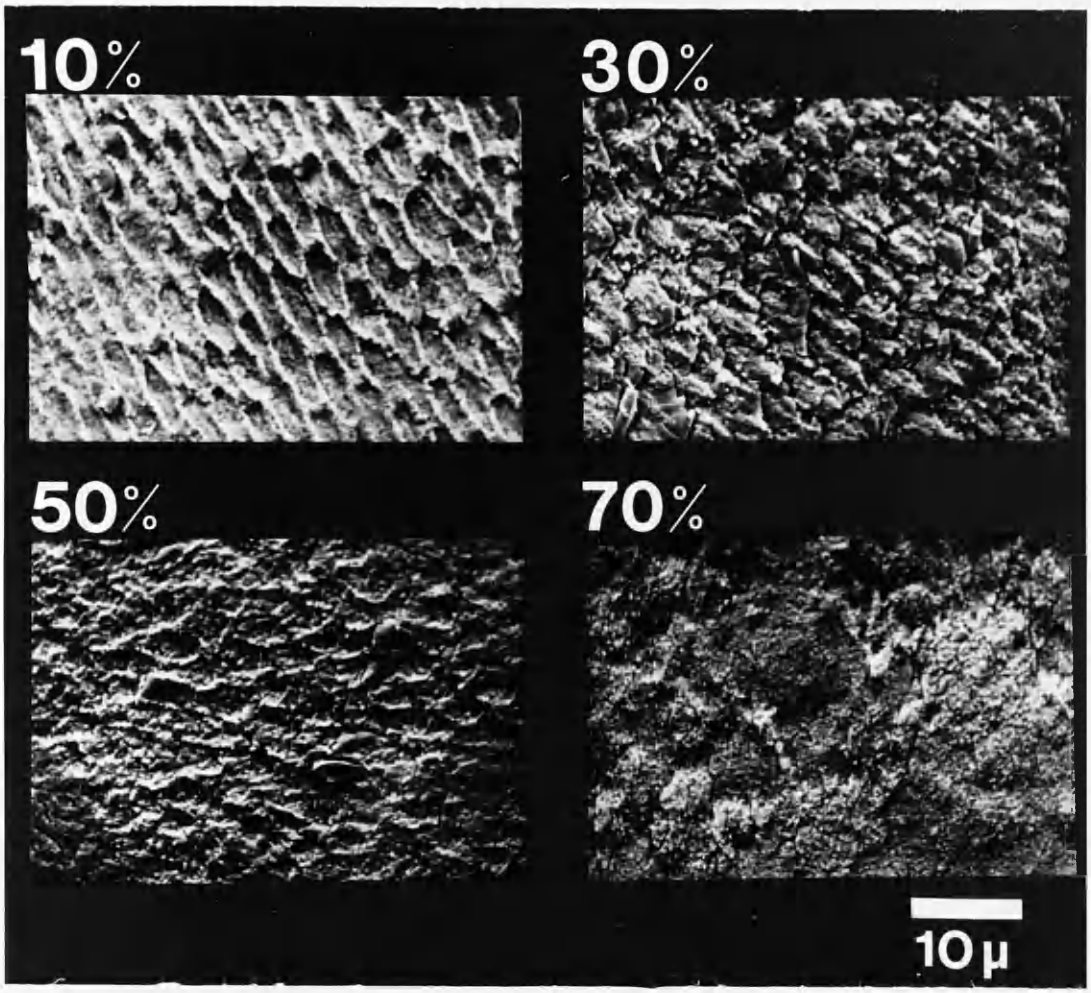
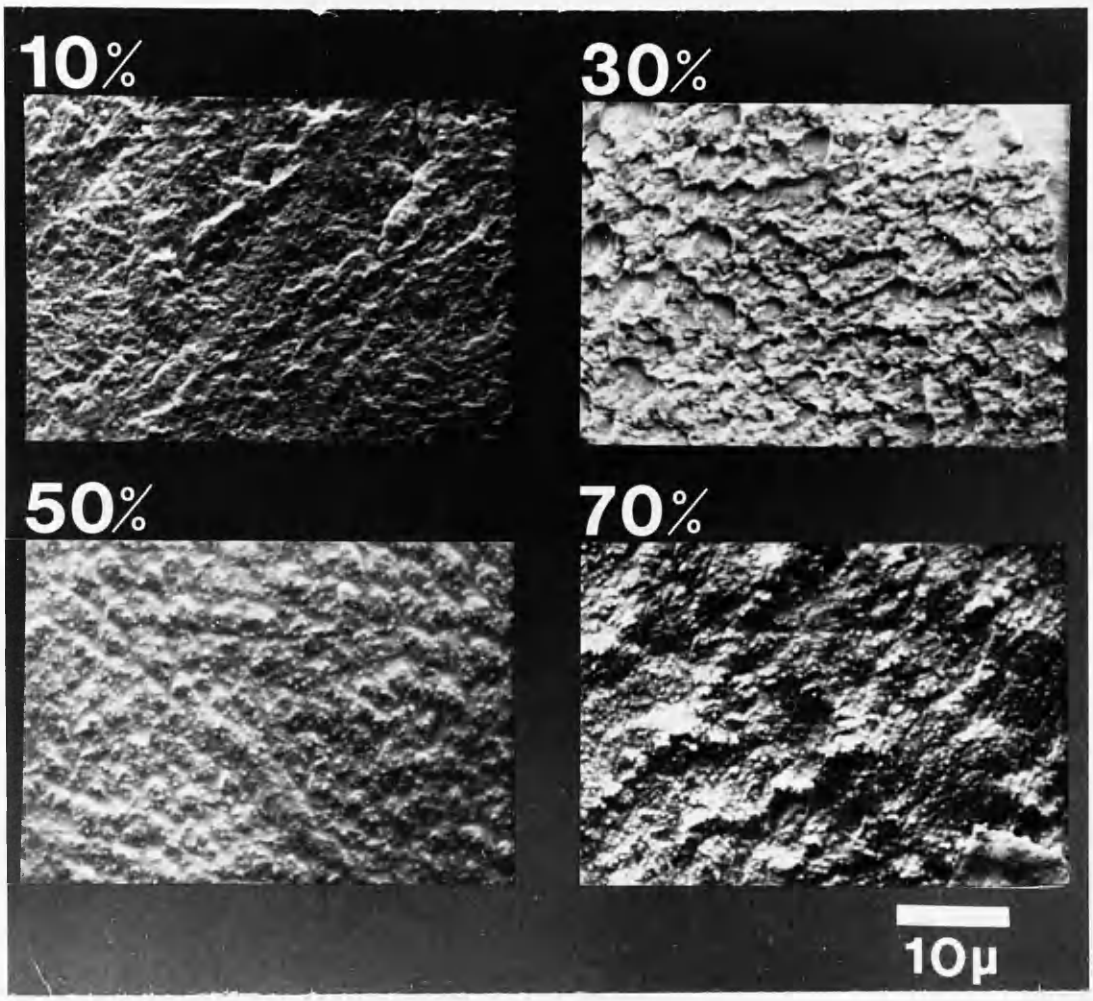

10%
30%
50%
70%
10 μ

Fig. 2.13 Scanning electron micrographs of sealant surfaces at areas of interfacial failure after tensile testing of adhesion to enamel, pre-conditioned for 60s with 10, 30, 50 and 70% w/w H_3PO_4.


10%
30%
50%
70%
10μ

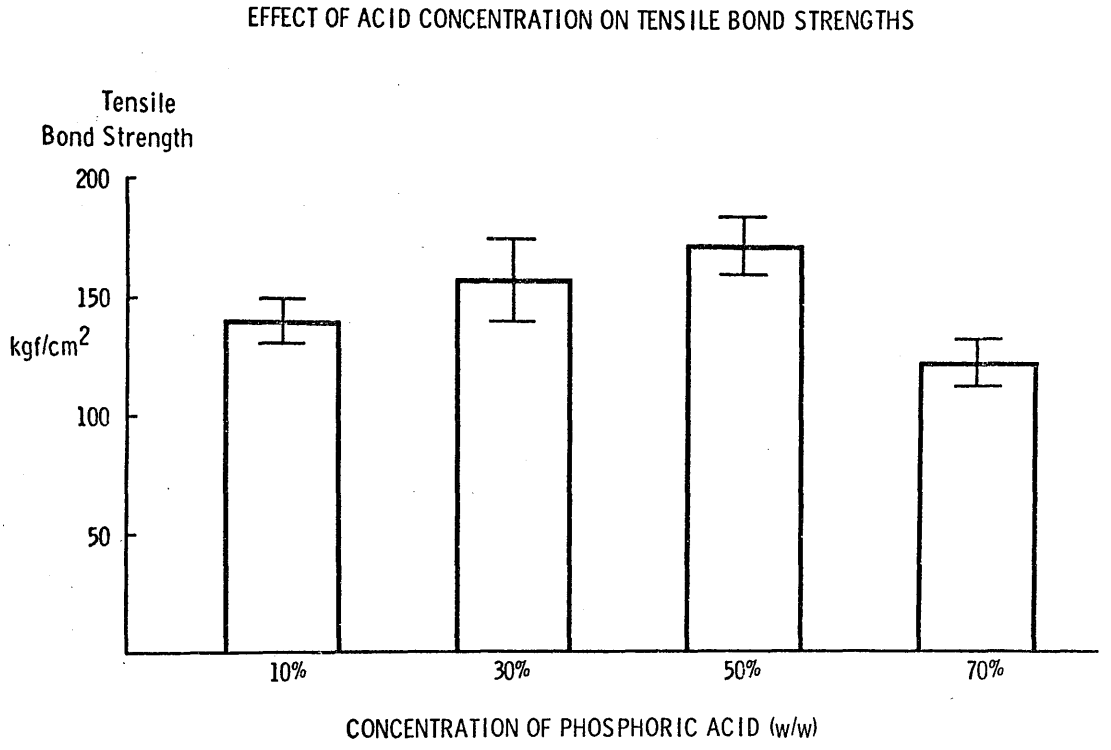


Fig. 2.14 Tensile bond strengths of Nuva-seal to bovine enamel conditioned for 60s by 10, 30, 50 and 70% H_3PO_4.

shown by Simmelink et al. (1974) that sealant also penetrates partially decalcified enamel on a more microscopic level. Using a transmission electron microscope they showed that not only did the sealant network penetrate the enamel at the prism level, but that the resin surrounded the crystals of hydroxyapatite, and penetrated and polymerised in their dissolved cores. Such microscopic penetration may be no less important to adhesion to etched enamel than the larger sealant "tags" which have been readily observed by optical and scanning electron microscopy.

Thus the high bond strengths found using a 70% w/w concentration of phosphoric acid may be explained, in the absence of the usual penetrating sealant "tags", by a more microscopic sealant network within the enamel surface. The presence of sealant "tags" when the lower concentrations of phosphoric acid were used, further enhanced the adhesion already possible in the absence of the tags. Under a tensile load these "tags" transfer stress from the sealant into the enamel via shear stresses along the "tag"/enamel interfaces. Since enamel has a much higher elastic modulus than sealant, most of the stress may be transferred from the "tag" into the enamel within a short distance from the main sealant/enamel bond surface. In this case, increasing the length of penetrating sealant "tags" would not necessarily affect the stress distribution near the bond surface, and consequently would not improve the bond strength. Thus using a 30% w/w concentration of phosphoric acid as proposed by Silverstone (1974), to increase the depth of sealant penetration into etched enamel may not greatly affect the adhesion attained.

Soetopo (1975) also concluded that high bond strengths were possible in the absence of "tags", following a series of tensile bond tests of adhesion to enamel etched by various concentrations of phosphoric acid. Williams, Fraunhofer and Winter (1976) confirmed that the use of 30, 50 and 70% w/w H_3PO_4 achieved similar adhesion

with Nuva-seal and Epoxylite 9075, although bond strengths were lower than reported here.

2.5 Conclusions

In this chapter physical factors relevant to the retention of fissure sealant in the clinical situation have been discussed. While the importance of macroscopic moisture contamination has been demonstrated, results suggest that prolonged drying of the etched tooth surface, may in fact, be unnecessary.

In the studies on the use of different concentrations of phosphoric acid etchant it was intended, not merely to discover the optimum concentration, but to understand why such an etched surface should result in greater adhesion. From the previous literature one would have expected that the more severely etched the surface the higher the bond strength. Although such a tendency was observed, even the more lightly etched surfaces achieved very high bond strengths. It is therefore suggested that the role of long "tags" of penetrating sealant into the etched enamel surface has been over-estimated. In particular one may question whether the presence of longer "tags" due to a greater degree of etching should be expected to result in better retention of the sealant.

As the mode of failure of u.v. sources may take the form of a prolonged period of gradually reducing intensity rather than a sudden failure, some objective monitoring procedure is necessary for checking the output of sources in clinical use to determine when replacement bulbs are required. Such a procedure is described in the next chapter.

CHAPTER THREE

EMISSION FROM ULTRA-VIOLET SOURCES USED TO POLYMERISE FISSURE SEALANTS

3.1 Introduction

The use of u.v. radiation to polymerise a pit and fissure sealant was first described by Buonocore (1970). A number of advantages over chemically initiated sealants were claimed. After the addition of 2% by weight benzoin methyl ether, the resin remained fluid indefinitely, allowing ample time for placement on pre-treated tooth surfaces. Application of u.v. radiation then produced a rapid set, in less than 30s.

The source used initially by Buonocore (1970) was a Spectroline lamp, with which the radiation from a 100W high pressure mercury discharge lamp was directed on to the tooth surface being treated by means of an intra-oral mirror. The u.v. source presently marketed with the Nuva-seal system is the Nuva-lite, with which filtered radiation from a 50W medium pressure mercury lamp is conducted to the tooth along a fused quartz light guide. The Alphaseal fissure sealant system uses a u.v. source, called the Alphalite, shown in Fig. 3.1. Here, the radiation from a 100W super-high pressure mercury discharge lamp is directed to the tooth along a bundle of quartz fibre-optics. This system was originally described in its prototype form by Rock (1974b) and Stephen et al. (1976) as TP2206 resin and the Quartzlite, shown in Fig. 3.2.

The tips of a Nuva-lite and Quartzlite are illustrated in Fig. 3.3. The fused quartz guide attached to the Nuva-lite has a diameter of approximately 10 mm, while the fibre-optic bundle supplied with the Quartzlite has an active diameter of 2·5 mm, and is protected by a

Fig. 3.1 The Alphalite u.v. source provided for use with the u.v. sensitized resin Alphaseal by the Amalgamated Dental Co., London.

Fig. 3.2 The Quartzlite u.v. source. This was the prototype for the Alphalite, and was originally used in conjunction with the resin TP2206, later called Alphaseal.

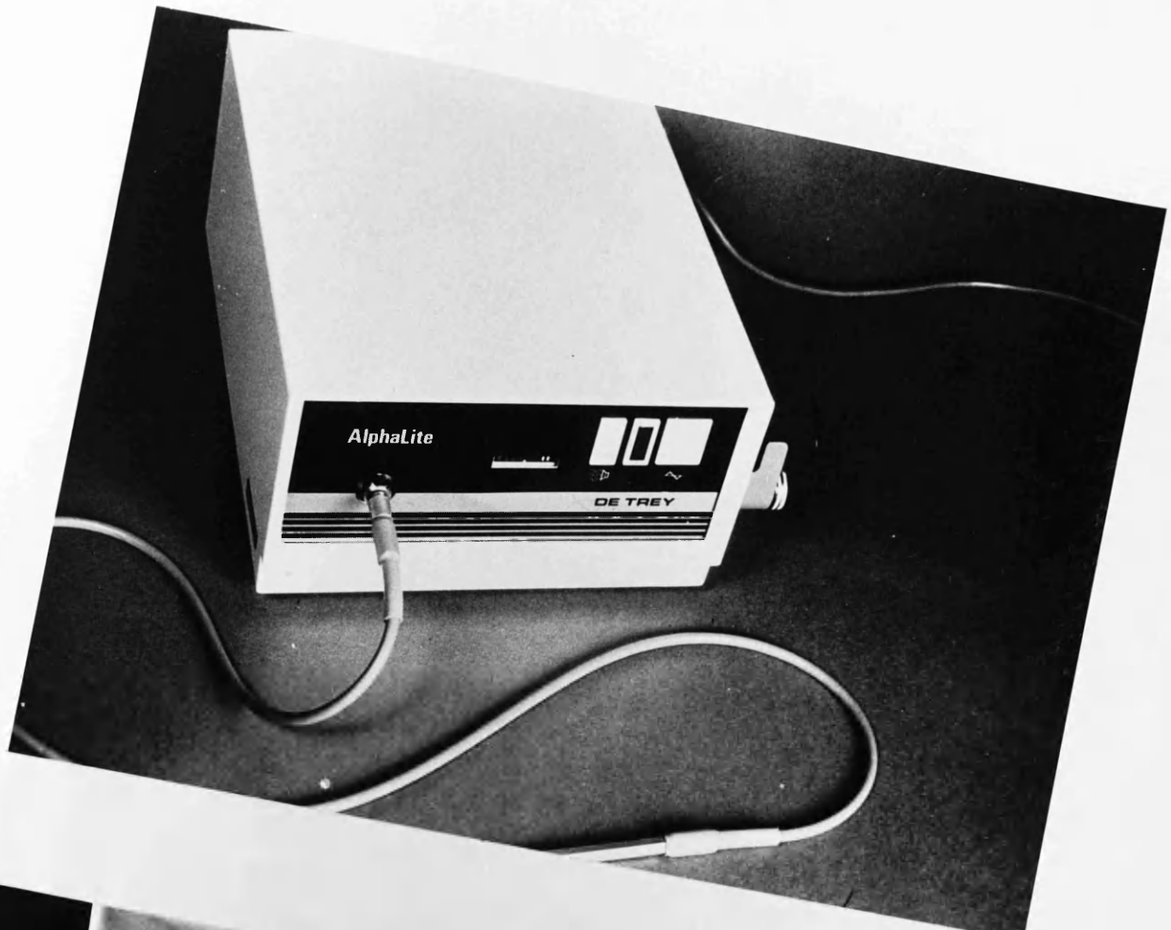
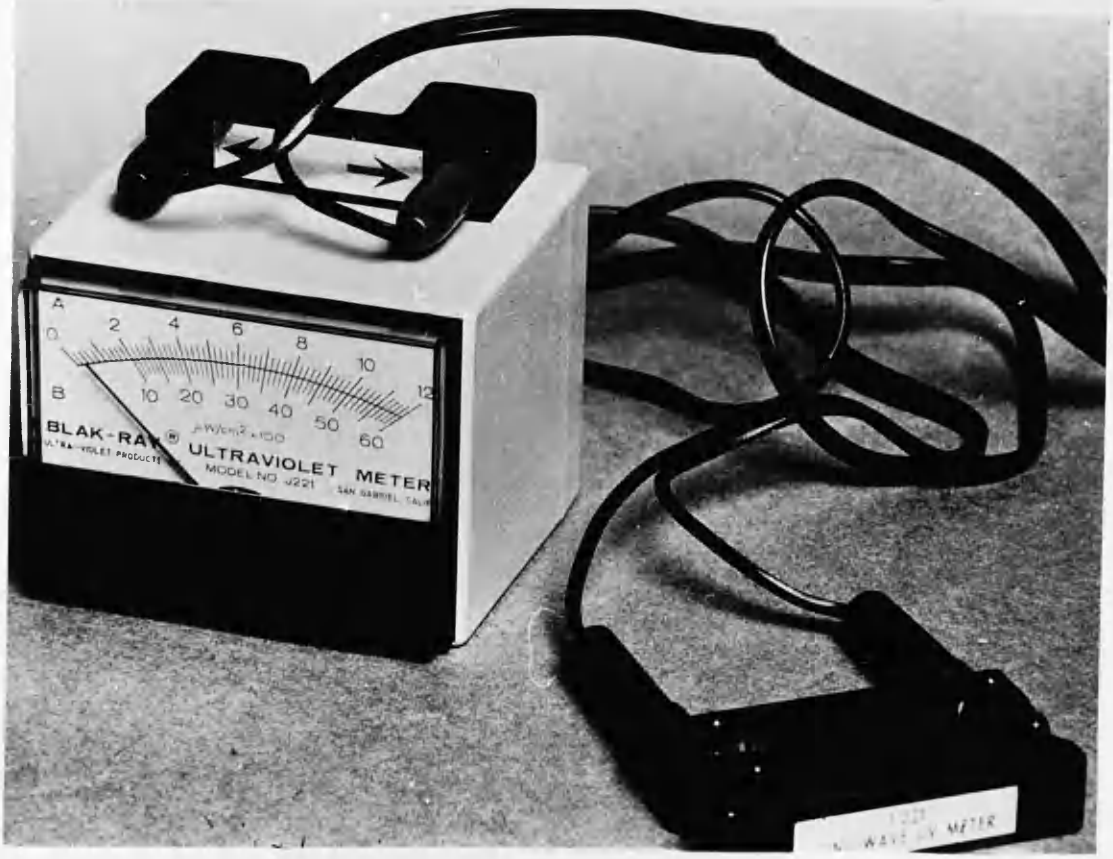

AlphaLite
DE TREY

Fig. 3.3 The light guides of the Nuva-lite and Quartzlite. Shown below is the fused quartz guide of the Nuva-lite, which has a diameter of approximately 10 mm. Shown above is the quartz fibre-optic bundle of the Quartzlite, which has an active diameter of 2·5 mm.

Fig. 3.4 The Blak-Ray Long-Wave U.V. Meter J221 with detachable sensor.


MM
mm 10 20 30 40 50 60 70 80 90 100


ULTRAVIOLET METER
MODEL NO 221

metal sheath of diameter 4·5 mm at its tip.

3.2 Materials and Methods

U.V. intensity measurements were made with a Blak-Ray u.v. meter shown in Fig. 3.4 (J221, UVP International, Inc.). A detachable sensor detected u.v. radiation of wavelengths between 300 and 400 nm, with a relative spectral response as shown in Fig. 3.5d. A comparison with Fig. 3.5a, b and c indicates that the peak sensitivity at 365 nm corresponds with the main u.v. emission from the Nuva-lite, Quartzlite and Spectroline sources, as given by Rock (1974b). Although a 15% tolerance is quoted by the manufacturers for the absolute value of the meter readings, the relative results reported here are more precise since all measurements were made with the same instrument.

The total output from each Nuva-lite was measured by locating the tip of its quartz guide in a circular aperture of diameter 10 mm cut in an aluminium screen covering the sensitive surface of the meter (Fig. 3.6). The output of u.v. radiation from the Quartzlite was found similarly, and the average intensity calculated on the basis of the illumination of a circular area of diameter 2·5 mm.

In order to measure the intensity distribution of radiation from the Nuva-lite, the u.v. sensor was fixed to the surface of the mechanical stage of a microscope and an aluminium screen with an aperture of diameter 1 mm fitted over the sensitive surface, as shown in Fig. 3.6. Using the calibrated controls of the stage to shift the sensor, intensity measurements were made across parallel planes, at various distances below the tip of a Nuva-lite guide. Thus the intensity distribution just below the guide, and the change in this distribution with distance from the guide's tip, were both investigated. Similar measurements were made on the distribution of u.v. radiation from the Quartzlite. However, because of the smaller area illuminated, an

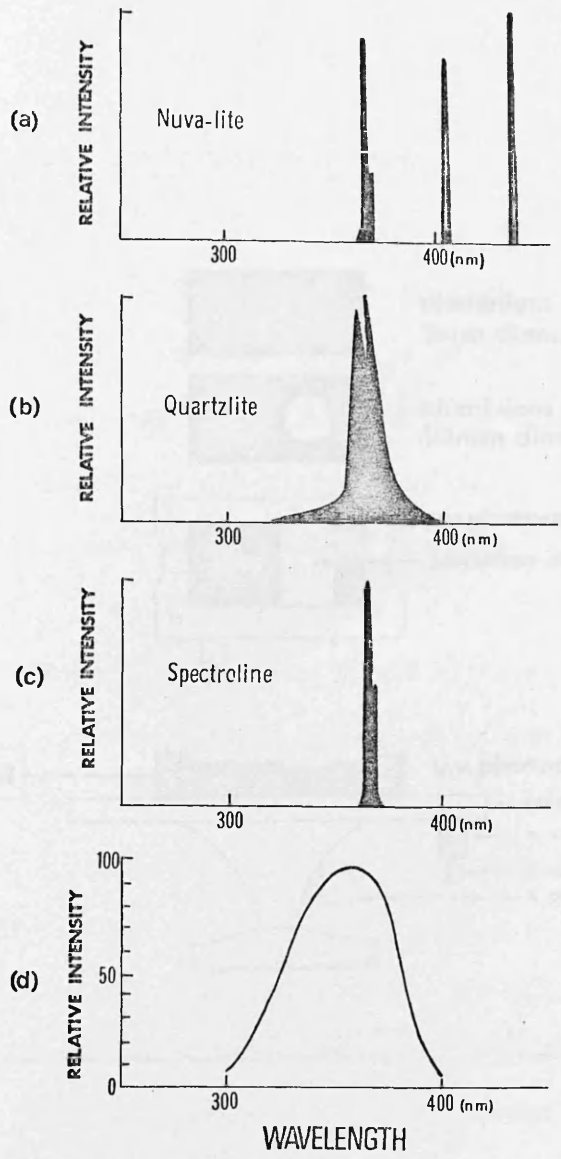


Fig. 3.5 The emission spectra of Nuva-lite, Quartzlite and Spectroline sources are shown in a, b and c resp. (Rock, 1974b). The relative spectral response quoted by the manufacturers for the Blak-Ray U.V. Meter J221 is compared in d.

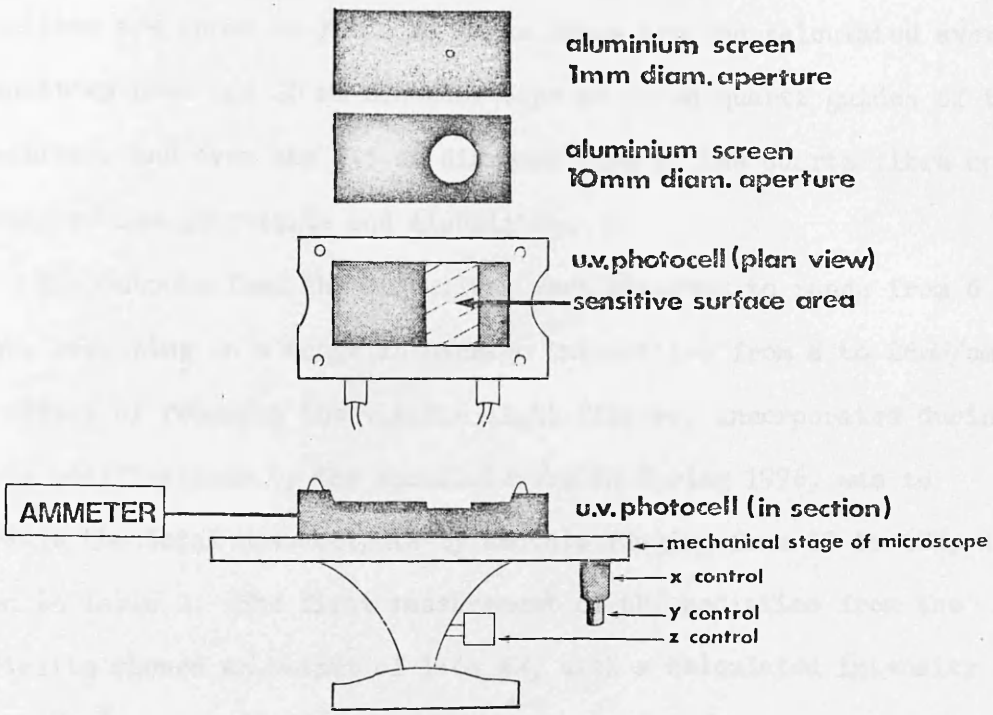


Fig. 3.6 The experimental arrangement used for making u.v. intensity measurements. The sensor of the Blak-Ray u.v. meter was fixed onto the mechanical stage of a microscope, and aluminium screens with circular apertures used to limit the area of the sensitive surface exposed.

aluminium screen with a smaller aperture (diameter 0·5 mm) was placed over the sensor.

3.3 Results

3.3.1 Total outputs and average intensities

The u.v. outputs from 12 Nuva-Lites, 1 Quartzlite and 3 Alphalites are shown in Table 2. Also shown are the calculated average intensities over the 10 mm diameter tips of fused quartz guides of the Nuva-lites, and over the 2·5 mm diameter tips of the quartz fibre optic bundles of the Quartzlite and Alphalites.

The outputs from the Nuva-lites were observed to range from 6 to 20 mW, resulting in a range in average intensities from 8 to 26mW/cm^2. The effect of removing the visible light filters, incorporated during safety modifications by the manufacturers in Spring 1976, was to increase the total u.v. outputs by amounts ranging from 10 to 62%, as given in Table 2. The first measurement of the radiation from the Quartzlite showed an output of 1·64 mW, with a calculated intensity of 33·4 mW/cm^2. A second measurement, after cleaning and re-aligning the lens system, showed an increased output of 3·02 mW, and an intensity of 61·5 mW/cm^2. The Alphalites had u.v. outputs which were similar to those from the various Nuva-lites, but the corresponding intensities were much greater.

3.3.2 Intensity distribution

The distribution of u.v. radiation within the circular areas illuminated, 1 mm below the tips of the Nuva-lite and Quartzlite, are shown in Fig. 3.7. The radiation from the Nuva-lite was mainly concentrated in a horse-shoe shaped band in the front half of the illuminated area, with the rear half receiving a much lower intensity. This general pattern was common to all Nuva-lites tested, although the

Table 2

U.V. outputs and intensities from 12 Nuva-lites, 1 Quartzlite and 3 Alphalites

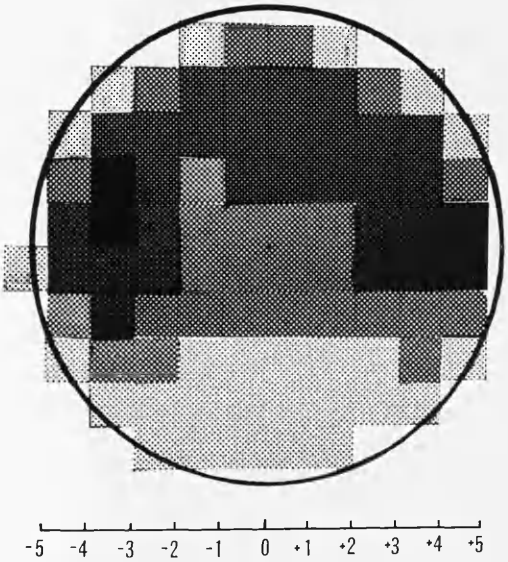
Type and Serial Number	Output mW	Intensity mW/cm^2	Increase on Removal of Filter (%)
Nuva-lite 14177	6.0 ± 0.5	7.7 ± 0.65	50
Nuva-lite 10673	7.1 ± 0.5	9.0 ± 0.65	13
Nuva-lite 19700	8.1 ± 0.5	10.3 ± 0.65	31
Nuva-lite 24393	8.1 ± 0.5	10.3 ± 0.65	25
Nuva-lite 19614	8.6 ± 0.5	10.9 ± 0.65	53
Nuva-lite 40624	9.1 ± 0.5	11.6 ± 0.65	33
Nuva-lite 24537	13.7 ± 0.5	17.4 ± 0.65	26
Nuva-lite 40606	14.7 ± 0.5	18.7 ± 0.65	10
Nuva-lite 40615	15.7 ± 0.5	20.0 ± 0.65	19
Nuva-lite 30389	17.2 ± 0.5	21.9 ± 0.65	62
Nuva-lite 30359	18.9 ± 1.0	24.1 ± 1.3	49
Nuva-lite 40747	20.2 ± 1.0	25.8 ± 1.3	15
Quartzlite[1]	1.64 ± 0.02	33.4 ± 0.4	
Quartzlite[2]	3.02 ± 0.11	61.5 ± 2.2	
Alphalite 1076	10.6 ± 0.5	216 ± 10	
Alphalite 1057	15.1 ± 0.5	308 ± 10	
Alphalite 1109	18.2 ± 0.5	361 ± 10	

Notes: 1. First measurement of the u.v. output from the Quartzlite.

2. U.V. output from the Quartzlite after cleaning and re-aligning the lens system.

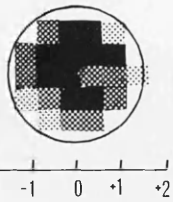
Fig. 3.7 The distributions of u.v. radiation over planes 1 mm below and parallel to the tips of the guides of a Nuva-lite and Quartzlite are represented by squares with different degrees of shading. The circles denote the extent of the visible radiation with the top and bottom of the circles corresponding to the leading and trailing edges of the guides, respectively.

NUVA-LITE



POSITION ACROSS DIAMETER (mm)

QUARTZLITE



POSITION ACROSS DIAMETER (mm)

KEY FOR U.V. INTENSITIES

0-15	mW/cm^2
15-30	mW/cm^2
30-45	mW/cm^2
45-60	mW/cm^2
60-75	mW/cm^2

extent of the asymmetry observed between front and rear halves varied from one unit to another. The intensity distribution of the improved u.v. emission from the Quartzlite was also found to be asymmetrical, at 1 mm below the tip (Fig. 3.7).

3.3.3 Variation of intensity distributions with distance along the u.v. beam

The distribution of u.v. radiation over surfaces at 0, 2, 5 and 10 mm below and parallel to the tips of a Nuva-lite and Quartzlite were compared. The intensities, measured along diameters of the two illuminated circles, from the leading edges to the rear edges, are shown in Fig. 3.8.

The concentration of radiation towards the front half of the area illuminated by the Nuva-lite was again observed, with this being particularly marked at 0 and 2 mm below the tip of the guide. However, at 5 and 10 mm below the tip this asymmetry was reduced as the radiation spread out more evenly over the illuminated area. The u.v. intensities measured were less the greater the distance from the guide tip. For example, the peak intensities measured along the diameters at 0, 2, 5 and 10 mm below the tip were 45, 40, 30 and 20 mW/cm^2, respectively.

Immediately below the tip of the fibre-optic guide attached to the Quartzlite the u.v. radiation was concentrated in a circular area of diameter 2·5 mm, with an intensity of about 50 mW/cm^2. At 2 mm below the tip, the diameter of the circular area illuminated was slightly larger at about 3·25 mm diameter, but the radiation remained concentrated at the centre with an intensity of approximately 40 mW/cm^2. At 5 and 10 mm below the tip, the circular areas illuminated were considerably larger, with diameters of about 4 and 6 mm respectively, and at both these distances the radiation was symmetrically distributed

about a central peak. Overall, the average intensity in the central area fell from approximately 50 mW/cm^2 immediately below the tip, to only 20 mW/cm^2, at a distance of 10 mm.

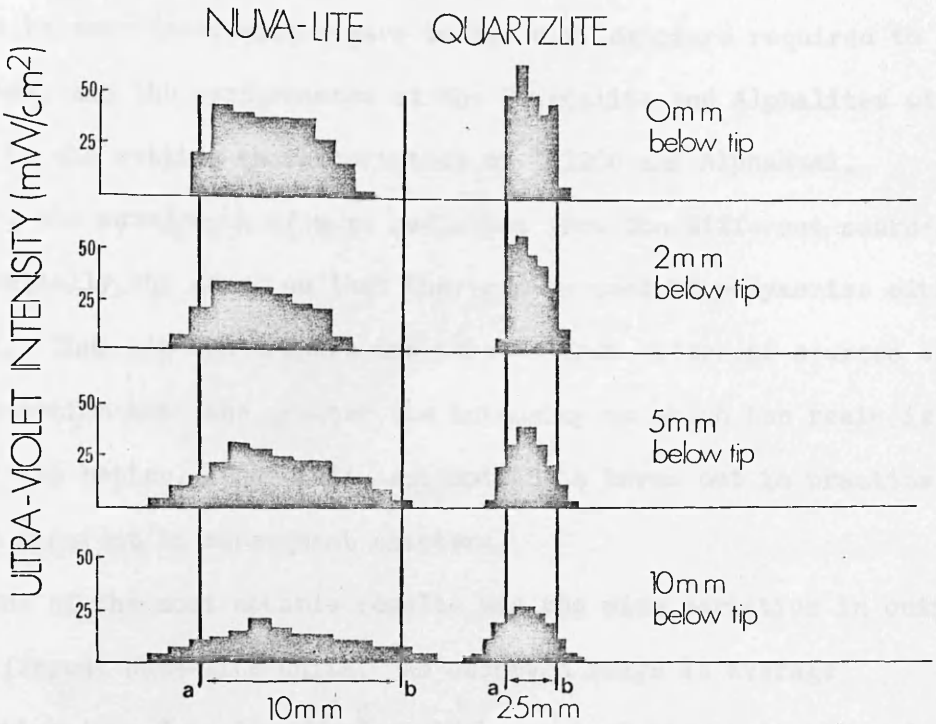


Fig. 3.8 The intensity distribution of u.v. radiation across diameters of the circular areas illuminated by the Nuva-lite and Quartzlite at 0, 2, 5 and 10 mm below the tips of their guides. The leading and trailing edges of the guides are represented by vertical lines marked a and b respectively.

3.4 Discussion

The effect of using different u.v. intensities to polymerise fissure sealants will be examined in detail in later chapters. The effectiveness of a particular u.v. source depends on the manner in which it is used, and on the setting characteristics of the resin being polymerised. Thus the performances of the Nuva-lites examined here have to be considered with regard to the u.v. exposure required to set Nuva-seal, and the performances of the Quartzlite and Alphalites with regard to the setting characteristics of TP2206 and Alphaseal. However, the wavelength of u.v. radiation from the different sources is essentially the same, so that they can be used to polymerise either sealant. Thus one can compare the outputs from different sources on the assumption that the greater the intensity to which the resin is exposed the better. That this assumption is borne out in practice will be apparent in subsequent chapters.

One of the most notable results was the wide variation in output from different Nuva-lite units. An observed range in average intensities from 8 to 26 mW/cm^2 could be part of the explanation for the inconsistent results which different operators have found in clinical trials with Nuva-seal. One reason for this large range in output is the gradual aging process which occurs in the u.v. sources used in Nuva-lites. This can result in units being used which emit considerable quantities of visible light but only low levels of u.v. radiation. In such a situation it would not be readily apparent to the operator that insufficient u.v. radiation was present.

Another factor noted to affect the performance of individual Nuva-lites was the angle of the bend in the quartz guide. This was by no means the same for all Nuva-lites examined and guides with more gradual bends were more efficient at transmitting the radiation. Indeed the output from one unit was increased by as much as 50% when

a guide with a particularly sharp bend was replaced by another with a more gradual bend.

Another factor of clinical relevance relating to the output from Nuva-lite units is the reduction due to the gradual accumulation of polymerised sealant on the tip of the quartz guide. Even seemingly insignificant quantities of cured resin on the tip of a guide were found to reduce the u.v. output by as much as 36%.

The first measurement of the u.v. output of the Quartzlite was made following its use in a clinical trial of TP2206 by Stephen et al. (1976). Further examination of this unit revealed some factors contributing to the low output. The lens system which directed the u.v. beam into the fibre optic was off alignment, and one of the lenses was partially obscured by a coating of red dust. Cleaning and re-aligning the lenses nearly doubled the u.v. output. Although the average intensity from the Quartzlite was greater than from Nuva-lites, the power emitted was considerably less, even after the lens system was corrected. The three Alphalites, which were examined at a later date, emitted as much radiation as the higher output Nuva-lites, and far more than their Quartzlite prototype. As a result of the smaller area illuminated, the intensities from Alphalites were much greater than from Nuva-lites.

To assess the effectiveness of the two types of u.v. sources in polymerising fissure sealants one has to take account of the distribution of the radiation emitted. In the clinical situation the asymmetrical distribution of radiation from the Nuva-lite would result in different areas of sealant receiving different intensities of radiation. When using a Quartzlite or Alphalite, the extent to which the different areas of sealant would receive different exposures to u.v. radiation would depend primarily on the particular scanning motion used, rather than on the intensity distribution. Each part of the sealant

would receive radiation only intermittently, and unless the source was sufficiently intense some sealant could receive inadequate exposure.

The distance maintained between the sealant and u.v. source also affects the intensity to which the resin is exposed. Moving away from the Nuva-lite, the u.v. intensity falls off as shown in Fig. 3.8. Therefore, when using this system it would be desirable to keep the tip of the guide as close to the sealant surface as possible. When using one of the fibre-optic units only a part of the sealant can be illuminated at a given time. Thus by holding the tip of the guide further from the sealant surface, the area illuminated may be usefully increased, since the amount of scanning motion required would be reduced. Hence the distance from the sealant surface may be less critical when using the fibre optic systems.

The results of investigations into the role of u.v. radiation in the setting of fissure sealants are presented in the following chapters, and polymerisation mechanisms of u.v. activated sealants are discussed in Appendix I.

would receive radiation only intermittently, and unless the source was sufficiently intense some sealant could receive inadequate exposure.

The distance maintained between the sealant and u.v. source also affects the intensity to which the resin is exposed. Moving away from the Nuva-lite, the u.v. intensity falls off as shown in Fig. 3.8. Therefore, when using this system it would be desirable to keep the tip of the guide as close to the sealant surface as possible. When using one of the fibre-optic units only a part of the sealant can be illuminated at a given time. Thus by holding the tip of the guide further from the sealant surface, the area illuminated may be usefully increased, since the amount of scanning motion required would be reduced. Hence the distance from the sealant surface may be less critical when using the fibre optic systems.

The results of investigations into the role of u.v. radiation in the setting of fissure sealants are presented in the following chapters, and polymerisation mechanisms of u.v. activated sealants are discussed in Appendix I.

CHAPTER FOUR

MICROHARDNESS STUDIES ON THE SETTING CHARACTERISTICS OF FISSURE SEALANTS

4.1 Introduction

Since the rate of set of u.v. activated sealants may depend on the u.v. intensity applied, the variation in the outputs of u.v. sources noted in the previous chapter, may be a factor contributing to the variable retention reported in different clinical trials. In this chapter the setting characteristics of Nuva-seal and Alphaseal are assessed using microhardness measurements after various exposure times to different u.v. intensities. For comparison, the setting behaviours of three chemically curing sealants were also investigated.

4.2 Materials and Methods

The microhardness of pit and fissure sealants was measured with a Wallace Micro Indentation Tester (H.W. Wallace Co. Ltd., Croydon, Surrey) suitable for assessing the hardness of rigid and semi-rigid plastics and the state of cure of synthetic resins. The use of this instrument and the effect of various testing parameters has been discussed in detail by Huggett (1975).

Each test specimen was mounted on a glass slide, as illustrated in Fig. 4.1. A circular hole of diameter 8 mm was cut in a square sheet of modelling wax, which was pressed on to one end of a glass slide, providing a 1·5 mm deep well which was filled with freshly prepared resin. A glass slip was placed over the sealant and pressed against the upper surface of the wax, providing a flat face for the sealant.

Samples of the u.v. polymerised sealants were set by exposure to radiation at 365 nm wavelength from a Blak-ray u.v. source (U.V.

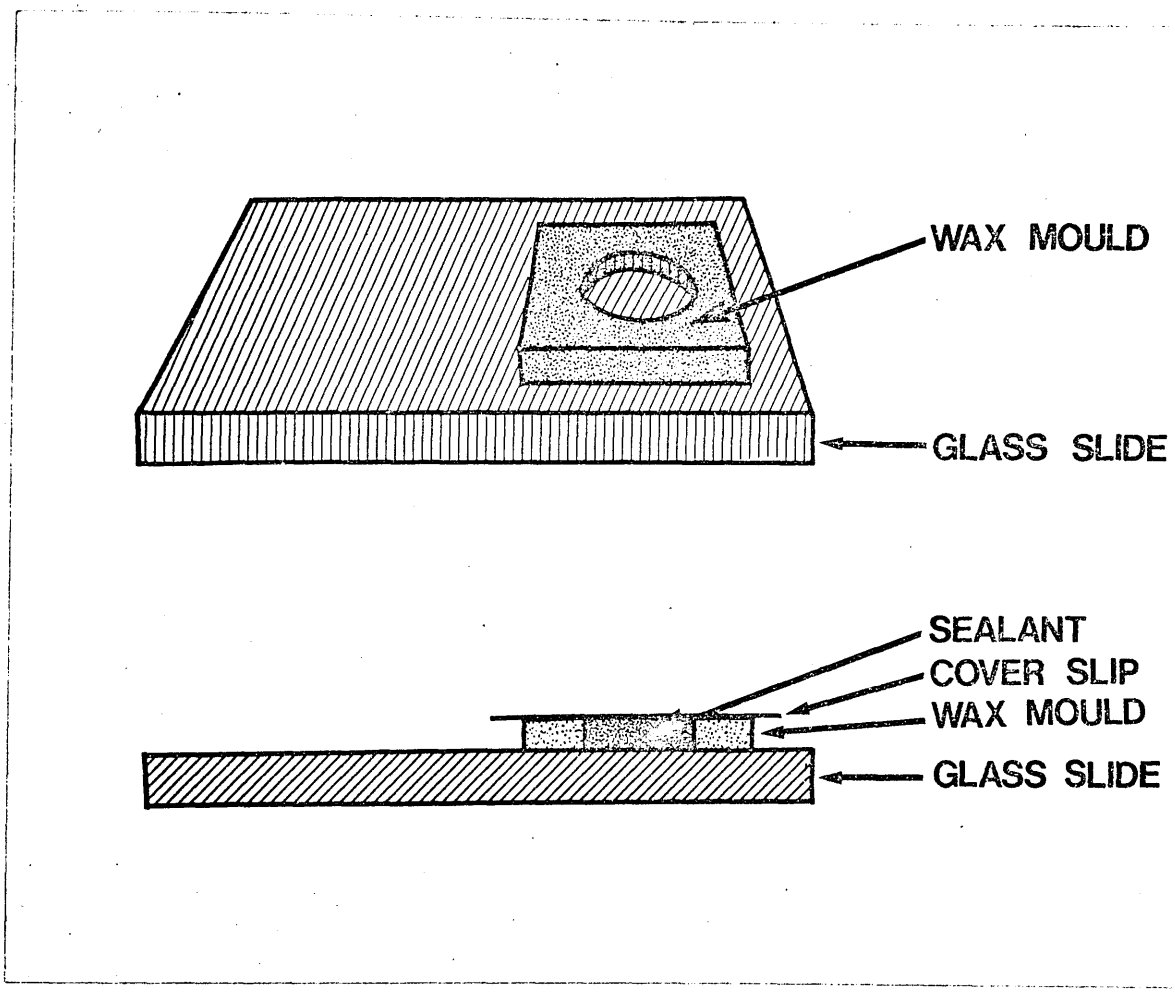


Fig. 4.1 Sample preparation for microhardness measurements on fissure sealants.

Products Inc., San Gabriel, California, U.S.A.) at intensities measured on a Blak-Ray Long Wave U.V. Meter J221 (U.V. Products Inc.). Nuva-seal samples were polymerised by exposure, for periods of 10, 20, 30, 40, 50 and 60s, to intensities of 5, 10 and 20 mW/cm^2. Since initial tests showed that Alphaseal required more u.v. radiation to set to a measurable hardness, longer exposure times of 30, 60, 90 and 120s at intensities of 20 and 30 mW/cm^2 were selected.

Four samples of the resins were set using each exposure time and intensity. Four samples each of Epoxylite 9075 (Lee Pharmaceuticals Co. Ltd.), Concise Enamel Bond (3M Co. Ltd.) and Aspa Cement (Amalgamated Dental Co. Ltd., London) were prepared by mixing the materials according to the manufacturers' instructions and filling wax moulds as shown in Fig. 4.1.

Nuva-seal and Alphaseal samples were tested immediately following exposure to u.v. radiation, while Epoxylite 9075 and Enamel Bond were maintained for 10 min, and Aspa Cement for 30 min at $37^{\circ}C$ before the first test. To assess each sample the cover slip was first removed with a blade to expose the sealant.

A primary load of 0·001 kg was applied, and the increase in surface penetration by a standard Vickers diamond indentor, on adding a further load of 0·3 kg for 30s, was measured to a precision of 0·25 μm. Ten such indentation measurements were made and the mean value used in the following formula to calculate the Woxen Hardness Number (Woxen, 1944).

$$Hw = \frac{(3{\cdot}783 \times 10^{-2})\ P_2 \left[1 - \sqrt{P_1/P_2}\right]^2}{(h_2 - h_1)^2}$$

where,

Hw = Woxen Hardness (kg/mm^2)

P_1 = Initial load (kg)

P_2 = Final load (kg)

h_1 = Indentation under initial load (μm)

h_2 = Indentation under final load (μm)

The specimens were stored dry at 37°C. Further measurements of the microhardness of the same samples were made at 1 hour, 24 hours, 1 day, 1 month and 3 months after the initial set.

Since it became apparent from initial tests that Alphaseal was not setting uniformly, a further series of samples of differing thickness was prepared to determine the effect of u.v. absorption on the setting of Alphaseal in depth. By inverting these samples, sealant in contact with the cover slip received an intensity reduced by absorption in passing through the depth of the sealant sample. After performing ten indentation measurements, the thickness of each sample was determined with a micrometer. Thus the variation in hardness with distance below the exposed sealant surface could be plotted. The reflection and absorption of radiation by the glass slide was measured with the Blak-Ray meter described in Chapter Three and intensity values adjusted to compensate.

4.3 Results

Shown in Fig. 4.2 is the microhardness reached by samples of Nuva-seal exposed to 5 mW/cm^2 for periods from 10 to 60s, where each point represents the mean of four samples. For a 30s exposure, a

hardness of 5 kg/mm^2 was initially recorded. Longer exposure up to 60s produced no increase in this initial hardness. Also shown is the increased hardness of the same samples stored for 1 hour, 1 day, 1 week, 1 month and 3 months after the u.v. exposure.

The corresponding results when the u.v. intensity was doubled to 10 mW/cm^2, are given in Fig. 4.3 which shows that the increased intensity resulted in a more rapid set, with a measurable hardness after only 15s. Samples exposed for longer periods were found to be slightly harder, and all samples showed considerable hardening over the 3 month test period.

The data in Fig. 4.4 illustrate the effect of doubling the intensity again, to 20 mW/cm^2. Here, the sealant set after only 10s, with samples being slightly harder after longer exposures. Once more, polymerisation continued throughout the 3 month period.

Compared in Fig. 4.5 is the microhardness recorded immediately after exposure to the three intensities, 5, 10 and 20 mW/cm^2, showing that the higher the intensity used, the faster was the set, and the greater the resulting hardness.

The data in Fig.4.6 detail the initial setting hardness of Alphaseal after exposure to a u.v. intensity of 20 mW/cm^2 for periods of 30, 60, 90 and 120s. Increasing the applied intensity to 30 mW/cm^2 produced little increase in the rate set of this material as shown in Fig. 4.7. For both intensities Alphaseal exhibited greater hardness as the exposure was continued up to 120s. All the specimens showed a marked increase in hardness during storage, with those which were initially softest hardening the most, so that after the first week, samples tended toward similar values, and showed only slight hardening with further storage up to 3 months.

Microhardness values for the undersurfaces of samples of Alphaseal of different thicknesses, exposed to 30 mW/cm^2 for 120s,

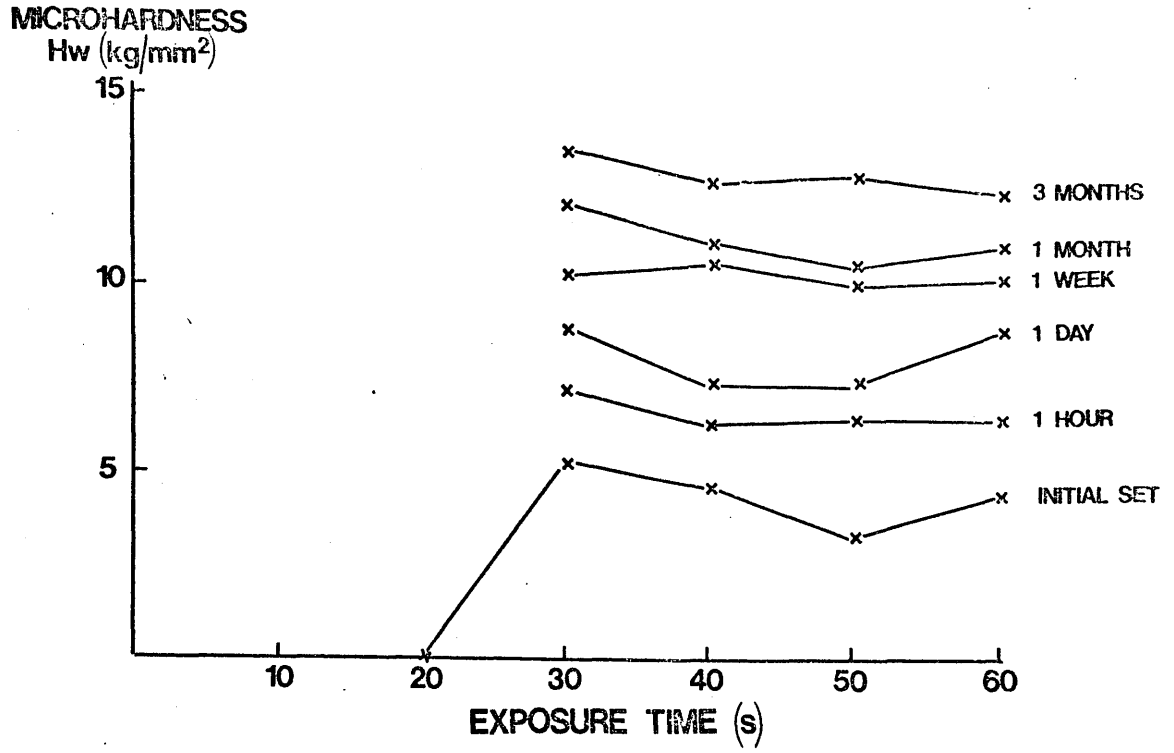


Fig. 4.2 Setting of Nuva-seal on exposure to an intensity of 5 mW/cm^2 of u.v. radiation.

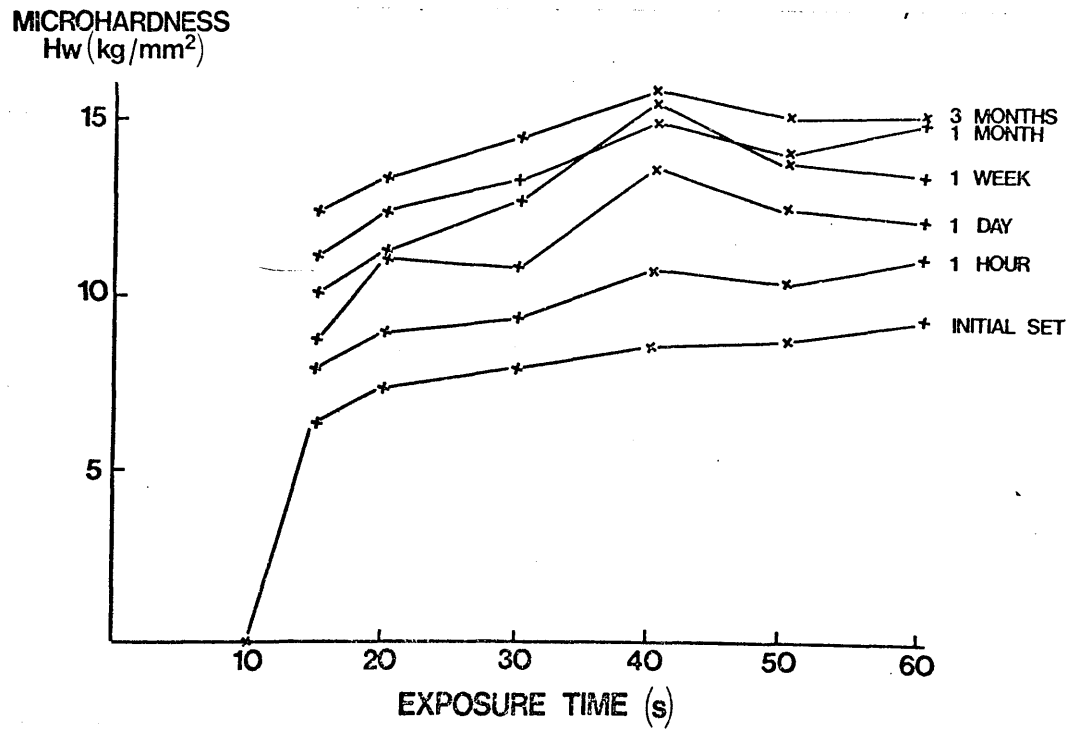


Fig. 4.3 Setting of Nuva-seal on exposure to an intensity of 10 mW/cm^2 of u.v. radiation.

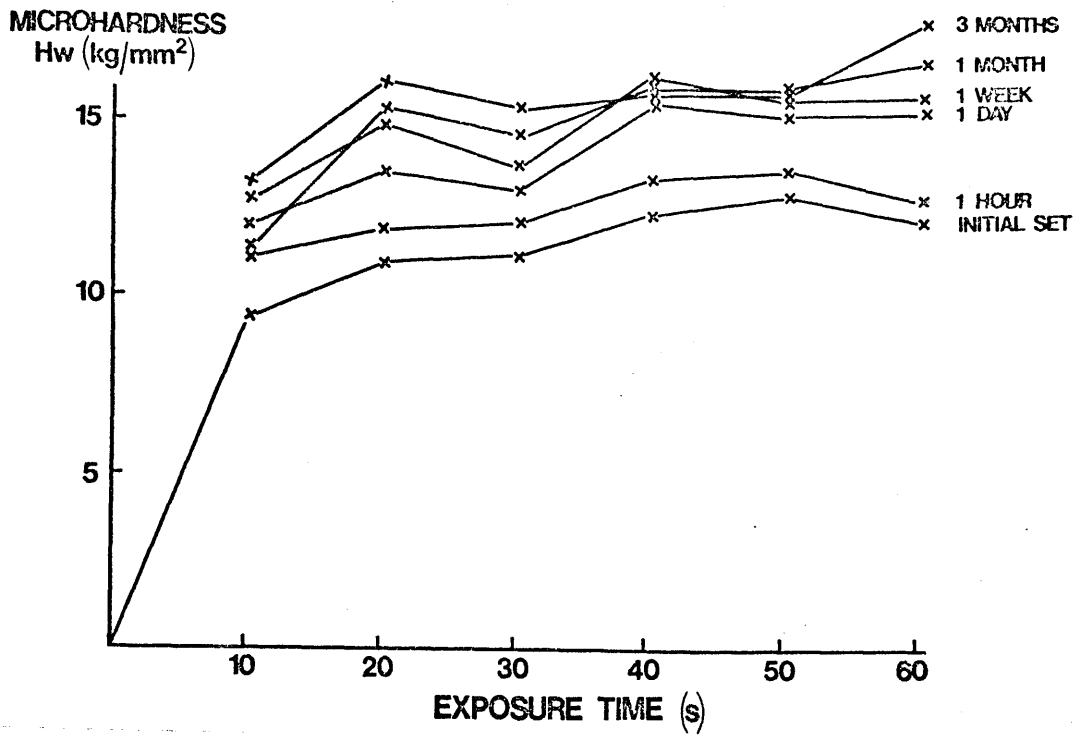


Fig. 4.4 Setting of Nuva-seal on exposure to an intensity of 20 mW/cm^2 of u.v. radiation.

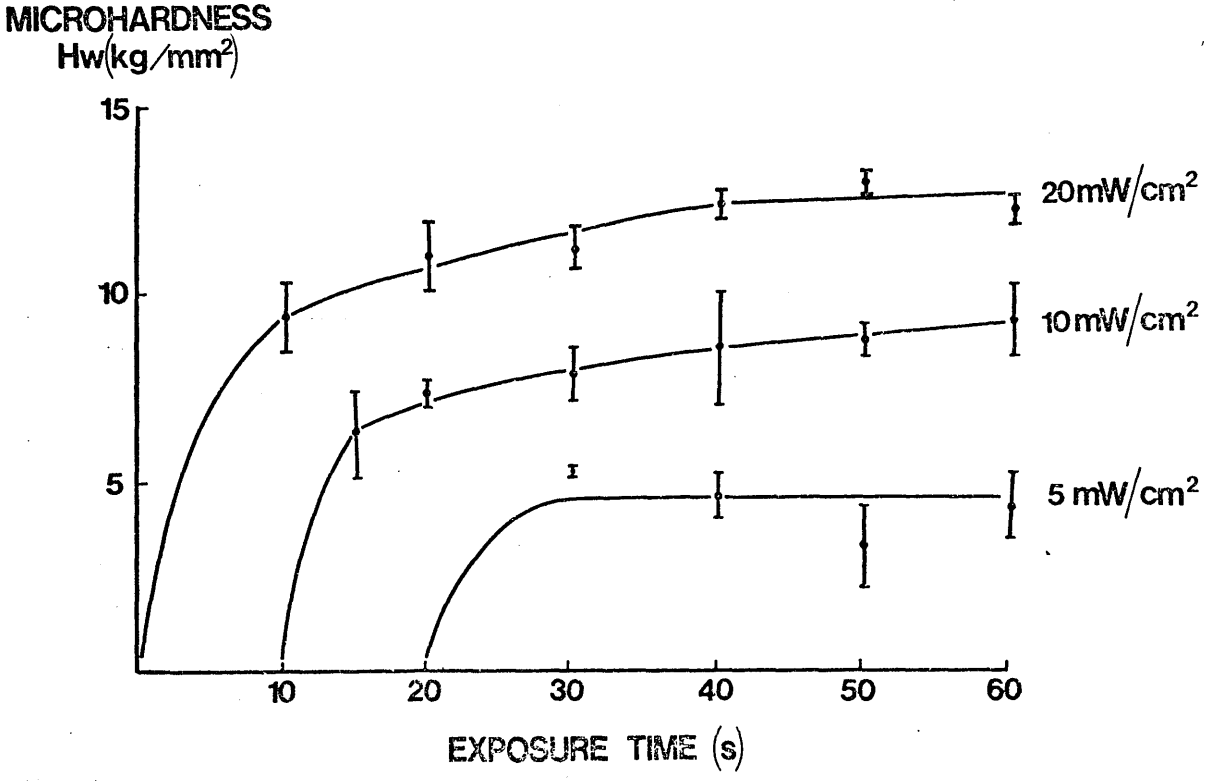


Fig. 4.5 A comparison of the initial hardness of Nuva-seal after exposure to intensities of 5, 10 and 20 mW/cm^2 of u.v. radiation.

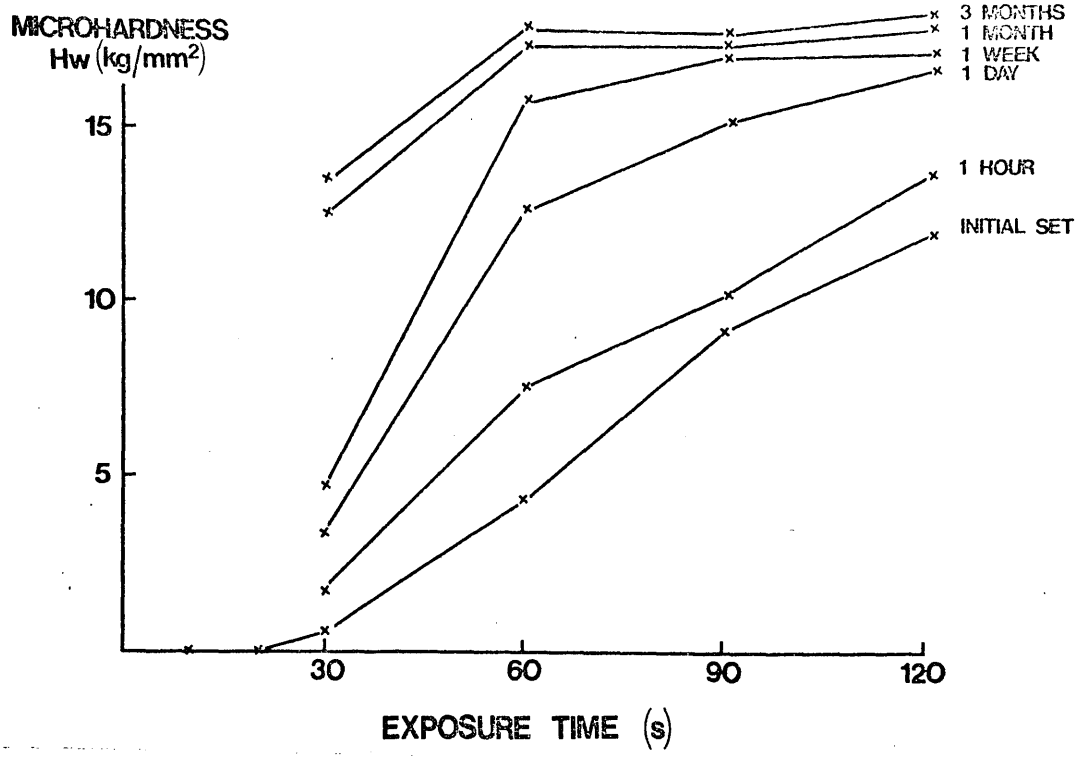


Fig. 4.6 Setting of Alphaseal on exposure to an intensity of 20 mW/cm^2 of u.v. radiation.

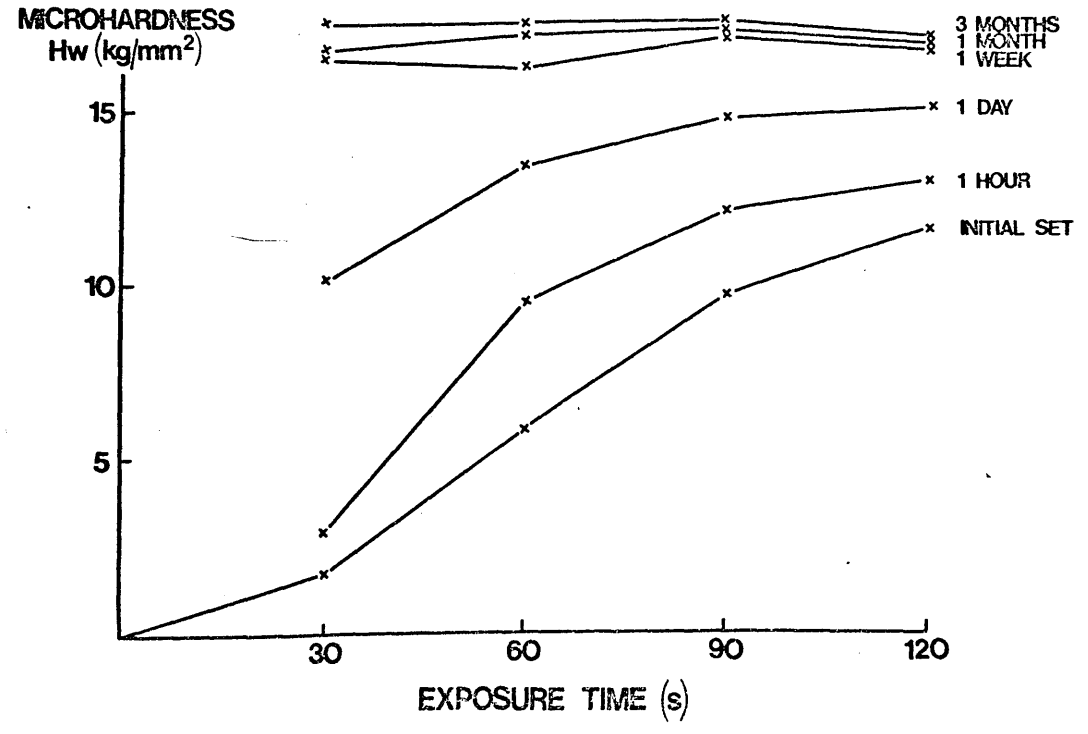


Fig. 4.7 Setting of Alphaseal on exposure to an intensity of 30 mW/cm^2 of u.v. radiation.

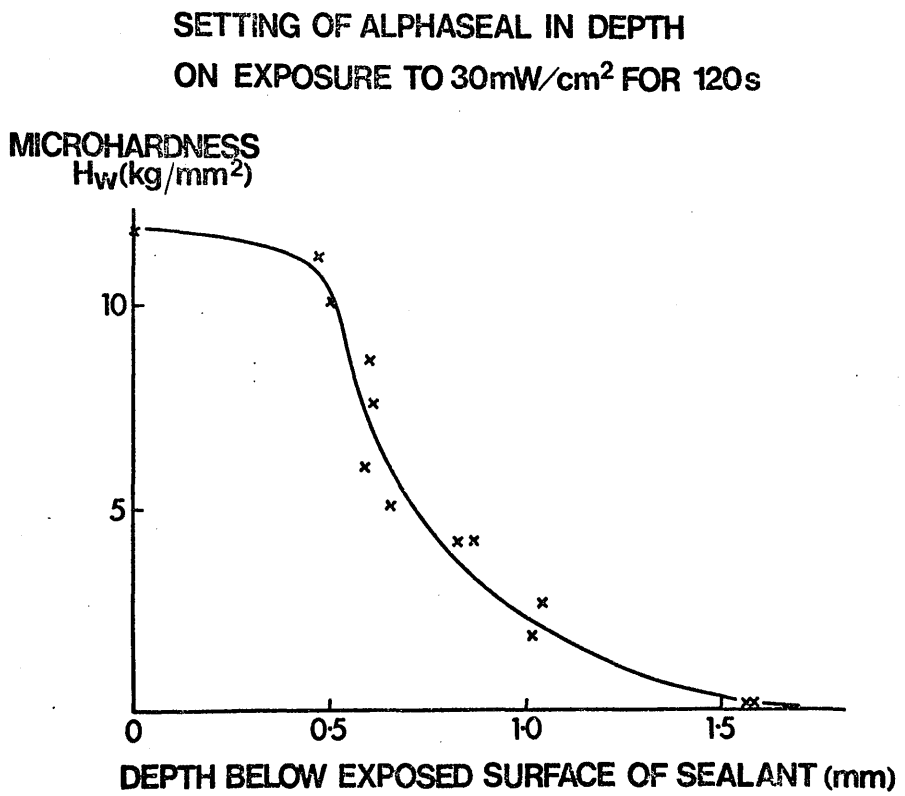


Fig. 4.8 Setting of Alphaseal in depth on exposure to an intensity of 30 mW/cm^2 of u.v. radiation for 120s.

Table 3

Microhardness of chemically polymerised fissure sealants.

	Epoxylite 9075 kg/mm^2	Concise Enamel Bond kg/mm^2	Aspa Cement kg/mm^2
Initial set	10.93 ± 1.15	$7.51 \pm .62$	$4.78 \pm .52$
1 Hour	11.65 ± 1.26	$11.02 \pm .26$	20.71 ± 3.32
24 Hours	$13.19 \pm .75$	$14.35 \pm .08$	45.41 ± 2.63
1 Week	$13.50 \pm .54$	$15.83 \pm .09$	54.76 ± 1.33
1 Month	$14.49 \pm .81$	$16.40 \pm .16$	$55.80 \pm .86$
3 Months	$14.49 \pm .97$	$16.49 \pm .29$	55.28 ± 1.31

are plotted in Fig. 4.8. At a depth of 0·5 mm the sealant had a hardness similar to that at the surface. However, samples of greater thickness revealed that the hardness of Alphaseal decreased with depth, beyond 0·5 mm, until at over 1·5 mm, only negligible values were recorded.

The microhardness values recorded over the three month test period, for the three chemically polymerising sealants are compared in Table 3.

4.4 Discussion

The results reported have confirmed the findings of Williams, Fraunhofer and Winter (1975b) and Watkins (1975) that sealants continue to increase in hardness long after their initial setting. Nuva-seal, Alphaseal, Epoxylite 9075 and Concise Enamel Bond tended towards similar values in the range 10 - 20 kg/mm^2 during the three month test period. Aspa Cement differed from the other sealants in having an initially slower set and higher final hardness of about 55 kg/mm^2. Such a relatively high value requires careful interpretation. In the Wallace Test used here the indentation was recorded while the sample remained under load, so that both plastic and elastic deformation of the test materials was detected. Due to the high elastic modulus of Aspa Cement, most of the indentation on this material arose from plastic deformation. The four other sealants have lower elastic moduli, so that additional indentation resulted from their elastic deformation.

Previous microhardness studies on fissure sealants have not investigated the effect of varying the u.v. exposure used to set sealants. In these tests on Nuva-seal, the use of higher intensities of u.v. radiation produced both a more rapid set and greater microhardness. For the two higher intensities used, a rapid initial increase was followed by more gradual hardening with continued exposure.

However it is important to note that a short exposure to a high intensity produced a harder set than could be achieved merely by prolonging illumination at a lower intensity. For example, a 10s exposure at 20 mW/cm^2 produced a greater hardness than a 60s exposure at 10 mW/cm^2.

These effects are of considerable relevance to the clinical results achieved with Nuva-seal, since a wide variation in Nuva-lite u.v. outputs was noted in Chapter Three. A Nuva-lite with a low output may fail to set all or part of the Nuva-seal covering a tooth. Even if the exposure was continued beyond the manufacturers' recommended periods of 20s for premolars and 30s for molars, the sealant would still only set to a reduced hardness.

The setting characteristics of Alphaseal were found to be markedly different from those of Nuva-seal. An exposure of some two minutes was necessary to set the resin to a hardness comparable to that reached by Nuva-seal in 10 to 20s. Increasing the intensity from 20 to 30 mW/cm^2 only produced a slightly faster set, and it would seem that very much higher intensities would be necessary to set the resin at a rate comparable to that of Nuva-seal.

During the preparation of these Alphaseal samples it became apparent that although a surface layer was setting rapidly, the samples were not setting completely in depth. Low microhardness values were recorded because the hard surface layer was not supported by the soft resin underneath. Measurements of the hardness of Alphaseal in depth confirmed these findings, suggesting that the u.v. radiation was not readily penetrating the sealant. This proposition has been confirmed by subsequent studies on the u.v. absorption by Nuva-seal and Alphaseal, presented in the next chapter.

Only two clinical trials have been reported with the Alphaseal fissure sealant system (Rock, 1974a; Stephen et al., 1976).

Both research workers used prototypes of the Alphalite called a Quartzlite and the resin was then known as TP2206. Rock found very promising results with 82% of the sealed teeth being completely sealed after one year. However, Stephen et al. found only 2.3% of the TP2206 applications were intact after one year. This second trial used two different Quartzlite units, one of which has since been examined and found to have an output of only 1.6 mW. This would have provided an average intensity of about 33 mW/cm^2 over a 2.5 mm diameter circle immediately below the tip of the quartz fibre-optic bundle used to transmit the radiation. It was shown in Chapter Three that the radiation from this fibre-optic diverges rapidly so that an intensity of about 15 mW/cm^2 would more likely have been applied in practice. Since a scanning procedure was necessary to cover the entire area of sealant during clinical application, each part of the sealant layer would have received this low intensity of radiation only intermittently. When one takes into account the manufacturer's suggestion that a 15 to 20s exposure time should be sufficient to set the resin, it seems hardly surprising that Stephen et al. (1976) found such poor sealant retention. In view of the low output from one of the Quartzlite units used in this trial, a scanning period well in excess of two minutes would have been necessary to achieve a hard set of the resin.

Later production models of the Alphalite discussed in Chapter Three had outputs far in excess of at least one of the units used by Stephen et al. (1976). The intensities produced by these new models are about 300 mW/cm^2 over the 2.5 mm diameter circular area immediately below the fibre-optic tip. Even after allowing for the spread in radiation below the guide tip and intermittent exposure due to the scanning procedure employed, Alphaseal may set sufficiently rapidly on exposure to these new Alphalites.

Thus the very poor sealant retention reported by Stephen et al. (1976) is probably explained by the extremely low output of the particular u.v. source used. Fortunately, the high outputs available from the new Alphalites should allow this problem to be overcome.

CHAPTER FIVE

ULTRA-VIOLET ABSORPTION BY NUVA-SEAL AND ALPHASEAL

5.1 Introduction

In the previous chapter microhardness measurements on Alphaseal demonstrated that surface layers set rapidly, but that setting in depth was slow. To assess whether this was due to the failure of the u.v. radiation to penetrate deeply into Alphaseal, the u.v. absorption characteristics of Nuva-seal and Alphaseal have been compared, and the contributions of the different constituents to the absorption by Alphaseal examined.

5.2 Materials and Methods

The absorption coefficients of Nuva-seal and Alphaseal were found by measuring the transmission of u.v. radiation at 365 nm wavelength through different depths of sealant. The radiation source used was an Alphalite with an output of 15 mW, and an u.v. emission primarily at 365 nm wavelength. The Blak-Ray U.V. Meter, described in Chapter Three, was used to measure the incident and transmitted intensities of radiation.

The measurement technique is illustrated in Fig. 5.1. A circular hole of diameter 8 mm was cut in a square sheet of model casting wax, and the wax pressed against a glass cover slip to provide a well into which sealant was poured. A second cover slip was then placed over the sealant and pressed against the wax. The intensity of u.v. radiation passing through a 5 mm diameter aperture in a steel screen over the sensitive surface of the u.v. meter was first measured. The prepared sealant sample was then placed over the aperture and the fraction of incident radiation passing through the sample recorded.

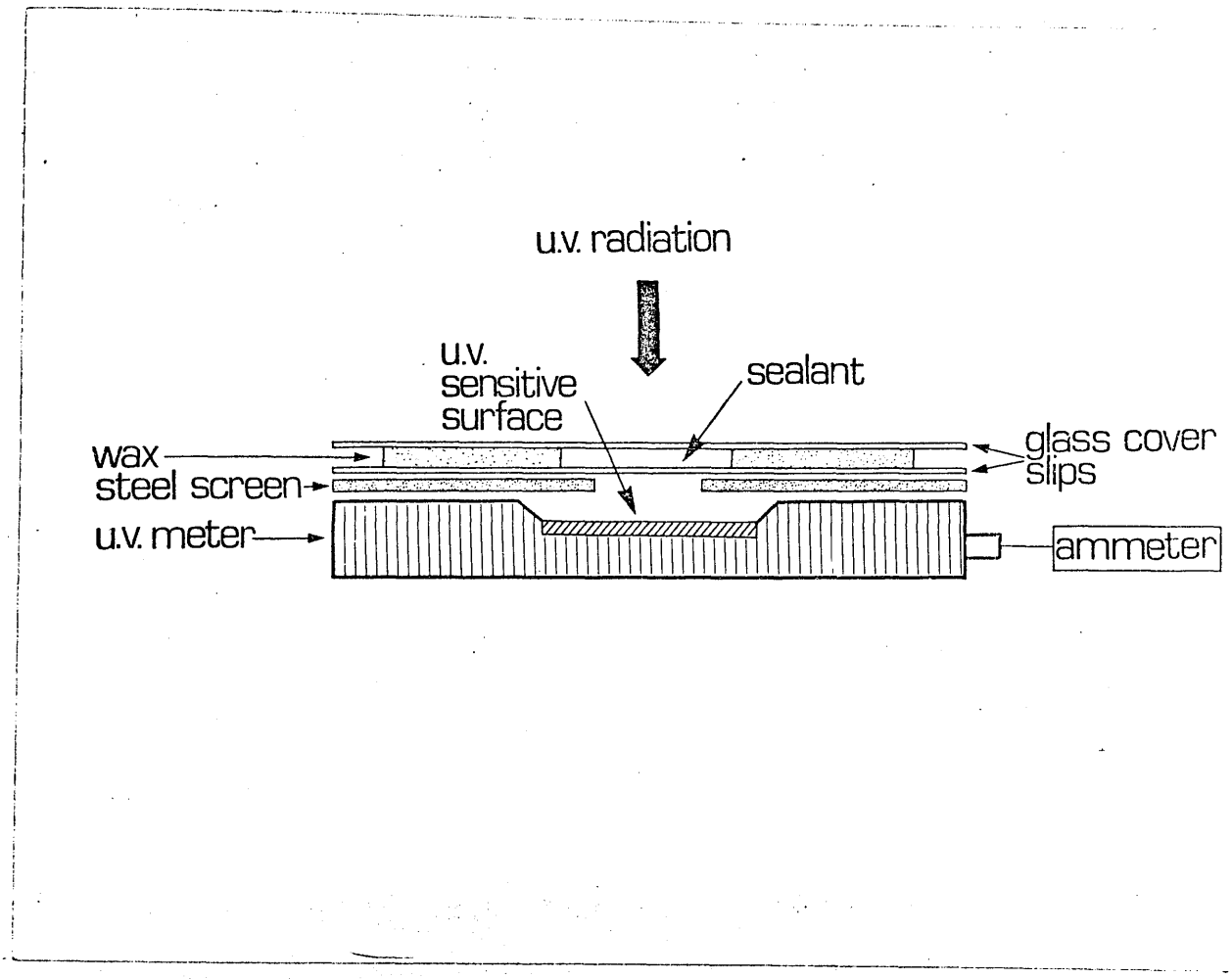


Fig. 5.1 Experimental arrangement for measuring the u.v. absorption by fissure sealants.

The absorption coefficients were calculated from the initial absorption on first exposure to the u.v. radiation and the change in transmission during polymerisation was also observed. Depths of sealant over 0.2 mm were produced using different thicknesses of wax, and for depths less than 0.2 mm one or more layers of adhesive tape were used in place of the wax. The depth of each sample was determined by removing the cover slips and measuring the thickness of the polymerised disc with a micrometer. After correcting for background infra-red radiation which was not absorbed by the sealant but was detected by the meter, and for reflections at the interfaces and absorption in the cover slips the transmitted intensities were plotted on a log scale, against the sample thickness in mm. The absorption coefficients were then calculated from the gradients of the resulting straight lines.

The absorption coefficient of Nuva-seal prepared according to the manufacturer's instructions, was first obtained, while that of Alphaseal was measured at catalyst concentrations of 1, 3 and 5% w/w, using normal resin which contains a u.v. absorbing fluorescent dye, and a batch of resin without this additive. For each catalyst concentration, approximately 250 mg of resin was dispensed into a P.T.F.E. mixing pot, and the exact weight of resin measured on a precision balance. The volume of catalyst necessary to obtain the required concentration was calculated, using the known density of the catalyst, and dispensed into the mixing pot from a micrometer syringe to a precision of 0·2 µl. The catalyst and resin were then mixed using a plastic spatula. The same batch of Alphaseal resin and catalyst was used throughout the study and all measurements of the transmission through different depths of sealant for a particular concentration of catalyst were carried out with the same resin mix. The change in absorption during polymerisation, at intensities typical of those used clinically was determined by measuring, over a period of 60s, the

transmission through 0·55 mm thick samples of Nuva-seal and Alphaseal using u.v. intensities of 20 and 250 mW/cm^2, respectively.

5.3 Results

Absorption by the Nuva-seal resin at 365 nm wavelength was not detected in the absence of the initiator. The transmission of the radiation through various depths of initiated Nuva-seal resin is shown in Fig. 5.2. The straight line fitted to the data indicates that the intensity I(x) at a depth x(mm) into the sealant is represented by the equation,

$$I(x) = I_o \, e^{-ax}$$

where, I_o = intensity of radiation entering the surface of sealant,

and, a = absorption coefficient (mm^{-1}).

The absorption coefficient of Nuva-seal was calculated to be $0{\cdot}165 \pm 0{\cdot}005$ mm^{-1}, from the gradient and the error in the gradient for the straight line best fitting the data in Fig. 5.2, using a least squares fit. The transmission of radiation through Alphaseal at catalyst to resin concentrations of 1, 3 and 5% w/w, with a u.v. fluorescent dye, is shown in Fig. 5.3. The increased transmission through samples of Alphaseal at the same catalyst concentrations where no fluorescent dye was present, is shown in Fig. 5.4. The absorption coefficients were calculated from the gradients of the straight lines best fitting the data in Figs. 5.3 and 5.4 and are shown as a function of the catalyst concentration in Fig. 5.5. The fluorescent dye made a constant contribution to the absorption coefficient of about 2·5 mm^{-1}, and the remainder of the absorption coefficient was proportional to the catalyst concentration, while the basic resin did not significantly absorb the radiation in its unpolymerised state. The absorption of both Nuva-seal and Alphaseal changed during polymerisation. The gradual reduction in

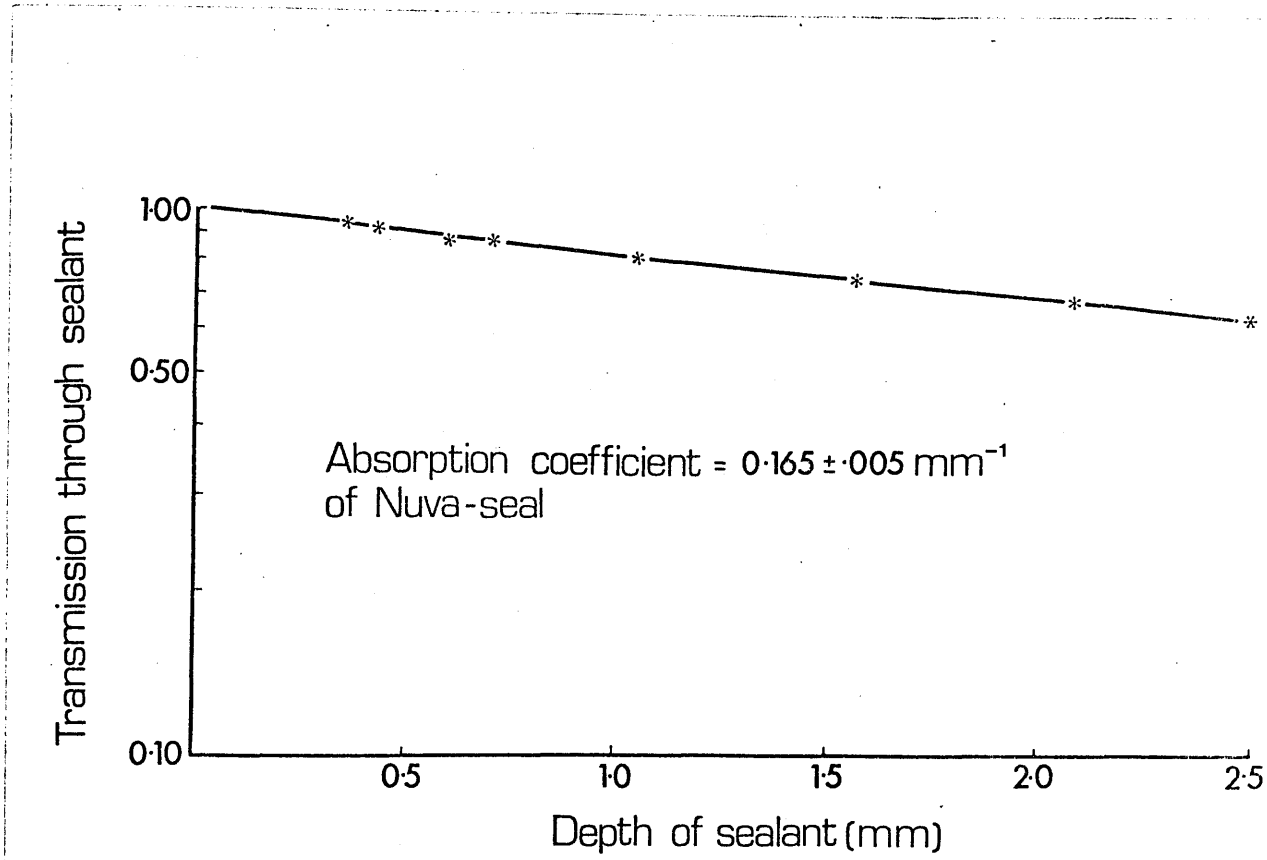


Fig. 5.2 Transmission of u.v. radiation at 365 nm wavelength through Nuva-seal.

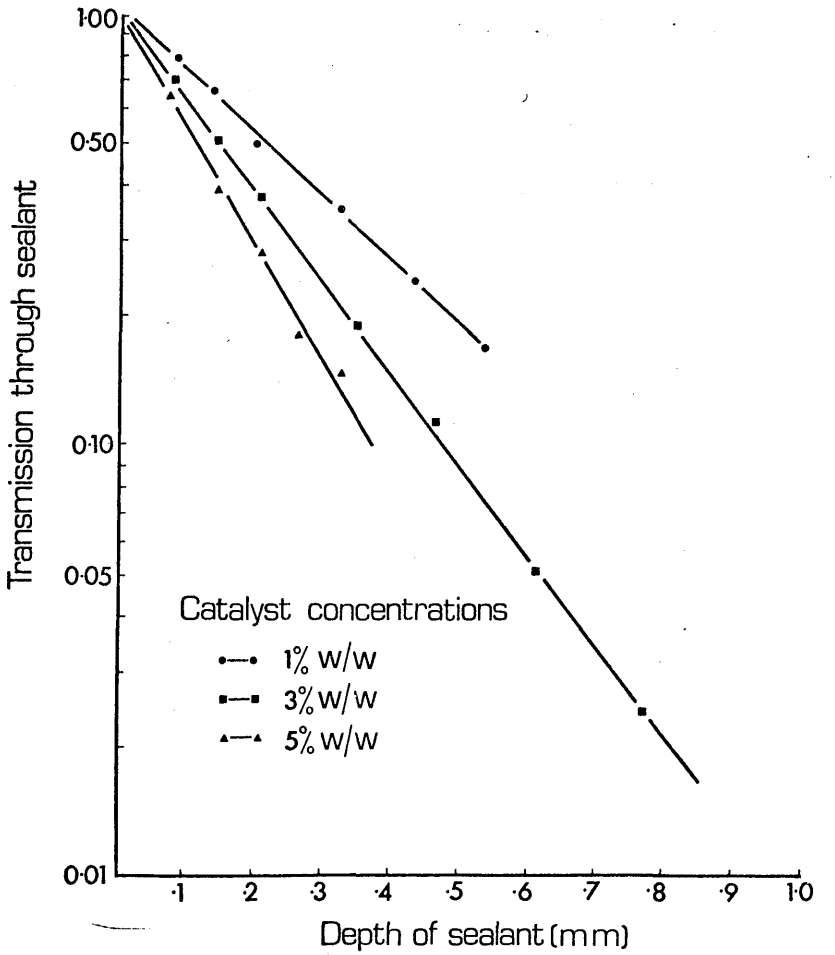


Fig. 5.3 Transmission of u.v. radiation at 365 nm wavelength through Alphaseal containing 1, 3 and 5% w/w catalyst, and a fluorescent dye.

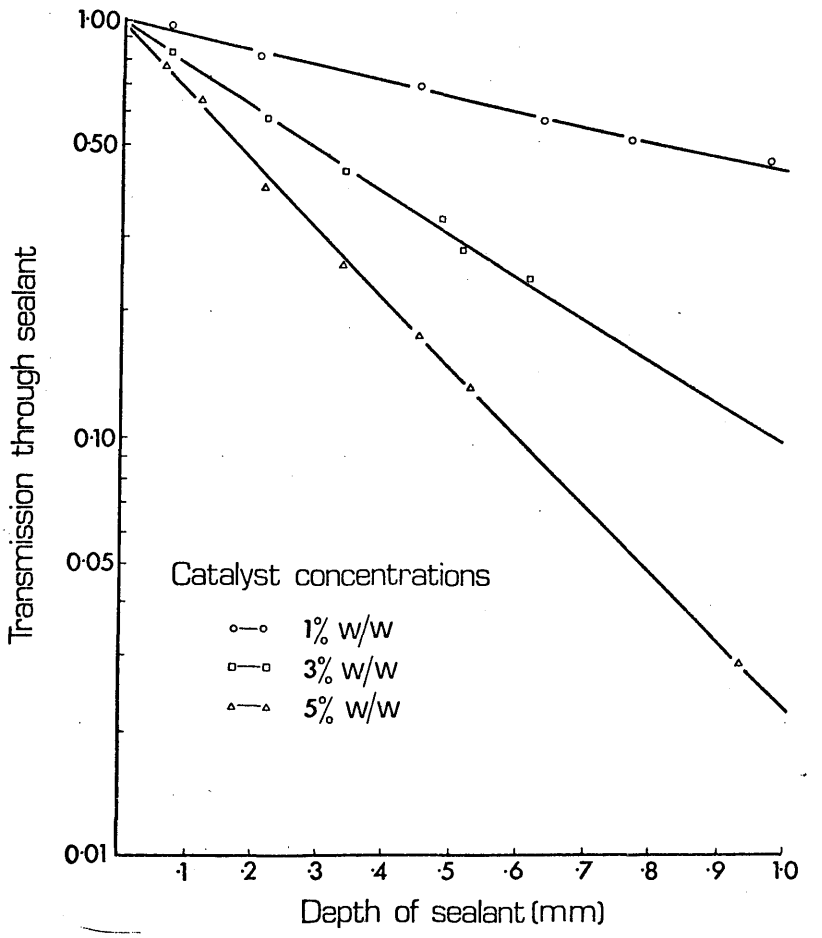


Fig. 5.4 Transmission of u.v. radiation at 365 nm wavelength through Alphaseal containing 1, 3 and 5% w/w catalyst, and without a fluorescent dye.

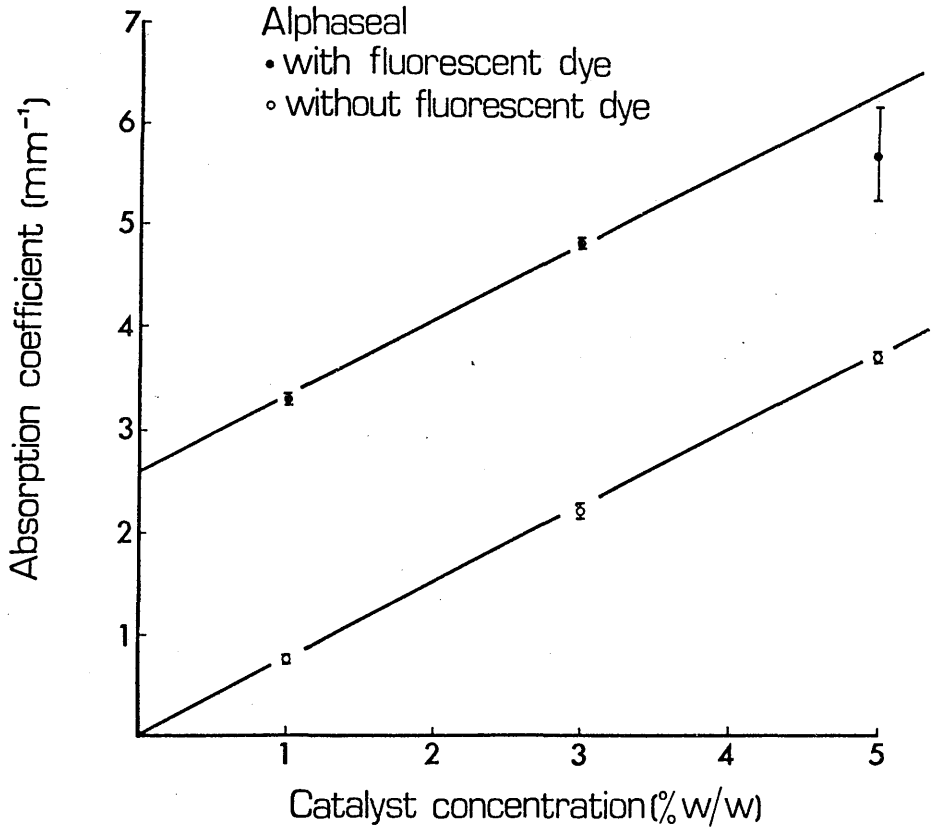


Fig. 5.5 The dependence of the absorption coefficient of Alphaseal at 365 nm wavelength, on the catalyst concentration, and the presence of a u.v. absorbing fluorescent dye.

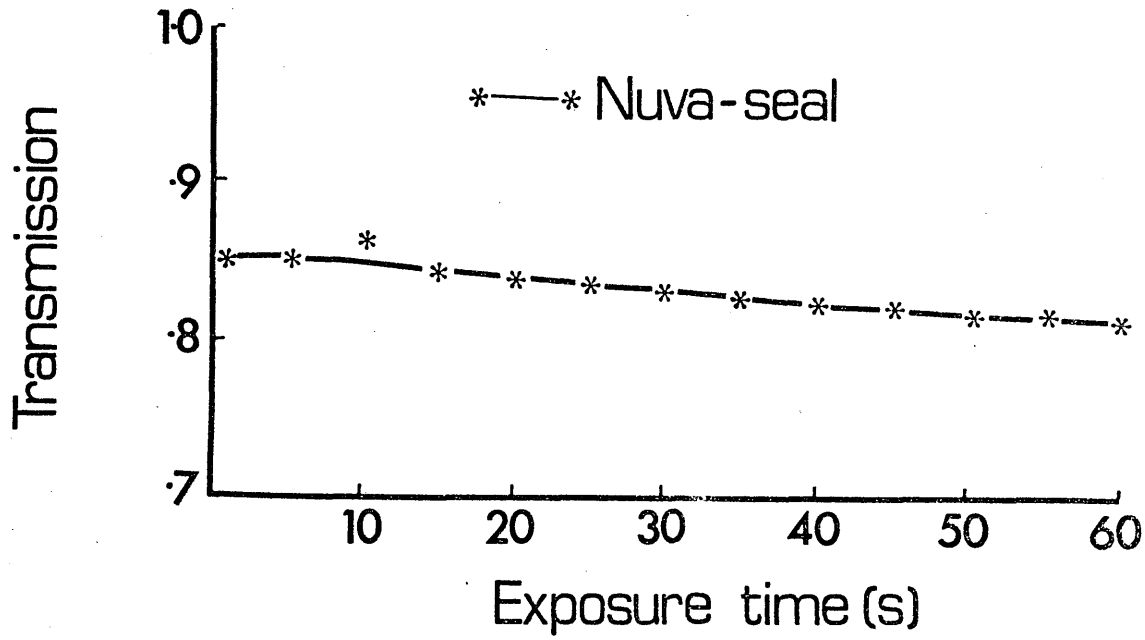


Fig. 5.6 The transmission of u.v. radiation at 365 nm wavelength, through a 0·55 mm thick sample of Nuva-seal, during exposure to an intensity of 20 mW/cm^2.

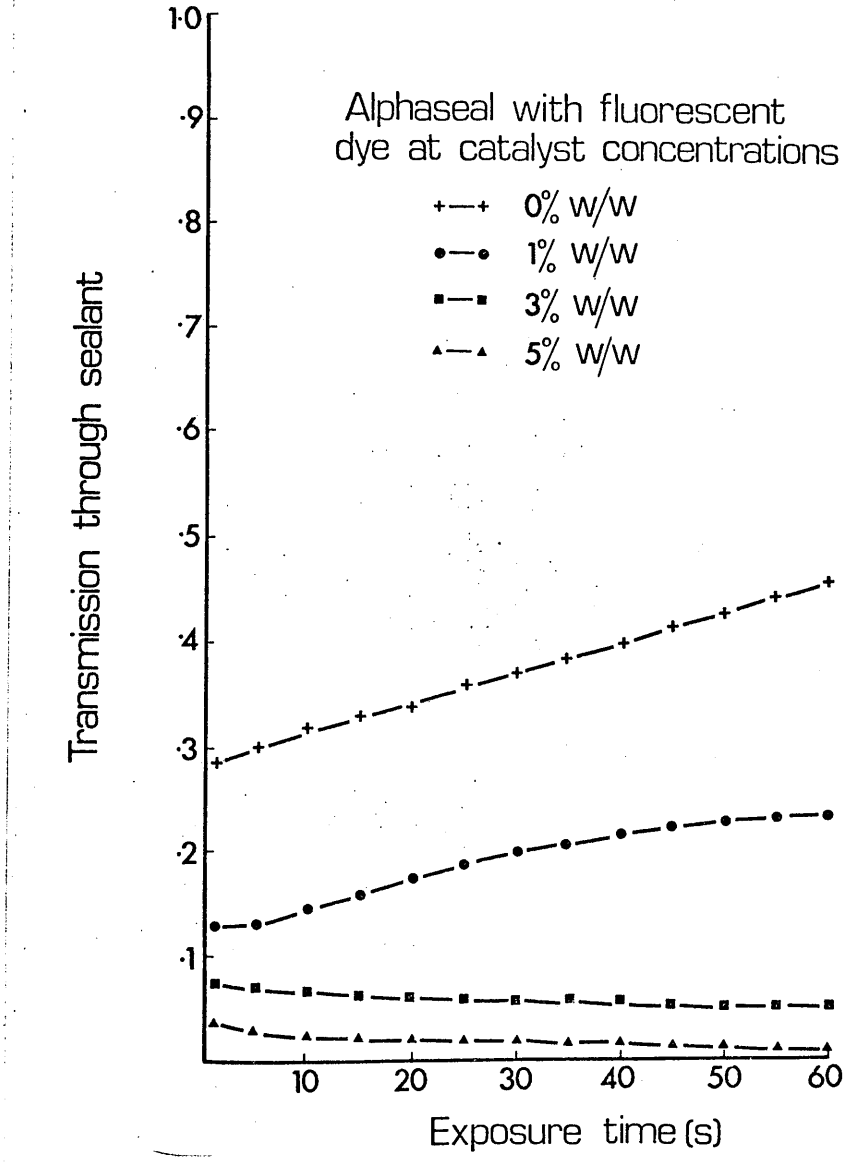


Fig. 5.7 The transmission of u.v. radiation at 365 nm wavelength, through 0.55 mm thick samples of Alphaseal, containing a fluorescent dye, and at catalyst concentrations of 0, 1, 3 and 5% w/w, during exposure to an intensity of 250 mW/cm^2.

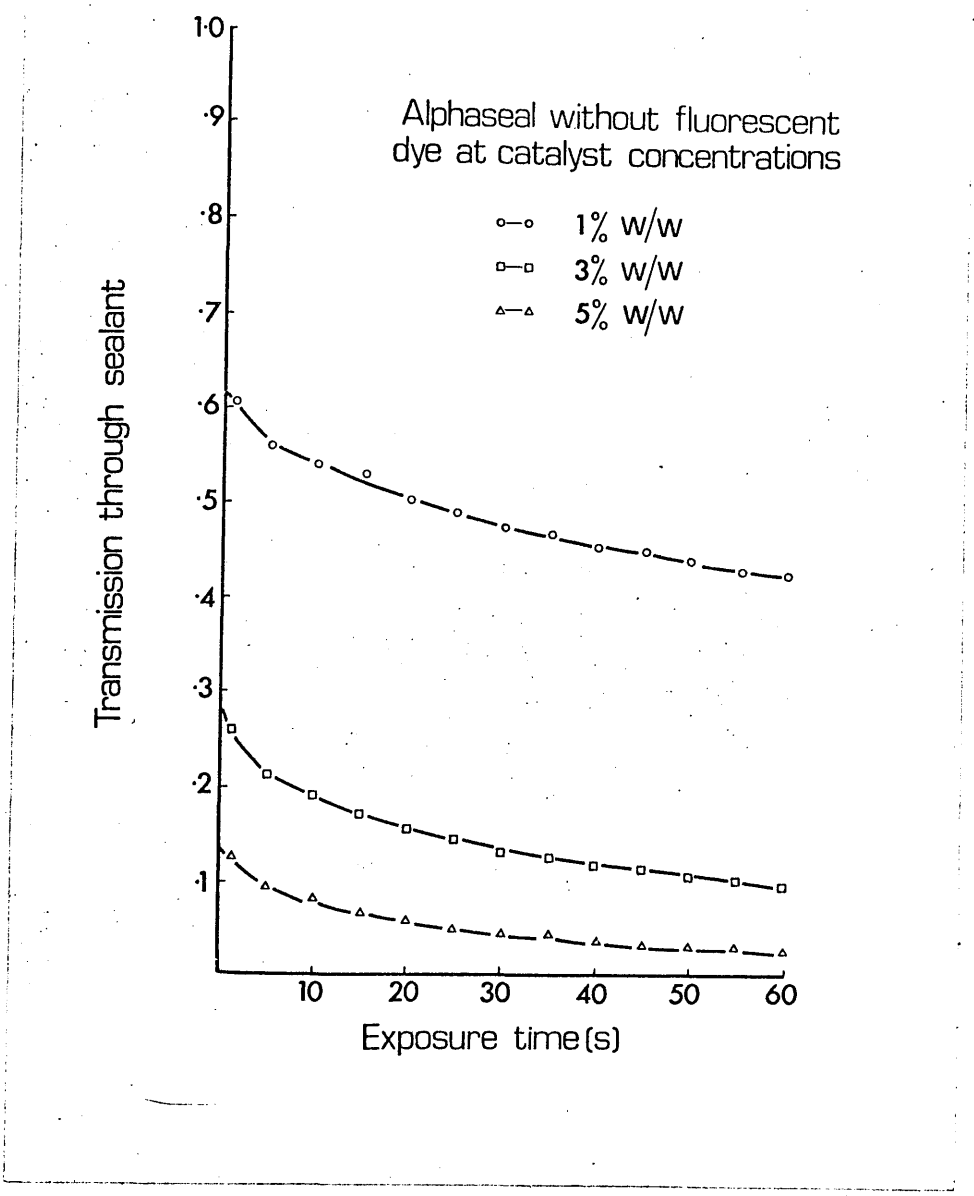


Fig. 5.8 The transmission of u.v. radiation at 365 nm wavelength, through 0.55 mm thick samples of Alphaseal, without a fluorescent dye, and at catalyst concentrations of 1, 3 and 5% w/w, during exposure to an intensity of 250 mW/cm^2.

transmission through a 0·55 mm thick Nuva-seal sample during a 60s exposure to a 20 mW/cm^2 intensity of radiation is plotted in Fig. 5.6. The transmission through the same thickness of Alphaseal exposed to 250 mW/cm^2 also changed during polymerisation, as shown for samples with and without the fluorescent dye in Figs. 5.9 and 5.8. In the absence of the fluorescer the absorption increased during polymerisation for each of the three catalyst concentrations tested. Where only the fluorescer was present in the resin the transmission increased steadily during the u.v. exposure. The addition of a 1% w/w concentration of catalyst still resulted in an increasing transmission during polymerisation. However at catalyst concentrations of 3 and 5% w/w the transmission decreased during polymerisation.

5.4 Discussion

Radiation at 365 nm wavelength has been found to penetrate Nuva-seal readily up to the depths normally required of fissure sealants. For example the intensity of radiation penetrating Nuva-seal to a depth of 2 mm is about 70% of that at the surface layer, and only a slight reduction in transmission was observed during polymerisation. Since the basic resin did not significantly absorb the radiation, the initial absorption coefficient of Nuva-seal was proportional to the catalyst concentration present. Although variations in the size of the catalyst drop used, would result in variations in the absorption of different bottles of mixed sealant, this would not greatly affect the clinical performance of Nuva-seal.

In the previous chapter microhardness measurements on Alphaseal showed that the hardness decreased below the exposed sealant surface. This effect can be explained by the strong absorption which prevents the activating radiation from penetrating Alphaseal to the depths normally required of sealants. For example the intensity at a depth

of 0·5 mm was only 6% of that at the surface. The u.v. fluorescent dye, incorporated to facilitate sealant detection after long periods in the mouth, accounts for a significant proportion of the absorption, while the remainder was attributed to the high concentration of catalyst present. Thus the removal of the fluorescent dye, or the substitution of one which does not absorb at 365 nm wavelength, and a reduction in the catalyst concentration could enable Alphaseal to set better in depth. (This assertion is confirmed in the next chapter).

The changes in transmission which occurred during the polymerisation of Alphaseal appeared to result from a combination of two effects. The polymerisation of the resin by the catalyst produced an increase in the absorption, while the presence of the u.v. fluorescent dye tended to increase transmission during u.v. exposure. The steady increase in transmission, where no catalyst was present suggested that the radiation was dissociating the fluorescent molecules. The concentration of catalyst added to the fluorescent resin determined whether the transmission rose or fell during polymerisation. In its standard preparation, at 5% w/w catalyst concentration, transmission declined during exposure to the u.v. radiation.

While radiation cannot penetrate deeply into Alphaseal the creation of a high concentration of free radicals at the surface may result in the diffusion of these radicals into the sealant where they may initiate polymerisation in regions where the intensity of u.v. radiation is low. In addition the polymer network can be expected to extend down into the bulk of the resin from the surface. Such extensions of polymerisation can have only a limited range, and the use of very high intensity u.v. sources would further extend the depth of set achieved.

The ultimate test of any sealant is its clinical performance. As yet only two studies of Alphaseal (TP2206) have been reported. The

degree of success achieved by Rock (1974a) suggests that Alphaseal is at least potentially effective. However the poor results found with TP2206 in Galloway by Stephen et al. (1976) require explanation. The prototype Quartzlite supplied for this latter trial had an output of 1·5 mW producing a peak intensity of about 30 mW/cm^2 which would not have been sufficient to adequately set the sealant. Currently available Alphalites however have outputs in excess of 15 mW, producing peak intensities over 300 mW/cm^2. By using such a high output source and applying only thin layers of Alphaseal, clinical results comparable to Nuva-seal might be achieved.

CHAPTER SIX

STUDIES OF THE SETTING BEHAVIOUR OF SEALANTS USING A NEW ACOUSTIC METHOD OF DETERMINING SET

6.1 Introduction

Microhardness tests described in Chapter Four were used to study the setting of fissure sealants, when polymerisation was well advanced. However, a great many measurements would be required to build up a complete picture of the setting of u.v. activated sealants by this method. Therefore, a new procedure for assessing the setting of photosensitive resins was devised, using the transmission of acoustic vibrations. The setting characteristics of different sealants have been compared, and the dependence of the setting of u.v. activated sealants on the applied intensity of radiation, examined. The rate of set of Alphaseal, prepared at different catalyst concentrations, has been examined, in view of the varied absorption of radiation by the resin, for the different formulations discussed in Chapter Five.

6.2 Materials and Methods

A new method for measuring the setting characteristics of pit and fissure sealant resins was devised, using the transmission of audio frequency vibrations. A schematic diagram of the system used is shown in Fig. 6.1, and the physical factors which determine the acoustic transmission across sealant samples are discussed in Appendix II. A photograph of the apparatus, with two steel cylinders aligned using retort stands, is shown in Fig. 6.2. The opposing end faces of the cylinders had flat headed steel pins fitted in holes cut in their centres. The two pin-heads were of 2 mm diameter and separated by a 1 mm gap. A vibration generator was bonded to the other end of one steel cylinder, and received a sinusoidal signal at 1 kHz from an audio frequency signal generator. A vibration transducer (accelerator type

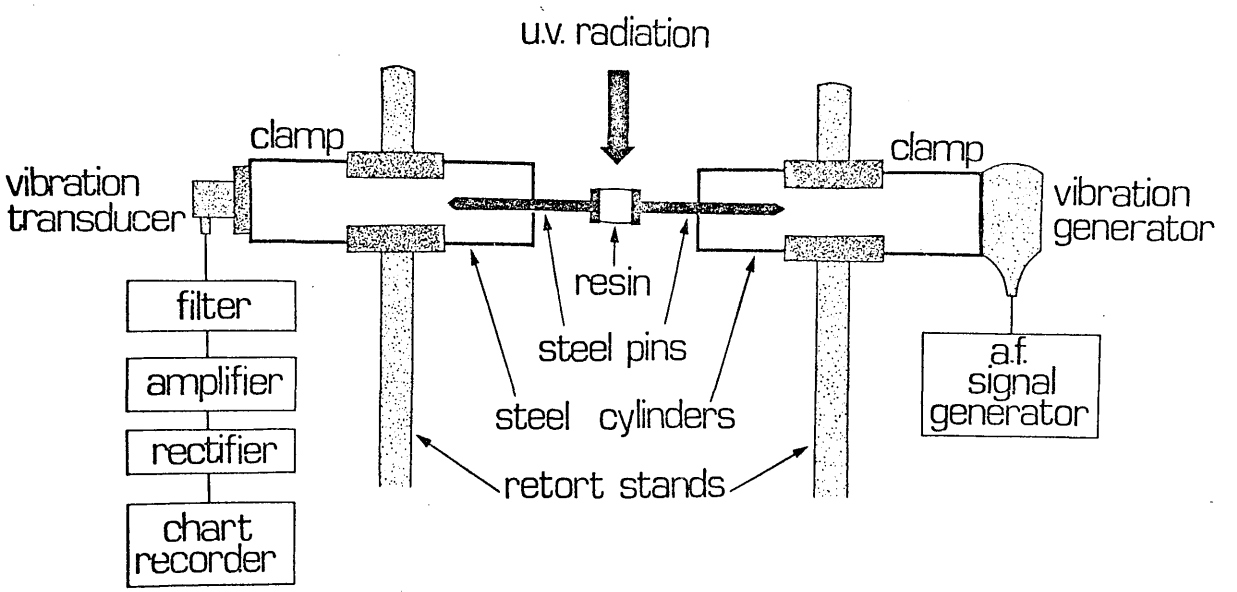
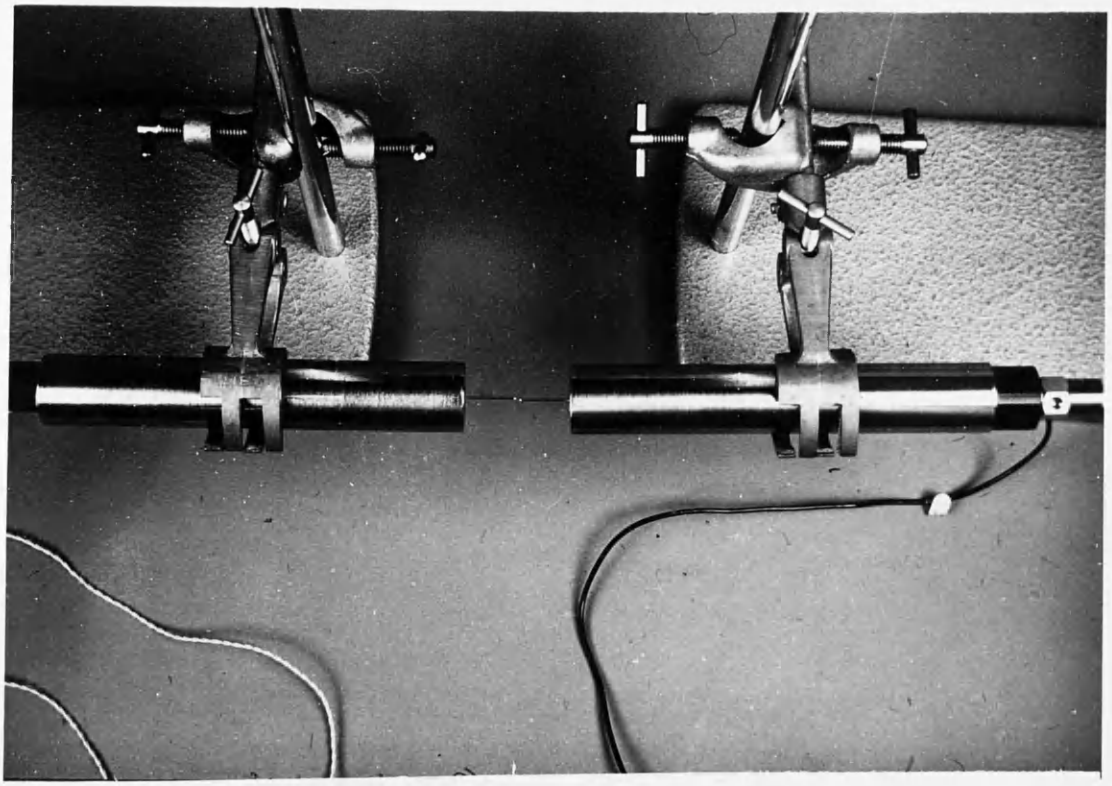


Fig. 6.1 Experimental arrangement for monitoring the setting of fissure sealants using the transmission of acoustic vibrations.

Fig. 6.2 Photograph of the arrangement of steel cylinders and pins shown schematically in Fig. 6.1. The vibration generator is bonded to the end of the left hand cylinder, and the vibration transducer is magnetically clamped to the end of the right hand cylinder.



4332, Bruel & Kjaer DK-2850, Naerum, Denmark) was magnetically clamped to the far end of the other steel cylinder. When the air space between the pin-heads was filled with resin the passage of vibrations could be detected by the transducer. The signal from the transducer was amplified by a sound level meter (type 2204, Bruel & Kjaer) and filtered at 1 kHz to eliminate background noise before being rectified and recorded on a paper chart recorder. The setting characteristics of three chemically initiated sealants were measured from the time of first mixing the resin, a few seconds before applying a drop of the mixture to the gap between the pin-heads. When u.v. sensitive sealants were tested the chart recorder was activated at the moment of exposing the resin between the pin-heads to a pre-arranged u.v. intensity from either a Nuva-lite, Alphalite or Blak-Ray 100W u.v. source (UVP International Inc., San Gabriel, California, U.S.A.). In all cases identical gain settings were used, and the chart record monitored the increase in the transmitted sound signal through the resin during the polymerisation process. To investigate the setting characteristics of the u.v. activated materials, sealant was placed between the pin-heads and the distance from the tip of an Alphalite adjusted to produce intensities from 5 to 120 mW/cm^2. From the setting curves obtained at each intensity, the initial rate of increase in acoustic transmission was measured in mV/s. The effect of varying the catalyst concentration and removing the fluorescent dye, on the setting of Alphaseal, was also investigated.

6.3 Results

The amplitudes of the transmitted vibrations recorded for 5 tests on each of five fissure sealants are compared in Fig. 6.3. The setting curves have been normalised to show the same transmission 10 minutes after the initial mixing of two of the chemically setting materials

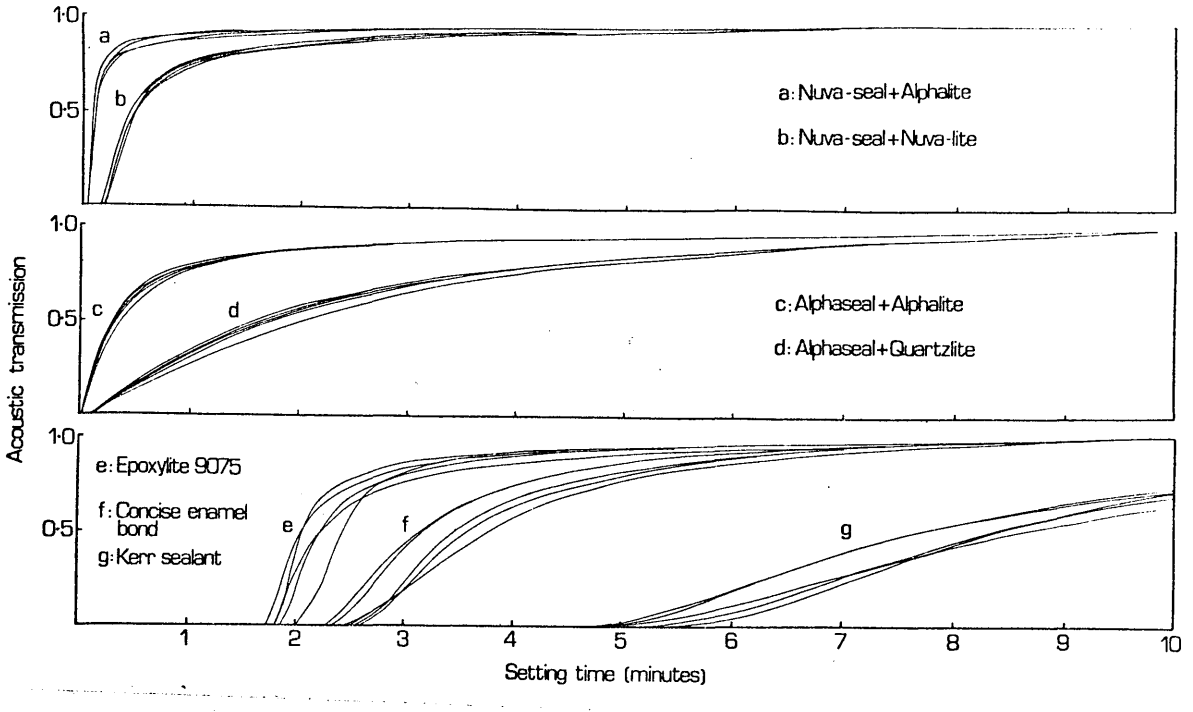


Fig. 6.3 Normalised acoustic transmissions through various fissure sealants during setting.

(a) Five Nuva-seal samples irradiated at an intensity of about 200-300 mW/cm^2, from an Alphalite with an output of 15 mW.

(b) Five Nuva-seal samples irradiated at an intensity of about 20-30 mW/cm^2, from a Nuva-lite.

(c) Five Alphaseal samples irradiated at an intensity of about 200-300 mW/cm^2, from an Alphalite with an output of 15 mW.

(d) Five Alphaseal samples irradiated at an intensity of about 40-60 mW/cm^2, from a Quartzlite with an output of 3 mW.

(e) Five Epoxylite 9075 samples.

(f) Five Concise Enamel Bond samples.

(g) Five Kerr Pit and Fissure Sealant samples.

(Epoxylite 9075 and Enamel Bond), and 10 minutes after beginning the exposure of Nuva-seal and Alphaseal to radiation. The transmission through Kerr Pit and Fissure Sealant (Sybron Corp., Romulus, Michigan, U.S.A.) was not normalised since the setting was not complete after 10 minutes.

Acoustic setting curves for Nuva-seal exposed to various intensities of u.v. radiation are shown in Fig. 6.4. The setting characteristics of an experimental filled fissure sealant Nuva-cote (L.D. Caulk Co.) are shown in Fig. 6.5. Corresponding setting curves for Alphaseal, prepared at 1% w/w catalyst concentration, without the fluorescent dye, are shown in Fig. 6.6. The peak gradients occurring at the start of these curves were used to determine the dependence of the initial rates of set of the three u.v. activated sealants on the u.v. intensity applied, and are plotted in Fig. 6.7. For all three sealants straight lines fitted to the data of Fig. 6.7 indicate that, the rate of set was proportional to $(\text{Intensity})^{0.85}$. From the relative positions of the straight lines in Fig. 6.7, it can be deduced that, for a given intensity, the rate of set of Nuva-seal was 3·5 times the rate of set of Nuva-cote and 1·9 times the rate of set of this preparation of Alphaseal. The initial rates of set of Alphaseal exposed to an u.v. intensity of 100 mW/cm^2, at various concentrations of catalyst and with and without the fluorescent dye are plotted in Fig. 6.8. The removal of the fluorescent dye resulted in an increase in the rate of set of Alphaseal at all catalyst concentrations, and a maximum rate was achieved in the absence of the dye at catalyst concentrations in the range 0·5 to 1·0% w/w.

The setting behaviour of the u.v. activated sealants when exposed to the same intensity (100 mW/cm^2) are compared in Table 4. The initial rates of increase in the acoustic transmission were obtained from Figs. 6.7 and 6.8, and the "final" acoustic transmissions were

estimated from setting curves in Figs. 6.4, 6.5 and 6.6. From this information the initial rates of set for the different materials were expressed in terms of percentages of the final transmissions and are presented in the third column of Table 4. Thus, at this intensity Nuva-seal set nearly 4 times as fast as Nuva-cote, and 13 times as fast as the usual preparation of Alphaseal. However, by preparing Alphaseal in the absence of the fluorescent dye and at a lower catalyst concentration, its setting rate was increased by a factor of 10.

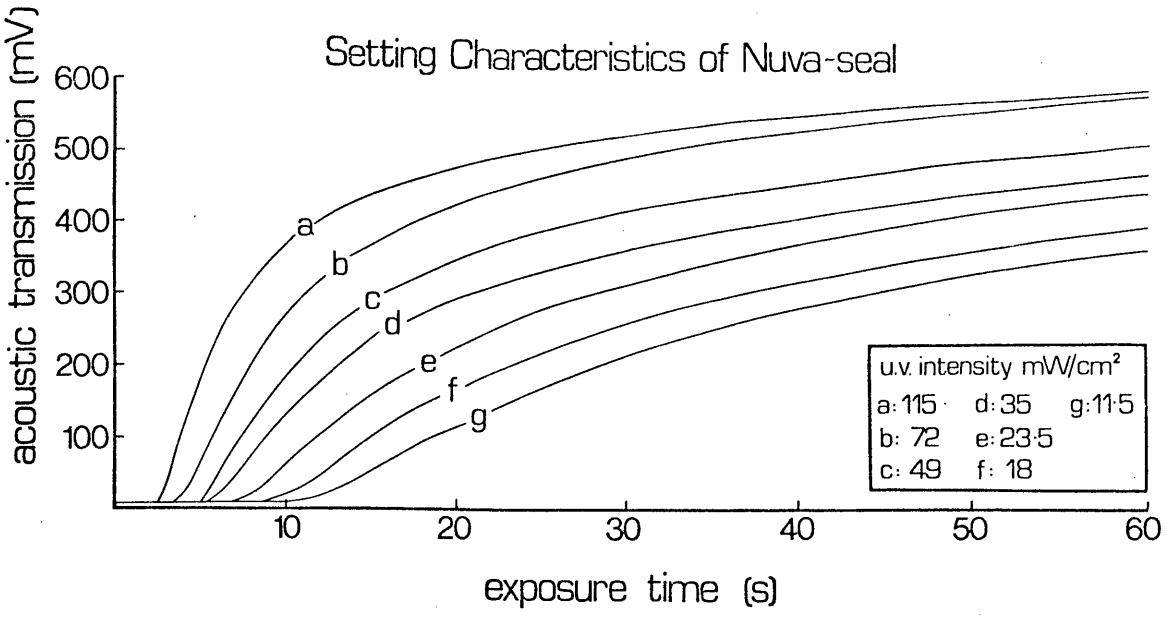


Fig. 6.4 Acoustic transmissions through samples of Nuva-seal exposed to different u.v. intensities in the range 11·5-115 mW/cm^2.

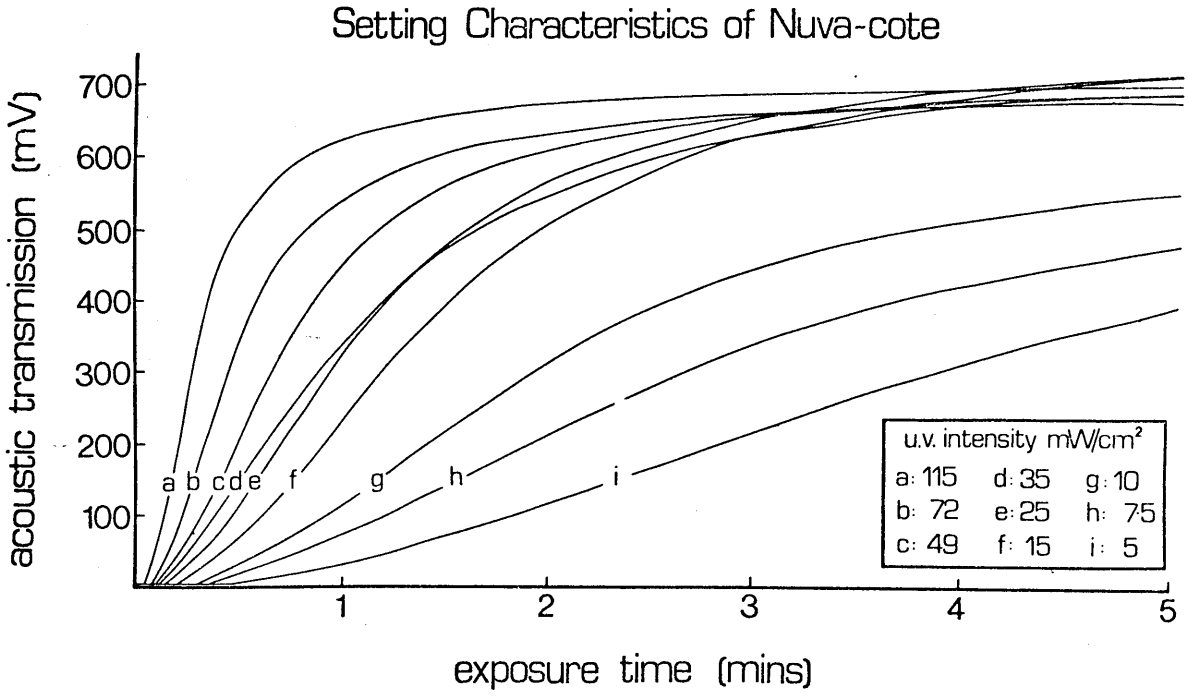


Fig. 6.5 Acoustic transmissions through samples of Nuva-cote exposed to different u.v. intensities in the range 5-115 mW/cm^2.

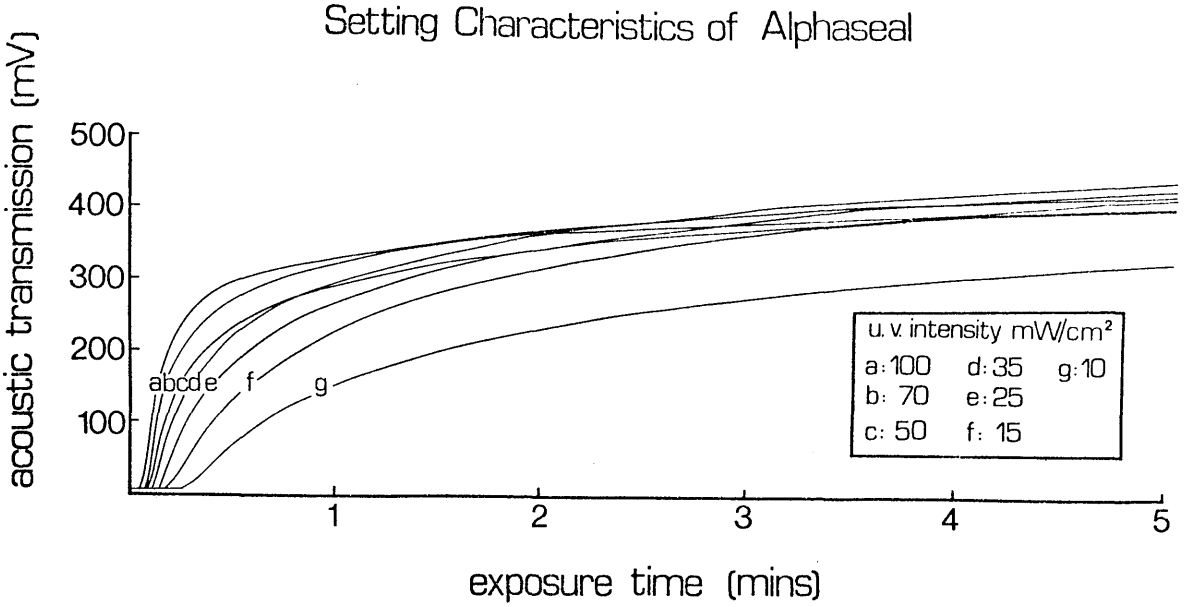


Fig. 6.6 Acoustic transmissions through samples of Alphaseal, prepared at a 1% w/w catalyst concentration without the fluorescent dye, on exposure to different u.v. intensities in the range 10-100 mW/cm^2.

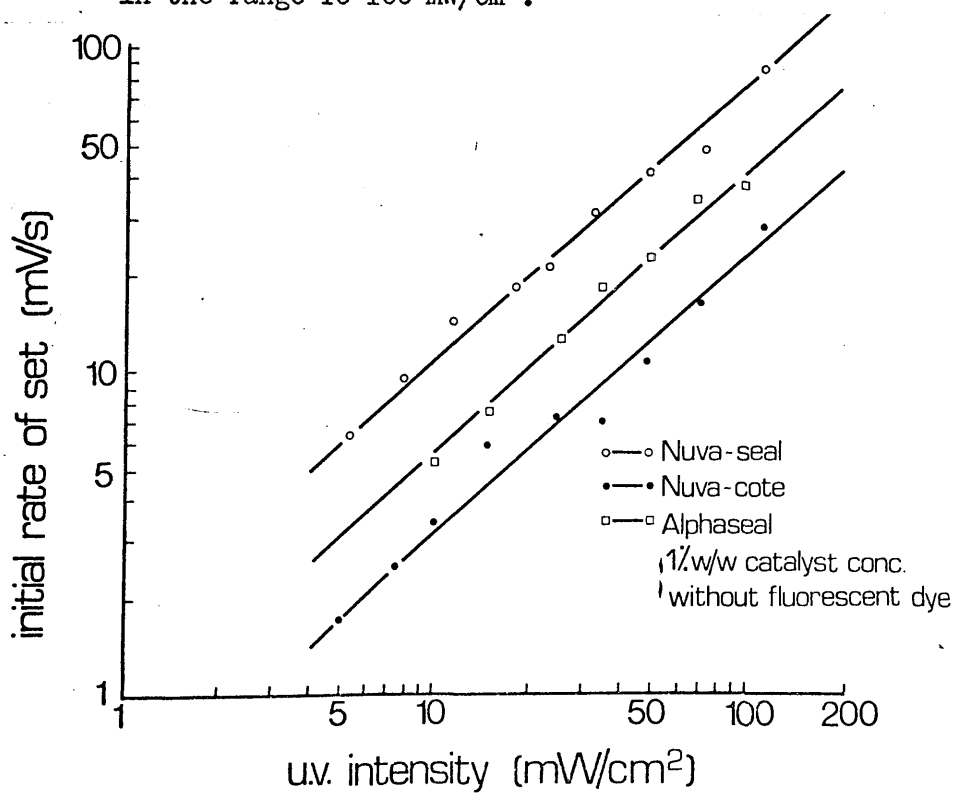


Fig. 6.7 Initial rates of set (i.e. initial peak acoustic transmissions) of Nuva-seal, Nuva-cote and Alphaseal (1% w/w cat. conc., no fluorescent dye) on exposure to different u.v. intensities.

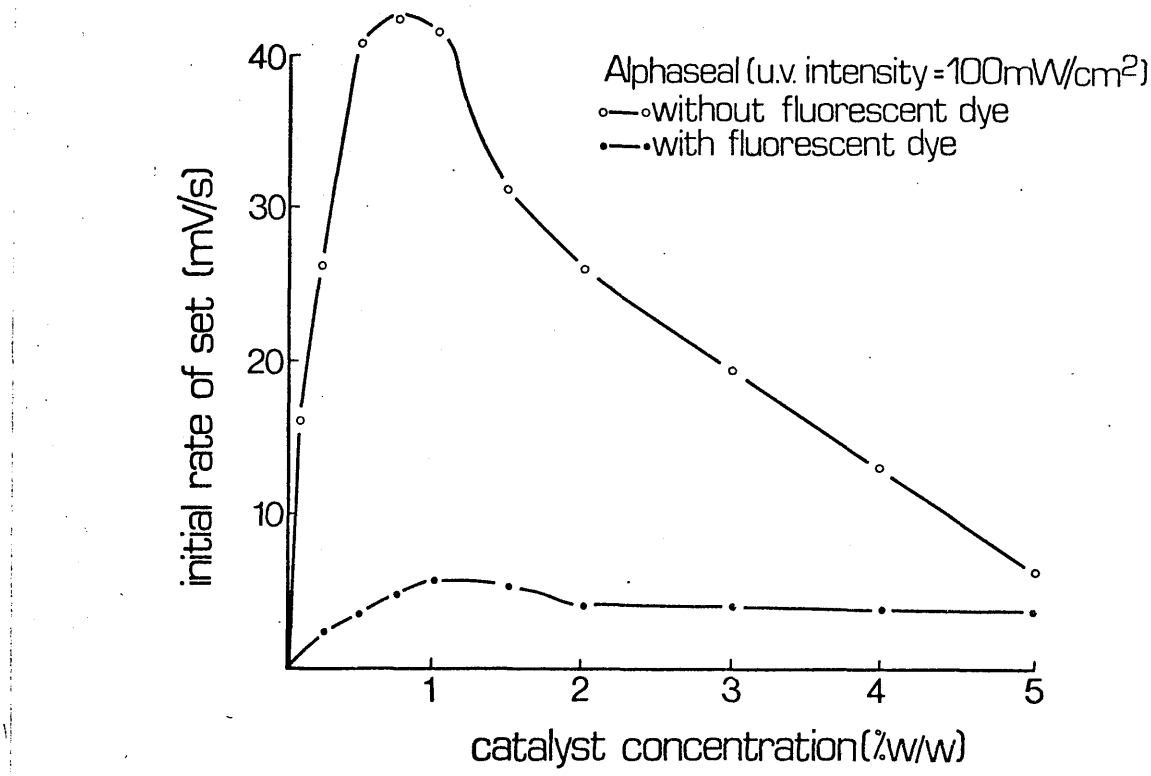


Fig. 6.8 Initial rate of set of Alphaseal prepared at various catalyst concentrations, with and without the fluorescent dye, on exposure to an u.v. intensity of 100 mW/cm^2.

Table 4

A comparison of the setting rates of u.v. activated sealants on exposure to an u.v. intensity of 100 mW/cm^2.

Sealant	Initial rate of increase in acoustic transmission ($mV.s^{-1}$)	Final acoustic transmission (mV)	Initial rate of set $\%.s^{-1}$
Nuva-seal	78	600*	13
Nuva-cote	23	700+	3.3
Alphaseal[1]	4	400+	1.0
Alphaseal[2]	42	400+	10.5

Notes: 1. Prepared at a catalyst concentration of 5% w/w and containing a fluorescent dye.

2. Prepared at a catalyst concentration of 1% w/w without the fluorescent dye.

* Approximate value attained after one minute.

\+ Approximate value attained after five minutes.

6.4 Discussion

This new method for assessing the setting of fissure sealants has yet to prove its full potential, and the limitations of the technique are still to be discovered. Consequently, some caution must be exercised when interpreting the results. The method by which the system worked was apparently straightforward. As the resins set, either by chemical initiation or through u.v. activation, their physical properties changed and the increased "stiffness" of the samples permitted vibrations to be transmitted across the resin gap between the pins. Thus the arrangement described, provided a continuous record of the changing physical state of the resins as they set. (This is discussed further in Appendix II).

The delayed setting of chemically initiated sealants (Concise Enamel Bond, Epoxylite 9075 and Kerr pit and fissure sealant) was apparent in Fig. 6.3. Concise Enamel Bond and Epoxylite 9075 set following a two to three minute delay, designed to allow placement of the sealant prior to viscosity changes due to polymerisation. However, the very long delay in the setting of Kerr pit and fissure sealant was greater than indicated by the manufacturers, suggesting that the batch of sealant tested was faulty. The more rapid setting behaviour of u.v. activated sealants on exposure to various u.v. sources was also demonstrated. The setting of Alphaseal immediately below the tip of an Alphalite (Fig. 6.3c) was similar to the setting of Nuva-seal when exposed to u.v. intensities of 20-30 mW/cm^2 below a Nuva-lite. However, when the u.v. radiation from an Alphalite, at intensities of 200-300 mW/cm^2, was used to set Nuva-seal it demonstrated a more rapid setting behaviour than Alphaseal (cf. Figs. 6.3a and c). The rate of set of Alphaseal under a high output Alphalite (15 mW), as shown in Fig. 6.2c, was much more rapid than when illuminated by the Quartzlite, with a 3 mW output, as shown in Fig. 6.3d. It was, therefore, apparent

from these preliminary findings that the rate of set of u.v. activated sealants depended on the u.v. intensity applied. Furthermore, Nuva-seal set more rapidly than Alphaseal for a given u.v. intensity. Subsequent experiments examined in more detail the setting of these two u.v. activated sealants and also Nuva-cote, showing that while there were considerable differences in their rates of set at any given intensity, all three materials had a similar dependence on the u.v. intensity.

The slower setting of Nuva-cote compared with Nuva-seal was probably due to the reduced penetration of u.v. radiation into Nuva-cote because of the incorporation of filler particles. The slow setting of Alphaseal in its usual constitution seemed also to stem from the high absorption of the radiation by the fluorescent dye and the high catalyst concentrations normally used. Removal of the dye from Alphaseal produced considerable increases in the rate of set at all catalyst concentrations tested, and was particularly marked at catalyst concentrations from 0·5 to 1·0% w/w, where peak setting rates were observed.

CHAPTER SEVEN

STUDIES ON SEALANT RETENTION USING AN IN VIVO REPLICATION TECHNIQUE

7.1 Introduction

When this project commenced it was thought desirable to prolong sealant retention in pits and fissures for as long as possible. However no attempt had then been made to study the mechanisms of sealant loss, which once understood might enable modifications in materials and application techniques to improve retention. As clinical trials normally only provide minimal information about sealant condition, an in-vivo replication technique was developed to provide a more detailed picture of the sealant at re-examination. This procedure was based on the methods employed by Saxton (1973) to study plaque formation. A low viscosity silicone impression material was used to produce epoxy resin replicas of the occlusal surfaces of teeth, at various times after sealing, and these models examined on a Scanning Electron Microscope. A similar technique has also now been reported by Douglas and Tranter (1975).

7.2 Materials and Methods

During the pilot study of Nuva-seal retention, discussed in the following chapter, impressions were taken at regular intervals from selected teeth. A small quantity of a low viscosity silicone resin (Silflo, Flexico Developments Limited, England) was placed on the dried occlusal surface of a sealed tooth and blown into a thin bubble-free layer. The bulk of the liquid silicone rubber was then applied via a single tooth impression tray, removed after four minutes and stored in a polythene bag. Within 24 hours the impression was rinsed

with distilled water, dried, and excess rubber trimmed off with a blade. The impression was then fitted inside a 10 mm length of polythene tubing of internal diameter 13 mm, and mounted on a card with a silicone adhesive (Silcoset 151, ICI Limited, U.K.), and stored at 50°C for 24 hours. Pre-mixed epoxy resin (Fluorochem Ltd., Glossop, Derbyshire) was then applied with a disposable syringe, and the sample stored for a further 48 hours at 50°C. The polythene tubing was removed and the epoxy resin copy of the sealed tooth mounted on a 13 mm diameter S.E.M. stub with conducting paint (Silver Dag, Acheson Colloids Company, Prince Rock, Plymouth). Prior to examination on a Phillips Stereoscan S.E.M., tooth models were coated with a 20 nm thick layer of gold, using a sputter coater. The models were also examined under optical microscopy, but photographic records were superior with the S.E.M., due to the better depth of field. Under normal conditions, magnifications of up to 2000 times were readily obtained. Incorporated into the display screen of the S.E.M. was a bar indicating the scale, which is included in the photographic records presented here.

7.3 Results

The condition of some selected teeth sealed during a pilot study of the retention of Nuva-seal was examined using the in-vivo replication procedure outlined. Fig. 7.1 shows the appearance of the distal part of the occlusal surface of an upper right first permanent molar before sealing, immediately after sealing and 16, 41 and 250 days later. The deep distal groove running towards the palatal surface of the tooth is evident in Fig. 7.1a. While sealing totally occluded this fissure from the oral environment as shown in Fig. 7.1b, after 16 days abrasion had made the appearance of the sealant rougher than the adjacent enamel, so that the two surfaces are easily distinguished in

Fig. 7.1c. After 41 days the extent of sealant cover was little reduced by further abrasion (Fig. 7.1d), but at 250 days the palatal extension of the sealant had been completely lost, with sealant remaining in only part of the distal fissure (Fig. 7.1e). This retained material is shown from the palatal side of the tooth, at the original magnification (Fig. 7.1f) and at higher magnifications in Figs. 7.1g and 7.1h. At these higher magnifications sealant is seen to be poorly adapted to enamel, and the entire palatal extension appears to have been lost by sealant fracture, rather than by gradual abrasion.

The condition of this tooth, 16 days after sealing, is seen in more detail in Figs. 7.2 and 7.3. Sealant cover on the distal fissure shows good marginal adaptation at this time, even under examination at the higher magnifications of Figs. 7.2b and 7.2c. The mesial part of the occlusal surface is shown at two different magnifications in Fig. 7.3 where a triangular block of raised sealant is an unusual feature only observed on this tooth. By the 12 month examination this tooth had lost all sealant cover and had an amalgam filling in the occlusal surface.

Fig. 7.4 shows the appearance of sealant on the distal and palatal fissures of an upper left first permanent molar, 14, 39, 140 and 249 days following application. Although the area covering the distal fissure was gradually reduced through abrasion, the sealant remained intact after 249 days (Figs. 7.4a, b, c and d). The palatal extension showed considerable abrasion losses, but still maintained an effective seal after 249 days (Figs. 7.4e, f, g and h). The mesial occlusal fossa of this tooth is shown for the same examination times in Figs. 7.5e, f, g and h. Again sealant was virtually intact at 249 days, with only slight loss from the mesial marginal grooves. After 12 months a mirror and probe examination judged sealant cover to be

complete on all surfaces of this tooth.

The distal fissure of another upper left first permanent molar is shown, 15, 39, 140 and 249 days after sealing in Figs. 7.5a, b, c and d. Here, sealant has been thickly applied and appears to have been hardly affected by abrasion. By contrast the mesial pit of this occlusal surface was only thinly coated with sealant, and Figs. 7.6a, b and c show its condition after 39, 140 and 249 days. Considerable loss was noted from the mesial marginal grooves, although the mesial pit remained sealed. However, after 12 months mirror and probe examination determined this tooth to be completely sealed on all sites.

Figs. 7.7a, b and c show the entire occlusal surface of an upper right first permanent molar, 14, 38 and 248 days after sealing. Resin was only thinly applied so that after the first few days abrasion it occupied only the bases of occlusal grooves. In Fig. 7.7a the sealant can be seen to be poorly adapted at the buccal extremity of the distal fissure. Sealant was found to be lost from this region after 38 days, and fragments of sealant were also lost from the mesial part of the occlusal surface (Figs. 7.7b and c). After 12 months sealant was graded as completely lost from this tooth by mirror and probe examination.

The condition of sealant on the mesial part of the occlusal surface of an upper right first permanent molar, after 31 days, is shown in Fig. 7.8. Incomplete sealant cover is shown in Fig. 7.8a at a nominal magnification of 20 times. The sealant fracture surface in a mesial marginal groove is examined at higher magnifications in Fig. 7.8b, c and d. The spherical globules observed are probably caused by air bubbles, trapped when the impression was taken. Sealant attachment to the enamel is studied on another part of this tooth in Fig. 7.8e, f, g and h at increasing magnifications. Poor adaptation to enamel is apparent with gaps of approximately 20 μm being observed (Fig. 7.8h).

The distal fissure of this tooth is depicted at different magnifications in Fig. 7.9e, f, g and h, and very poor adaptation is again apparent with the sealant edge being separated from the enamel in some regions, by as much as 40 μm.

The condition of sealant applied to premolar surfaces was generally much superior to that found on molars. An instance of sealant detachment from the surface of a premolar is shown in Figs. 7.9a, b, c and d. After 38 days in the mouth, a small length of sealant edge had separated from the enamel by about 10 μm (Fig. 7.9d). However adaptation over the rest of the tooth surface was excellent. The condition of the sealant on this upper left first premolar is shown in Fig. 7.10, after 16, 38, 142 and 251 days in the mouth. The mesial aspect of the tooth is depicted in Figs. 7.10a, b, c and d, while the distal aspect is seen in Figs. 7.10e, f and g. Although there has been slight loss from the surface of the sealant, the fissures remained well covered after 251 days. That the depth of sealant over the central fissure has been little reduced by wear, during the eight month study period, is apparent from the mesial profiles of this tooth in Fig. 7.11.

The second premolar adjacent to this first premolar has also been examined and the condition of its sealant cover after 38, 142 and 251 days is shown in Figs. 7.12a, b and c. Although some of the sealant bulk has been lost through abrasion, it continues to provide excellent cover after 251 days. No premolar was judged to have lost any sealant cover at the annual mirror and probe examination.

(a)

(e)

(b)

(f)

(c)

(g)

(d)

(h)

Fig. 7.1 The distal fissure of an upper right first permanent molar is shown before sealing, immediately after sealing, and 16, 41 and 250 days after sealing in a, b, c, d and e, resp. A palatal view of the remaining sealant is shown, after 250 days, at nominal magnifications of 20, 50 and 100 times, in f, g and h, resp.





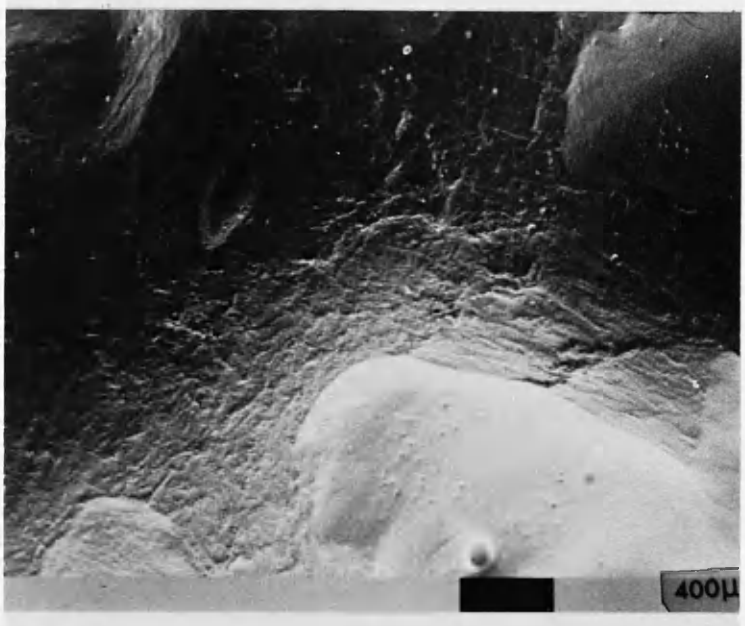



(a)

(b)

(c)

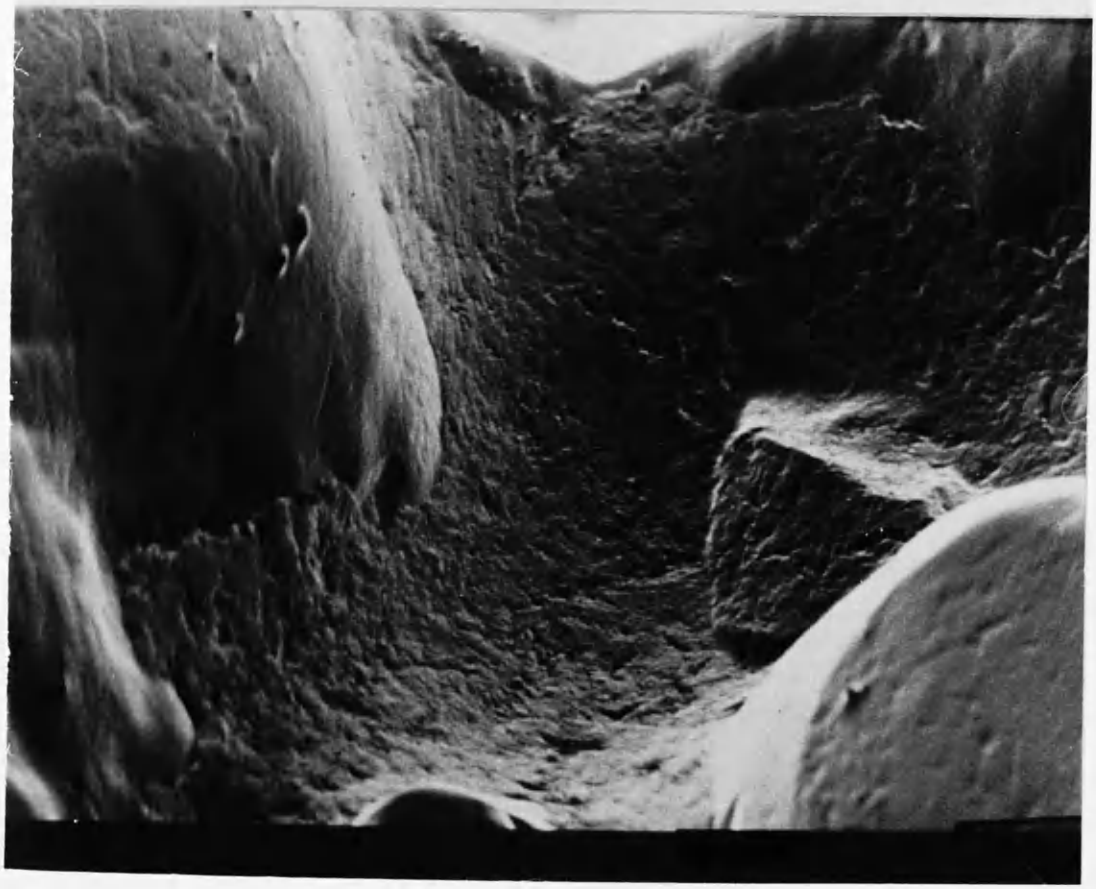
Fig. 7.2 The sealant on the distal fissure of the tooth studied in Fig. 7.1, is shown 16 days after sealing, at nominal magnifications of 20, 50 and 100 times, and a, b and c resp.


400µ

(a)

(b)

Fig. 7.3 The appearance of sealant, after 16 days, in the mesial part of the occlusal surface of the tooth studied in Figs. 7.1 and 7.2, is shown at nominal magnifications of 20 and 50 times, in a and b, resp.



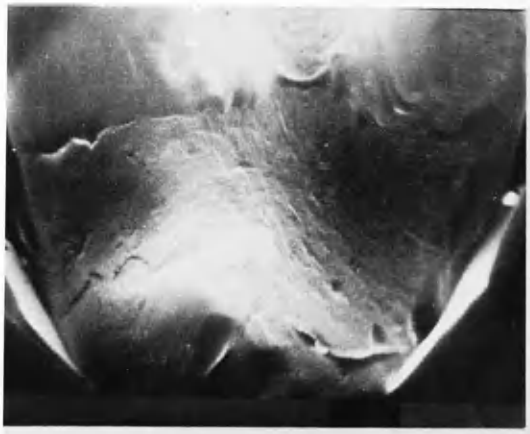
(a) (e)

(b) (f)

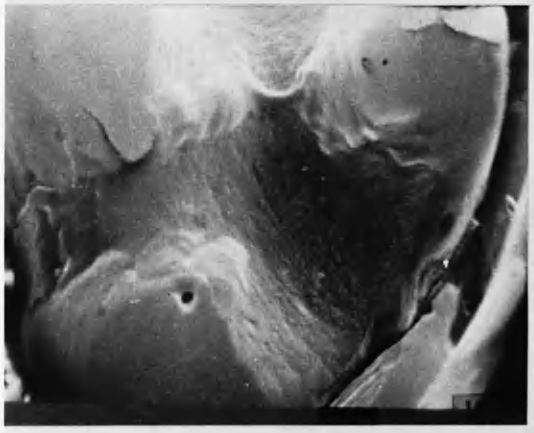
(c) (g)

(d) (h)

Fig. 7.4 The appearance of sealant in the distal fissure of an upper left first permanent molar, 14, 39, 140 and 249 days after sealing, is shown in a, b, c and d, resp. Sealant in the palatal fissure of the same tooth, 14, 39, 140 and 249 days after sealing, is shown in e, f, g and h, resp.








(a) (e)

(b) (f)

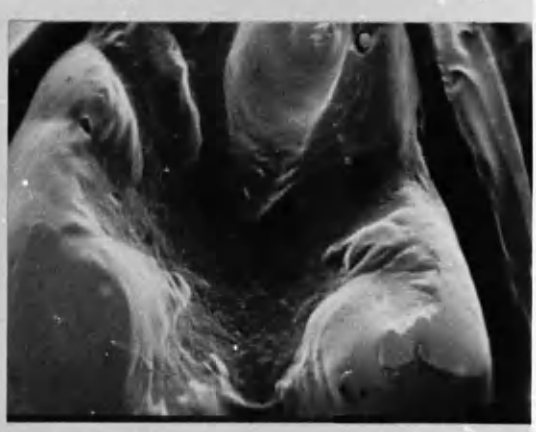
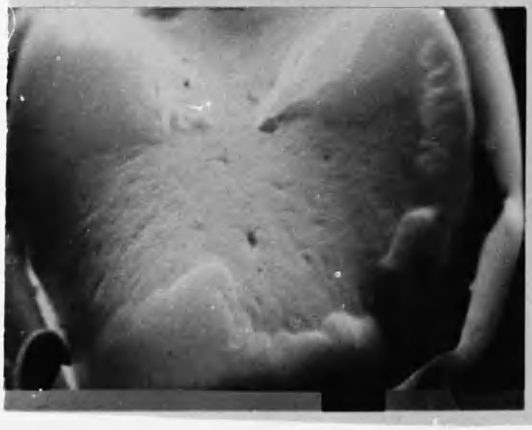
(c) (g)

(d) (h)

Fig. 7.5 The mesial part of the occlusal surface of the tooth studied in Fig. 7.4, is shown, 14, 39, 140 and 249 days after sealing, in e, f, g and h, resp. The distal fissure of another upper left permanent molar is shown, 15, 39, 140 and 249 days after sealing in a, b, c and d, resp.






(a)

(b)

(c)

Fig. 7.6 The mesial part of the occlusal surface of the tooth where distal fissure is examined in Fig. 7.5, is shown, 39, 140 and 249 days after sealing, in a, b and c resp.

(a)

(b)

(c)

Fig. 7.7 The occlusal surface of an upper right first permanent molar, 14, 38 and 248 days after sealing, is shown in a, b and c, resp.

(a)

(e)

(b)

(f)

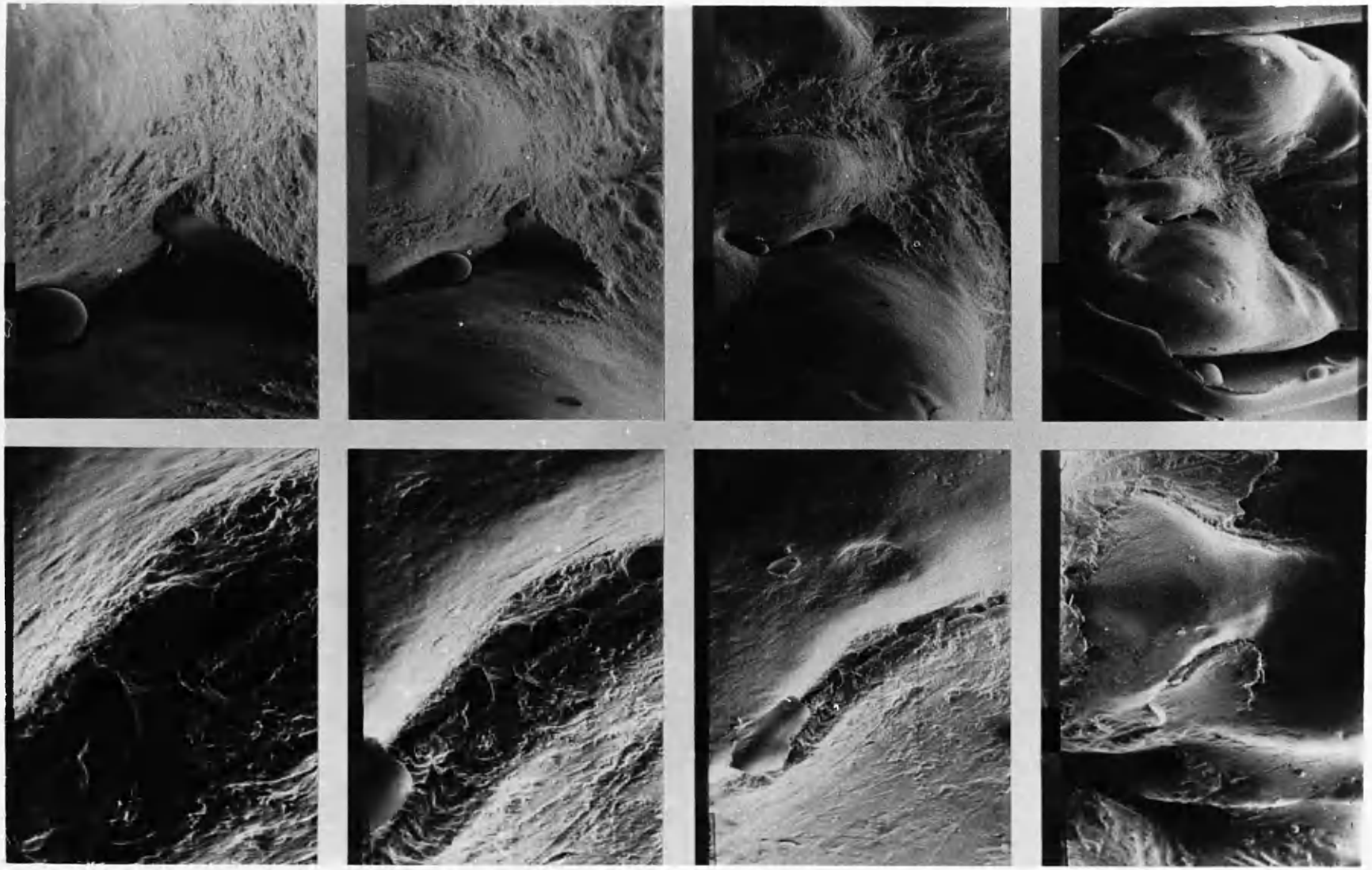
(c)

(g)

(d)

(h)

Fig. 7.8 Sealant in a mesial marginal groove of an upper right first permanent molar is shown, 31 days after sealing, and at nominal magnifications of 20, 50, 100 and 200 times, in a, b, c and d, resp. Sealant, at the same time after application to this tooth is shown, on the mesial side of the oblique ridge, at nominal magnifications of 50, 200, 500 and 1000 times in e, f, g and h, resp.



(a)

(e)

(b)

(f)

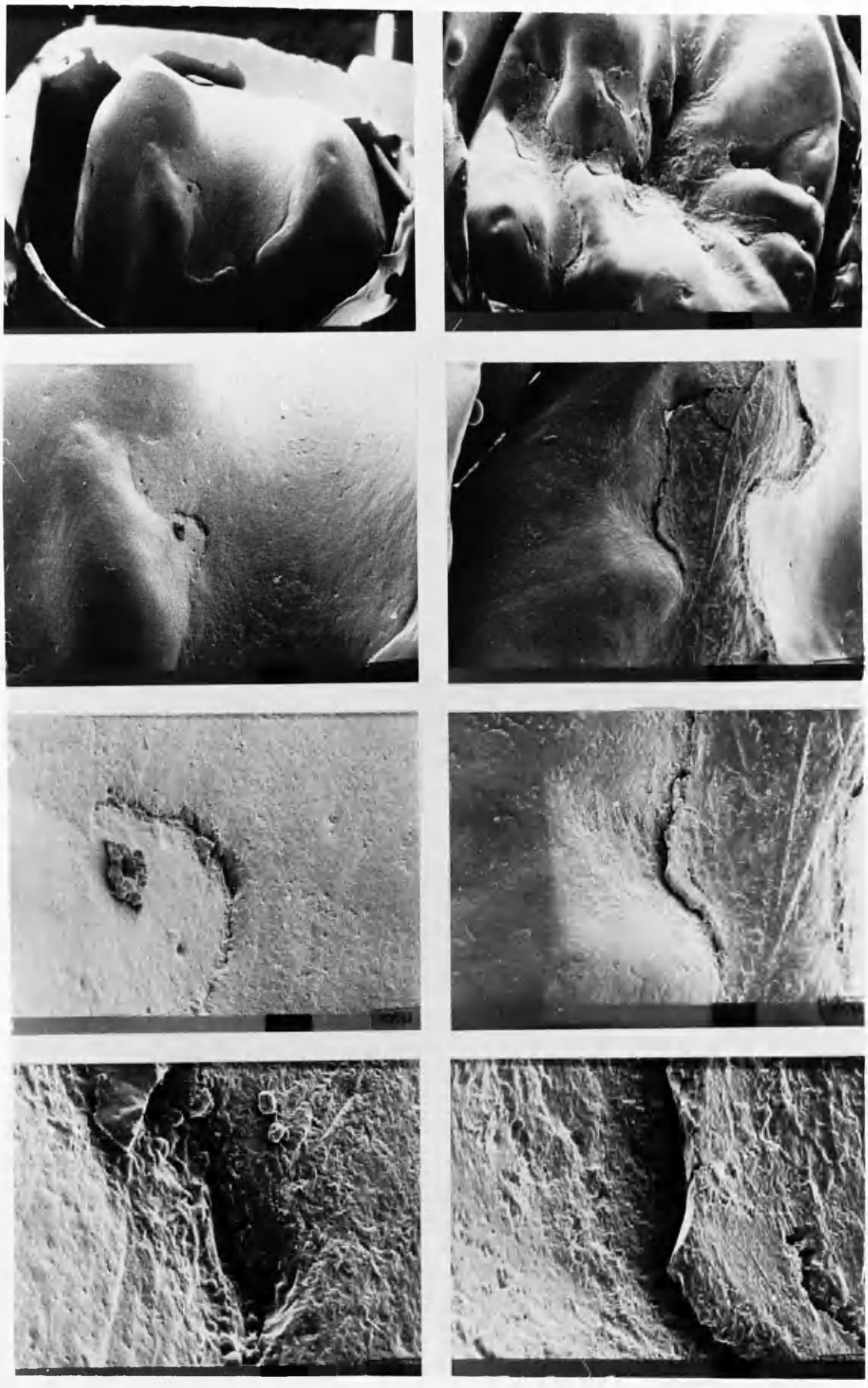
(c)

(g)

(d)

(h)

Fig. 7.9 An upper left first premolar is shown, 38 days after sealing, and at nominal magnifications of 20, 50, 200 and 1000 times, in a, b, c and d, resp. The distal fissure of the tooth examined in Fig. 7.8 is shown, 31 days after sealing, and at nominal magnifications of 20, 50, 100 and 500 times, in e, f, g and h, resp.



(a)

(e)

(b)

(c)

(f)

(d)

(g)

Fig. 7.10 The mesial aspect of the upper left first premolar studied in Fig. 7.9, is shown, 16, 38, 142 and 251 days after sealing, in a, b, c and d, resp. The distal aspect is shown, 16, 142 and 251 days after sealing in e, f and g, resp.

(a)

(b)

(c)

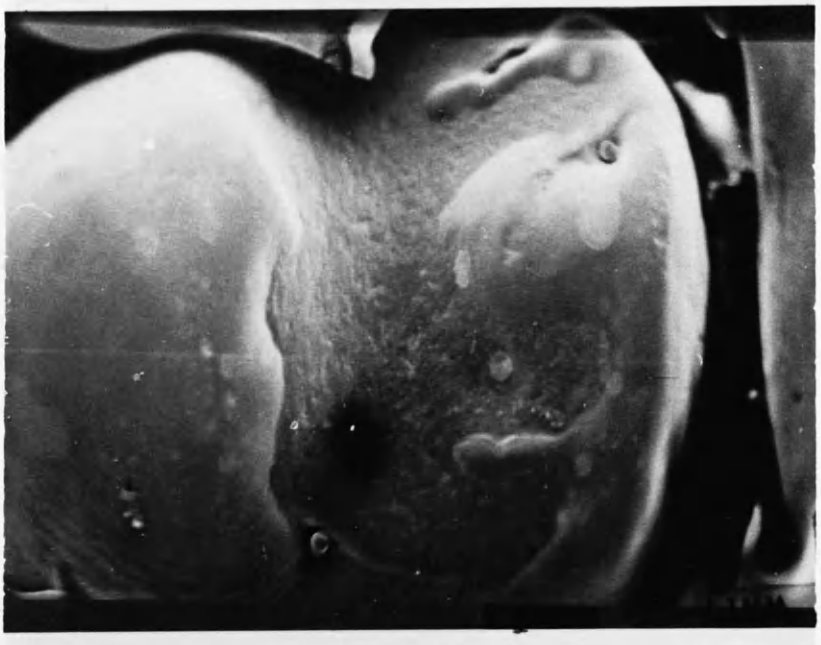
Fig. 7.11 The upper left premolar shown in Figs. 7.9 and 7.10, is shown in mesial profile, 16, 142 and 251 days after sealing, in a, b and c, resp.

(a)

(b)

(c)

Fig. 7.12 The second premolar proximal to the first premolar studied in Fig. 7.9, 7.10 and 7.11, is shown 38, 142 and 251 days after sealing, in a, b and c, resp.



7.4 Discussion

The photographs shown here were selected from replication studies on a total of 28 teeth. While it cannot be claimed that the teeth selected were necessarily typical, they provide some insight to the prevailing mechanisms of sealant loss.

One striking feature of the study was the different condition of sealant applied to premolars and molars. This may be due to the different occlusal forces and abrasion to which these two types of teeth are subjected. The better retention of sealant on premolars than molars has been frequently observed in previous clinical trials, as has already been discussed in Chapter One.

A second feature of the results was that more thickly applied sealant appeared to survive occlusal forces better than thinner layers, although the evidence for this assertion was not conclusive. Certainly the amount of sealant initially placed on the teeth varied greatly from one tooth to another, although it was applied by the same operator. One would also expect that considerable variations would occur between operators, so that the retention of sealant in different clinical trials could be affected.

One mechanism of sealant loss was the gradual removal of sealant from the exposed surface by abrasion. This appeared to be the main factor which would affect the retention of sealant on premolars. However, on molars, substantial fragments of sealant were also lost due to fractures, which in some instances may have been related to the separation of part of the sealant from the enamel. Such a lack of adhesion could have been caused by a faulty application technique, which permitted moisture contamination or incomplete polymerisation at the sealant/enamel interface.

One may conclude that provided a correct application technique is used, sealant retention could be greatly improved by altering the

bulk physical properties without necessarily improving adhesion at the sealant/enamel interface. Improved abrasion resistance would clearly be beneficial, as would an increase in the mechanical strength of the material, to prevent fracturing under occlusal forces.

CHAPTER EIGHT

CLINICAL TRIALS USING A MODIFIED SEALING TECHNIQUE

8.1 Introduction

A wide variation in the retention of Nuva-seal in different clinical trials was shown in Chapter One. Examination of the caries incidence in teeth which had lost all or part of their sealant covering showed that good sealant retention was essential for long term caries prevention. A major objective of this project has been to determine the causes of sealant failure and to outline improvements in sealing technique to optimise retention. Information which has been gathered on the adhesion of sealant to etched enamel, the exposure required for u.v. setting sealants and experience with the materials and equipment resulted in a number of modifications to the standard application procedure. To validate these laboratory conclusions, a detailed clinical technique was outlined and its effectiveness evaluated in clinical trials.

8.2 Fissure Sealing Technique

8.2.1 Tooth cleaning

Teeth were thoroughly cleaned with a non-fluoride prophylactic paste and a rotary brush. The paste was washed off with a 3-in-1 syringe, and a sharp probe used to remove traces of paste from fissures.

8.2.2 Tooth isolation

Upper teeth were isolated by placing No.1 cotton wool rolls in the buccal sulcus while an assistant used a high velocity aspirator to remove water and saliva. Lower teeth were isolated using No.1 cotton wool rolls in the buccal and lingual sulci and a child size flanged

saliva ejector. These smaller cotton wool rolls were more comfortable for the patient and allowed a clear view of the tooth during treatment.

8.2.3 Acid etching

Fresh acid was placed in a dappen's glass for each patient and applied with cotton pellets for a timed period of 60s. To remove acid and dissolved enamel following etching, a jet of water squeezed from a polythene bottle by hand pressure, was used to soak the tooth surface. This water was removed with an aspirator, and the procedure repeated until six separate rinses of the etched tooth surface had been completed. Soaking the tooth in this manner was more acceptable to the patient than the conventional 3-in-1 water spray which tended to irritate the throat and interrupted the procedure, with consequent contamination of the etched surface. Once rinsing was completed dry cotton rolls were placed over the original rolls, which were then removed from beneath.

8.2.4 Tooth drying

The etched tooth was dried for about 20s with an air jet, free from either oil or water contamination. Great care was exercised at this stage to avoid salivary contamination, by guarding the tooth with a mouth mirror to prevent lingual contact, while the assistant continued aspiration. Should saliva contact the etched enamel a rapid deposition of protein could have taken place which might not be removed by washing or re-etching (Meurman, 1976). If any salivary contamination was observed, sealing was postponed to a later date.

8.2.5 Application of sealant

In developing the application technique in the laboratory air bubbles were found to be accidentally incorporated into the sealant.

Since it was believed that these could adversely affect sealant retention, a technique was devised to avoid their incorporation. The initiated sealant was mixed slowly for 30s, producing as few air bubbles as possible. A drop of sealant was then applied to the tooth surface using a flat, plastic instrument instead of the camel hair brush supplied by the manufacturers. A probe was then run along the fissures prior to activation with u.v. radiation in an attempt to remove any trapped air. The latter process has also been used by Ulvestad (1973). Further sealant was added to produce the maximum thickness which would not interfere with the occlusion, since studies on sealant loss in Chapter Seven had indicated that a thicker layer would be better retained than a thin layer.

8.2.6 Application of u.v. radiation

To ensure optimum polymerisation, the output of the Nuva-lite was checked prior to the trial, using the Blak-Ray U.V. Meter as described in Chapter Three. Throughout the trial further measurements determined that the output was always sufficient to supply an average intensity in excess of 20 mW/cm^2. The sealant was exposed to the u.v. radiation for a timed period of 45s, by moving the tip of the Nuva-lite across the tooth surface, as close to the sealant as possible. This process maximised the u.v. intensity and compensated for the uneven distribution of radiation at the quartz tip. Following each treatment the quartz guide was cleaned with chloroform to prevent the accumulation of polymerised sealant.

8.3 Design of Clinical Trials

To assess the technique outlined above, permission was obtained for a clinical trial using children from Garnetbank Primary School adjacent to Glasgow Dental Hospital. Sealing was carried out by a dental

auxiliary aided by her regular dental assistant. Since neither operator had any previous experience with sealants, they were able to learn the procedure outlined above without having to modify pre-conceived ideas. Both received laboratory instruction and sealing practice on extracted teeth during the first few days of training. Twelve children of the staff of the Department of Clinical Physics and Bio-Engineering were then treated during a supervised two week practice session.

A small pilot study was initiated, in which 16 ten-year-old and 6 six-year-old children had 54 caries-free first permanent molars and 20 caries-free premolars treated. These teeth were examined by a probe and mirror within a few days of sealing and subsequently at 1, 4, 8 and 12 months after sealing using a blind examination technique. Retention of sealant was assessed separately for the occlusal and buccal surfaces of lower first permanent molars, and for the mesial fossa, distal fissure, and the palatal extension of the distal fissure in upper first permanent molars. Premolars were assessed for the coverage of pits and fissures on their occlusal surfaces as a whole. Each site was recorded as having 'complete,' 'partial' or 'no sealant' retention. If any part of the fissure pattern was uncovered, then that site was recorded as having partial retention, even if the majority of the fissures remained sealed.

When the pilot study showed that this sealing technique resulted in good retention, a larger clinical trial was initiated. Here, 78 children aged from six to ten years had any caries-free permanent molars sealed over a three week period. Re-examinations were completed at 1, 2, 4, 6, 9 and 12 months after the sealing period.

8.4 Results

8.4.1 Pilot trial

Sealant retention, up to 12 months, on the different surfaces

sealed in the pilot trial are shown in Table 5. After one year 88% of occlusal surfaces examined in lower first permanent molars were wholly covered by sealant, while 82% of the mesial fossae and 79% of distal pits in upper first permanent molars were found to be completely sealed. The few buccal and palatal sites treated showed poorer retention. Sealant was completely retained in all premolars during the study.

8.4.2 Second trial

The retention of sealant in the 241 sites treated on 157 first permanent molars in 78 children is shown, for examinations up to 12 months, in Table 6. Due to children leaving the area the number of sites available for examination fell from 241 at the one month re-examination to only 194 at the end of the study year. After one month all the sealant was intact. Subsequent examinations revealed that a few tooth surfaces gradually lost some sealant covering with 93·3% of treated surfaces fully covered after 12 months. At that time only 3·1% of sites examined had totally lost their sealant, while the remaining 3·6% had partial coverage. In most cases these had lost sealant from only a small fraction of the total fissure pattern.

Table 7 shows the pattern of retention for the different tooth sites. At the one month examinations all the surfaces were intact. Mesial and distal sites which were later found to have only partial retention, were generally completely uncovered at subsequent examination. However, none of the occlusal sites in lower first permanent molars had lost all of their sealant covering after 12 months, although 5 sites were found to have partial loss.

The sealant status on surfaces of first permanent molars for the different age groups of children is shown in Table 8. Although there was little difference with regard to sealant loss in the first 6 months, by the 9 month examination the loss sustained by the 6 and 7 year-olds

Table 5

Retention of Nuva-seal on different tooth sites, on upper and lower first permanent molars and premolars sealed during the pilot trial, and examined at times indicated.
(C=Complete retention; P=Partial retention; L=Sealant lost)

	2 weeks			1 month			4 months			8 months			12 months		
Sealed sites	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.
Mesial Fossa	28	-	-	28	-	-	25	2	1	25	1	2	23	3	3
Distal fissure	28	-	-	28	-	-	25	2	1	24	3	1	22	2	4
Palatal fissure	10	-	1	9	-	2	8	1	2	8	-	3	6	-	5
Occlusal surface	25	-	-	25	-	-	24	1	-	23	2	-	22	1	2
Buccal pit	5	-	-	5	-	-	4	-	1	3	-	2	3	-	2
First premolar	13	-	-	13	-	-	13	-	-	12	-	-	12	-	-
Second premolar	7	-	-	7	-	-	7	-	-	6	-	-	6	-	-
All Sites	116	-	1	115	-	2	106	6	5	101	6	8	94	6	16

Table 6

Retention of Nuva-seal on all first permanent molar occlusal sites at examination times indicated.

RETENTION	1 month	2 months	4 months	6 months	9 months	12 months
Complete	241 (100%)	238 (99.2%)	215 (98.2%)	210 (97.7%)	186 (94.9%)	181 (93.3%)
Partial	-	2 (0.8%)	4 (1.8%)	4 (1.8%)	7 (3.6%)	7 (3.6%)
Lost	-	-	-	1 (0.5%)	3 (1.5%)	6 (3.1%)

Table 7

Retention of Nuva-seal on 'mesial' and 'distal' aspects of upper, and occlusal surfaces of lower first permanent molars at examination times indicated.

(C = Complete retention; P = Partial retention; L = Sealant lost)

Sealed occlusal site		1 month			2 months			4 months			6 months			9 months			12 months		
		C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.
'Mesial'	n	78	-	-	78	-	-	70	1	-	68	-	1	60	1	2	59	1	3
	%	100%	-	-	100%	-	-	98.6%	1.4%	-	98.6%	-	1.4%	95.2%	1.6%	3.2%	93.7%	1.6%	4.7%
'Distal'	n	75	-	-	73	1	-	66	1	-	64	1	-	57	1	1	54	2	3
	%	100%	-	-	98.7%	1.3%	-	98.5%	1.5%	-	98.5%	1.5%	-	96.6%	1.7%	1.7%	91.5%	3.4%	5.1%
Occlusal	n	88	-	-	87	1	-	79	2	-	78	3	-	69	5	-	68	4	-
	%	100%	-	-	98.9%	1.1%	-	97.5%	2.5%	-	96.3%	3.7%	-	93.2%	6.8%	-	94.4%	5.6%	-

Table 8

Number of first permanent molar sites showing different categories of sealant retention for the various age-groups at examination times indicated.

(C = Complete retention; P = Partial retention; L = Sealant lost)

	1 month			2 months			4 months			6 months			9 months			12 months		
Age (yr)	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.	C.	P.	L.
6	48	-	-	47	1	-	39	2	-	40	2	-	33	3	-	31	4	1
7	45	-	-	45	-	-	37	2	-	37	1	1	35	1	3	34	1	4
8	53	-	-	52	1	-	47	-	-	47	-	-	41	-	-	41	-	-
9	46	-	-	46	-	-	43	-	-	39	-	-	31	1	-	32	-	-
10	49	-	-	48	-	-	48	-	-	47	1	-	46	2	-	43	2	1
All	241	-	-	238	2	-	215	4	-	210	4	1	186	7	3	181	7	6

was found to vary significantly, using a chi-square test, from that of the 8, 9 and 10 year-olds ($P < 0{\cdot}05$) and this pattern was more noticeable after one year ($P < 0{\cdot}001$), with 10 of the 13 sites which had partially or totally lost sealant cover to be found in the younger age group.

8.5 Discussion

In these trials, which were initiated to determine whether Nuva-seal could be successfully applied to first permanent molars of young children using an optimised sealing technique, exceptionally high retention rates have been found. Retention in premolars and the occlusal sites of lower first permanent molars and the mesial and distal sites in upper first permanent molars was good in the pilot trial and better in the second trial. However, in both studies the few buccal and palatal sites treated showed poor retention, confirming findings in previous trials (Cons et al., 1976).

The second trial was carried out to confirm the indication from the pilot study that first permanent molars could be sealed effectively with this technique, even under the more difficult conditions prevailing in the small mouths of six to seven-year-old children. The method used was essentially that employed in the pilot trial, but particular emphasis was placed on applying as thick a layer of sealant as possible. Here, sealant retention was higher than in the pilot study, but retention in younger children was slightly worse than in older children. Good retention in the teeth of these young children is important, since the rapid onset of caries in first permanent molars after eruption makes it very desirable to seal these teeth as early as possible (Lewis and Hargreaves, 1975; Stephen et al., 1976; Crabb, 1976). However, it is generally recognised that first permanent molars in young children are the most difficult sites to seal, due to problems

of isolating these teeth in their confined position at the rear of a small mouth.

The advantage gained by following the technique described here can be judged by a comparison with the retention of Nuva-seal in other clinical trials. Twelve out of thirty trials with Nuva-seal which were studied, specified retention data in first permanent molars using a comparable assessment technique. The percentage of teeth in these trials which were wholly covered at various times after sealing were discussed in Chapter One and plotted in Fig. 1.2. Only Helle (1975) has reported better retention than found in the second trial, with all the other studies revealing a greater rate of loss. Many reports on Nuva-seal trials have not specified the retention of sealant in the first permanent molars, and were therefore not suitable for direct comparison with results found here. However, in some cases the retention was poor in both premolars and molars as a whole, indicating that molar retention was probably much worse. For example, Stiles et al. (1976) reported 61% retention after 6 months, Harris et al. (1976) found as little as 19% complete retention in one treated population after one year, and Whitehurst and Soni (1976) found less than 5% of the treated sites remained covered after one year.

8.6 Conclusions

It has been shown that by following a careful application procedure it is possible to obtain excellent retention of Nuva-seal even in first permanent molars in six-year-old children. Previous trials may have achieved less success because of,

(a) inadequate isolation

(b) inadequate washing of the etched tooth

(c) oil or water contamination from the air supply

(d) incomplete polymerisation due to exposure to too low an intensity of u.v. radiation, or for too short a period

(e) use of faulty materials

(f) use of thin layers of sealant

(g) incorporation of air bubbles into the sealant

A successful sealing technique requires meticulous care and attention to detail. However, it is also important that knowledge of the properties of the sealant materials and equipment should be used to train operators to use the best techniques possible. Further improvements on results reported here will probably not stem from new advances in the application technique, but from the development of sealants with better bulk physical properties.

APPENDIX I

POLYMERISATION MECHANISMS OF U.V. ACTIVATED SEALANTS

Photosensitizers have been added to fissure sealant resins to allow rapid polymerisation on exposure to long-wavelength u.v. radiation. Three systems employing u.v. activation have been mentioned in the dental literature (c.f. Chapter One), Nuva-seal, Alphaseal and the experimental products ESPE 717 and 71729. Only Nuva-seal and Alphaseal were initially included in these studies, since supplies of ESPE products were not available from the manufacturers. During the final year of the project an experimental u.v. activated sealant containing filler particles (Nuva-cote, L.D. Caulk Co. Ltd.) became available for study. The u.v. emission from Nuva-lite and Alphalite sources has already been discussed in Chapter Three. However, the significance of the results found there can only be assessed in the light of the setting behaviours of the two corresponding materials Nuva-seal and Alphaseal, which have been investigated using two new techniques. In Chapter Four, the microhardness of Nuva-seal and Alphaseal on exposure to various u.v. intensities was presented. In Chapter Six a new method was described which relied on the passage of vibrations to assess the set.

However, the setting behaviour of an u.v. activated resin can be predicted from theoretical considerations, and in this appendix the effect of the u.v. intensity on the polymerisation of such resins is discussed.

Kinetics of photopolymerisation

The polymerisation of u.v. activated resins, such as the fissure sealants Nuva-seal and Alphaseal, can be considered as proceeding by four distinct steps,

(a) the production of primary radicals,

(b) the initiation of chain radicals,

(c) the propagation of chain radicals,

(d) The termination of chain radicals.

Each of these steps are discussed in turn using the following symbols

(M) = concentration of monomer

(P) = free radical polymer chain concentration

(A) = concentration of initiator

(A*) = concentration of primary radicals

I_a = u.v. intensity absorbed

R_p = rate of polymerisation

K_a = activation constant

K_i = initiation constant

K_p = propagation constant

K_t = termination constant

The primary radicals are produced by the absorption of photons of sufficient energy to dissociate initiator molecules into free radicals, at a rate given by

$$\text{activation rate} = K_a(A)I_a \qquad (1)$$

The remaining stages of the polymerisation process can be described by the Mass Action Law, which states that the rates of reaction are proportional to the concentrations of the reacting species. Thus the primary radicals combine with monomer molecules to initiate chain radicals at a rate given by,

$$\text{rate of initiation} = K_i(A^*)(M) \qquad (2)$$

If it is assumed that the rate of growth of a chain radical is

independent of its length, a single propagation constant is applicable, and (P) represents the sum of the concentrations of the different lengths of growing polymer chains. Thus

$$\text{propagation rate} = K_p(P)(M) \qquad (3)$$

The growth of the radical chains may be terminated by the interaction of two growing radical chains, so that

$$\text{termination rate} = K_t(P)^2 \qquad (4)$$

This may represent either the combination of the radicals or disproportionation. In addition to these processes termination could also occur by the interaction of chain radicals with primary radicals, or by direct combination of primary radicals. However the simplest situation is assumed, here, with termination occurring by the interaction of chain radicals. Of one assumes a steady state condition, where the rate of change of the concentrations of radicals is much less than their rates of production and destruction, then

$$\frac{d(A^*)}{dt} = 0 \qquad (5)$$

$$\frac{d(P)}{dt} = 0 \qquad (6)$$

To maintain condition (5) the rates of activation and initiation must be equal (neglecting the recombination of primary radicals). To maintain condition (6) the rates of initiation and termination must be equal. Hence,

$$K_a(A)\, I_a = K_i(A^*)(M) = K_t(P)^2 \qquad (7)$$

$$P = \left[\frac{K_a(A) I_a}{K_t}\right]^{\frac{1}{2}} \qquad (8)$$

Substituting equation (8) into equation (3)

$$R_p = \frac{K_p K_a^{\frac{1}{2}}}{K_t^{\frac{1}{2}}} (M)(A)^{\frac{1}{2}} I_a^{\frac{1}{2}} \qquad (9)$$

Thus the rate of polymerisation in a photosensitized reaction can be

expected to have a direct dependence on the monomer concentration and a square root dependence on the initiator concentration and the radiation intensity. The form of the dependence on the initiator concentration and radiation intensity has been confirmed experimentally by Chinmayanandam and Melville (1954) for benzoin-sensitized photo-polymerisation of methyl methacrylate using radiation at 360 nm wavelength. Chen and Huang (1969) also used benzoin to polymerise methyl methacrylate with radiation at 297, 313 and 365 nm wavelength, confirming the results found by Chinmayanandam and Melville for the rate dependence on initiator concentration and intensity, as well as finding a square root dependence on the monomer concentration.

Chain lifetimes

This approach can also be used to determine free radical chain lifetimes, where

τ = average lifetime of radicals

= concentration of active radicals/rate of their disappearance

$$= \frac{(P)}{K_t(P)^2} = \frac{1}{K_t(P)}$$

Now using the expression for (P) in equation (8), one finds

$$\tau = \left[K_t K_a (A) I_a\right]^{-\frac{1}{2}} \qquad (10)$$

Thus the lifetime of chain radicals is expected to be inversely proportional to the square roots of the intensity and initiator concentration.

Molecular weight of products

The molecular weight (M.W.) of the products can be predicted using expressions (3) and (4), so that

$$M.W. = \frac{\text{rate of propagation}}{\text{rate of termination}} \times m, \quad \text{where } m = \text{molecular weight of monomer.}$$

$$= \frac{K_p (P) M}{K_t (P)^2} m = \frac{K_p (M)}{K_t (P)} m$$

Now substituting for the expression for (P) in equation (8)

$$M.W. = \frac{K_p}{(K_t K_a)^{\frac{1}{2}}} \times \frac{(M)}{(A)^{\frac{1}{2}} I_a^{\frac{1}{2}}} \times m \qquad (11)$$

Thus the molecular weight and therefore the mechanical properties of the polymer structure can be expected to depend on both the intensity and the initiator concentration.

The gel effect

Once a large proportion of the monomer has been incorporated into polymer chains, polymerisation might be expected to continue at a reduced rate due to the reducing concentration of monomer. However polymerisation also produces an increase in viscosity which reduces the mobility of the free radical polymer chains, so that a "gel" effect reduces the termination rate. In terms of the above theory, this represents a reduction in the value of K_t. From equations (9), (10) and (11) the rate of polymerisation, chain lifetimes and molecular weight can all be expected to increase due to the "gel" effect.

One consequence of this is that in the early stages of polymerisation chain lifetimes are quite short. Thus polymerisation comes to a rapid halt if illumination is interrupted. However, once the viscosity rises, the gel effect increases chain lifetimes, so that polymerisation may continue for some time after illumination is cut off. This has particular relevance to the setting of Alphaseal in the clinical situation where illumination is applied only intermittently, due to the scanning motion of the Alphalite. Thus polymerisation may continue during the periods when the resin is not being irradiated.

Conclusions

The theory presented here can be expected to apply in detail, only for the early stages of polymerisation where steady state conditions would be applicable, and it is in this region that it has been verified experimentally. However with fissure sealants one is concerned with the progress of polymerisation beyond this region. Thus any predictions about the setting of sealants using the above theoretical approach require experimental confirmation. Nonetheless this simple theory provides an insight into the chemical processes involved, and illustrates that the u.v. intensity can be expected to affect both the rate of set and final physical properties of u.v. activated sealants.

APPENDIX II

TRANSMISSION OF ACOUSTIC VIBRATIONS THROUGH FISSURE SEALANTS DURING POLYMERISATION

In Chapter Six an acoustic method of monitoring the setting of fissure sealants was described. Experiments showed that changes in the physical properties of the sealants during polymerisation, increased the acoustic transmission across the resin samples. The rate of increase in the transmission provided a measure of the rate of set of the sealants. This was particularly useful when comparing the setting rates achieved by the same u.v. sealant under different intensities of u.v. radiation. In this Appendix the physical changes which determined the increase in acoustic transmission are discussed.

In the experimental arrangement, a gap between two steel pins was filled by fissure sealant. A continuous longitudinal acoustic signal of amplitude, v_1, was sent along one steel pin, and the amplitude of the vibrations transmitted into the other pin, v_3, was measured. Since the characteristic acoustic impedence of the resin was very much less than that of steel, multiple reflections could be expected to occur between the resin/steel interfaces. The reflection loss for a similar system used to damp the transmission of sound vibrations has been discussed by Beranek (1971) using results which were derived by Cremer (1967) by the application of the theory of multiple reflections. It was shown that the fraction of the incident acoustic energy transmitted (T) is given by

$$T = \left| \frac{v_3}{v_1} \right|^2 = \frac{1}{1 + \left[\frac{wz}{2s} \right]^2}$$

Where, for this arrangement v_1 = incident amplitude of vibration

v_3 = transmitted amplitude of vibration

w = frequency

Z = acoustic impedence of steel

s = stiffness of resin sample

$= EA/d$

A = cross-section area of resin

d = width of resin gap

E = elastic modulus of resin

If reflection losses are large, so that T is much less than unity, then

$$T = \left|\frac{v_3}{v_1}\right|^2 \doteq \left[\frac{2s}{wZ}\right]^2 = \left[\frac{2EA}{wdZ}\right]^2$$

$$\Rightarrow \left|\frac{v_3}{v_1}\right| \propto s \propto E$$

Hence under these circumstances the fraction of the incident amplitude which is transmitted is proportional to the stiffness of the resin sample, and also therefore to the elastic modulus for a given sample length. Thus in the liquid state the stiffness of the resin would be virtually zero, so that the transmission would be negligible. In practice this was confirmed, since the acoustic signal across the liquid sealant was not detectible above the background noise. As the resin set the stiffness of the resin sample increased so that the transmission also increased. This change was readily detected by the apparatus described in Chapter Six.

However, this simple one dimensional model does not fully explain the behaviour of the experimental system. Despite the fact that the resin has a much lower acoustic impedence than steel, the theory predicts complete transmission once the resin is set, due to the shortness of the gap compared to the wavelengths involved. In practice

the experiments discussed in Chapter Six showed that the transmission was certainly less than unity for Nuva-seal and Alphaseal since higher transmissions were noted with Nuva-cote. This is probably explained by the greater stiffness of Nuva-cote, due to the incorporation of filler particles.

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