J. Dent. Ass. S. Afr. Vol. 30, No. 7. pp. 595 - 601

Laboratory evaluation of the tensile adhesive bond strength of a composite dental restorative system containing a surface — active comonomer to enamel and dentine

D H Retief

Dental Research Unit of the University of the Witwatersrand and the South African Medical Research Council, Johannesburg

SUMMARY

A major problem in restorative dentistry is the lack of adhesion of dental materials to tooth structure. Bowen (1965a) synthesized a surface-active comonomer, the reaction product of N-phenylglycine and glycidylmethacrylate, to serve as a coupling agent between dental restorative materials and tooth structure. A new composite restorative material, De Trey Cosmic, which incorporates Cosmic Bond (Bowen's surface-active comonomer) in a metered aerosol, has recently been introduced to the profession. The tensile adhesive bond strengths of Cosmic to untreated and primed enamel and dentine surfaces were determined. All of the 30 experimental bonds prepared on untreated enamel and dentine surfaces failed spontaneously under the experimental conditions employed in this investigation. Nine of the test specimens prepared on enamel surfaces primed with Cosmic Bond failed spontaneously and a mean tensile adhesive bond strength of 0,8 ± 0,4 N mm ² was obtained for the remaining 6 specimens. Only one of the 15 experimental bonds prepared on primed dentine failed spontaneously and a mean tensile adhesive bond strength of 3,4 ± 1,9 N mm ² was recorded. Examination by scanning electron microscopy of the enamel and dentine aspects and Cosmic surfaces of fractured bonds on primed surfaces, revealed evidence of some interaction at the interfaces of fractured bonds that recorded higher bond strengths.

OPSOMMING

'n Groot probleem in die herstellende tandheelkunde is die kleefgebrek tussen tandheelkundige stowwe en tandstruktuur. Bowen (1956a) het 'n oppervlakaktiewe komonomer gesintetiseer, 'n reaksie-produk van N-fenielglisien en glisidielmetakrielaat om as 'n koppelagent tussen tandheelkundige herstelmateriale en tandstruktuur te dien. 'n Nuwe samegestelde herstellingsmateriaal, De Trey Cosmic, wat die Cosmic Bond (Bowen se komonomer wat oppervlakaktief is) in 'n gemete aërosol ingelyf het, is onlangs aan die professie vrygestel. Die vaskleefkrag en trekvastheid van Cosmic teenoor onbehandelde en voorbereide glasuur- en dentien-vlakke is vasgestel. Die dertig eksperimentele bindings wat voorberei is op onbehandelde glasuur- en dentien-vlakke het onder die eksperimentele omstandighede in hierdie ondersoek spontaan misluk. Nege van die proefmonsters in hierdie ondersoek wat voorberei is op glasuur-vlakke wat met Cosmic Bond voorberei is, het spontaan misluk en 'n gemiddelde vaskleefkrag en trekvastheid van 0,8 ± 0,4 N mm ² is met die oorblywende ses monsters verkry. Slegs een van die vyftien eksperimentele bindings wat op voorbereide dentien uitgetoets is, het spontaan misluk en 'n gemiddelde vaskleefkrag en trekvastheid van 3,4 ± 1,9 N mm ² is aangeteken. Ondersoek onder die skandeer-elektronmikroskoop van die glasuur- en dentienaspekte en die Cosmic-oppervlakke van gebreekte bindings op voorbereide oppervlakke het getoon dat daar 'n mate van wisselwerking was van die intervlakke van gebreekte bindings wat hoër bindingsterktes aangeteken het.

INTRODUCTION

As water is always present on the tooth surface and it actually functions as a liquid adhesive, a dental adhesive material has to compete with it for the bonding sites on the hydroxyapatite surface. The adsorption energy of a monolayer of water on the hydroxyapatite surface is from 18-21 kilo calories/mole (Beebe, 1961). These figures imply that a tooth surface cannot be dried completely by clinical procedures. The ideal filling material must also be successfully competitive with water over long periods because of the fluid flow that occurs from the pulp of the tooth to the enamel surface (Bergman, 1963; Linden, 1968) and constant exposure to the moist oral environment.

The presence of water in the oral cavity can be the precursor of chemical activity at the adhesive/adherend interface which will eventually dislodge the bond. In addition, many dental restorative materials absorb water leading to swelling and dimensional changes in their bulk. This will lead to the development of stress concentrations at the interface which will have an adverse effect on the bond strength.

Rose *et al* (1955) found that bonding between many dental restorative materials and dentine and enamel was destroyed by exposure to water. As recently as 1968, Gwinnett and Matsui reported that none of the materials available to the dental profession consistently maintained adhesion to tooth structure in the oral environment.

Bowen, (1965a) concluded that the achievement of strong and stable adhesion between a dental restorative material and enamel and dentine required a waterproof bonding mechanism. He postulated that a coupling agent containing polar groups which were theoretically capable of chelating with surface calcium atoms in mineralized dental tissues, and additional groups, capable of copolymerization with the resin systems of dental restorative materials, should give improved bonding of the materials to tooth surfaces. He coined the term surface-active comonomer for this type of coupling agent. Bowen (1965a) reported the synthesis of such a coupling agent, the reaction product of N-phenylglycine (NPG) and glycidylmethacrylate (GMA). The formula of the NPG-GMA adduct [N-(2-hydroxy-3-methacryloxypropyl)-N-phenylglycine] is shown in Fig. 1. He used a 5% solution of the coupling agent in ethanol and reported that the NPG-GMA solution promoted bonding to a significant degree when used as an intermediary between a methacrylate restorative system and dentine. He found, however, that after the NPG-GMA solution had aged for 2 months with intermittent exposure to fluorescent light, it was no longer able to produce reliable bonding. He suggested that a small amount of polymerization inhibitor should be added to the surface-active comonomer solution to improve its shelf-life.

In a subsequent investigation, Bowen (1965b) reported that pretreatment of dentine surfaces with 0,01 N HCl and 0,01 N NaOH followed by rinsing and blotting prior to the application of the NPG-GMA coupling agent, substantially increased the bond strength of the methacrylate restorative material to dentine. To further elucidate the mechanism of action of the NPG-GMA coupling agent, Bowen (1965c) determined the bonding of a direct filling resin to various substrates. These included human dentine and enamel and single crystals of natural mineral fluorapatite. He selected fluorapatite as a substrate because it contains no organic matter and closely resembles hydroxyapatite, the principal inorganic constituent of enamel and dentine. Since the highest bond strengths were recorded on fluorapatite, Bowen concluded that collagen and other organic constituents present in dentine and enamel were not responsible for the mechanism of bonding obtained with the NPG-GMA surface-active comonomer. He found that one of the resin formulations containing vinylsilane-treated fuzed silica particles as a reinforcing filler, gave significantly higher bond strengths than the unfilled control resin.

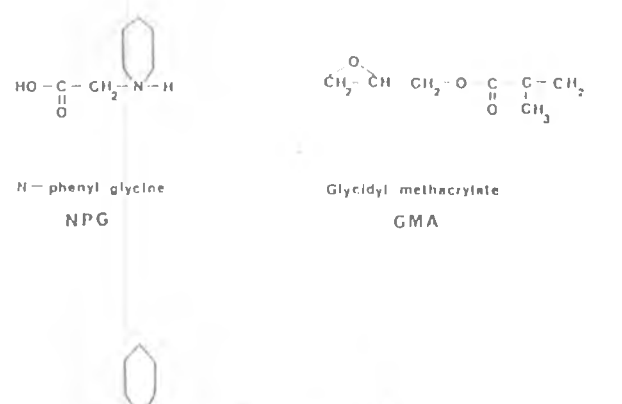

Fig. 1. Formula of N-phenylglycine (NPG) and glycidylmethacrylate (GMA) adduct.

The NPG-GMA adduct did not improve bonding between a dental resin and surfaces of bovine tendon which consists predominantly of collagen (Bowen, 1965d). The results of these investigations suggested that the mechanism by which the NPG-GMA coupling agent improved bonding between a methacrylate resin and hard tooth tissues may be primarily by interaction with the mineral phase rather than the organic phase of the tooth's structure. In a hypothetical schematic diagram, Bowen (1965e) demonstrated how the various phases in a composite material containing silane-treated siliceous filler might be connected to tooth mineral by chemical bonds via the NPG-GMA surface-active comonomer. The interaction between a composite material consisting of BIS-GMA resin and inorganic filler coated with a methacrylate organofunctional silane and tooth structure based on Bowen's hypothesis is depicted in Fig. 2.

A new composite restorative material "De Trey Cosmic"[1], which incorporates Bowen's surface-active comonomer in a metered aerosol, has recently become commercially available. The filler consists of silane-coated micronized barium glass and the resin is a urethane-acrylate monomer. The surface-active comonomer solution, Cosmic Bond, is deposited on the cavity walls prior to the placement of the restorations. The manufacturers claim that the comonomer reacts with the calcium ions of the tooth

[1]Amalgamated Dental Company, 26 Broadwick Street, London W1, England.

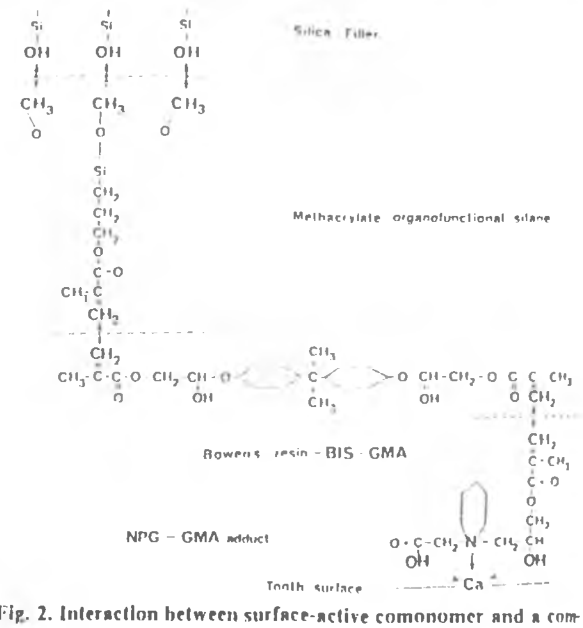

Fig. 2. Interaction between surface-active comonomer and a composite material consisting of BIS-GMA resin and inorganic filler coated with a methacrylate organofunctional silane and tooth structure based on Bowen's hypothesis.

surfaces by a process of chelation and forms a chemical union with the Cosmic paste as it polymerizes. The silane coupling agent further bonds the filler particles to the resin. Adhesive bonding tests were conducted with Cosmic, with and without Cosmic Bond, by attaching orthodontic buttons to the enamel and dentine surface of natural, extracted teeth. The teeth were immersed in water at 37°C and the tensile adhesion determined after 24 h and 28 days in an Instron test machine. An average bond strength of 10 kg/cm² was recorded for Cosmic on both enamel and dentine without Cosmic Bond. An increased bond strength of 24 kg/cm² was obtained on enamel and 19 kg/cm² on dentine pretreated with Cosmic Bond after the specimens had been immersed in water for 24 h. The bond strengths of the specimens immersed in water for 28 days were not significantly decreased (De Trey Cosmic. The First Complete Composite System).

The physical and mechanical properties of De Trey Cosmic restorative material have recently been investigated by Bailey, Shovelton and Wilson (1973). Incisor teeth were mounted in acrylic resin and the exposed labial enamel surfaces reduced in a lathe so that a circle of enamel with a diameter of 5 mm was obtained on each specimen. The enamel surfaces were sandblasted and the tests performed after the material had been allowed to set for one h at room temperature. Only two assemblages were used for each test to assess the enamel-Cosmic-enamel and enamel-primer-Cosmic-primer-enamel interfacial strengths. The authors reported that it appeared that a relatively high one-hour bond strength was obtained whether or not the primer was used.

Chandler *et al* (1974) carried out a clinical evaluation of a tooth-restoration coupling agent. The clinical effectiveness of the NPG-GMA coupling agent was tested by the restoration of 54 Class III and Class IV cavity preparations pretreated with a 5% acetone solution of NPG-GMA containing stabilizers. For comparative purposes, a similar number of restorations were placed in cavities in matched teeth pretreated with acetone alone. The composite restorative material used was a crosslinked methacrylate polymer as the resin binder and a combination of radiopaque, barium fluoride-containing glass and spheriodized fuzed silica particles as the inorganic reinforcing filler. The restorations were examined regularly and evaluated by three dentists over a period of 3½ years. The results indicated that the restorations placed over the NPG-GMA coupling agent had improved margins and a significantly higher number associated restorations were rated better compared to those placed over the acetone treated surfaces.

The purpose of this investigation was to determine the tensile adhesive bond strengths of Cosmic, with and without Cosmic Bond, to polished enamel and dentine surfaces under controlled experimental conditions. Both the Cosmic and corresponding enamel and dentine aspects of fractured experimental bonds were examined by scanning electron microscopy (SEM) in an attempt to determine the site and mechanism of failure.

MATERIALS AND METHODS

Freshly extracted, sound human maxillary central incisors were used in this investigation. The teeth were cleaned and stored at -4°C until required.

1. Tensile bond strengths of Cosmic with and without Cosmic Bond to polished enamel and dentine surfaces

The tensile disruptive force required to break an experimental bond was used as a measure of the tensile adhesive bond strength of Cosmic to polished untreated and primed enamel and dentine surfaces. The technique used in this investigation was developed by Hanke (1968) and modified by Phillips, Swartz and Rhodes (1970). The preparation of the experimental bonds was fully described in a previous paper (Retief, 1974). Thirty test specimens were prepared on polished enamel surfaces and a similar number on polished dentine surfaces. The Cosmic restorative material was mixed according to the manufacturer's instructions. Cosmic Bond was applied with the metered aerosol to 15 enamel and dentine specimens respectively, prior to the preparation of the experimental bonds. The restorative material was applied directly to the remaining polished enamel and dentine surfaces.

The experimental bonds were allowed to set at room temperature for 15 min and then immersed in water at 37°C for 24 hours. The specimens were mounted in a test jig which was designed to allow proper alignment of the specimens in the testing machine and to eliminate as nearly as possible all forces other than tensile during the application of the load. As Instron Table Model 1026 tensile testing machine[2] was used, a loading speed of 0,05 cm min⁻¹ applied and the force required to break an experimental bond automatically recorded. The tensile adhesive bond strengths were calculated and expressed in newtons per square millimetre ($N\ mm^{-2}$). [$1\ Kg\ cm^{-2} = 0{,}0981\ N\ mm^{-2}$].

2. Scanning electron microscopy of enamel and adhesive aspects of fractured bonds

After failure of the test specimens, some of the teeth were removed from the cold-curing resin moulds and the corresponding adhesive stubs were separated from the ball mountings with a diamond disc. The specimens were mounted on aluminium stubs, coated with silver in an Edward Coating Unit, Model E12E4[3], and viewed in a Cambridge Stereoscan S4[4] SEM. The instrument was operated at 30 kV and the beam/specimen angle varied to obtain the best surface projection.

RESULTS

None of the specimens prepared by the direct application of Cosmic to polished enamel and dentine surfaces showed any adhesion. All these experimental bonds failed during immersion in water at 37°C. Nine of the 15 specimens prepared on polished enamel surfaces pretreated with Cosmic Bond became dislodged

[2]Instron Limited, High Wycombe, England.
[3]Edwards Ltd., Cramley, Kent, England.
[4]Cambridge Scientific Instruments Ltd., Cambridge, England.

during the period of water immersion. Only one of the 15 experimental bonds on dentine pretreated with Cosmic Bond failed prior to testing in the Instron machine. The mean tensile adhesive bond strengths obtained are presented in Table I. Only the results obtained with the specimens which did not fail during incubation in water at 37°C were taken into account when calculating the mean values. The mean tensile adhesive bond strengths of Cosmic to polished enamel and dentine surfaces pretreated with Cosmic Bond were 0,8 $\pm$ 0,4 N mm^{-2} and 3,4 $\pm$ 1,9 N mm^{-2} respectively.

An enamel surface polished with 600 grit silicon carbide paper showed criss-cross striations produced by the abrasive particles when viewed in the SEM (Fig. 3). The enamel aspect of a Cosmic experimental bond on an untreated polished enamel surface which failed spontaneously during water immersion is shown in Fig. 4. The abrasive marks are clearly defined and debris particles are scattered on the surface.

The adhesive aspect of the same bond is a mirror image of the enamel aspect (Fig. 5). The experimental bond of Cosmic on a polished enamel surface pretreated with Cosmic Bond which gave the highest bond strength (1,46 N mm^{-2}) was selected for viewing in the SEM. The enamel aspect of the fractured bond revealed fine abrasive marks and well defined fracture areas in the enamel (Fig. 6). The adhesive aspect of the same fractured bond exhibited a veiled appearance with a band of fractured enamel on the surface (Fig. 7). The bonds on pretreated enamel surfaces which failed spontaneously or re

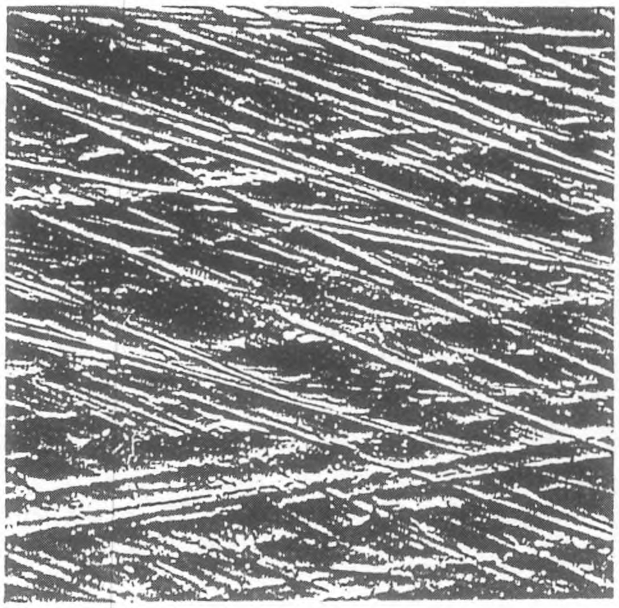

Fig. 3. Enamel surface polished with 600 grit SiC. SEM X 2000.

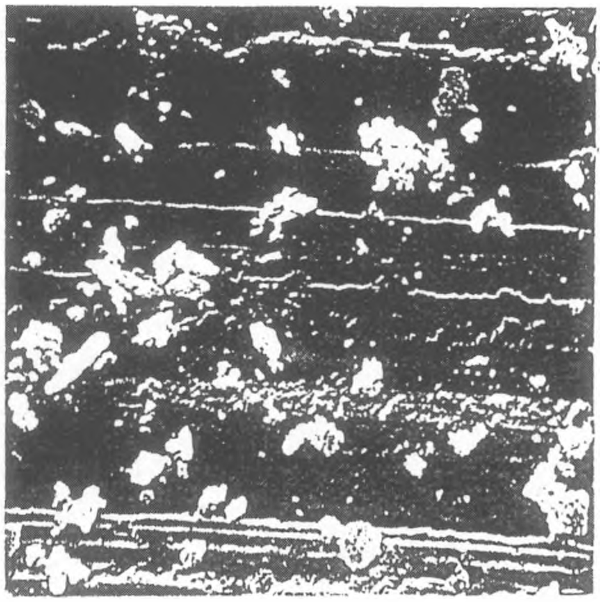

Fig. 4. Enamel aspect of fractured Cosmic experimental bond on untreated polished enamel which failed spontaneously during water immersion. SEM X 2000.

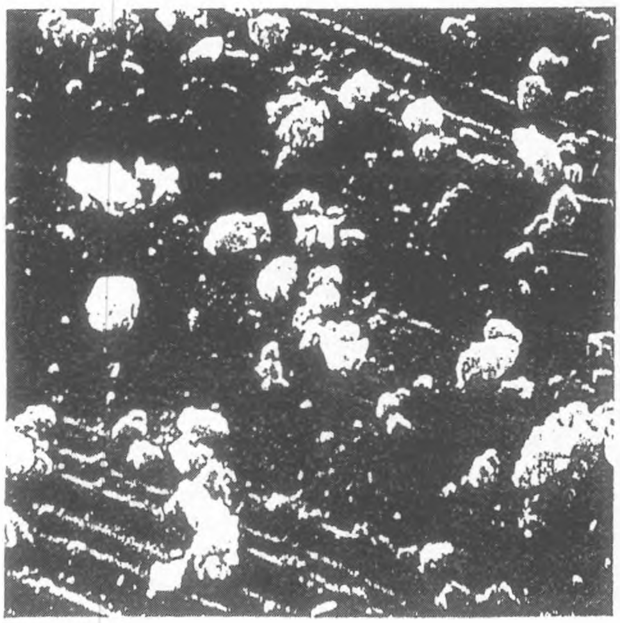

Fig. 5. Adhesive aspect of fractured bond depicted in Fig. 4. SEM X 2000.

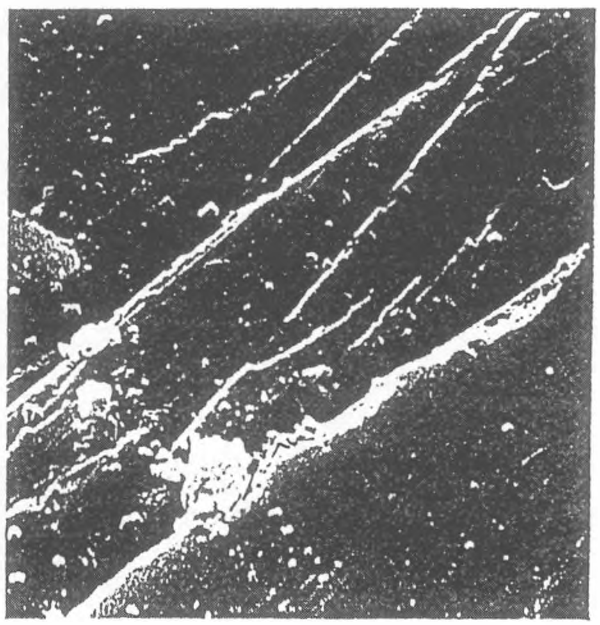

Fig. 6. Enamel aspect of fractured Cosmic experimental bond on polished enamel surface pretreated with Cosmic Bond. Bond strength 1,46 N mm^{-2}. SEM X 2000.

corded low tensile bond strengths had similar appearances to those shown in Figs. 4 and 5 respectively.

A dentine surface exposed by polishing with a 600 grit silicon carbide abrasive disc revealed a grooved surface with some longitudinally cut dentinal tubules exposed (Fig. 8). The dentine aspect of an experimental bond prepared with Cosmic on untreated polished dentine and which failed spontaneously, showed an abraded surface with debris particles (Fig. 9). The Cosmic interface of the same bond revealed a similar profile with, in addition, small holes on the surface probably caused by air bubbles entrapped within the restorative material during mixing (Fig. 10). The experimental bond prepared on a polished dentine surface pretreated with Cosmic Bond which recorded the highest tensile adhesive bond strength (8,7 N mm^{-2}) was again selected for viewing in the SEM. The dentine aspect of the fractured bond (Fig. 11) was similar to that observed on a polished untreated dentine surface (Fig. 9). The adhesive aspect of the same fractured bond, however, had a relatively featureless appearance (Fig. 12). The dentine aspects of fractured experimental bonds which recorded lower adhesive bond strengths were all similar to those depicted in Figs. 9 and 11. The Cosmic aspect of all these bonds had a veiled, featureless appearance, similar to that shown in Fig. 12.

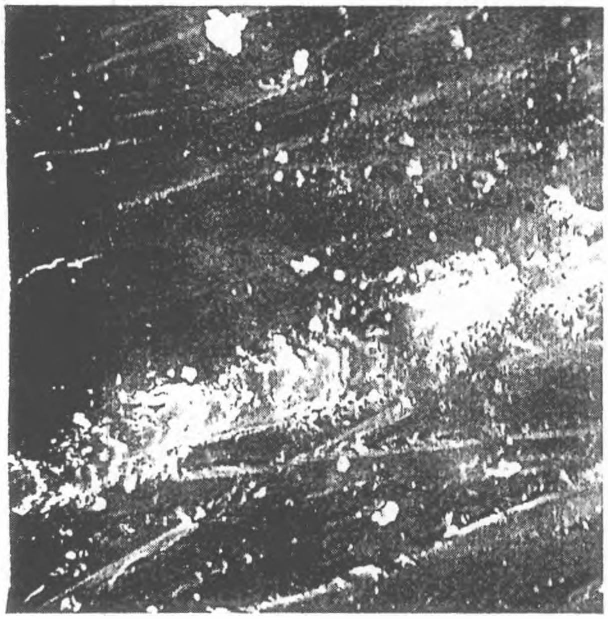

Fig. 7. Adhesive aspect of fractured bond shown in Fig. 6. SEM X 2000.

Fig. 8. Dentine surface polished with 600 grit SiC. SEM X 2000.

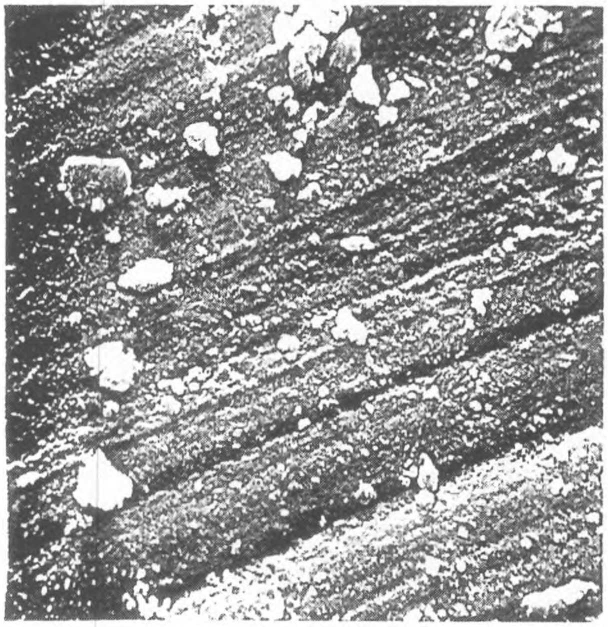

Fig. 9. Dentine aspect of fractured Cosmic experimental bond on untreated polished dentine which failed spontaneously during water immersion. SEM X 2000.

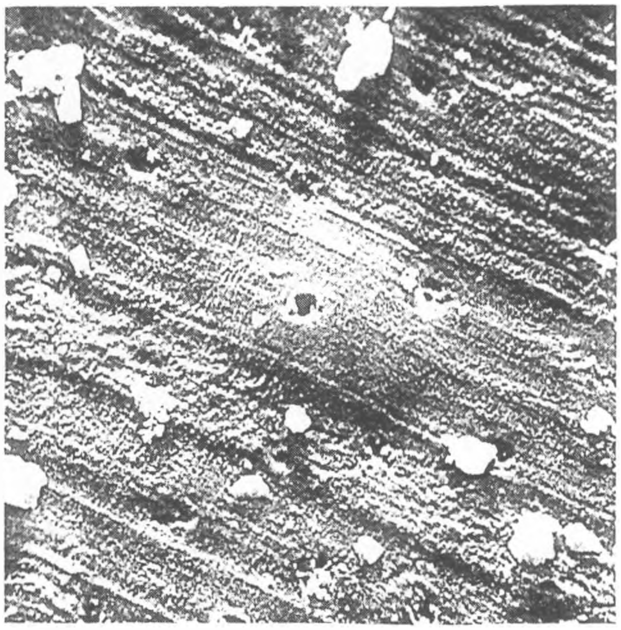

Fig. 10. Adhesive aspect of fractured bond shown in Fig. 9. SEM X 2000.

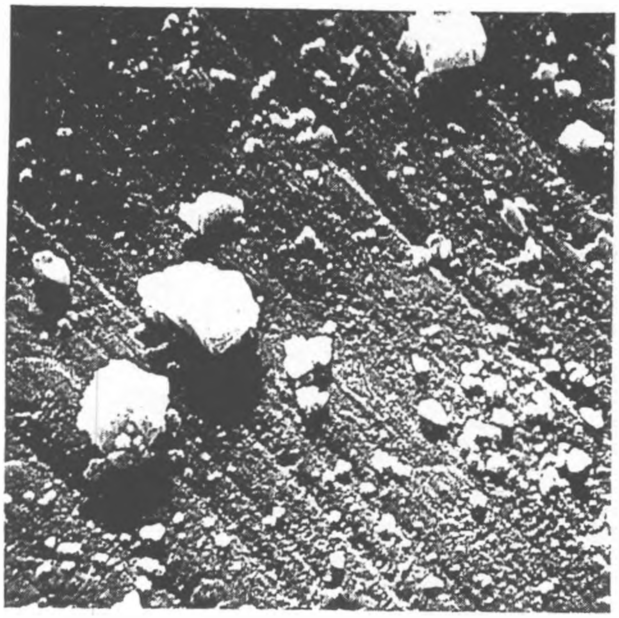

Fig. 11. Dentine aspect of fractured Cosmic experimental bond on polished dentine surface pretreated with Cosmic Bond. Bond strength 8,7 N mm^{-2}. SEM X 2000.

Fig. 12. Adhesive aspect of fractured bond depicted in Fig. 11. SEM X 2000.

TABLE 1

Mean tensile adhesive bond strengths of Cosmic to polished enamel and dentine surfaces pretreated with Cosmic Bond

Adherend surface	Number of specimens*	Mean tensile adhesive bond strength ± S.D. N mm^{-2}	Coefficient of variation %
Polished enamel pretreated with Cosmic Bond	6	0,8 ± 0,4	50,0
Polished dentine pretreated with Cosmic Bond	14	3,4 ± 1,9	56,4

*Fifteen test specimens prepared on each adherend surface. Only the number which did not fail spontaneously recorded.

DISCUSSION

In this investigation, I was not able to confirm the claim of the manufacturers that Cosmic adhered to untreated enamel and dentine surfaces, nor the observations of Bailey *et al* (1973) that relatively high bond strengths were obtained on enamel whether the primer was used or not. All the 30 experimental bonds prepared with Cosmic on untreated polished enamel and dentine surfaces failed during water immersion at 37°C overnight.

A mean tensile adhesive bond strength of 0,8 ± 0,4 N mm^{-2} was obtained for the 6 experimental Cosmic bonds prepared on primed enamel surfaces that did not fail prior to testing. This was considerably lower than the 24 kg cm^{-2} (2,35 N mm^{-2}) reported by the manufacturers. The mean tensile adhesive bond strength of the 14 Cosmic bonds prepared on primed dentine recorded in this study (3,4 ± 1,9 N mm^{-2}) was, however, substantially higher than the average value of 19 kg cm^{-2} (1,86 N mm^{-2}) reported by the manufacturers in their technical bulletin. Unfortunately they did not describe under what experimental conditions their test bonds were prepared and stored prior to testing. The bond strength of dental restorative materials is usually higher to enamel than dentine (Bowen, 1965a). In this study, however, higher bond strengths were recorded on primed dentine than on primed enamel.

A major problem encountered during the synthesis of the NPG-GMA adduct and its subsequent solution in organic solvents, was the prevention of polymerization of the coupling agent (Bowen, 1974). This may be overcome by the incorporation of stabilizers in the NPG-GMA solution. Polymerization of the NPG-GMA adduct will adversely affect the reactivity of the surface-active comonomer.

The manufacturers claim that the Cosmic restorative system has been specifically designed to overcome marginal leakage and surface roughness. Although the high glass filler content has reduced the co-efficient of thermal expansion to a value near to that of natural tooth substance, polymerization shrinkage is still significant. Whether the integrity of the marginal seal will be maintained and marginal leakage eliminated in the oral environment with adhesive bond strengths of Cosmic to primed enamel and dentine surfaces as obtained in this study, will require further investigation.

Interpretation of the surface appearances of the enamel, dentine and Cosmic aspects of fractured experimental bonds obtained with the SEM, proved to be difficult. The enamel (Fig. 4) and dentine (Fig. 9) aspects of the fractured bonds on untreated polished tooth surface were very similar to polished enamel (Fig. 3) and polished dentine (Fig. 8) surfaces respectively. The restorative aspects of these fractured bonds (Figs. 5 and 10) were mirror images of the respective substrate surfaces. These observations indicated that no interfacial interaction occurred between Cosmic and untreated polished enamel and dentine surfaces and hence would explain the zero bond strengths obtained with these specimens.

The enamel aspect of the fractured Cosmic bond on a primed enamel surface (bond strength 1,46 N mm^{-2}) showed areas of fracture within the enamel (Fig. 6). The corresponding restorative aspect of the fractured bond revealed a band of fractured enamel on the Cosmic surface (Fig. 7). This indicated that some interaction had occurred between the coupling agent, enamel and the restorative material.

The dentine aspect of the fractured bond on a primed dentine surface that recorded a tensile adhesive bond strength of 8,7 N mm^{-2} (Fig. 11) was similar to the dentine aspect of a fractured bond prepared on untreated polished dentine that showed no adhesion (Fig. 9) and a dentine surface polished with 600 grit SiC paper (Fig. 8). The restorative aspect of the same fractured bond, however, had a relatively featureless appearance (Fig. 12). It appeared that the Cosmic Bond primer adhered to the Cosmic surface thus masking its surface. In view of the relatively high adhesive bond strength obtained in this specimen, one would have expected more evidence of a reaction at the primer-polished dentine interface (Fig. 11).

The term tensile adhesive bond strength has been used in this paper because it implies the testing of adhesion as opposed to mechanical bonding at the interface. It has been suggested that the surface-active comonomer has the ability to form a chemical bond with tooth structure.

ACKNOWLEDGEMENTS

The author wishes to thank P. Grant Smith for supplying the Cosmic restorative materials used in this study. I also wish to thank the staff of the Electron Microscope Unit of the University of the Witwatersrand, Miss D. Bagley and Miss E. Vieira of the Dental Research Unit for their assistance with the photography and Miss B. Slack for typing the manuscript.

REFERENCES

Bailey, A.R., Shovelton, D.S. & Wilson, H.J. (1973) A new composite restorative material. An evaluation. *British Dental Journal*, **135**, 311-317.

Beebe, R.A. (1961) Gas adsorption studies on bone mineral. In *Adhesive Restorative Dental Materials*, ed. Phillips, R.W. Ch. III. pp. 75-83. Indiana: Owen Litho Service.

Bergman, G. (1963) Techniques for microscopic study of enamel fluid *in vitro*. *Odontologisk Revy*, **14**, 1-7.

Bowen, R.L. (1965a) Adhesive bonding of various materials to hard tooth tissues. II. Bonding to dentin promoted by a surface-active comonomer. *Journal of Dental Research*, **44**, 895-902.

Bowen, R.L. (1965b) Adhesive bonding of various materials to hard tooth tissues. III. Bonding to dentin improved by pretreatment and the use of surface-active comonomer. *Journal of Dental Research*, **44**, 903-905.

Bowen, R.L. (1965c) Adhesive bonding of various materials to hard tooth tissues. IV. Bonding to dentin, enamel and fluorapatite improved by the use of a surface-active comonomer. *Journal of Dental Research*, **44**, 906-911.

Bowen, R.L. (1965d) Adhesive bonding of various materials to hard tooth tissues. V. The effect of a surface-active comonomer on adhesion to diverse substrates. *Journal of Dental Research*, **44**, 1369-1373.

Bowen, R.L. (1965e) Development of an adhesive restorative material. In *Adhesive Restorative Dental Materials* — **II**, ed. Austin, R.H. *et al.* Ch. IV. pp. 225-231. Washington: Public Health Service Publication No. 1494, 1966.

Bowen, R.L. (1974) Personal communication.

Chandler, H.H. *et al.* (1974) Clinical evaluation of a tooth-restoration coupling agent. *Journal of the American Dental Association*, **88**, 114-118.

Gwinnett, A.J. & Matsui, A. (1967) A study of enamel adhesives. The physical relationship between enamel and adhesive. *Archives of Oral Biology*, **12**, 1615-1620.

Hanke, G. (1968) *Screening Test Methods and their Application in Measuring Adhesion of Selected Dental Resins*. M.S. Thesis, Indiana University School of Dentistry.

Linden, L. (1968) Microscopic observations of fluid flow through enamel *in vitro*. *Odontologisk Revy*, **19**, 349-365.

Phillips, R.W., Swartz, M.L. & Rhodes, B. (1970) An evaluation of a carboxylate adhesive cement. *Journal of the American Dental Association*, **81**, 1353-1359.

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

Rose *et al.* (1955) The screening of materials for adhesion to human tooth structure. *Journal of Dental Research*, **34**, 577-588.