DESIGN AND EVALUATION OF ALUMINA/FELDSPAR RESIN

INFILTRATED DENTAL COMPOSITE MATERIALS

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ABSTRACT

Introduction: Incorporating a feldspar chemical bond between alumina filler particles is expected to increase the wear resistant and flexural strength properties, while reducing flexibility of dental composites.

Aims and Objectives: An investigation was carried out to evaluate the influence of the feldspar chemical bonding between alumina filler particles on wear, flexural strength and flexibility of experimental alumina/feldspar dental composites. It was hypothesized that wear resistance and flexural strength would be significantly increased with increased feldspar mass, while flexibility was expected to decrease.

Methods: Alumina was chemically sintered and bonded with 30%, 40%, 50% and 60% feldspar mass, silanized and infiltrated with UDMA resin to prepare the dental restorative composite material specimens.

Results and conclusions: Significantly higher wear resistant characteristics resulted with increased feldspar mass ($p<0.5$). Improvements in flexural strength characteristics as the feldspar mass was increased was not statistically different ($p>0.5$). Flexibility characteristics as the feldspar mass was increased was not statistically different ($p>0.5$). The alumina/feldspar specimens showed lower flexibility (mm displacement) than SR ADORO® ($p<0.05$). Feldspar chemical bonding between the alumina particles may improve on the wear resistance and Flexibility of alumina/feldspar composites when compared to SR ADORO®. This study evaluated the influence of a chemical feldspar bond between alumina filler particles.

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CHAPTER ONE

INTRODUCTION

When one adds up the benefits of small contributions to the body of knowledge; all contributing efforts should be encouraged

1.1 INTRODUCTION

Global recognition of the poor aesthetic and potential toxic effects of amalgam has resulted in increased replacement with dental composites. Amalgam has increasingly been replaced with composites even in the posterior area where amalgam exhibits more desirable wear characteristics than composite restorations (Kahler *et al.*, 2006; Marghalani and Al-jabab, 2004). This shift is significant not only to the dentist patient relationship, but also to the potential impact in global dental education. Not only are composite restorations competing with the use of amalgam but there is the potential for composites to completely replace amalgams as restorative materials in dentistry. The Nijmegen Dental School in North America has already replaced teaching of amalgam restorative materials completely with composite aesthetic restoratives and more dental schools may follow this trend (Roeters *et al*., 2004). Initially used mainly as restoratives for small restorations the use of composites has increased to include indirect replacements for large cusp bearing restorations, crowns and bridges. With the increased use of dental composites in large stress bearing areas and for amalgam inlay replacements, limited data on some properties such as wear flexibility and flexural strength of dental composites contained in dental literature is cause for concern (Ferracane, 2006).

The purpose of this *in vitro* study is to design and evaluate alumina/feldspar resin infiltrated composite materials in order to increase wear resistance while maintaining adequate flexural strength and flexure resistance in dental

composite restorations. This will provide additional data about the potential success of dental composites with regard to wear, flexibility and flexural strength. The aim is further to produce a material that will increase wear resistance of dental composites by improving the bonding mechanism in order to improve confidence in using large posterior dental composite restoratives.

The success of dental composite materials depends largely on silane bonding ability to provide adhesion between the filler material and the matrix (Asmussen and Peutzfeldt, 2003). Without adequate adhesion being provided by silane the filler particles act as stress concentrators that weaken the material (Bae *et al*., 2001). Limited success and the need to improve on silane bonding are of concern because deterioration of the silane bond with time results in weakening of the composite material (Blomlof *et al*., 2001; Bolsen and Kern, 2003). Weakening of the composite material would influence the wear since it was also found that there is a positive correlation between wear resistance and impact strength (Bonilla *et al*., 2001). Since silane bonding agents pose certain problems such as slow degradation and discoloration, improved bonding agents should be investigated (Blomlof *et al*., 2001; Bolsen and Kern, 2003). Regardless of these problems few attempts have been made to improve the interfacial silane-glass filler bond (Choi and Ferracane, 1999).

Historically the initial dental composite materials did not provide the wear resistant properties sought for posterior stress bearing areas (Degrange and

Roulet, 1997; Dietschi *et al*.,1994 ; Ellakwa *et al*., 2002 ; Goracci *et al*., 1996; Gordon e*t al*., 2000). With time the surface of these materials deteriorated substantially due to wear, and fatigue (Appert, 1999; Choi and Ferracane, 1999; Göhring *et al*., 2002). Although wear resistance of dental composites have improved the surface deterioration combined with polymerization shrinkage still contributes to caries and pulpal injuries around the restoration due to micro-leakage (Fraunhofer *et al*., 1988; Hagenbush, 1997; Hardy, 2001). More recent controversy has arisen concerning the ability of dental composite materials to resist wear in cusp supporting, stress bearing large posterior restorations. Posterior restorations have been reported to provide acceptable wear resistance in posterior stress bearing areas for small restorations (Hashimoto *et al*., 2002). However insufficient evidence exists to support acceptable wear resistance of dental composite materials for large cusp bearing restorations since wear of small restorations is less significant (Hashimoto *et al*., 2002). Adhesive bonding with silane further restricts desirable wear characteristics of dental composites to a maximum particle size between 1.3-1.5μm and other bonding agents should be introduced into dental composites to determine whether improved adhesion to larger particles will provide the solution to this restriction (Bonilla *et al*., 2001; Jones, 1983; Jooste *et al*., 1997; Jagger *et al*., 2000).

Flexural strength and wear resistant characteristics are expected to increase with feldspar chemical bonding between larger filler particles (between 5 to 50μm, Knobloch *et al*., 1999). The feldspar chemical adhesion between alumina particles is expected to be superior to silane adhesion used in the

inhomogeneous micro-filled dental composite material SR ADORO® (Ivoclar, Lichtenstein). Based on the above information, this investigation was carried out to compare flexural strength, flexibility and wear of macro particle alumina/feldspar resin infiltrated composites with flexural strength, flexibility and wear of the SR ADORO® composite material.

The objective was to increase wear resistance while maintaining adequate flexural strength and flexure resistance in dental composite restorations by:

(i) Designing and constructing alumina/feldspar resin infiltrated composite materials that include a chemical bond (by sintering the alumina or incorporating feldspar) in order to improve on wear resistance of SR ADORO®.

(ii) Incorporating silane with sintered alumina resin infiltrated composite material and evaluating the wear of alumina resin infiltrated materials as compared to SR ADORO® commercial composite material

(iii) Incorporating 30% feldspar mass without adhesive bonding agents in alumina/feldspar resin infiltrated composite material and evaluating the wear of alumina/feldspar material with SR Link® as compared to SR ADORO® commercial composite material

(iv) Incorporating 30%-60% feldspar mass in alumina/feldspar resin infiltrated composites with silane and evaluating the wear of alumina/feldspar material as compared to SR ADORO® commercial composite material

(v) Incorporating 30% feldspar mass and SR Link® in alumina/feldspar resin infiltrated composite material and evaluating the wear of alumina/feldspar material with SR Link® as compared to SR ADORO® commercial composite material

(vi) Comparing the results of the 30% feldspar mass resin infiltrated composites, 30% feldspar mass with silane, and 30% feldspar mass with SR Link® in order to determine the influence of the adhesive bonding agents, on the wear of alumina/feldspar resin infiltrated composite material

(vii) Comparing the results of the feldspar mass variations with silane as compared to SR ADORO® commercial composite material

(viii) Testing alumina/feldspar resin infiltrated composite materials in order to evaluate their flexural strength results as compared to the SR ADORO® commercial composite material

(ix) Testing alumina/feldspar resin infiltrated composite materials in order to evaluate their flexibility results as compared to the SR ADORO® commercial composite material

(x) Comparing all the results of the composite materials evaluated in order to recommend which alumina feldspar material has the most desirable design, wear resistance, flexibility and flexural strength characteristics.

Success of this study will depend on:

(i) Matching the thermal expansion of the alumina and feldspar in order to prevent fracture during processing techniques (Yamamoto, M. 1985)

(ii) Providing adequate pore size in the fired alumina/feldspar material to facilitate resin infiltration

(iii) Increasing wear resistance with alumina/feldspar composites as compared to SR ADORO®

(iv) Obtaining similar or higher flexural strength values with alumina/feldspar composites as compared to SR ADORO®

(v) Obtaining similar or lower flexibility values with alumina/feldspar composites as compared to SR ADORO®

(vi) Obtaining an alumina/feldspar composite material with the most desirable design, wear resistance, flexibility and flexural strength characteristics, if all three property requirements are desirable when compared to SR ADORO®.

CHAPTER TWO

LITERATURE REVIEW

Every new revelation stems from something old.

2.1 INTRODUCTION

Evaluating wear[1] resistant properties of dental composite materials highlights factors that have influenced the success of dental composite materials in dentistry (Lionjua *et al*., 2003; Gordon *et al*., 2000; Rueggeberg, 2002; Yoshida *et al*., 2001; Ferracane, 2006; Dietschi *et al*., 1994). Controversy surrounding the acceptance of dental composites for posterior application has continued for over a decade (Ferracane, 2006). In the past, some manufacturers of dental composites promoted their products on the grounds of being the ideal wear resistant material for posterior restorations (Personal communication with Gary Hockley (2000), Ivodent, Cape Town and with Zarius Marks (1998), Nova Dental, Johannesburg). Literature on the other hand indicated that the ideal composite material to resist wear for large stress bearing posterior restorations still needed to be developed (Ferracane, 2006; Gordon *et al*., 2000; Oh *et al*., 2002; Suzuki and Leinfelder, 1994; Yoshida *et al*., 2001). Manufacturer advertising to promote their composite products as aesthetic restoratives (to replace amalgam) needs to be balanced by performance analysis.

In a search to solve the controversy surrounding posterior composite wear Ferracane (2006) indicated that although wear of small restorations were no longer seen to be of clinical concern, the wear of dental composites is still of clinical concern for large stress bearing restorations. Additional improvements are therefore required on performance and longevity of cusp-replacing resin composite restorations (Fennis, *et al*., 2005). Posterior dental composites result in higher

[1] Wear refers to the loss of material due to a sliding action of different materials against each other (Anusavice, 2003). For the purpose of this study three body wear was used where loose abrasive particles slide between two surfaces.

flexure and fracture than anterior composites and as a result wear, flexibility[2] and flexural strength[3] performance needs to be assessed.

The success of dental composite materials depends largely on silane adhesive bonding that prevents weakening of the composite material by coupling the filler particles to the resin (Ho and Marcolongo, 2005). Silane bonding agents unfortunately pose certain problems such as slow degradation which influences the strength and wear performance characteristics of the composite material with time (Yoshida *et al*., 2002). Regardless of the problem of slow degradation of silane, few attempts have been made to improve the interfacial silane-glass bond (Yoshida *et al*., 2002). As a result the need to improve the wear, flexure resistance and flexural strength of dental composites by introducing a strong chemical bond between alumina[4] particles with the addition of feldspar should be considered.

[2] For the purpose of this study flexibility is the amount of deformation that the material undergoes (mm) at the proportional limit (Anusavice, 2003).
[3] Is the force per unit area at the point of fracture of a test specimen subject to flexural loading (Anusavice, 2003). For the purpose of this study flexural strength is measured at the proportional limit of the specimens during utilization of a three point bending test whereby flexural stress is induced in the porcelain.
[4] Porcelain is classified as aluminous once it is composed of a glass matrix phase with 35% or more aluminum oxide, by volume (Van Blarcom, 1999).

2.2 DENTAL COMPOSITES

2.2.1 Introduction

For the purpose of this study, a composite refers to a dental material formed from two different compounds that are not soluble when combined and therefore need special coupling consideration. Composites consist of filler particles with more desirable physical properties that are incorporated into, though not soluble with the resin. These filler particles significantly improve the matrix material if they are bonded well to the resin matrix (Anusavice, 2003; Blomlof *et al*., 2001; Craig *et al*., 2000; Ellakwa *et al*., 2002; Hashimoto *et al*., 2002; Rosa and Perdigao, 2000; Sadan *et al*., 2003; Worm and Meiers, 1996).

Dental composites are generally accepted as consisting of a compound of two or more distinctly different materials. This definition has resulted in some confusion since ceramic composite and resin composite materials have been classified together and yet are considered as two comparatively different types of materials. Ceramic composites are generally referred to as ceramic or porcelain while they are occasionally referred to as composites (Fleming *et al*., 2006). The term composites interchanged with resin composite in reference to resin composite restoratives is on the other hand generally accepted because of the difficulties in coupling two distinctly different materials that are insoluble in each other together (Anusavice, 2003).

2.2.2 History, description and use of dental composites

Dental resins were the precursors to the development of dental composite restorative materials (composites). Globally, methacrylate-based chemistry still forms the basis of the most current restorative dental resin materials (Hussain *et al*., 2005). Since the late 1800's when natural resins such as horns, hoofs, gutta-percha, vulcanite, celluloid and insect exudates such as shell lacca (Shellack) were available, nothing impacted as largely on the market up to 1931 as when the first commercial polymethyl methacrylate (PMMA) resin (Plexiglass®) was introduced (Rueggeberg, 2002). Although not used in dentistry Plexiglass® was a precursor to Vernonite® (1936) a heat cured polymethyl methacrylate that could be used for inlays, crowns and fixed partial dentures.

By 1946 PMMA was used in approximately 95% of the denture market (Rueggeberg, 2002). PMMA was adapted for further use with the development of dental composites. The inclusion of urethanes, as a series of monomers that adds toughness and flexibility to the urethane backbone chain, formed a good basis for epoxy materials to be made. Replacing the epoxy group with methacrylate groups (in the 1960's) resulted in the first successful monomer system for composite materials, bis-GMA (Rueggeberg, 2002).

Most research and development of dental composite materials have only been done since 1965 with short chain polymers like urethane-dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) or decandiol-di-methacrylate added to the matrix in order to improve cross linking chains and physical properties of bis-phenol-glycidal-methacrylate (Rueggeberg, 2002). Shinohara *et al*., (2001); Suzuki

and Leinfelder, (1994) found that the amount of base monomer greatly influenced double bond conversion, unreacted monomer fraction, and crosslinking. Increasing the base monomer concentration decreased the double bond conversion, increased the leachable fraction, and decreased the crosslinking and network formation. Replacing bis-GMA with UDMA has many advantages. Bis-GMA polymers possess higher leachable amounts of unreacted monomer, while UDMA mixtures result in more crosslinking than the bis-GMA mixtures (Floyd and Dickens, 2006).

The success of dental composite materials depends largely on silane bonding that allows for good adhesion between the filler material and the resin matrix. Without adequate adhesion being provided by silane bonding the filler particles act as stress concentrators that weaken the mechanical properties of the material (Anusavice, 2003).

The need to improve on silane bonding has however been observed because the silane bond deteriorates with time and water sorption resulting in a weakening of the composite material (Zandinejad *et al*., 2006; Shajii and Santerre, 1999). Bagheri *et al*., (2007) states that, "*degradation of dental composites is caused by leaching of residual monomers, organic substances, filler particles and ions*". Deterioration that often occurs gradually by a process of erosion, abrasion or fatigue is the final result of chemical degradation (Koichi *et al*., 2004). The depth of degradation is dependent upon a complex function of the physical and chemical characteristics of the resin, the filler, and the silane coupling (Bagheri *et al*., 2007).

Dental composites absorb water after a period of use (Lassila *et al*., 2002). The water content further decreases resin strength and silane bond strength. Palin *et al*., (2005) discovered that a significant decrease in bi-axial flexure strength and associated increase in filler particle exfoliation was identified for composite materials that permitted higher water sorption. Weakening of the composite material would influence the wear of dental composites, especially if particle exfoliation is experienced, since it was found that there is a positive correlation between wear resistance and impact strength (Shi *et al*., 2004).

Filler particles were incorporated in the resin to improve wear resistance and impact strength. Initially filler particles were produced from grinding quartz or glass, to produce traditional composites with a range of particles from 0.1 to 100 μm. Small particle hybrid composites (Figure 1), with the majority of filler particles between 0.5 and 3.0 μm were introduced thereafter, in order to provide improved surface finish (reduced roughness). Filler particles less than 0.1 μm were used to fill in spaces between larger particles and provide improved surface and wear resistant characteristics (Anusavice, 2003; Venhoven *et al*., 1996).

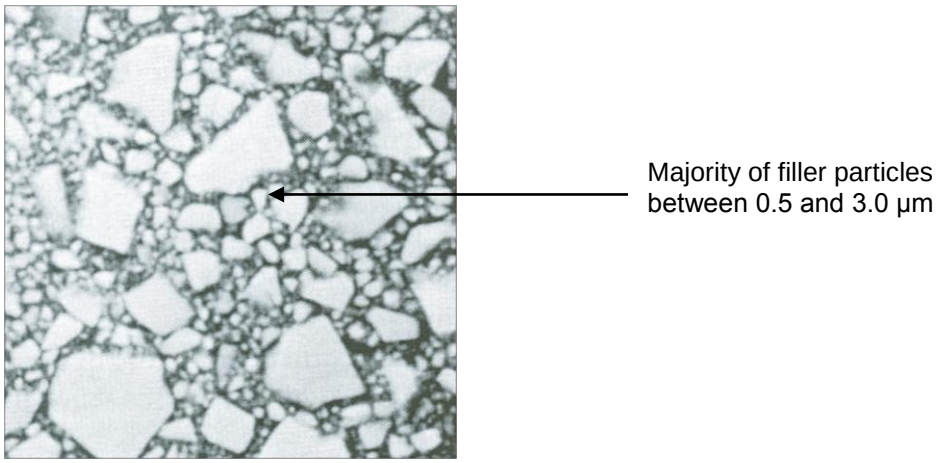

Figure 1 Small particle hybrid composite filler (*Adapted from Anusavice 2003*)

There appeared to be a critical maximum value of filler particle size between 1.3-1.5 μm that reduced wear of these early small particle hybrid dental composites, because the food fibers were unable to penetrate the inter particle spaces (Venhoven *et al*., 1996). Establishing the need to reduce the filler particle size resulted in dental composites that use micro-filler particles in the nanometer range (Figure 2), in order to further improve wear resistance and surface finish (Shi *et al*., 2004). The nanometer range particles stem from micro filled composites that used colloidal silica precipitate particles of 0.04 μm (40 nm).

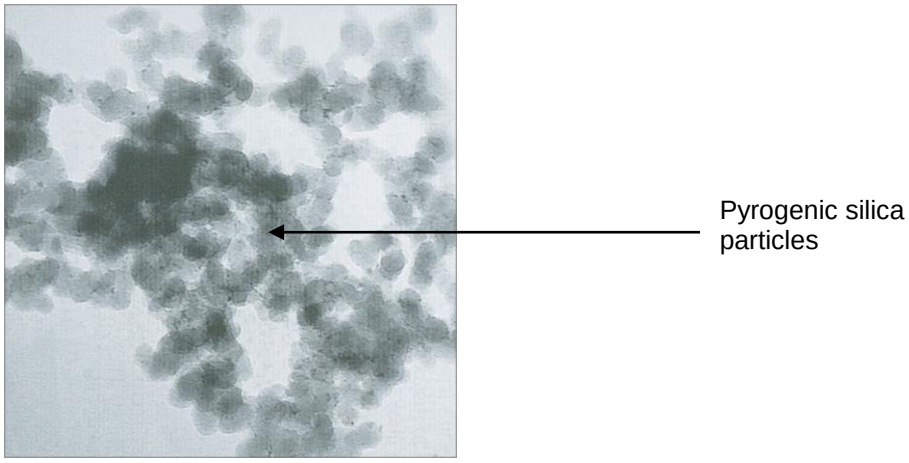

Figure 2 Agglomerate of 40 nanometer range pyrogenic silica particles

(*Adapted from Anusavice 2003*)

Surface finish is improved after wear with micro particles composites, however on fracture the filler particles pull out of the resin (Figure 4) resulting in a rough surface (Figure 3). The roughness of a composite surface after wear is of clinical importance rather than the roughness after fracture. Fracture resistance of micro filled composites, though slightly lower than macro and small particle hybrid composites, are considered to be acceptable while wear resistance is of concern

for large, posterior, stress bearing restorations (Anusavice, 2003; Whitters *et al*., 1999).

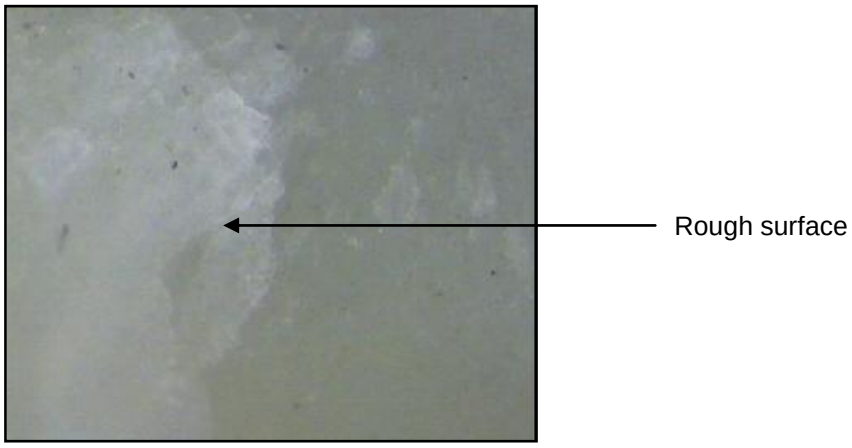

Figure 3 Rough surface as a result of fractured micro-filled dental composite (SR ADORO®)

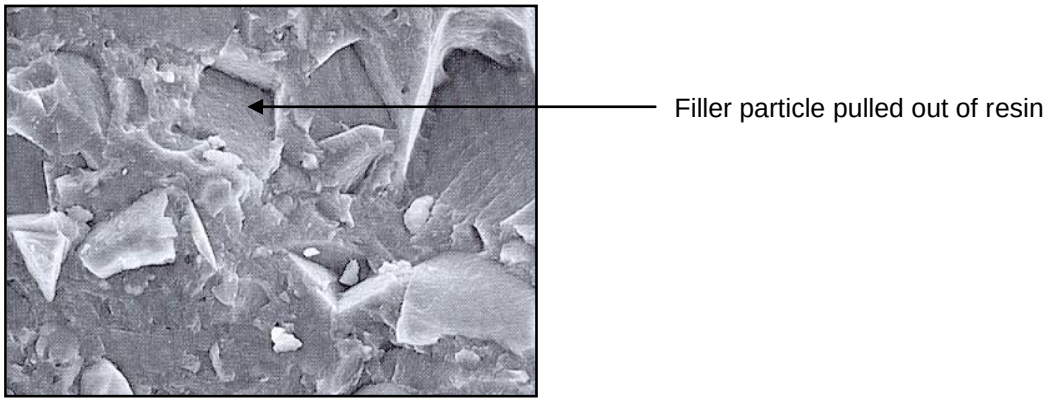

Figure 4 High magnified image of a fractured micro-filled composite surface with filler pulled out of the resin resulting in a rough surface (*Adapted from Anusavice 2003)*

Current generation composite resins are classified into hybrids and microfills, based on filler size differences. Hybrids contain a major fraction of the filler in the micron size range (with an average particle size of 0.4–3 μm) while the remaining filler is substantially below the submicron size range (microfill particles of

approximately 0.04–0.06 μm size range). Two additional sub-classifications are recognized within the wide filler particle size range of the main filler fraction of hybrids namely: small particle systems (also referred to as midifilled, with an average particle size >1 μm) and universal composite systems (also designated as minifills with an average particle size <1 μm) (Vaidyanathan *et al*., 2003). These trends and the availability of a large variety of dental composite materials have resulted in the global use of dental composites as routine fixed restorative materials for resins based cements, inlays, posts, crowns and bridges.

2.2.3 History and problems with adhesive bonding

Success in dental composites is due to the silane adhesive bonding agent[5]. There is sufficient evidence to show that fillers would weaken the composite material without silane adhesive bonding (Anusavice, 2003; Zandinejad *et al*., 2006; Shi *et al*., 2004; Shajii and Santerre, 1999). The particles would act like stress concentrators that initiate separation of the different types of material (Anusavice, 2003).

Resin materials will not normally bond to glass since the surface of glass readily adsorbs water. This reduces the wet ability of resin to glass resulting in a poor bond. Pioneer work in dental composite materials involved overcoming this weak link with silane. Silane contains a bipolar molecule (Figure 5) consisting of a methacrylate group at one end and a silanol group at the other (Degrange and Roulet, 1997). This permits the methacrylate group to bond to the resin while the

[5] The bonding agent refers to any material used to promote adhesion or cohesion between two different substances (Van Blarcom, 1999).

silanol group bonds to the glass, as a result of the condensation reaction of the silane as it dries (Degrange and Roulet, 1997).

More recently multifunctional urethane and thioether(meth) acrylate alkoxysilanes have been developed as dental restorative bonding materials for the synthesis of inorganic–organic copolymer ormocer composites, with filler particles in the nanometer range. The methacrylate groups are available for photochemically induced organic polymerization while the alkoxysilyl groups of the silane allow the formation of an inorganic Si—O—Si network (Figure 5) by hydrolysis and polycondensation reactions (Papadogiannis *et al*., 2007). Understanding of the silane bonding mechanism for bonding resins to non etchable filler materials (such as zirconia and alumina) is however limited and additional tests involving silane bonding with these materials, as well as efforts to improve these bonding mechanisms, are required (Matinlinna *et al*., 2006).

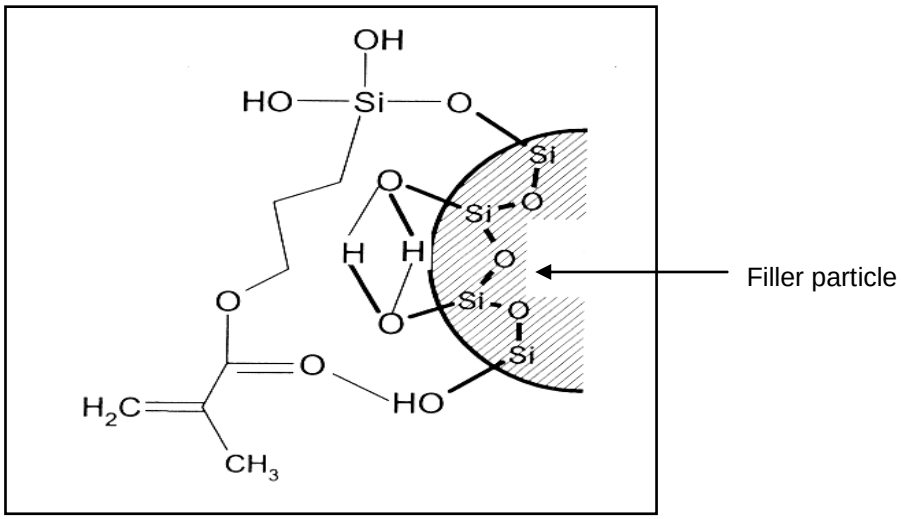

Figure 5 Chemical structure of (-methacryloxypropyltrimethoxy silane with Si—O—Si network (*Adapted from Anusavice, 2003*)

Adhesive bonding initiated from methyl metacrylate adhesion between restorative materials and tooth structure that relied on micromechanical adhesion from the presence of surface irregularities and undercuts, as well as weak chemical adhesion. Bonding two insoluble materials together is a complex process that has thwarted efforts to improve on the bonding mechanism of silane and leaves many unanswered questions. Methyl methacrylate will not normally bond to glass because the surface of glass readily adsorbs water. This reduces the wet ability (forces of attraction) of resin to glass (that repel each other like water on wax) resulting in a poor bond. (Degrange and Roulet, 1997).

Improved chemical adhesion resulted by replacing methyl methacrylate with silane or halophosphors bipolar molecules. Wet ability of the adhesive to the irregular surface of the substrate was extremely important. Any air or vapour has to escape before the advancing adhesive (Derange and Roulet, 1997). Surface irregularities not only improved mechanical adhesion, but chemical adhesion as well. The surface area and wet ability is improved when a rough tooth surface is provided. Acid etching to roughen the tooth surface decrease the surface energy for bonding and increase the wet ability by the adhesive agent (Derange and Roulet, 1997).

Applying the same principles formulated for silane adhesive bonding to tooth structure resulted in success of dental composite materials. Acid etching of the glass filler materials enhances the adhesive bond further, in a similar manner to acid etching of a tooth surface (Blomlof *et al*., 2001). The surface treatment for glass should include, priming the surface, rinsing in clear water, drying and applying the bonding agent. The benefits of phosphoric acid, originally used by

Buonocore, who first proposed the acid–etch technique in 1955, are still successfully applied (Rosa and Perdigao, 2000).

Hashimoto *et al*., (2002) further found that acetone is extremely effective to decontaminate the filler after phosphoric acid priming, just before the adhesive is applied (Hashimoto *et al*., 2002). If the filler particles are not free of contaminants the bond between the glass and the bonding agent will not be very effective (Yoshida *et al*., 2002). Agglomerations of micro fillers where many filler particles are directly bonded to each other are used to increase the filler content of composites. These agglomerates improve the physical properties of the material when bonded with silane.

Silane bonding agents pose certain problems such as slow degradation and discoloration. Improved bonding agents should be developed and tested to overcome the problem of slow degradation of silane. However attempts to improve the interfacial silane-glass bond have provided little overall benefit (Yoshida *et al*., 2002). Even though Yoshida *et al*., (2002) and Hashimoto *et al*., (2002) succeeded in improving the silane bond by applying an acetone primer to the glass prior to the silane treatment, this has not solved the problem of slow degradation of silane in oral fluids.

Weakening of silane bonding involved in composites comprised of large restorations and bridges, increases further with functional loading and bonding of material layers, to form the bulk of the restoration. Unfortunately literature does not report much on evaluation of adhesive bonding involving a composite bridge system. Li *et al*., (2004) reports, that clinical observations and experimental investigations have shown, that for direct composite resin-bonded bridge

structures, debonding at the interface between the abutment and pontic is the main mode of failure. An unexpected interfacial debonding can lead to entire failure of the bridge, even though the reinforced-fibre may prevent the pontic from dismounting completely.

Additional knowledge about the adhesion strength, initiation and progression of debonding still needs to be gained (Li *et al*., 2004). Without this knowledge, success in composites, due to improving the silane bonding mechanism will remain limited (Choi and Ferracane, 1999).

Silane bonding agents should be improved on since they deteriorate with time, but no other bonding agent has been introduced as a silane replacement for composites. Hagenbush, (1997) reports on the development of SR Link®, a bonding agent based on phosphoric acid ester with a methacrylate function that was produced in order to overcome the problems of silane stability when bonding composite materials to a metal alloy (Matsumura *et al*., 2001). The phosphoric acid ester group of the molecule is described as a strong acid that reacts with the substrate to form a phosphate. This compound forms passive layers on a metal surface, resulting in an inert layer due to the drying process and bonding of the methacrylate group with the resin.

SR Link® is highly water repellent which increases its bonding potential, since it contains a monomer with aliphatic hydrocarbon. Although SR Link® was formulated for resin to metal bonds it might be tested as a bonding agent to glass as well. It must be stressed that SR Link® was not developed for resin to glass adhesion. Silane was on the other hand not developed for metal to resin adhesion, but is used extensively for this purpose, as well as its original resin to glass

application. Although SR Link® would be used outside of its intended purpose, if used to replace silane in dental composite materials, it may with the restrictive use of silane be worth testing as a more stable composite coupling agent.

2.2.4 An overview of the problems associated with wear of resin restoratives

Anatomical form of natural teeth may be preserved with opposing resin restorative materials, however undesirable vertical dimension changes from high values of wear of resin restoratives result in potential permanent undesirable changes to the temporomandibular joint (Shillingburg, 1997). Longevity and functional efficiency of resin restorations are also reduced as a result of excessive wear of restorations. In order to prevent these problems, artificial restorations need to remain more efficient than natural teeth because the biting force derived from artificial teeth is greatly reduced (Hardy, 2001). If the cusp tips of artificial teeth wear down and become flat the biting force is reduced to the extent that the chewing efficiency is negligible, and only soft foods can be taken. Careful selection of restorative materials and the need to introduce improved materials with regard to wear resistant properties need therefore be considered (Oh *et al*., 2002).

Historically dental composite materials did not provide the wear resistant properties sought for posterior stress bearing areas (Gordon *et al*., 2000; Yoshida *et al*., 2001; Dietschi *et al*., 1994). Excessive force exerted on the dental composite material over time, produced fatigue (Figure 6) related to the amount of wear (Koichi *et al*., 2004; Callaghan *et al*., 2006). Whitters *et al*., (1999) found that, the

main improvements required for current resin composites are polymerization shrinkage and insufficient wear resistance under high masticatory forces.

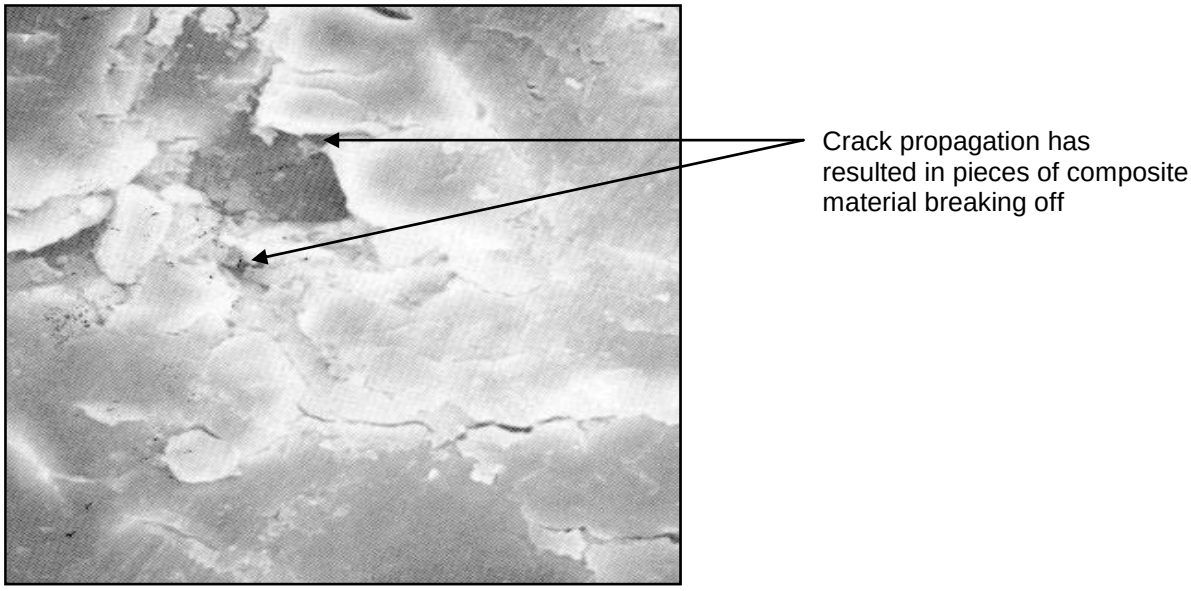

Figure 6 Wear facet from occlusal contact between micro-filled composite and enamel cusp (*Adapted from Anusavice 2003*)

Although initially applied extensively in the anterior region where the biting force is reduced, composites were used with limited success for posterior restorations (Anusavice, 2003; Yoshida *et al*., 2001). Even with new improvements and developments in dental composite materials continually taking place, it has been difficult to determine whether the ideal wear resistant characteristics for these materials have been produced for all composite applications (Gordon *et al*., 2000; Heintze *et al*., 2005; Luo *et al*., 2000; Rueggeberg, 2002; Thordrup *et al*., 2001; Yoshida *et al*., 2001; Zantner *et al*., 2004; Ferracane, 2006; Callaghan et al., 2006; Bolsen and Kern, 2003).

More recently dental composite materials have been reported to provide acceptable wear resistance in posterior stress bearing areas for small restorations (Ferracane, 2006). Insufficient evidence to support acceptable wear resistance from the same dental composite materials for larger restorations, suggests that wear for small restorations is less significant, since it was found that the material need not resist wear in functional cusp areas (Ferracane, 2006).

As far back as 1999 it was reported that the longevity and quality of Class I and Class II resin composite restorations was rated ‘good’ while wear characteristics of composites were undoubtedly considered undesirable (Whitters *et al*., 1999). The lack of clinical data on performance and longevity of cusp-replacing resin composites (Fennis *et al*., 2005) and the high variation in their wear test results (Heintze *et al*., 2005) are indicators that continued evaluation of posterior composite performance should be done, before they are pronounced suitable for all posterior applications (Fennis *et al*., 2005; Nagasiri and Somsak, 2005; Vallittu, 2004).

2.2.5 Factors that influence wear of dental composites

Attempts to improving the wear and mechanical properties of acrylic resins has resulted in composite restorative materials. In order to improve wear resistance filler particles with more desirable physical properties are introduced into the resin. These filler particles significantly improve the matrix material if they are bonded well to the resin matrix with silane. (Anusavice, 2003).

Initially filler particles were produced from grinding quartz or glasses, to produce a range of particles from 0.1 to 100 μm. These were called macro-particles. Filler particles less than 0.1 μm were called micro-fillers. Micro-fillers were used to fill in spaces between the macro-fillers since smaller particles that fill in the spaces between the larger particles provide a continuous distribution of particles, with the most compaction of filler (Anusavice, 2003).

More desirable wear resistant results were initially achieved with micro-filler composites than with macro-filler (Venhoven *et al*., 1996; Dietschi *et al*., 1994). In recent years nano-particles and hybrid nano-particles–micro-particles as fillers have been reported to further increase wear resistance (Condon and Ferracane, 2002; Shi *et al*., 2004; Beyth et al., 2006).

Wear of dental composites is an extensive field of research and the questions regarding how dental composite materials can be improved further to be more suitable for posterior stress bearing restorations, is still applicable (Anusavice, 2003; Ferracane, 2006). To find some answers to these questions the factors that control wear resistant properties must be considered. According to Anusavice, (2003) the wear resistant properties are controlled by the following composite properties:

(i) Porosity

(ii) Stability of adhesive bonding agent

(iii) Resin matrix-type and curing

(iv) Percentage filler content

(v) The size and type of filler particles.

It stands to reason that improving these properties where possible will result in increased wear resistance of dental composite materials; therefore each point will be discussed individually as presented by Anusavice, (2003).

(i) Porosity is caused by incorrect mixing and curing of the resin material, rather than the size and shape of the filler particles. Ensuring that porosity is reduced is therefore related to correct mixing and curing techniques. Porosity is greatest in a two paste system that must be mixed by hand. Light cured composites in a syringe also result in small areas of porosity. Porosity from mixing and curing cannot be eliminated completely in practice.

(ii) Stability of the adhesive bonding agent differs significantly from methacrylate or dimethacrylate solutions first used for adhesive bonding in dentistry to silane and silane agent derivatives now used. Increasing the bonding mechanism between filler particles and the matrix is an important consideration in improving the wear resistant properties of composites.

(iii) Wear resistance due to the resin depends on the strength of polymer links with cross linking providing improved wear resistance to the material. In addition the degree of monomer conversion differs between heat cured and light cured resin materials with heat cured materials showing a greater conversion factor. Light cured composite materials only produce a sufficient degree of conversion if the material is cured in thin layers. Using heat cured composite in conjunction with light curing is therefore more beneficial in maximising the degree of conversion.

(iv) The percentage filler content is directly related to wear resistance of composites. As the filler content is increased wear resistant properties are increased, on condition that they are bonded well in the matrix. There is a saturation point of filler content after which the filler decreases the wear resistance of composites.

(v) The size and type of filler particles affects wear resistance markedly. This is a major component of dental research presently. Presently micro particles in the nanometer range provide the greatest wear resistance. This may be because the macro particles are too large to be secured adequately in the composite material by silane adhesives.

Wear is a natural process that occurs whenever surfaces move in contact (Nagarajan *et al*., 2004). *In vivo* wear of composites occurs at both occlusal contact and contact free sites. The length of time during which the surfaces are in motion attributes to wear. Clinically, occlusal contact area wear is of the greatest concern with composites (Yap *et al*., 2002).

Fatigue is the general cause of material failure (Al-Turki *et al*., 2007) from wear of restoratives. Fatigue wear occurs as a result of formation and propagation of subsurface microcracks (Figure 6) when two surfaces move under dynamic load (Mair, 1992). Failure from wear is explained by Yap *et al*., (2002), “When one surface slides over another, there is a zone of compression ahead of the motion. Plastic deformation[6] of the material causes a zone of tension behind the motion. Dissipation of this energy nucleates cracks which eventually spread laterally to the

[6] Deformation refers to the change of form or shape of an object (Van Blarcom, 1999).
Applied forces produce stresses that can cause plastic strain. Plastic deformation is reversible and will recover when the stress is eliminated (Anusavice 2003)

surface as a result of repeated cycles. Eventually a small area of the surface material becomes surrounded by a network of linked cracks and a fragment is subsequently displaced or lost. In filled materials, the subsurface cracks may propagate either through the filler particles or around the interface" (Yap *et al*., 2002).

Wear patterns and an understanding of the resistance of restorative materials to wear, have become more complex. Previous theories that wear resistance was directly proportional to hardness of dental materials have been discarded (Mandikos *et al*., 2001). The property of wear resistance is complex, with no direct relationship between hardness (Lynch *et al*., 2003) and wear resistance, except in limited materials of similar composition (Anusavice, 2003; Craig *et al*., (2000); Oh *et al*., 2002). Mandikos *et al*., (2001) and Callaghan *et al*., (2006) did establish that significant relationships were observed between depth of wear and hardness in a comparison of wear resistance and hardness of certain dental composites. Dental composite materials therefore fall within the unique and limited range of materials that provide a relationship between hardness (Sharkey *et al*., 2001) and wear resistance. The fatigue wear mechanisms for composites however have not been well provided in literature (Yap *et al*., 2002).

Rough surfaces, high loads, high sliding speeds and the type of opposing surface as well as three body wear with saliva and food particles as the third body have been reported to influence the wear resistant properties of the materials due to an increase in the coefficient of friction (Bonilla *et al*., 2001; Oh *et al*., 2002; Tanoue *et al*., 2000; Zantner *et al*., 2004; Ferracane, 2006; Turssi *et al*., 2005; Callaghan *et*

al., 2006). Acidic and or alkali chemical environments may also play a role in composite wear.

Acid or alkali fluctuations are detrimental to wear resistance of enamel and dental ceramics, therefore the pH must remain as neutral as possible to reduce wear of these materials (Oh *et al*., 2002). Since literature does not supply desired information regarding the influence of pH on wear of dental composite materials, investigations to provide additional data for pH influence on composite wear should be undertaken.

Increased wear of dental composites that are softer than ceramics can be expected from extreme pH changes, but little supporting data is available. It was observed that acetic acid, propionic acid, or lactic acid with pH values found in saliva produced a softening influence (Asmussen, 1984). Van Groeningen *et al*., (1986) observed surface degradation of dental composites placed *in vivo* that were not subjected to stresses and report that these findings are especially relevant for attrition of composites under stress bearing conditions *in vivo*. Ortengren, (2000) found that pH affected the water sorption and solubility behaviour and therefore may be detrimental to longevity of dental composites.

Attin *et al*., (1998) showed that abrasion in the acidic environment was significantly higher compared with neutral conditions. It has been reported that abrasion due to tooth-brushing of dental composites is undesirable and varies in accordance with the material (Tanoue *et al*., 2000). These studies are not directly related to wear and resultantly confidence of wear performance of posterior dental composites is reduced, due to insufficient relevant data (Ferracane, 2006).

Degrange and Roulet, (1997) suggested that the maximum sized particles of traditional composites should be in a range of 3.0 to 5.0 μm, to ensure relatively low abrasive wear while allowing adequate filler loading and compaction of macro-particles to improve the mechanical properties. Microfilled composites that improved wear resistance exhibited poor adhesion between procured polymer blocks and the matrix resulting in reduced flexural strength, localised wear and marginal breakdown.

Attempts to solve the problem of strength reduction as filler particle size decreased resulted in hybrid composites, consisting of micro and macrofilled particles. Hybrid inter-locking of two completely different shaped micro and macro particles improved the wear resistance due to the materials ability to resist extrusion of filler particles, when combined with the correct adhesive bonding. Improved wear resistance was attributed to microfiller that provided closer compaction of micro and macro- filler particles (Derange and Roulet, 1997).

Silane bonding improved wear resistant properties of composites, since it prevented the filler particles from breaking away from the resin matrix. Nano-hybrid filled composites provide even better filler compaction and wear resistance as a result of silane bond efficiency in procuring the filler particles (Shi *et al*., 2004; Beyth *et al*., 2006; Condon and Ferracane, 2002). Besides the reliance on silane to bond the different materials (in a dental composite) together, wear resistance also relies on the type of load or opposing surface. The material must be able to resist the most abrasive material it may encounter which presently would be ceramic

materials (Won-suck *et al*., 2002). Composites materials must be able to resist wear and maintain a smooth surface after being subjected to cyclic wear loads.

The nanoparticles provide smoother composite surfaces after wear since pitting and rough surface structure after wear are reduced. Pitting results from depressions as the glass filler is etched away with time in the oral fluids. Rough surfaces are increased by depressions after wear since the softer resin wears away to a larger extent than the traditional size filler material (Anusavice, 2003). Nanoparticles wear away within the agglomerate of particles formed in the material leaving a smoother surface (Anusavice, 2003) (Figure 7a and 7b). Agglomerates of nanoparticle fillers (Figure 2) are more resistant to extrusion while providing wear resistance to the material than macro particles, which may extrude, thereby increasing surface roughness.

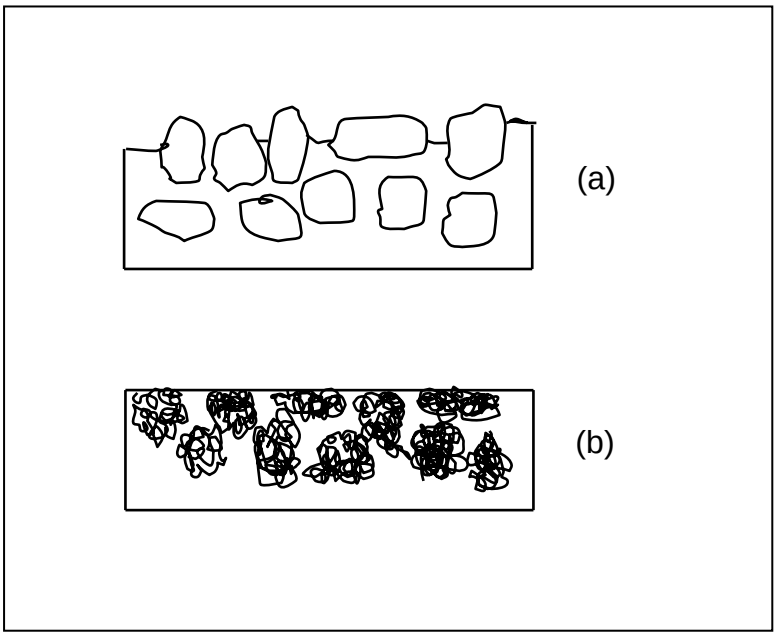

Figure 7 Schematic depicting surface wear of dental composites with macro particles 7(a), and agglomerates of nanoparticles 7(b)

The drive to obtain acceptance of dental composites from the American Dental Association (ADA) has been largely supported by manufacturers who have commissioned numerous wear and overall performance studies (Van Niewenhuysen *et al*., 2003). Relatively high acceptable wear rates made it easy for most dental composites to comply with the ADA requirements.

Recently dental composite materials have been reported to provide acceptable wear resistance in posterior stress bearing areas for small to moderate sized restorations (Ferracane, 2006). Long-term studies suggest that dental composite materials in two to three surface preparations are only considered a significant concern when the patient has abnormal occlusal habits, such as clenching and bruxing (Ferracane, 2006). These studies do not consider the importance of striving to meet optimum wear resistance of proven restorative materials such as gold and amalgam and suggest inferior acceptable limits are accepted (Van Niewenhuysen *et al*., 2003).

Insufficient studies exist to support acceptable wear of posterior dental composite materials for full crowns or other extensive restorations without actually assessing this parameter (Anusavice, 2003; Ferracane, 2006). The overall conclusion that dental composite materials for full crowns are acceptable can further not be accepted in light of the large variation of results from different studies (Ferracane, 2006). It was further discovered that surface pitting as a result of wear and oral conditions indicated that the surface finish of glass filled composites such as Artglass®, bellGlass® and Targis® for full crowns, are less than desirable. This has

resulted in certain materials such as Targis® being withdrawn from the market and replaced with SR ADORO® (Lendenmann, 2003) composite materials (Personal communication with Gary Hockley, Ivodent 2004).

Failure of large dental composite restorations as a result of wear or fracture still occurs (Pagliarini *et al*., 2000) and justifies the need for continued wear testing of dental composites (Ferracane, 2006). Ferracane, (2006) concludes that the wear resistance of dental composite restoratives is no longer considered to be a major concern for most restorations. However the limited information available suggests that it may still be a concern for large restorations in direct occlusal contact, or for those patients with bruxing and clenching behavior. This conclusion may be supported as long as due consideration is given to the problems with wear analysis of dental materials (that reduce life expectancy when compared to other fixed restorative materials).

Until sufficient information is available regarding large restorations in direct occlusal contact and for crowns and bridges, the potential wear problems should not be ignored. The assessment of wear in clinical studies (Palin *et al*., and Fleming *et al*., 2005), the prediction of wear for new materials based on *in vitro* test methods, and the improvement of methods for quantifying wear remain important concerns for dental researchers (Ferracane, 2006).

2.2.6 Problems related to in vitro wear testing of dental composites

Although a variety of *in vitro* wear tests have been used in order to speed up the assessment of dental composites wear, the need for their continued use has been

questioned (Ferracane, 2006). This is because literature contains conflicting reports on the ability to simulate the wear of composites since *in vivo* wear resistance (Dahl and Øilo, 1994) of resin based dental restorative materials is complex in nature (Mandikos *et al*., 2001).

In a comparison of the wear resistance of ten indirect dental restorative materials in five wear simulators, it was found that relative ranks of the materials varied tremendously between the test centers (Heintze *et al*., 2005). Until *in vitro* wear testing machines are universally accepted to provide consistent results, designing and evaluating new wear testing machines and their ability to simulate *in vivo* wear will be beneficial. The failure prevalence of large dental composite restorations due to wear or fracture, further justifies the need to continue with accurate, reproducible *in vitro* wear testing of dental composite materials (Ferracane, 2006).

2.2.7 Relevant wear studies of dental composites

To date, studies in wear resistance (Lee, 2006; Callaghan *et al*., 2006; Ármalo and Vale 2005; Ellakwa *et al*., 2002; Jagger *et al*., 2000; Suzuki and Leinfelder, 1994; Xu, 2003) have been directed mainly at comparing and evaluating wear resistant patterns of existing dental composite materials, improving wear resistance by changing the filler content, introducing changes to the resin matrix to improve physical properties, or improving the adhesive bond between filler and matrix.

A study using electron beam irradiation was shown to improve wear resistance of dental composites by reducing chain entanglement. However, the resultant color effects were found to be detrimental (Behr *et al*., 2005). Yap *et al*., (2002)

investigated the effects of cyclic loading on occlusal contact area wear and the possible presence of fatigue wear mechanisms in four composite resins (Silux, Z100, Ariston and Surefil) using a reciprocal compression-sliding test apparatus. They reported that some restorative materials exhibit fatigue wear while others exhibit deep microcrack formation with extended cyclic loading. Deep microcrack formation (Loughran, 2005) may precipitate failure despite the low wear observed. They concluded that care should be exercised when selecting materials for posterior stress-bearing areas.

Ferracane and Condon, (1999) found that signficant wear of composites was produced at the attrition margin. Enamel degradation at the margin was similar to composite degradation. The microfills, and to a lesser extent the minifills, showed more marginal breakdown than the midifill composites. Marginal breakdown showed an inverse correlation with fracture toughness. Ferracane and Condon, (1999) also reported that microfill composites and certain small particle hybrids (minifills) are more susceptible to marginal degradation than posterior composites with coarser filler particles, i.e. midifills. A current hypothesis is that inadequate fatigue resistance is responsible for the accelerated wear and marginal degradation of composites that contain a greater percentage of filler particles less than 1.0 μm (Yap *et al*., 2002). Yap *et al*., (2002) also reported that microfilled resins show excellent wear resistance, but experienced a higher incidence of fracture than composites with larger particle size.

Applequist and Meiers, (1996) suggested that glass/ceramic inserts be placed in composite restorations in order to improve the wear resistant properties of

posterior restorations, but as was confirmed by Sjögren *et al*., (2000) fractures or flaking of restorations as well as rough or pitted surfaces between the composite and insert, reduced the success of such restorations. More desirable wear results were achieved with micro filler composites than with macro filler (Zantner *et al*., 2004; Venhoven *et al*., 1996). Nanoparticles and hybrid nanoparticles–microparticles as fillers continue to set the standard for wear resistance of dental composites (Shi *et al*., 2004; Beyth *et al*., 2006; Condon and Ferracane 2002).

The shape of filler was also found to be of significant interest with regard to the properties of dental composite materials. Irregularly shaped particles provided better wear resistance than smooth round fillers (Turssi *et al*., 2005; Venhoven *et al*., 1996; Callaghan *et al*., 2006). Jagger *et al*., (2000) tried to replace chopped fiber reinforcement with polyethylene beads, only to discover that the beads were not as effective. The strengthening mechanism due to fiber reinforcement through the incorporation of whiskers or fibers appears to be more effective as a result of their desirable shape and orientation (Ellakwa *et al*., 2002; Bae, 2001; Bolsen and Kern, 2003; Xu, 2003; Li *et al*., 2004).

Fiber reinforcement depends on variables such as type of fibers, percentage of fibers in the matrix, modulus and distribution of the fibers, fiber length, orientation and form (Fennis *et al*., 2005). Callaghan *et al*., (2006) found that fiber length of 3 mm performed better than short 1.5 mm fibers under all load conditions and provided a comparable wear rate to a particle-filled dental composite. Increasing the length of the fibers increased the wear resistance of the specimen. The advantages of whiskers on their own as composite filler material have been shown

with regard to physical properties only and have not derived commercial benefit (Xu, 2003).

Hybrid composites that are formed by combining two types of filler materials, fall into a category of composites that are superior to those containing only one type of filler material. In light-polymerized composites, the agglomeration of different sized particles resulted in a smoother polished finish, and greater strength benefits were achieved by producing more compaction between filler particles (Anusavice, 2003; Papadogiannis *et al*., 2007). Ormocer composites (organic modified ceramics) combines inorganic constituents such as glass with organic (polymer) constituents to provide a packable (Tantbirojn *et al*., 2003) composite material with handling properties of a hybrid composite material (Papadogiannis *et al*., 2007). Experimental results performed by Shi *et al*., (2004) also indicated that the frictional coefficient and wear rate of epoxy can be reduced at rather low concentration of nano-alumina.

Extensive research efforts to improve composites have not solved problems with wear and marginal leakage. Marginal leakage has largely been attributed to the curing shrinkage of dental composite materials (Sakaguchi *et al*., 2004; Versluis *et al*., 2004; Dauvillier *et al*., 2003) and additional testing of the influence of wear on marginal leakage, of large posterior composite fillings should also be done (Ferracane and Condon, 1999). Marginal leakage can be influenced by the stiffness of the composite material and its influence on tooth structure (Tam and Pillair, 2000). The stiffness of dental composite materials should ideally match that of the tooth structure to which it is to be bonded in order to withstand compressive, flexural and shear stress (Katz *et al*., 2007). The adhesive bonding of composites

to dentine increases the strength and stiffness of the tooth and thereby reduces fracture susceptibility.

Stiffer materials such as ceramic cores are more desirable since they provide more protection to the underlying dentin, but are more susceptible to radial cracking as a result of a brittle material that supports more of the flexural load (Lawn *et al*., 2004). Resin composite restorations in turn alters the location of greatest stress, from the base of the cavity where fractures often initiate in teeth with amalgam restorations, to the interface between the resin-composite and the enamel at the cavity surface when a tooth is in function, thereby reducing the occurrence of tooth fracture (Kahler *et al*., 2006).

Increased composite stiffness in composites is desirable since this will resist flexure from functional loading better (Pfeiffer *et al*., 2003). Soft composites run a higher risk of marginal leakage that may occur from flexural and shearing stresses[7] as a result of wear (Figure 8), that causes deformation to the bonded composite around the margins (Bonilla *et al*., 2001; Choi and Ferracane, 1999). The problem with composite restorations is the occurrence of secondary caries as the `composite material breaks down around the margins (Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Worm and Meiers, 1996; Behr *et al*., 2005).

[7] Shearing stress refers to the internally induced force that opposes the sliding of one plane on an adjacent plane or the force that resists a twisting action (Van Blarcom, 1999).

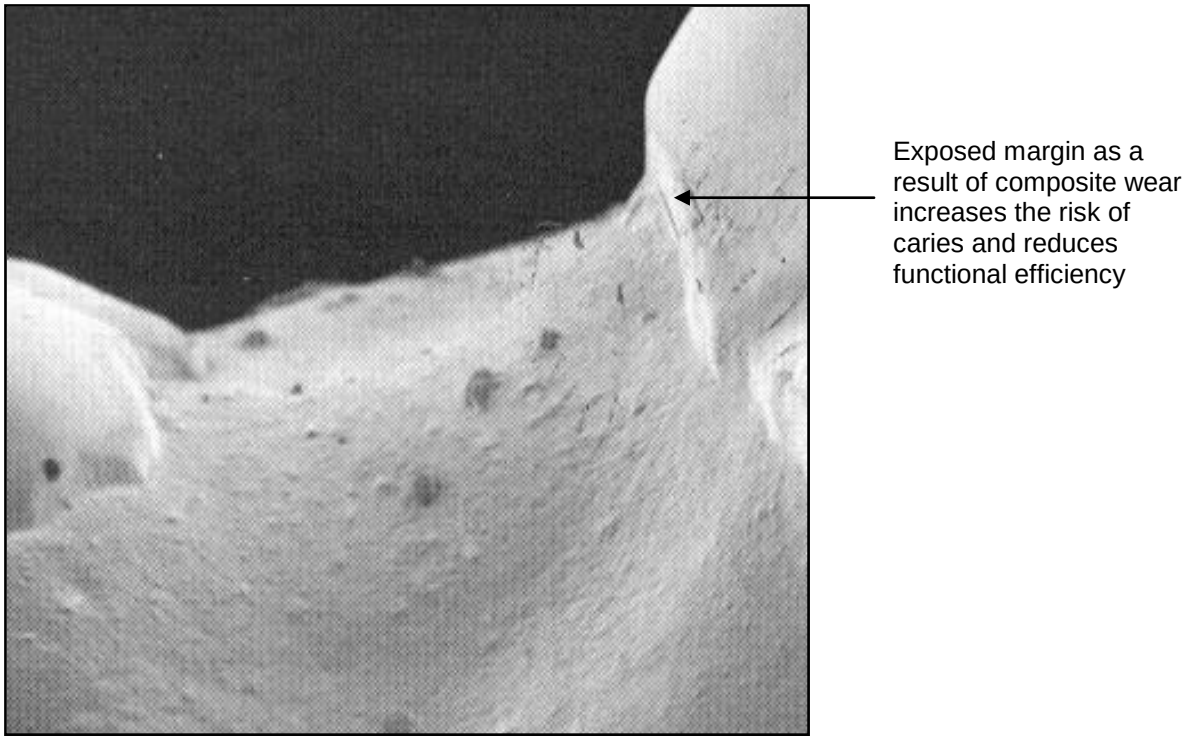

Figure 8 Wear of a small particle composite (*Adapted from Anusavice 2003*)

According to Kleverlaan and Feilzer, (2005) considerable efforts have not resulted in rendering any of the contemporary bonding systems able to maintain reliable bonding between the resin composite and the tooth structure, resulting in poor marginal adaptation and recurrent carries. Added to this, significant differences in gap-free margins result with different bonding systems (De Munck *et al*., 2005) (Frankenberger and Tay, 2005).

Restoration on fracture, are simply replaced to prevent further clinical problems. However, when a composite material breaks down around the margins, it is difficult to detect clinical problems such as secondary caries and the health of a tooth may be jeopardized as a result of delayed treatment. Factors that prevent flexure and

improve wear resistant properties should therefore be tested with regard to preventing clinical problems and preventing marginal leakage, marginal breakdown, and chipping of dental composite restorations (Splieth *et al*., 2003).

Fillers have been incorporated largely to increase wear resistance of composites but the influence of cross linked polymers (Leifelder, Undated Brochure) that also improve wear resistance and strength of resin materials should not be ignored. Duravite® artificial teeth which consist of a cross-linked polymethyl methacrylate resin, do not contain an inorganic filler material, yet exhibited similar wear resistance to Ivoclar Orthosit denture teeth even though Ivoclar Orthosit teeth incorporated 0.04 µm pyrogenic silica filler particles (Con Jooste *et al.,* 1997).

2.2.8 Importance of longevity of dental composites

Attempts at improving longevity of dental composites have concentrated on the marginal performance as expressed by De Santis *et al*., (2005) who found that the long-term prognosis of a restored tooth depends on the sealing efficacy of the restorative material. With concentrated efforts to improve on composite shrinkage, the influence of wear on longevity of dental composites has not featured well in literature. Few studies have been conducted to establish factors that influence the longevity of dental composites (Anusavice, 2003).

Although the performance of posterior composite restorations have improved during the last decade the mean age of dental composites are reported to be 7.5 years only (Burke *et al*., 2001; Thordrup *et al*., 2001). Studies report between 32.6% - 62.5% failure rate of dental composites ranging from small non stress related inlays to full posterior crowns (Anusavice, 2003; Bolsen and Kern, 2003).

The mean life expectancy of dental composites have been much lower than that of metal ceramic restorations (25% failure after 15 years) and amalgam restorations (10% failure after 10 years, Anusavice, 2003; Bolsen and Kern, 2003).

The success of dental composites have been jeopardized as a result of marginal or bulk fracture due to fatigue and are reduced to a lower mean lifespan especially in cusp and stress bearing posterior regions (Thordrup *et al*., 2001). Small to moderate restorations were reported to be more successful within a one to three year period (Luo *et al*., 2000; Türkün *et al*., 2003). Preventing marginal stresses in dental composites as a result of shrinkage, undesirable stiffness and wear are important considerations for preventing fatigue and increasing longevity. Polymerisation contraction and the mismatch in mechanical properties of dental composites with tooth structure result in stresses at the restoration-tooth interface (Kahler *et al*., 2006). Development of stresses at the restoration-tooth interface was shown to be detrimental to longevity of dental composite restorations. The stiffness of the composite material needs to more closely match the tooth structure otherwise stresses at the restoration tooth interface occurs (Ensaff *et al*., 2001).

According to van Dijken *et al*., (2006) reasons for composite failure were secondary caries, material deterioration and cusp fracture. The majority of the failures occurred after 3 years, however most studies are within the 3 year period. Secondary caries occurred between 4 and 6 years and are not solved by the incorporation of fibers that result in very rough surface characteristics. Rodolpho *et al*., (2006) found the clinical performance of posterior resin composite restorations evaluated was acceptable after 17-year evaluation. However, the failure of resin composite restorations in molars, Class II, and large restorations was higher and

therefore still problematic (Rodolpho *et al*., 2006). Insufficient information on the variety of dental composite materials, together with longevity variation values, indicate the need for greater scientific support, before dental composite restorations can be regarded as suitable for use in posterior stress bearing areas (Ferracane, 2006, Thordrup *et al*., 2001, Anusavice, 2003, Bolsen and Kern, 2003, Ensaff *et al*., 2001).

2.2.9 Flexural strength and flexibility testing of dental composites

Occlusal forces cause undesirable flexure of bridges (Shillingburg, 1997). The amount of displacement and force, that can be withstood are significant considerations when posterior forces are considered for bridges. Three or four point bending tests are therefore routinely employed to test flexural strength and rigidity of dental materials (Al-Turki *et al*., 2007; Walker *et al*., 2006; Tanimoto *et al.,* 2006). According to Walker *et al*., 2006 the flexural modulus (*E*) and flexural strength (*S*) can be obtained using the following equations:

1. $E=FL^3/4bh^3d.10^{-3}$ (measured in GPa)
2. $S=3PL/2bh^2$ (measured in MPa)

where *L* is the support span length (mm), *b* the specimen width (mm), *h* the specimen height (mm), *F* the load (N), *d* the deflection (mm) at load *F*, and *P* is the maximum load (N) resulting in failure.

Little information exists on flexibility as a measurement of dental composites since modulus of elasticity of dental composites is rather measured in order to establish

the stiffness of materials (Marghalani and Al-jabab, 2004; Chung *et al*., 2004; Asmussen and Peutzfeldt, 2002; Xu *et al*., 2003; Condon and Ferracane, 2002). To quote Dyer *et al*., 2005, "*Modulus of elasticity (E) is the ratio of stress to strain within the elastic range, according to Hooke's Law it is a measure of the stiffness, defined by the slope of the stress-strain (load-deformation) curve linear segment before plastic deformation*". Therefore, the higher the *E*, the stiffer or less flexible the material will be.

Testing for the modulus of elasticity is problematic for dental composites because the ISO specifications, to obtain the modulus of composite materials (ISO, 1993) require a length of 25 mm which requires the material curing to be overlapped because of restricted curing areas with specimen construction that is larger than tooth size (Palin *et al*., 2003; Palin *et al*., 2005; Hussain *et al*., 2005; Al-Turki *et al*., 2007; Walker *et al*., 2006; Tanimoto *et al.,* 2006). The performance of these long specimens may as a result not be the best comparison for dental composite restorative materials and the author of this literature review supports the notion that flexibility standards of smaller specimens be examined as a more practical comparative tool to test composite rigidity (Palin *et al*., 2003; Palin *et al*., 2005; Hussain *et al*., 2005; Al-Turki *et al*., 2007; Walker *et al*., 2006; Tanimoto *et al.,* 2006).

2.2.10 Flexural strength and flexibility influence on marginal leakage of dental composites

Flexural strength is one of the most important properties of restorative dental materials (Sherrer *et al*., 2003; Anusavice, 2003) because it is an important parameter in relation to the liability of fracture (Lucena-Martin *et al*., 2001) of restorations under occlusal load (Asmussen and Peutzfeldt, 2003). Chipping and bulk fractures are major causes for clinical failure of composite restorations. Failure is most likely preceded by a slow sub-critical propagation of internal flaws (Loughran *et al*., 2005).

Fatigue fractures of composites in clinical use were found to be a common reason for time related failure with frequent reports of damage to bulk, cusp, or marginal fractures of restorations (Lohbauer *et al*., 2003; Burke *et al*., 2001). Cyclic fatigue of dental composites may result in individual loading cycles well below the ultimate strength of the material. The cumulative effects of cyclic loading results in crack formation and propagation that leads to fracture. Bulk fatigue characteristics of composites, are most commonly evaluated by subjecting the material to three-or four-point flexural loading (Al-Turki *et al*., 2007).

The degradation of composites during clinical applications is a limiting factor to increased usage of dental composites. The stress induced during polymerization enhances the degradation effect and may also produce microcracks, which enhance fatigue failure. Aging time and oral conditions are among other factors that accelerate degradation of dental composites. Specimens that were stored dry were stronger than specimens that were stored wet even if they were dried before being tested. Significant decrease in flexural strength and fracture toughness was

also observed after aging a glass-reinforced composite in water for 6 months (Al-Turki *et al*., 2007).

The influence of loading (Marquis *et al*., 2000) is expected to be significant as well since occlusal function results in flexure as well as cyclic loading. Al-Turki *et al*., (2007) discovered significantly lower flexure strength for specimens that were flexure loaded as opposed to contact loaded specimens. For the flexure loaded specimens, the aging, load, and the media were all significant while the number of cycles had no significant effect. For the contact loaded specimens the media, aging, and cycles completed, had a significant effect, but no effect was observed for the different cycling loads. Al-Turki *et al*., (2007) concluded that the decrease in flexure strength from flexure loading was mainly affected by the aging media, whereas, the decrease from contact loading was attributed mainly to the number of cycles. Flexural strength and flexural modulus of dental composites are increased by incorporating ceramic fillers. Stronger and more porous fillers provide higher flexural strength (Zandinejad *et al*., 2006; Shi *et al*., 2004).

Lack of information regarding functional performance and design considerations of composite materials, for large restorations, is still of concern. Even with whiskers that significantly improve the properties of dental composites the use of composites in large stress-bearing applications such as direct posterior restorations involving cusps, indirect crown and multiple-unit restorations is not advocated without risk (Xu *et al*., 2003).

The relatively high brittleness and low fracture resistance of current composites still hinder their use for large stress-bearing restorations (Xu *et al*., 2003). Dyer *et al*., (2005) observed that, fiber-reinforced composite (FRC) dental applications as

replacement for metal-ceramic and all-ceramic crowns and bridges (Fleming, 2005) have not yet been proven to be successful with long-term clinical trials. One of the reasons for reduced confidence in the use of FRC material is a lack of information regarding the effect of design parameters on the mechanical performance of a composite prosthesis.

Design influence as a result of flexibility of composites and resultant marginal leakage due to the plasticity of resin should be studied more extensively (Mesquita *et al*., 2006). The stiffness of dental composite materials should be as high as possible in order to withstand compressive, flexural and shear stress (Anusavice, 2003). Marginal leakage that occurs from flexural and shearing stresses is increased as a result of wear that causes deformation to the bonded composite around the margins (Bonilla *et al*., 2001; Choi, and Ferracane, 1999).

High inorganic filler content has been shown to present lower shrinkage values and higher stiffness (Braga *et al*., 2005). Higher stiffness and flexural strength values prevent the composite material from breaking and flexing around the margins. The relationship between polymerization shrinkage stress and marginal integrity (Figure 9) has been demonstrated, *in vitro* (in class V restorations, Braga *et al*., 2005).

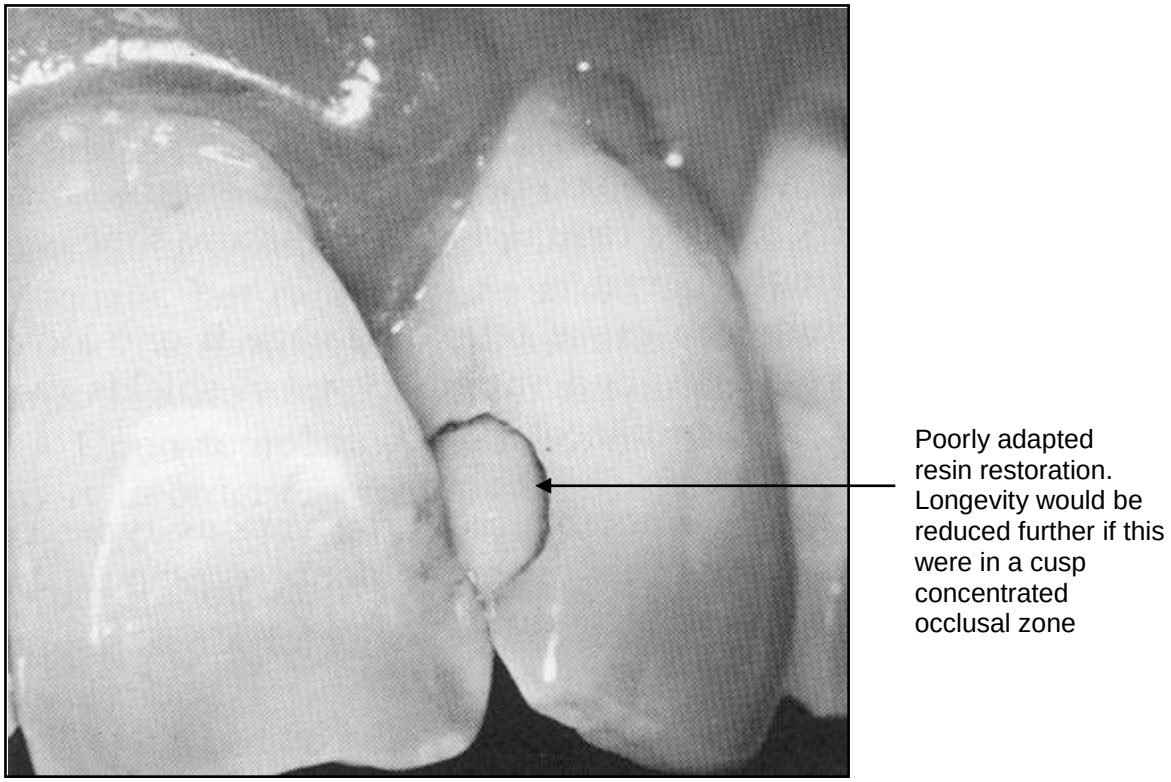

Figure 9 Clinical resin restorative with marginal leakage from poor marginal adaptation (*Adapted from Anusavice 2003*)

Correlation of marginal leakage due to the plasticity of resin that increases flexibility of composites around the margins has been limited when compared to composite shrinkage stress analysis (Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Wahab and Shaini, 2003; Worm and Meiers, 1996). Factors that prevent flexure and increase flexural strength should be applied more extensively in order to help prevent marginal leakage. Kleverlaan and Feilzer, (2005) observed that polymerization shrinkage, contraction stress, elastic modulus, and flow are important factors in determining the final properties of the resin composite. Only polymerization shrinkage in relation to elastic modulus and polymerization contraction stress in

relation to elastic modulus, and filler load and type of resin composite had been evaluated. Further evaluation by Kleverlaan and Feilzer (2005) resulted in linear correlation observed for:

(i) contraction stress and shrinkage

(ii) contraction stress and tensile modulus, and

(iii) shrinkage and tensile modulus.

Influence of flexure on marginal leakage of dental composites bridges is not included in literature as an important consideration. Pastila *et al*., (2007) studied the influence of water on the elastic properties of dental composites and concluded that absorption of water has the effect of changing the yield limit of the matrix rather than the elastic properties.

In order to improve on the strength by improving the resin component Floyd and Dickens, (2006) report that improvements in the resin base by increasing the base monomer, improved flexural strength and decreased volumetric shrinkage of composites. Replacing bis-GMA or TEGDMA by UDMA also causes an increase in flexural strengths of the resin matrix. This may be explained by the degree of conversion of the polymer matrix or the ability of the urethane linkage to form hydrogen bonds in the copolymer. Restricted sliding of the polymer segments relative to each other is presumed to cause the resultant increase in flexural strength (Floyd and Dickens, 2006).

Zandinejad *et al*., (2006) sought improvements from the filler component rather than the resin and found that composites containing glass–ceramic fillers increased flexural strength and modulus significantly more than conventional glass

fillers. The incorporation of nano-alumina particles also led to increased flexural modulus and flexural strength of composites. The wear performance of the composites did not however correlate with the mechanical properties although a positive correlation between wear resistance and impact strength was observed (Zandinejad *et al*., 2006).

Tanimoto *et al*., (2006) found that flexural strength of composite resins decreased with increasing filler particle size. Since dental composites cannot withstand heavy occlusal forces, many ways have been introduced to reinforce them, such as using fibers or whiskers as reinforcing agents (Xu *et al*., 2000; Xu *et al*., 2002; Xu *et al*., 2003; Xu, 2003; Xu, 2006). Similar findings were observed by Papadogiannis *et al*.; (2007) discovered that compressive strength, hardness, flexural strength and modulus of elasticity all increased when filler volume fraction of dental composites was increased and shrinkage decreased as filler volume fraction increased. However there was some evidence that strength begins to decline at very high filler levels (greater than 60% volume) even though the modulus of elasticity continued to increase as more filler was incorporated (Papadogiannis *et al*., 2007).

Körber and Ludwig (1983) showed that biting forces above the fracture resistance of glass fiber reinforced composites can occur during the mastication process. This highlights the need to maximize the flexural strength of dental composite materials while maintaining a rigid restorative structure. Fischer *et al*., (2004) reports on the following strategies that have been developed to maximize the loading capacity of polymer-based dental bridges:

(i) Kobayashi *et al*., (2000) succeeded by adding ceramic micro-fillers into the polymer matrix

(ii) Bischoff, (1996) and Shuman, (2000) strengthened the material by inserting glass fibers into the restoration. Glass fiber insertion has been successfully used with the commercial product Targis/Vectris (Ivoclar, Liechtenstein) (Clunet –Coste 1997). Application of fiber reinforced composites (FRC) do however not prevent undesirable fractures in cuspal coverage restorations and as with wear, the biggest problem with fracture resistance of dental composites, is for cusp replacement, stress bearing restorations (Fennis *et al*., 2005)

(iii) Loose *et al.,* (1998) and Vallittu (1998) also report that that bisGMA bridges reinforced by unidirectional orientated glass fibers showed a significantly higher fracture strength than bridges without fibers

(iv) Fischer *et al*., (2004) succeeded in increasing the load capacity of resin based bridges from 515 to 1603 N by ceramic bar reinforcement.

The flexural strength is dependent on design principles that optimize both tooth and composite thickness. Nagasiri and Chitmonkolsuk (2005) showed that the survival probability increased with greater amounts of coronal tooth structure remaining, with endodontically treated molars. This may be because not only flexural strength but stiffness of a material increases as the thickness of the material increases (Anusavice, 2003).

Design considerations for dental composites are largely dependent on the need for adequate space, that will permit adequate stiffness of the restoration, as well as

the need for adequate retention and resistance to dislodging forces on cementation (Nissan *et al*., 2001). Flexibility of composites is especially important since flexure from forces on the unsupported top-middle area of the bridge will tend to displace the most extreme mesial and distal margins of the bridge. The area of the margin affected may change depending on the direction of the force. However the concentration of stress will still be referred to the marginal area.

Poor analysis regarding the influence of flexibility (Vaidyanathan and Vaidyanathan, 2001; Vaidyanathan and Vaidyanathan, 2003) on composite margin integrity restricts knowledge on optimum bridge design. Studies to date do not appear to analyze the influence of flexibility of composite materials on marginal integrity in order to improve bridge design. The need to reduce flexibility is however acknowledged by Visvanathan *et al*., (2007) who report that, improving flexural modulus and flexural strength will yield better marginal integrity**.** Bridge flexibility and influence on marginal seal can be visualized by viewing forces acting on a beam (representing a bridge) suspended between two stands (representing tooth support of the bridge) (Figure 10).

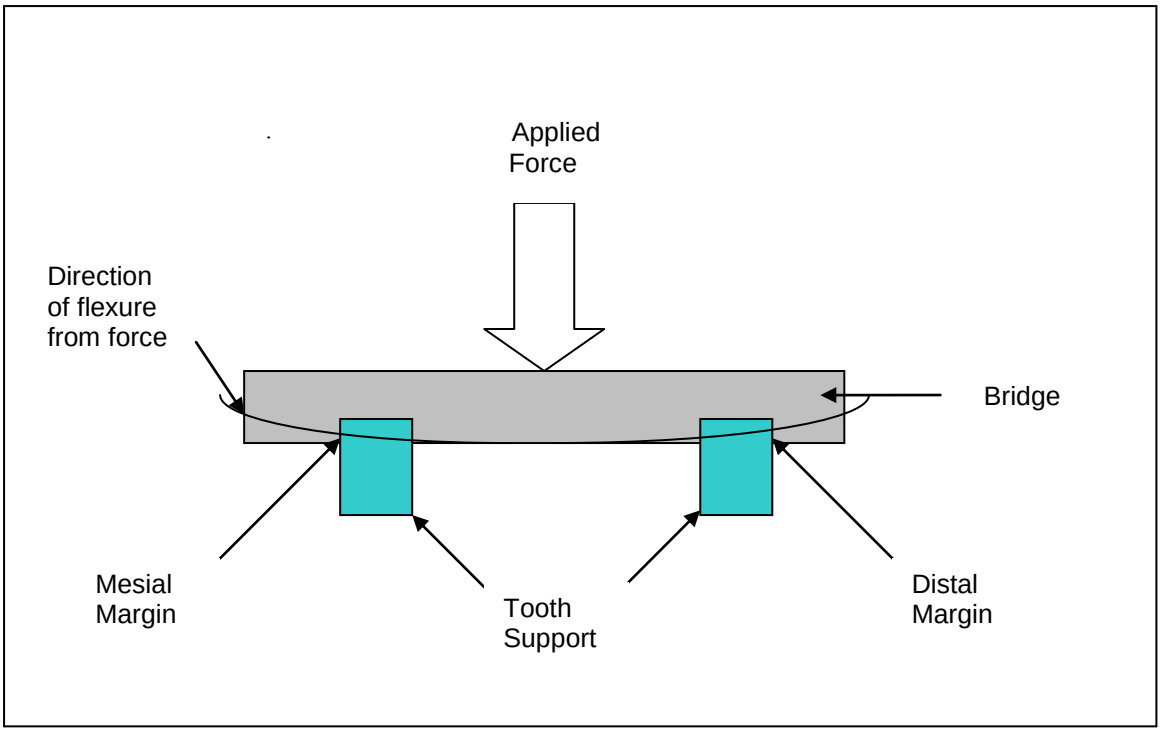

Figure 10 Schematic depicting the likely displacement (of mesial and distal margins) of a bridge

2.2.11 Control of marginal shrinkage of dental composites

If all possible causes for marginal leakage are not evaluated thoroughly and solutions sought, the problem of marginal leakage will persist. The extent of research contributed to solving the problem of marginal leakage without success suggests that the numerous efforts to prevent marginal leakage of dental composites, of which reducing resin shrinkage is foremost, may not be the critical factors to solving the problem.

If curing shrinkage was the main contributing factor, then feldspar ceramic restorations that shrink between 20-40% when fired would not provide the marginal seal that they have been able to for decades (Yamamoto, 1985). Manufacture layering techniques are able to compensate for shrinkage and produce accurate

margin adaptation with indirect dental composite restorations, yet longevity of marginal integrity of these materials is still less desirable and improvements more frequently sought.

Studies that evaluate shrinkage do not consider compensation techniques (during inlay placement) to reduce shrinkage (Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Wahab and Shaini, 2003; Worm and Meiers, 1996). It stands to reason therefore that if bulk application of a material that shrinks does not include compensation techniques, the material will shrink towards the centre of the material mass (Figure 11) and not regain the volume loss. If it is desirable that the outer form is re-established after bulk shrinkage then additional material must be added at the edges. Unfortunately addition of material around the margins of an inlay is less problematic with indirect composite restorative technique.

For an indirect technique the author of this literature review recommends that either a thin outer layer of material (for which shrinkage can be compensated readily) with less resultant bulk shrinkage, is applied in a ring formation that will resist inward shrinkage forces. Thereafter additional layers can be cured towards the centre, thereby reducing bulk shrinkage towards the centre of the restoration (Figure 12). Alternatively the bulk centre of the composite inlay can be cured, removed and trimmed if necessary before reseating it in a thinner composite layer supplied around the marginal area (Figure 13).

Unfortunately techniques to control composite shrinkage are not easy to apply with composites that flow on application. Material properties and manipulation

techniques may need re- evaluation; however improved composite application may reduce flow of composite material that contribute to increased shrinkage stress. The latest CAD/CAM technology further makes it possible to mill exact desired restoration sizes before final placement, of indirect composite restorations. Reducing polymerization shrinkage may be an important consideration, but with improved placement techniques need not be viewed as the unsolvable link to marginal leakage of dental composites.

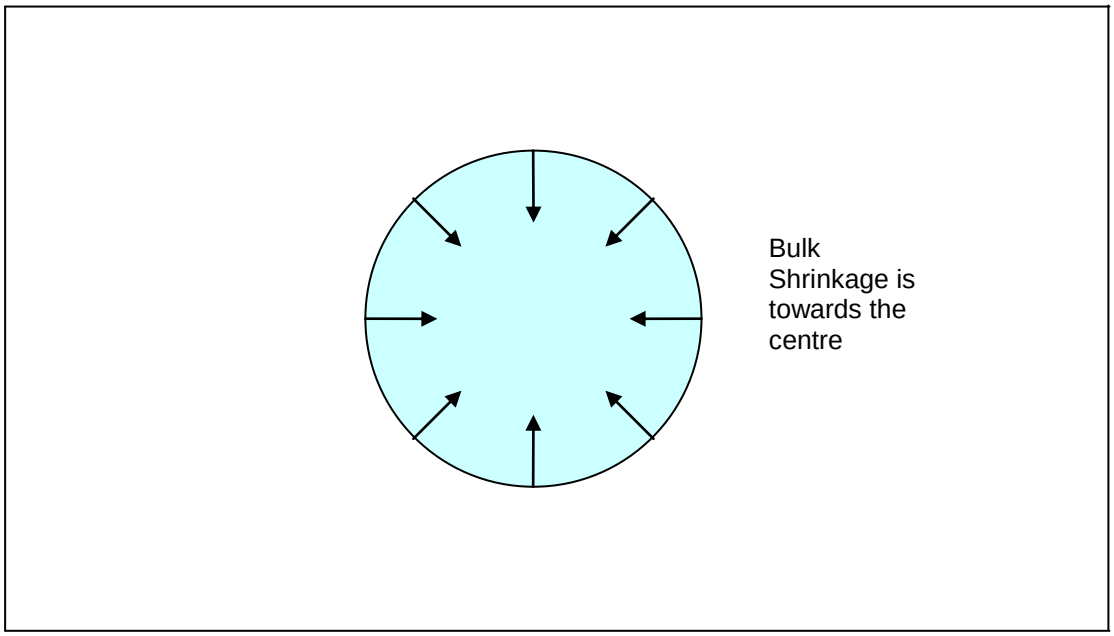

Figure 11 Schematic depicting bulk polymerization shrinkage of dental composite material (arrow direction) that results in reduced overall size of the outer edge

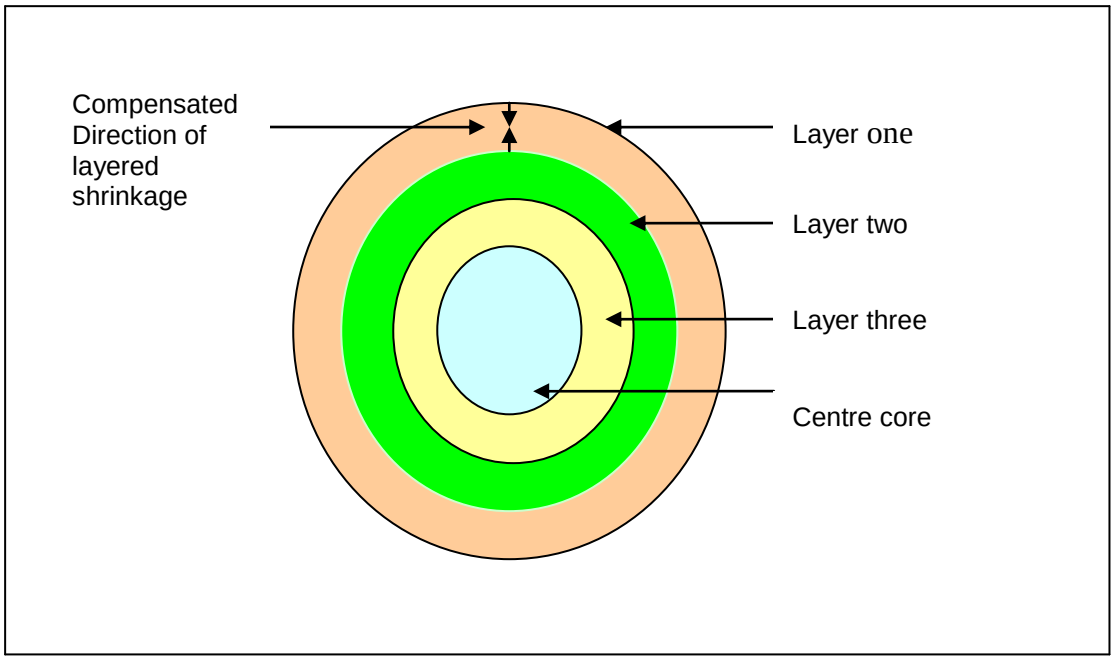

Figure 12 Schematic depicting compensating layering and curing technique to reduce bulk polymerization shrinkage of dental composites

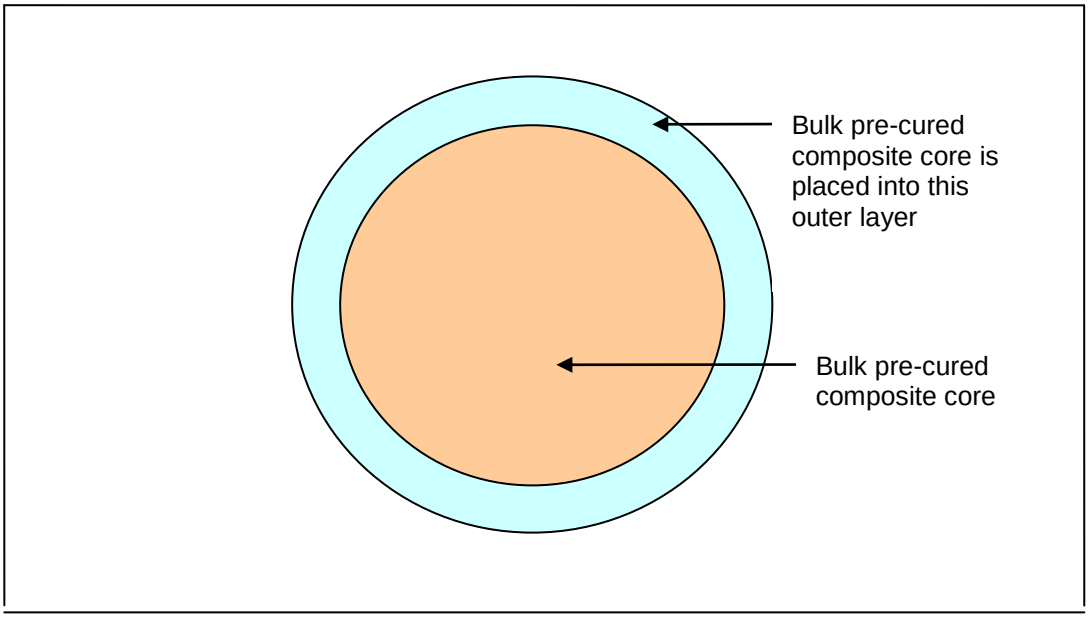

Figure 13 Schematic depicting compensating shrinkage of outer composite layer into which a bulk pre-cured composite core is seated

2.2.12 Design considerations for composite bridges

Composite bridges encompass all the design perimeters of metal ceramic and full metal bridges, including margins, connectors and pontics. The design perimeters are dependent on availability of space and the law of beams (Figure 14). Although composite bridges are not of exact symmetrical design as a bridge the law of beams still applies when a force is applied (Shillingburg,1997). According to Shillingburg (1997) the following principles apply:

(i) The flexure of the bridge increases proportionally to the cube of the increase in the length of the bridge. If the length is doubled, 8 times the flexure will therefore result and if tripled in length the bridge will flex 27 times more. The greatest area of significance are the connectors which are the thinnest part of the representative beam area of a bridge, therefore flexure or fracture may be greater in the connector area.

(ii) The flexure is also inversely proportionate to the cube of the height of the connectors. Thus if the vertical thickness is halved the flexure will again be 8 times greater etc.

(iii) The flexure increases proportionally to the inverse of the width of the connectors, thus by halving the connector width, the value of flexure will double.

(iv) Corrugated lingual directed connectors with U rather than V shaped design further resists flexure and increases flexural strength.

The need to reduce flexibility is also reliant on the amount of available space for adequate material thickness. Feedback from dental technicians has shown the most common complaint to be the lack of space to achieve desired shade and

strength characteristics (Kersten 1988). Correct preparation design must be adhered to so that sufficient space for the composite material is permitted, especially at the connector, margin and occlusal areas.

The margins need special preparation attention to provide bulk thickness at this inevitable thinner finishing section (Yamamoto, 1985). Generally it is accepted, that while ideally all ceramic design principles can be adopted composite thickness should be increased where possible due to increased flexibility of composites as compared to ceramic materials (Preston, 1984). Figure 14 illustrates a full porcelain crown restoration and shows the problems of working with space constraints. Due to the shape of a dental composite restoration, there are different amounts of space available at different areas of the restoration. The reduction of tooth structure should ideally be (Yamamoto, 2005):

(i) 1.0 mm at the margins

(ii) 1.5 mm at the middle third of the crown

(iii) 2.0 mm at the cusp area.

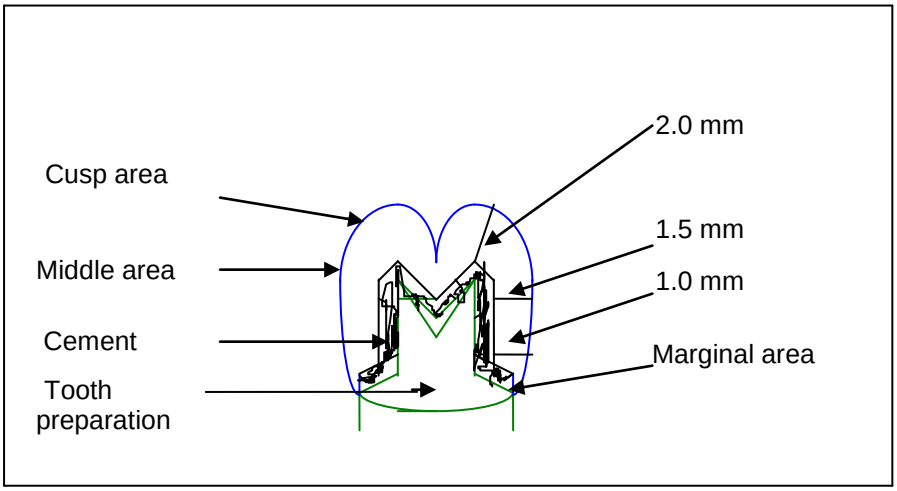

Figure 14 Schematic depicting a hypothetical tooth and the zones where available space for composite restorations varies

The recommended composite thickness may be applied to these areas (Le Roux, 2001). The margin preparation is particularly important to prevent flexure or fracture of the composite restoration. At the margins it is possible to provide sufficient space for the restoration by preparing a deep chamfered preparation. Beveled margins are unfortunately often employed. Beveled margins allow less space proportionately to the increase in the angle of the bevel. Theoretically, beveled margins increase the marginal seal improving the longevity of the restoration, but practically many aspects such as distortion of thin composite sections, chipping of fine edges or loss of fine edges due to finishing procedures, may occur.

Inadequate space availability combined with the increased demand in the ability of the dental technician to reproduce accurate thin sections in dental composites, prevents this bevel margin preparation from being optimal. Using the correct shaped bur to obtain a deep chamfer of at least 1.0 mm at the tooth margin (1.4 mm -1.6 mm round tip, parallel bur) (Figure 15) as well as allowing sufficient space for the connectors and occlusion areas in order to resist flexure, are important considerations for dental composite restorations (Le Roux, 2001).

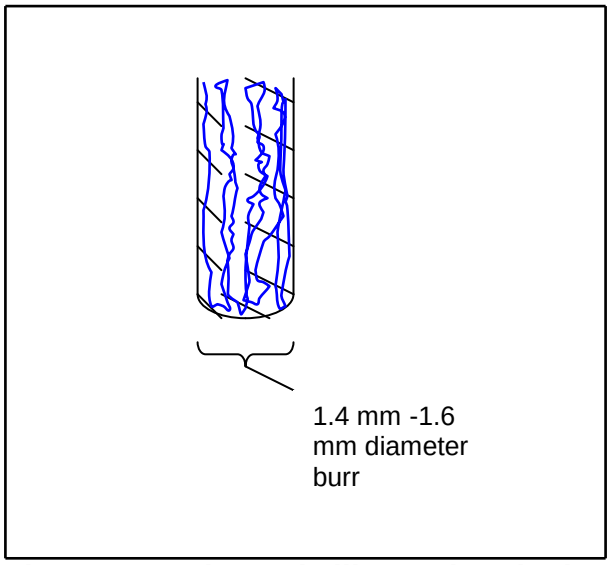

Figure 15 Schematic illustrating the bur design required for a deep chamfered margin

Li *et al*., (2004) reports on the growing interest of *in vitro* experimental investigation of fixed posterior inlay bridges fabricated with a fibreglass-reinforced system, in order to determine if they possess adequate fracture strength and marginal adaptation, under clinical conditions. With regard to direct fibre-reinforced dental bridges, the effect of span-to-thickness ratio on flexural properties of fibre-reinforced composite used for dental, both the conditions of constant thickness and constant support span were studied. Based on experimental investigations, the absolute load bearing capabilities were higher than the ones expected, although the calculated flexural strengths and moduli are lower at shorter support span. This finding suggests that the presence of fibres within the bridge could be capable of supporting considerably higher loading than the composite material properties allow (Li *et al*., 2004).

Li *et al*., (2004) supplied some relevance to bridge design. They used finite element analysis (FEA) in order to quantify specific stress and displacement

distributions anywhere within the analysis domain. The advantage of finite element (FE) modelling is its capability of parametric studies (Awadalla *et al*., 1992). Once the fundamental FE model is appropriately verified, it can conveniently evaluate the influence of geometry and parameter variations such as shape and materials properties. Unfortunately, little FEA has been applied to direct composite fibre-reinforced dental bridge structures (Li *et al*., 2004).

According to Li *et al*., (2004) the fundamental composite bridge model consists of five different material parts:

(i) abutment dentin

(ii) crown (enamel)

(iii) composite pontic

(iv) reinforced fibre

(v) adhesive layers on abutment/pontic, abutment/fibre and pontic/fibre interface.

Each tooth is in static and dynamic contact with the adjacent tooth at each side of it as well as with the opposing teeth. Normally, there exists at least a 13 μm clearance space between adjacent teeth. When such a 13 μm clearance space is closed due to a certain level of compressive bite force, a positive contact relationship between the adjacent teeth and dental structure can be developed. Under such a situation, the dental structure would be partially supported by the contact forces coming from its adjacent teeth.

With FEA limited to linear elastic evaluation Li *et a*l., (2004) found that the highest stress levels are distributed from the pontic to the connectors. The high marginal

stress was not counted as a failure stress because the strength of abutment is generally much higher than those in the connector of the bridge. Instead, the stress concentration in the connectors indicated a higher possibility of failure due to the lower strength in adhesives.

Finite element analysis applied to bridges by Li *et a*l., (2004) unfortunately focused on material failure and not marginal seal failure which is more critical (see Section 2.8 and 2.10). Farah *et al*., (1989) found that the bridge also resulted in higher tensile stresses distal to the abutment teeth. From a stress standpoint bridges resulted in more uniform stress distribution around the abutments and an increase in the tensile stress distal to the abutments. This is probably a result of the mesial inclination and movement of posterior teeth that may influence marginal adaptation (Shillingburg, 1997).

Young and Thompson, (1991) found that tilting of the molar abutment, induced additional stress on the mesial side of the root, while the presence of a fixed prosthesis markedly reduced the magnitude and distribution of stress in the periodontium. When considering the cantilever action of a bridge Awadalla *et al*., (1992) also found that a cantilever pontic creates considerable compressive stress[8] on the abutment nearest to the pontic and produces tensile stress on the abutment farthest from the pontic. High stress concentrations were observed by Yang *et al*., (1996) around the connectors of the fixed prosthesis and the tooth closest to the cantilever. Reduced bone support also increased the deflection and stress concentrations while splinting the teeth together resulted in a reduction in

[8] Compressive stress refers to the internally induced force that opposes the shortening of a material in a direction of the stress; any induced force per unit area that resists deformation caused by a load that tends to compress or shorten a body (Van Blarcom, 1999).

displacement and stress. Increased abutment support and decreased pontic function was therefore advised.

2.2.13 Alumina/feldspar resin infiltrated composites

Alumina has successfully been used to strengthen dental ceramics for decades. Since as early as 1965, McLean (cited in Jones, 1983) incorporated up to 50% alumina in feldspar in order to strengthen the core material for full ceramic crowns (Fleming *et al*., 2005). In-Ceram® material (Vita, Germany) increased alumina content above 50% through a process of sinter firing fine alumina powder before glass infiltration to form a core material with improved strength (Fleming *et al*., 2006) (Lawn *et al*., 2004).

In-Ceram® material must be covered with feldspar since the glass will etch forming pits in the material if exposed to pH fluctuation of oral fluids. In-Ceram® exhibits high flexural strengths values between 300 and 700 MPa in order to resist fracture, as a result of the brittle nature of ceramics. Clinically, radial cracks form in In-Ceram® because of its brittle properties.

Radial cracks tend to remain subsurface and close up when unloaded making them difficult to detect by surface inspection alone. Radial cracks are especially dangerous in thin crown structures and in prolonged loading (Lawn *et al*., 2004). Although composites do not require as high flexural strength as ceramics to resist fracture because they are slightly less brittle, flexural strength should be maximized as far as possible. Extensive investigation into alumina as a strengthening filler material for composites has been unsuccessful because silane

bonding of alumina to resin has posed more problems than silane bonding of resin to glass (Matinlinna *et al*., 2006).

Feldspar chemical adhesion between alumina particles should be evaluated as an replacement for silane adhesion of SR ADORO®. SR ADORO® is a newly developed, wear resistant, microfilled (nanoparticle range), light/heat-curing veneering composite for full coverage veneers and partial veneers (Zandinejad *et al*., 2006).

2.2.14 Improving success of dental composites

Attempts at improving the success of dental composite materials, have highlighted areas in which dental composites need to be improve. Wear studies have attempted to provide lower wear values. Flexural strength studies have attempted to increase the flexural strength and rigidity. Studies to improve the bonding agents between filler and resin have resulted in improving but not replacing silane. Numerous studies have been directed to reduce the polymerization shrinkage of composite materials.

The potential to replace amalgams as restorative materials with composites in dentistry (Roeters *et al*., 2004) combined with the many problems experienced with dental composites highlights the need for continued investigation regarding the performance of the various composite materials. Dental composite materials are constantly being tested and developed in order to produce the desirable properties of wear, strength, rigidity, shrinkage, aesthetics, surface finish and cementation. Concentrated efforts are exerted to condone the use of dental composites for

posterior stress bearing areas (Van Niewenhuysen *et al*., 2003). Limited relevant data however suggests that all composites materials may not be well presented, while generalized statements regarding their success are made (Ferracane, 2006).

Problems with wear and marginal integrity that persist need to be solved and research directives may need to be re-evaluated (Ferracane, 2006; Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Wahab and Shaini, 2003; Worm and Meiers, 1996). Compensation techniques to reduce polymerization shrinkage should be considered as well as other relevant factors that influence marginal seal such as flexibility. Based on the literature review, it is recommended that factors that decrease flexibility while increasing flexural strength and wear resistant properties should be improved in order to prevent marginal leakage and chipping of dental composite restorations while preserving tooth structures as far as possible.

Silane bonding agents should be improved on since they deteriorate with time. Further investigation is required regarding silane bonding between non etchable fillers (such as alumina and zirconium) and resin materials. In addition, further studies to determine the influence of pH on the wear of dental composites due to time related silane deterioration within the composite material would be of benefit.

Improvements in dental composite materials have resulted in wear resistant properties that are more suitable for posterior restorations than the early composite materials. Accurate reproducible *in vitro* wear tests on dental composite materials

however still needs to be performed for large cusp bearing restorations. The complex nature and continued problems of wear of composite restorations require further solutions for large composite restorations before they can be considered to possess the ideal wear resistant properties.

CHAPTER THREE

MATERIALS AND METHODS

When solving a problem the question is: What is the best way of searching for the solution with the available resources? Research methodology is the process of finding the answer.

3.1 METHODOLOGY

This study involved three components:

(i) The material design component that involved trial and error application in order to produce alumina/feldspar resin infiltrated materials for comparative analysis

(ii) The experimental component, that provided data on wear, flexure and flexural strength of alumina/feldspar and SR ADORO® composite materials

(iii) Comparative component that included design and experimental phase analysis in order to make recommendations regarding the overall performance of the composite materials evaluated.

3.1.1 The shape of the specimen

The specimens' shape allowed flexibility and flexural strength tests to be performed after the specimens were subjected to *in vitro* cyclic loading in a Minimet® polishing machine (Buehler Ltd, Illinois, USA), After subjecting each specimen to *in vitro* cyclic loading (for the same duration of time and under the same load) accurate *in vivo* flexibility and flexural strength performance was simulated.[1]

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[1] The reason *in vitro* performance was evaluated was because some restorative materials exhibit fatigue-wear while others exhibit deep micro-crack formation with extended cyclic loading. Care should therefore be exercised when selecting materials for posterior stress-bearing areas. If the material is not subjected to cyclic loading before flexural strength tests are performed a higher strength value than is realistic may be obtained. This is because micro-crack formation decreases the strength of composite and ceramic materials and may precipitate failure despite the low wear observed (Yap *et al.,* 2002).

3.1.2 Sample size

The sample size required to validate statistical analysis (from the central limit theorem) and obtain a normal distribution of results is 30 or above. Eight specimen groups were identified. An alphabetical letter was ascribed to each group. Thirty wear repetitions of each specimen group were performed for specimen Group A, B, C, D, E and F. The total of all specimens, including Group F (the SR ADORO® commercial control group) was 180.

In order to determine whether wear of alumina/feldspar composites could be improved further by varying the percentage feldspar mass in finer increments, the performance of 40% and 50% feldspar mass (Group H and I) were tested as well. Additional specimen groups (H and I) were included in the research design as follows:

(i) n=10 for Group H (50% feldspar mass)

(ii) n=10 for Group I (40% Feldspar).[2]

The total additional specimens was 20 resulting in a total sample size of n=200, including the SR ADORO® commercial control Group. [3]

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[2] Only 10 specimens for each additional group (H and I) were prepared and tested. Having established certain trends (with 30 specimens for alumina/feldspar specimen groups A to E) 10 specimens for each additional specimen group (H and I) was sufficient since the results were consistent.

[3] The original research focus was on alumina/feldspar composite design in order to improve wear resistance of dental composite materials, therefore the need to test flexural strength and flexibility of the composite specimens was only considered later. Since micro-crack formation may precipitate failure despite the low wear observed (Yap *et al.,* 2002) the decision was taken to include flexural strength data on the strength of alumina/feldspar specimens after cyclic loading. As a result of the decision to include flexural strength tests flexibility testing with the Instron® 44 was performed at the same time. Ten specimens from group C, H, and I (that showed similar or less wear than SR ADORO®) as well as ten SR ADORO® specimens were subjected to flexural strength and flexibility testing.

3.2 ALUMINA FELDSPAR MATERIAL DESIGN

3.2.1 Matching the coefficient of thermal expansion (CTE) of the alumina and feldspar

Chemical bonding was incorporated between filler particles since the traditional size alumina particles extruded readily. The chemical bond resulted from Vitadur® feldspathic porcelain (Nova Dental, Johannesburg) that was observed to be CTE compatible. The linear coefficient expansion of a material is defined as, “the relative linear change in length per unit of initial length during heating of a solid per ^{o}K within a specified temperature range” (Anusavice, 2003). As the alumina/feldspar composites constituted a new material concept in dental composites, the curved (s-shaped) CTE and temperature relationship for ceramic materials, require that bonding compatibility of the alumina and feldspar, be confirmed by observation. Stress formation is inevitable between ceramic materials of different composition and changes in accordance with the firing cycle (time and temperature ranges) selected, which influence leucite crystal precipitation of feldspathic ceramics. Ceramic materials such as alumina possess a lower CTE than metal alloys and are not subject to crystalline changes, resulting in the need to select a compatible feldspar material. Compatible CTE for the specified firing cycle (Table 1) could only be reported should consistent results (absence of crack formation) in the bonded materials occur, since unacceptable CTE differences would result in crack formation (Yamamoto, 1985).

3.2.2 Porous alumina/feldspar material design

The alumina/feldspar specimens that were prepared for data collection contained the same basic porous alumina/feldspar resin infiltration design (Figure 16). The bonding between the alumina were altered between specimen groups by varying the percentages of feldspar mass added to alumina and/or including silane or phosphate adhesive bonding agents. As the feldspar mass was increased the pore size between alumina particles decreased slightly but did not prevent resin infiltration of the materials if feldspar mass was below 60% mass.

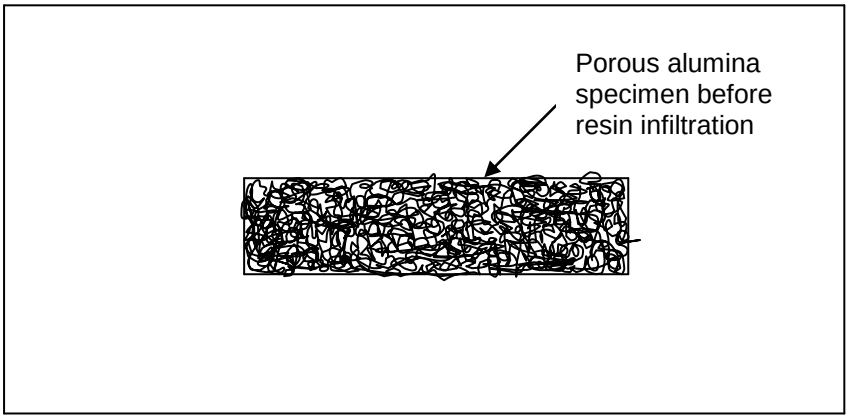


Figure 16 Schematic depiction of basic rectangular porous alumina specimen design [4]

The percentage feldspar added to alumina influenced the bond strength and pore volume. High feldspar mass reduced the pore size between alumina particles. Reduced pore size influenced resin infiltration, thus 24 hours was allowed to ensure complete infiltration of all specimens.

[4] The condensed mass of ground alumina particles (up to 50μm) mixed with feldspar resulted in a chemical bond between the particles of the aluminous porous structure once fired. This permitted UDM resin to flow through the pores of the material, thereby combining the properties of ceramic and resin materials.

The chemical bond between alumina was formed by feldspar since it reached a liquid phase at 1100^{o}C and was drawn toward the contact areas. On solidification the pores (between alumina particles) remained open and resin infiltration was possible (Figure 17).[5]

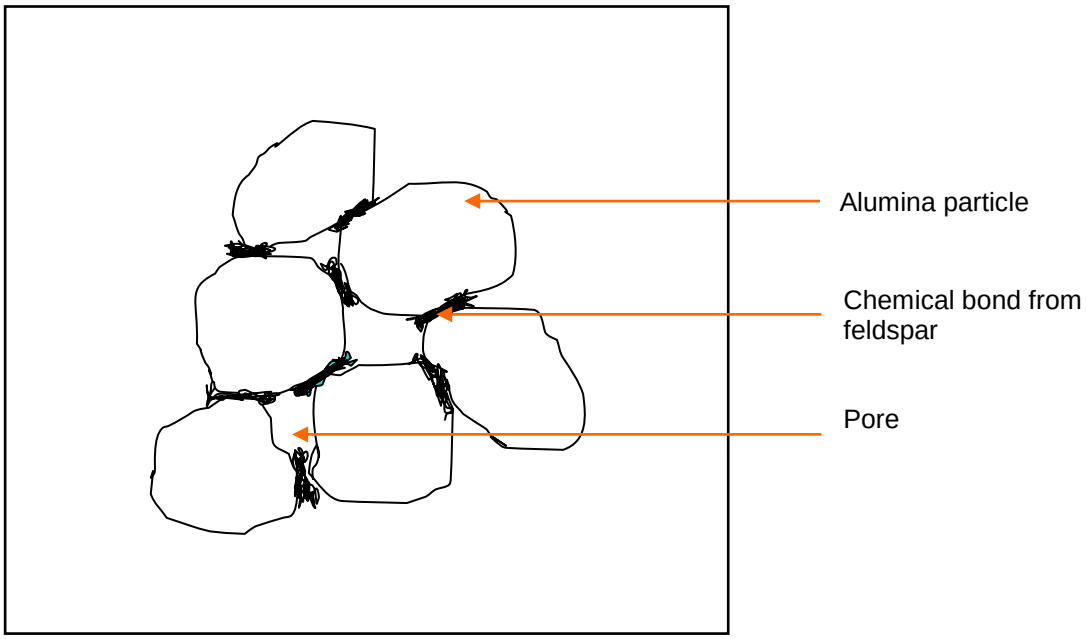


Figure 17 Schematic depiction of the chemical bond formed between the alumina particles.

3.2.3 Specimen preparation

Alumina sand was ground using a small ceramic mortar and pestle to produce a mixture of macro particles.[6] One gram of 50μm alumina sand was ground for one minute to produce the desired alumina particle size (≤50μm). The ground alumina

[5] If too much feldspar was added (above 60% mass) the pores become blocked and complete resin infiltration was not possible.
[6] Large alumina particle (up to 120 um) were first tried but these specimens produced rough surfaces after wear and too large a pore size resulting in poor retention of the resin material during the curing process.

particles were then placed in a plastic bag and shaken to obtain a uniform mixture of particles. Grinding resulted in better compaction with a smoother surface finish.

Varying amounts of feldspar were added to the ground alumina to provide the desired alumina/feldspar mass ratio for each specimen group. The feldspar was added by % mass required.[7] The alumina/feldspar mass ratio was determined using a Mettler Toledo® electronic scale.

The alumina/feldspar mix was applied in a depth gauge (Figure 18).[8] The depth gauge allowed the correct amount of alumina/feldspar to be built up so that after firing the specimens had a desired thickness of 2.0 mm. The depth gauge was set 0.2 mm deeper than the required 2.0 mm to compensate for slight shrinkage of the edges of some specimens. This was necessary because the center of the specimens shrunk up to 0.15 mm more than the edges and the specimens had to be trimmed with a barrel shaped diamond bur (Edenta®, Switzerland) to provide a constant 2.0 mm thickness of alumina/feldspar specimens.

The alumina/feldspar powder was mixed with water to a creamy paste consistency and applied with a brush into the brass rectangular container (5.8 mm X 19 mm) attached to the vernier measuring device of the depth guage. The particles were condensed by tapping the sides of the container and removing the excess water, with the aid of a hair dryer. The rectangular shaped alumina specimens were carefully pushed out with the plunger and fired on a honeycomb firing tray (1100^{o} C

[7] If 50% feldspar mass was required then 50g feldspar was added to 50g alumina etc.
[8] The depth gauge used by Le Roux (2001) was an adaptation of a similar instrument used by Somers (1998), the difference being that the specimens were rectangular in shape, unlike those of Somers, which were round specimens. However the principle to obtain accurate height (thickness) of specimens remained the same.

for ten minutes) to produce a fired porous structure of consistent specimen thickness (2.0 mm). The firing cycle is indicated in Table 1.

Cycle	Temperature	Time
Holding time:	600^{o}C	3 minutes
Temp increase:	68^{o}C	6 minutes
Final temp:	1100^{o}C	10 minutes
Vacuum:	600^{0}C to 1020^{0}C	15 minutes

Table 1 Firing cycle for alumina/feldspar specimens

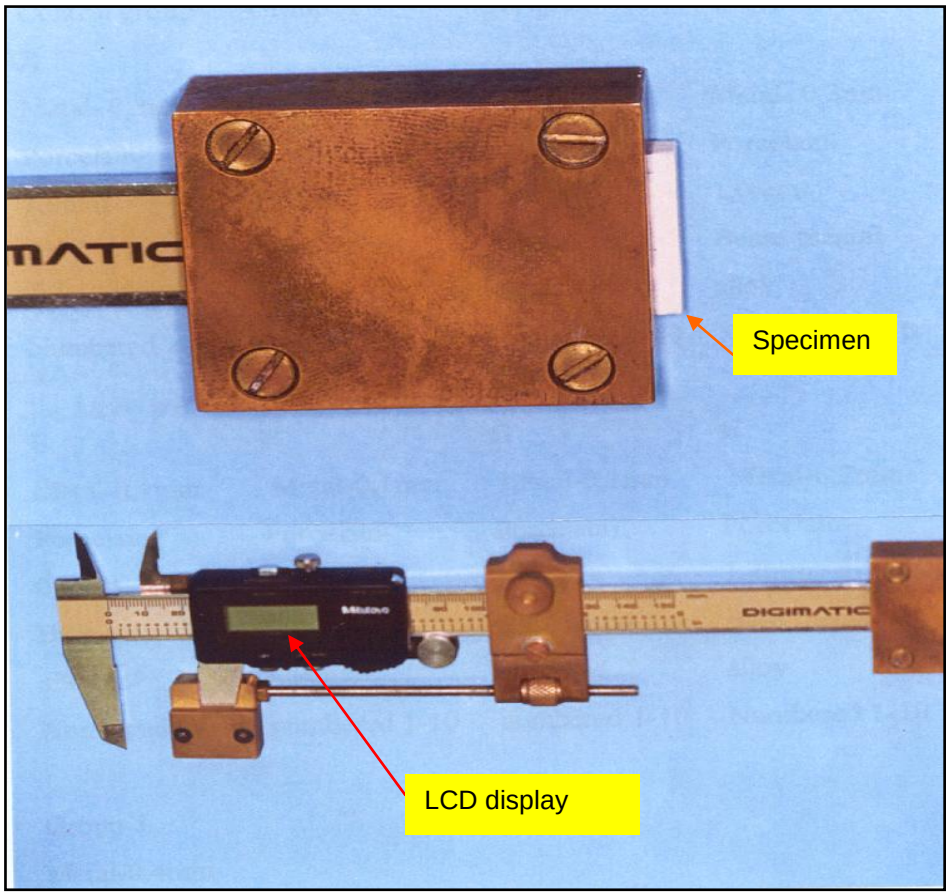


Figure 18 Depth gauge for the alumina/feldspar layering technique

The depth gauge consisted of a modified digital vernier with an LCD display and accuracy of 0.01 mm. The modified digital vernier was linked to the manufactured portion of the depth gauge which consisted of a rectangular shaped box conforming to the specimen size and a piston that matched the rectangular shape perfectly. The rectangular box and piston were made of brass to prevent corrosion that might interfere with alumina/feldspar properties on firing. The depth gauge had a locking mechanism, which allowed it to be locked into position during the application of porcelain to allow repeated results and measurements. A fine adjusting screw was incorporated to allow fine adjustments and reduce the time taken in reaching exact measurements.

A hair dryer was used to dry the porcelain and the excess porcelain above the outer surface of the gauge was leveled off with a straight flat metal edge. The specimens were removed and fired in batches of 10 in a Vacumat 300 porcelain furnace (Vita, Germany) (Figure 19).

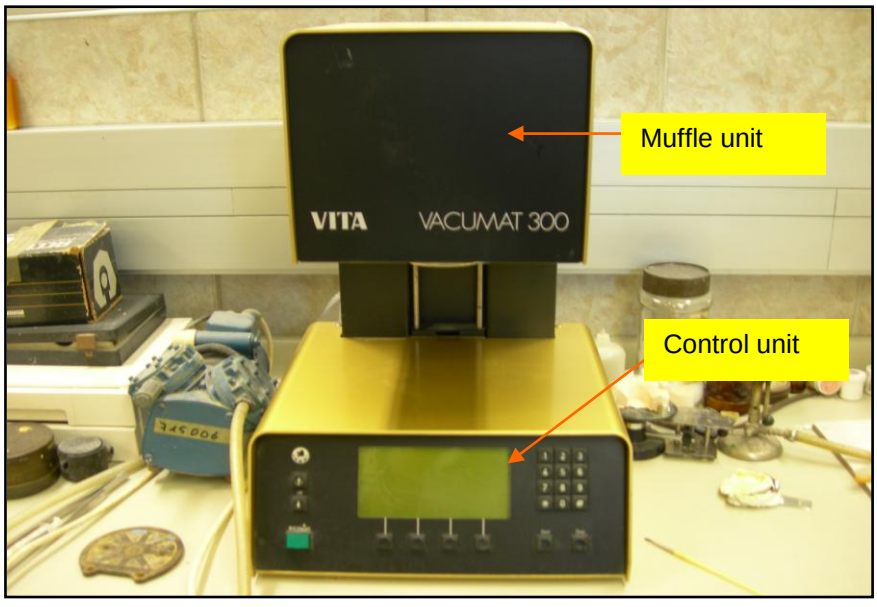


Figure 19 Vacumat 300 porcelain furnace

Specimens that included adhesive bonding agents were treated with 3 drops of silane (Monobond S®, or SR LINK®, Ivoclar Lichtenstein) and allowed to dry for 12 hours before being infiltrated with UDM resin for a further 24 hours to ensure complete infiltration of the pores. The drying silane process allowed even consistent layers of the adhesive bonding agent before UDM resin infiltration. UDM resin infiltration from gravity and capillary action was obtained by placing excess resin on top of each specimen.

Excess UDM was wiped from the specimens after infiltration using tissue paper before they were cured in a Sharp® R-341C microwave oven set to 1000W. Each specimen was cured for four minutes on a ceramic plate (specimens were turned at one minute intervals).

The specimens were prevented from bonding to the ceramic plate upon which they were cured by having the specimens rest on 2 raised ridges on the ceramic plate (Figure 20).

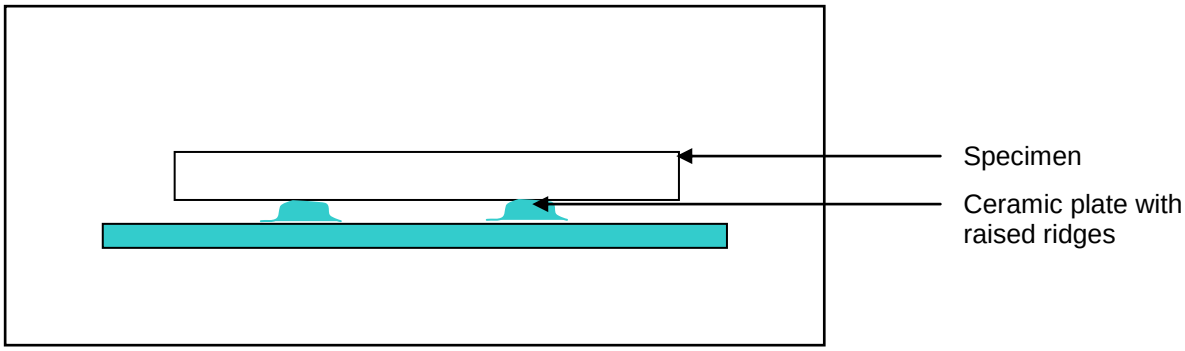


Figure 20 Schematic depiction of specimen placement for curing process.

After UDM curing the specimens were ground to obtain a consistent 2.0 mm thickness with a barrel shaped (code- E09000) diamond bur (Edenta, Switzerland)

using a Kavo K10 grinder (Nova dental, South Africa) (Figure 21). The specimens were measure with a standard dental laboratory thickness measure to ensure accurate desired results (Figure 22) (Nova dental, South Africa).

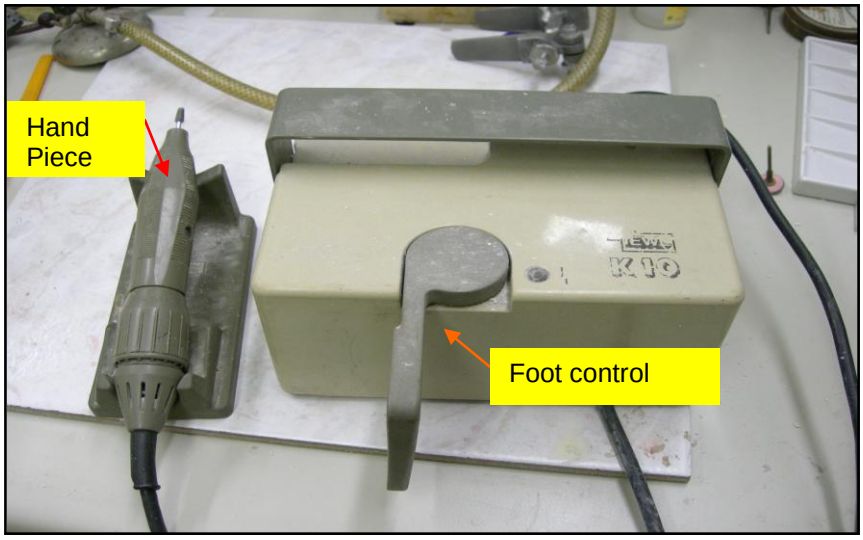


Figure 21 Kavo K10 grinder with barrel shaped diamond burr

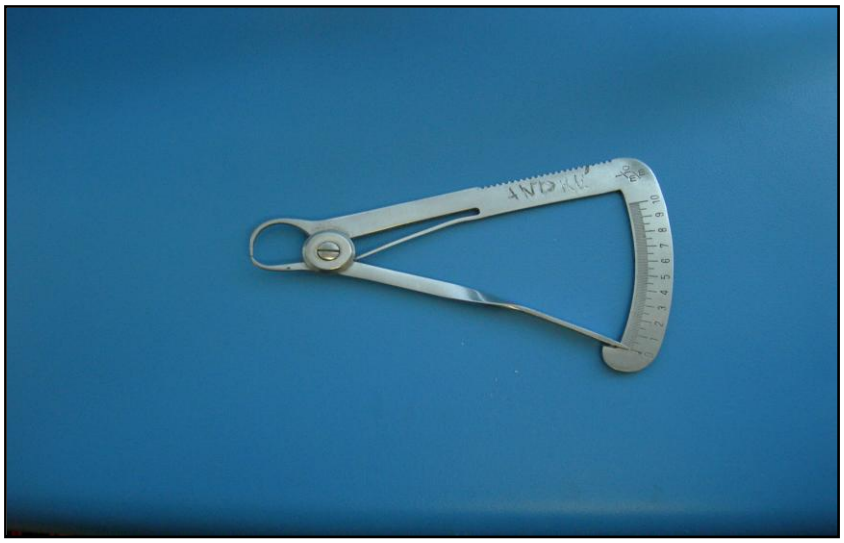


Figure 22 Thickness measurement gauge

3.3 NUMBERING AND IDENTIFICATION OF SPECIMENS

Each specimen could be identified by the letter and number allocated to it. The letter identified the sample group while the number identified specimens in the sample group (Table 2).

Group	Description	Numbering
Group A	**Sintered alumina with silane adhesive bonding**	A1-A30
Group B	**30% Feldspar with alumina and no adhesive bonding**	B1-B30
Group C	**60% Feldspar with alumina and silane adhesive bonding**	C1-C30
Group D	**30% Feldspar with alumina and silane adhesive bonding**	D1-D30
Group E	**30% Feldspar with alumina and SR Link® adhesive bonding**	E1-E30
Control Group-F	**SR ADORO® Commercial composite With silane adhesive bonding**	F1-F30
Group H	**50% Feldspar with alumina and Silane adhesive bonding**	H1-H10
Group I	40% Feldspar with alumina and Silane adhesive bonding	I1-I10

Table 2 Sample groups and numbers

Each specimen was stored in a photograph spool container that was numbered according to the relevant specimen group letter and specimen number (Figure 23).

Figure 23 Containers with Group (A) specimens numbered 1-30

Each specimen group was designed to obtain specific primary data. The data required corresponding to the specimen groups to provide the required data are shown in Table 3.

Data required	Specimen groups to provide required data
Surface structure analysis of SR ADORO® and alumina/feldspar composite materials using the Nikon SMZ800 Microscope.	Selection of photographic material from Group A to E, H and Group I
Wear values in mm for four variations in chemical (Feldspar) bond of alumina/feldspar composite materials.	Group C, D, H and I
Wear values in mm for two variations in adhesive bonding agents of alumina/feldspar composite materials.	Group D and E
Wear values in mm without adhesive bonding agents in alumina/feldspar composite materials.	Group B
Wear, Flexibility and flexural strength values for SR ADORO® commercial composite material (the control)	Group F
Strength values for three variations in chemical (feldspar) bonding between the alumina particles of alumina/feldspar composite materials.	Group C, H and I
Flexibility values for three variations in chemical (feldspar) bonding between the alumina particles of alumina/feldspar composite materials.	Group C, H and I

Table 3 Data required corresponding to specimen groups to provide the required data.

3.4 WEAR TESTING OF COMPOSITES SPECIMENS

Three body wear was performed on each specimen using dentifrice (Mentadent P® Micro Granules; Unilever Durban) as the third body.[9] The dentifrice was mixed with water (10g water with 90g dentifrice) to form the consistency of a polishing slurry (that would simulate *in vivo* use).[10]

3.4.1 The Minimet® polishing machine

The Minimet® polishing machine patent was chosen for its unique polishing action. Each specimen to be tested was placed in the same acrylic mould attached to the Minimet® polishing machine and subjected to the same polishing program. The Minimet® polishing machine (Figure 24) was set on speed 4 and time setting 1. The specimens were placed under the minimum load setting (10-15N when not functioning).[11]

[9] Mentadent P® dentifrice (with micro granules) is formulated to assist with the smooth polishing and cleaning of teeth by displacing impacted food particles and therefore need to have a similar action as food particles. The advantage of the micro granules in Mentadent P® over food particles is that after a removing and taking the position of food particles they dissolve leaving the teeth smooth and clean.

[10] Dentifrice with micro granules was chosen since the micro granules would brake up much like food and therefore not be too abrasive while assisting with the polishing action. The use of Mentadent P® Micro Granules allowed consistent application as apposed to saliva that varies in pH, composition and consistency from day to day and cannot be stored easily.

[11] Load of the Minimet® polishing machine is varied (increases and decreases) due to the design of the polishing mechanism. Higher settings than setting 1 resulted in a frictional grating sound formed (even with the third body dentifrice in place) between the specimen and graphite surface that would not simulate natural attrition.

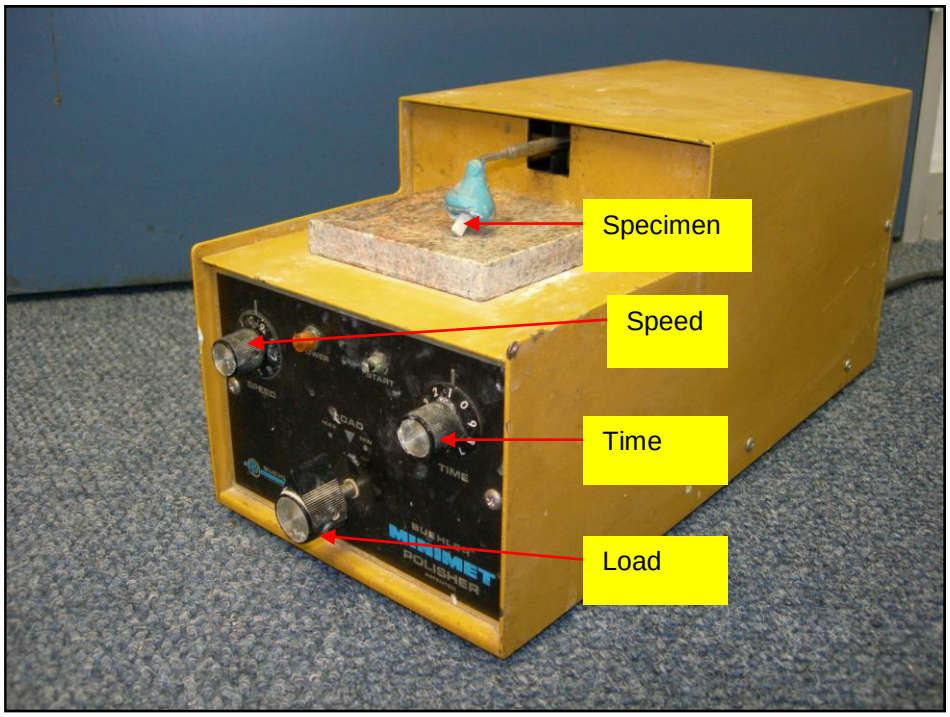


Figure 24 Minimet® polishing machine with a specimen in place.[12]

3.4.2 Measuring wear with the use of the Nikon® SMZ800 Microscope

The wear depth of specimens was measured in mm by imaging the worn area under a Nikon® SMZ800 Microscope (Figure 25). The resultant 22 x magnification was confirmed by measuring the distance between two points of a 1 mm magnified rule and applying the formula *Magnification= LMO/LO*[13]

Although it was possible to measure the actual length of the worn area of specimens with a thickness gauge it was easier and more accurate to measure the

[12] The specimens were placed in the same acrylic mould that was attached to the Minimet® polishing machine resulting in each specimen being tested at the same contact angle of 45^0.

[13] Where LMO is the length of magnified object and LO is the actual length of the object.

magnified area of each worn surface in mm (Figure 26) and then to convert it back to actual values by dividing by 22 (the magnification).[14]

Figure 25 Nikon SMZ800 Microscope connected to a computer

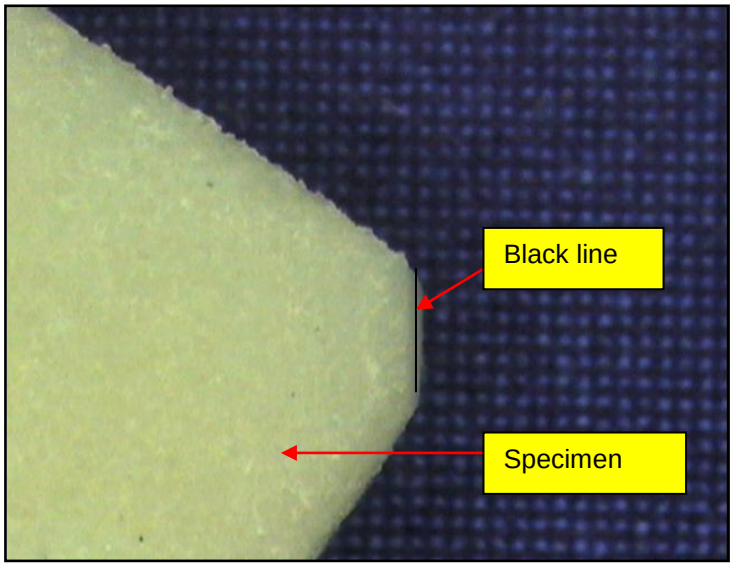


Figure 26 Magnified (22x) image of worn specimen[15]

[14] Measured values were within 1.0 mm error (This was confirmed by measuring the worn specimens twice and evaluating the variation of results). When 1.0mm is divided by 22 (magnification) a 99.95% level of confidence is established. This method is accurate since the eye can easily measure within 0.5mm accuracy with an mm rule, which divided by 22 gives a 99.98% level of confidence. Even with a 2.0 mm measurement error which is not likely to occur with an mm rule 99.91% level of confidence would still be achieved because of the high magnification.

[15] The black line depicts the length of the wear that was measured.

3.4.3 Specimen orientation for wear testing

The specimen was orientated in such a way as to have one corner contact the graphite surface at a 45^0 angle to facilitate measuring the wear of a 90^0 cusp (Figure 27a and Figure 27b).

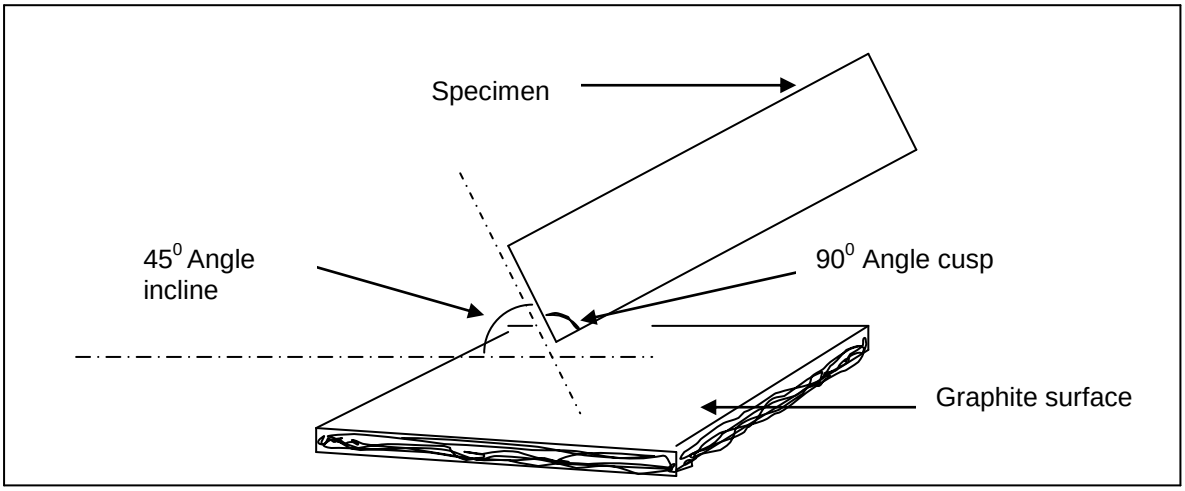


Figure 27(a) Schematic depiction of the position of the specimen to the graphite surface.

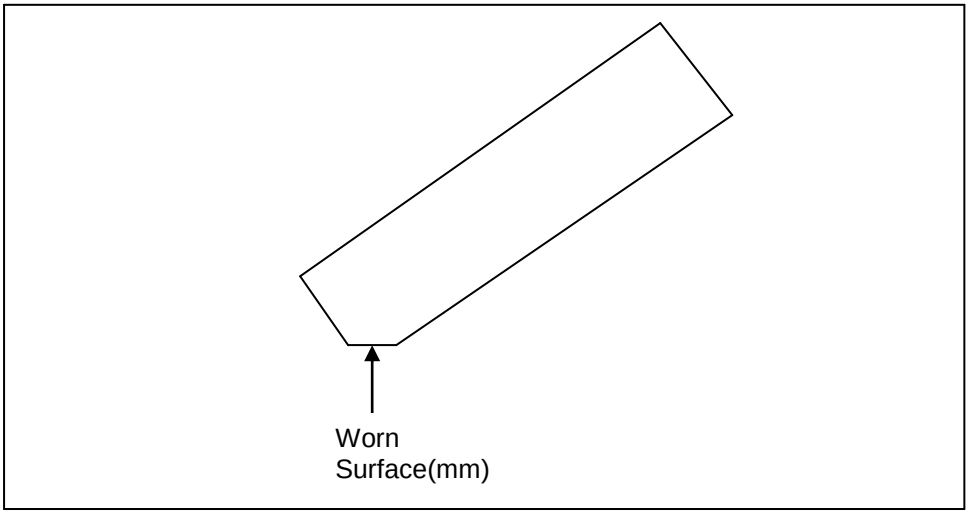


Figure 27(b) Schematic depiction of a worn specimen surface after testing

3.5 FLEXURAL STRENGTH AND FLEXIBILITY TESTING

The 3 point bending tests were conducted by means of a universal testing machine (Instron® 44, Apollo Scientific cc South Africa) operating at 95% confidence level. Specimens to be tested remained at 2.0 mm height for comparable results and were suspended between 10 mm supports. The mean width of specimens was 5.2 mm with acceptable 0.2 mm deviation resultant from firing shrinkage.

The Instron® 44 (Figure 28) was set so that specimens fractured at a constant speed of 10 mm per minute. This speed was reduced in comparison to *in vivo* function in order to obtain consistent results. The specimens were placed between two smooth metal plates 10 mm apart, to allow free rotation during the flexure strength test (Figure 29). At the point that the specimens fractured (first fatigue value) a reading of force resisted (N) and amount of flexure (mm) was recorded for each specimen.

Mean flexural strength value (*S*) was obtained for each specimen group (from the mean N value) that was tested using the following formula:

1. $S=3PL/2bh^2$ (measured in MPa)[16]

[16] Where *L* is the support span length (mm), *b* the specimen width (mm), *h* the specimen height (mm), *F* the load (N) at a convenient point on the straight line portion of the curve, *d* the deflection (mm) at load *F*, and *P* is the maximum load (N) resulting in failure (Walker *et al*., 2006). Specimen shape conformed to the law of beams. Flexibility of each specimen was therefore dependent upon the force, width and distance between the beam support rather than the length of the beam (Figure 29). The small variations in wear from the same cyclic loading between specimen groups could therefore be ignored during flexural strength and flexibility testing because the worn surface was not positioned between beam supports and the test values were dependent on distance between beam support *(L*) rather than specimen length.
Strength and flexibility tests were only performed on specimens that exhibited similar wear as SR ADORO® resulting in testing of the same sample size and specimen shape for comparison.

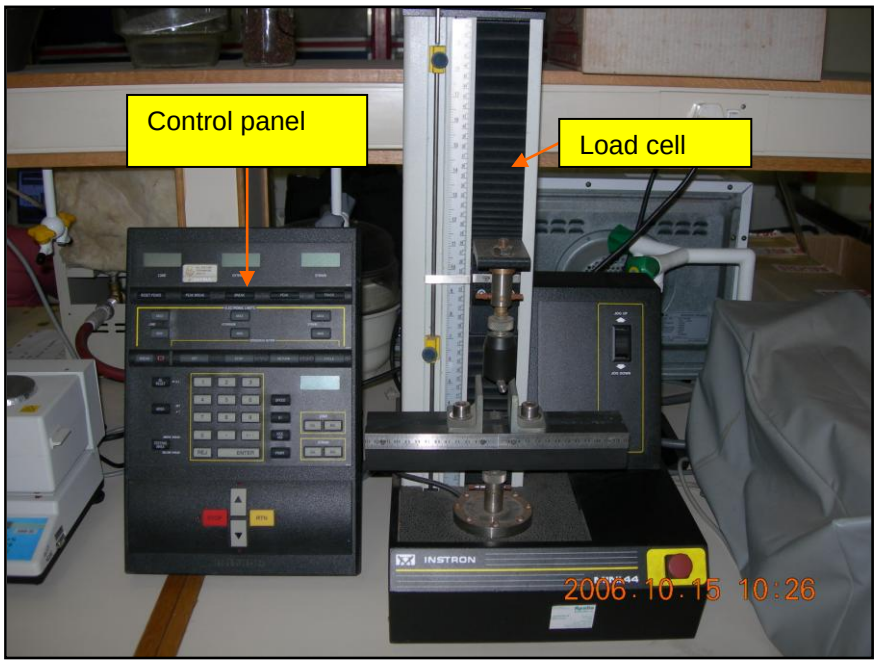


Figure 28 Instron® 44 Testing Machine

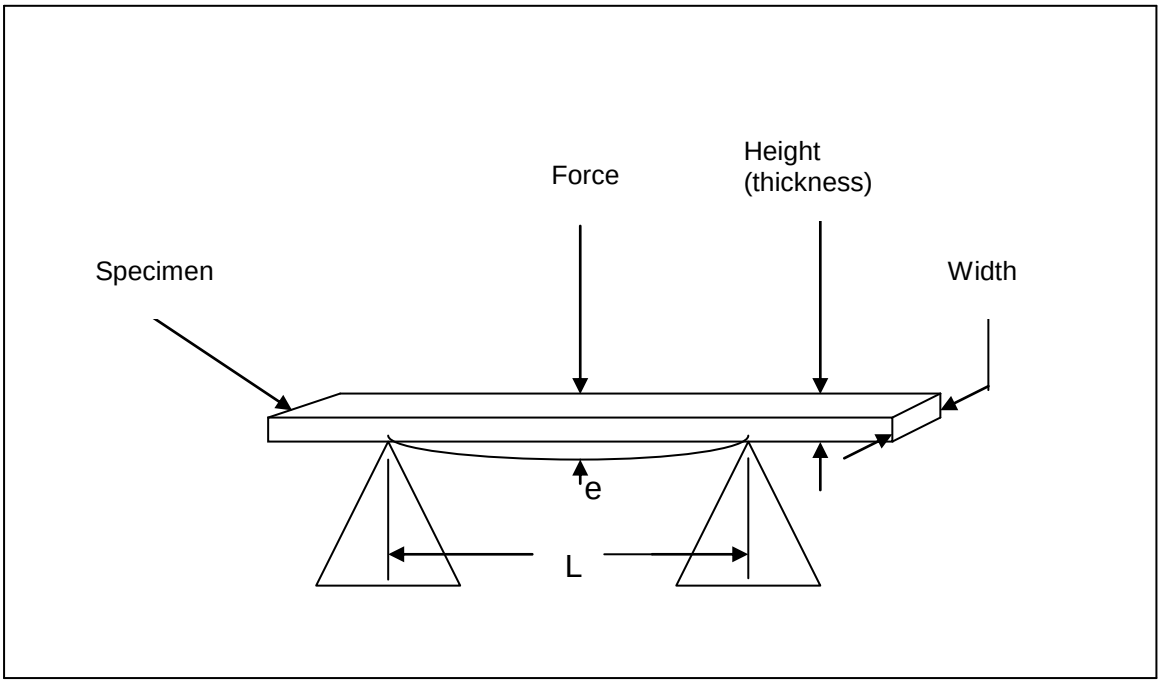


Figure 29 Schematic depiction of the flexural direction of a beam (e) relative to height, width and the distance between the beam support (L)

3.5.1 Cross breaking

Consistency of cross breaking values are increased if the span to depth ratio is greater than ten to one with a 5:1 span to depth being on the border line (Jones, 1983). The specimens were 5.0 mm wide, suspended between 10 mm supports and 2.0 mm thick. The span to depth ratio was greater than 5:1 and cross breaking results were accurate.

3.5.2 Free rotation of specimens

Test specimens should be free to rotate on loaded points, to allow consistent strength measurements without obstruction (Jones, 1983). By allowing the specimens to rest freely on either end of the smooth metal supports, they could freely rotate, so that the fracture strength alone, and no other external restrictive force component, was measured. Since the specimens possessed a smooth finish they could freely rotate with minimal friction, to allow consistent results.

3.6 OBTAINING THE ALUMINA/FELDSPAR COMPOSITE MATERIAL DESIGN

The final aim of this study was to compare all the results (quantitative and qualitative) in order to recommend which alumina/feldspar composite material provided optimum improvements when compared to Group F. Qualitative design comparisons of success were evaluated by surface analysis of the pore size of each specimen group.

Quantitative comparisons were made with data that resulted from wear, flexural strength and flexibility values for each specimen group. The data were analyzed and interrelated using the one-way ANOVA to compare the performance of each specimen group with SR ADORO®. Ad hoc statistical analysis allowed comparisons between all specimens groups. The specimen group that provided the lowest wear, highest flexural strength and lowest flexure values was considered as compared to SR ADORO® for potential commercial use.

3.7 THE DATA

3.7.1 Primary data

In order to design and evaluate alumina/feldspar composite materials the following data were obtained:

(i) Magnified surface imaging of SR ADORO® and alumina/feldspar composite materials

(ii) Wear values (mm) of alumina/feldspar composite materials for four variations in chemical (feldspar) bond

(iii) Wear values (mm) of alumina/feldspar composite materials for two variations in adhesive bonding agents

(iv) Wear values (mm) of alumina/feldspar composite materials without (feldspar or adhesive) bonding agents

(v) Wear, flexibility and flexural strength values for SR ADORO® commercial composite material (the control)

(vi) Strength values for three variations in chemical (feldspar) bonding between the alumina particles of alumina/feldspar composite materials

(vii) Flexibility values for three variations in chemical (feldspar) bonding between the alumina particles of alumina/feldspar composite materials.

The data were analyzed and interrelated using the one-way ANOVA and Post Hoc statistical methods in order to establish comparable values for wear resistance, strength and flexibility. Comparable values with SR ADORO® were required in

order to establish potential wear, flexibility and flexural strength success of alumina/feldspar composites (experimental materials).

3.7.2. Criteria governing the admissibility of data

The following criteria were applied to the data collection:

(i) Only data recorded by the following research equipment were used:

- Universal Minimet® machine (Buehler Ltd. USA)
- Instron® 44 testing machine (Apollo Scientific cc, South Africa)
- Nikon® SMZ800 Dissecting Microscope (Nikon, South Africa)

(ii) Only alumina composite specimens made by the researcher in the dental laboratory at the Dental Services Department, DUT, were used

(iii) Only SR ADORO® composite tabs manufactured and supplied by Ivoclar (Germany) were used to produce the SR ADORO® (control) specimens.

3.8 RATIONALE FOR EXPERIMENTAL ALUMINA/FELDSPAR RESIN INFILTRATED DENTAL COMPOSITES

Alumina has successfully been used to strengthen dental ceramics for decades. Since as early as 1965, Jones (1983) incorporated up to 50% alumina in feldspar in order to strengthen the core material for full ceramic crowns (Fleming *et al*., 2005). From the literature on alumina/feldspar resin infiltrated dental composites it appears that this is the first time these experimental materials have been designed and evaluated.

Wear, flexural strength and flexibility of alumina/feldspar composite materials were evaluated and compared to SR ADORO®. Before commencing with this study Targis (Ivoclar Vivadent) was one of the most wear resistant dental composite materials. During the design of the alumina/feldspar resin material, Ivoclar Vivadent introduced SR ADORO® onto the global market. The increased wear resistant properties of SR ADORO® warranted using this material as a control. The challenge for alumina feldspar composites to improve on wear resistant properties of a commercial dental composite was increased by using SR ADORO® material.

3.8.1 Rationale for choice of SR ADORO®

SR ADORO® was evaluated as a control because it was the latest commercial dental composite material (for the duration of this study) that showed improved wear resistant properties over Signum®, Solidex®, Gradla®, Dialog®, belleGlass®, Enamel plus®, Cristobal®, Targis®, and Sinfony® dental composite materials

(Lendenmann 2003). Primary data gathered from SR ADORO® material was compared to the alumina/feldspar resin infiltrated materials to evaluate their potential for success as dental composite materials.

The SR ADORO® replaced Targis® material (Ivoclar, Lichtenstein) which will not be available in future, as a result of deteriorating surface structure from time related function.[17] The wear resistant properties of the SR ADORO® composite material has been improved on in comparison to Targis and other dental composite materials. Without primary data on SR ADORO® accurate comparisons of alumina/feldspar with commercial composite materials to determine potential decrease in wear would not have been possible.

SR ADORO® was chosen as a control because it performed well above the ADA standard specifications for wear resistance. The importance of choosing a composite with high wear resistance is seen because relatively high acceptable wear rates made it easy for most dental composites to comply with the ADA requirements (Van Niewenhuysen *et al*., 2003). These requirements do not consider the importance of striving to meet optimum wear resistance of proven restorative materials such as gold and amalgam and suggest inferior acceptable limits are accepted (Van Niewenhuysen *et al*., 2003).

3.8.2 Rationale for choice of specimen design

The need to test wear of composites in stress bearing, cusp design (Willem *et al*., 2005; Hardy, 2001; Anusavice, 2003; Ferracane, 2006) was considered, during specimen design. Specimens were orientated so that wear of a 45^0 angled

[17] Personal communication with Gary Hockley (product representative), Ivodent, Cape Town.

specimen resembling a 90^{o} cusp angle could be evaluated (Fig 27a). The extent to which a cusp would resist being worn flat is an important consideration to maintain desirable occlusal function (Hardy, 2001).

3.8.3 Rationale for choice of Alumina filler

Alumina filler was used in the alumina/feldspar composite design as a result of the need to test performance of non-etchable composite fillers (Matinlinna *et al*., 2006). Alumina is an opaque, white material that is ideal for posterior aesthetics. The alumina/feldspar composite materials were selected in order to increase wear resistance of dental composite materials. Alumina was selected in order to evaluate a non-etchable filler material with high wear resistant and strength improving properties. Properties of alumina were further desirable because it is a stable material that does not shrink or melt when fired to 1100^{o}C (This is an important consideration in the design of the experimental material since feldspar is required to melt at 1100^{o}C). Alumina particles therefore could remain porous and permit UDM resin infiltration.

3.8.4 Rationale for using the Minimet® polishing machine

The patented Minimet® polishing machine (Buehler LTD, Illinois, USA) was used in consideration of the need to explore reliable *in vitro* wear simulators. Confidence in the performance of *in vitro* wear simulators for ISO standards needs to be established (Mandikos *et al*., 2001; Heintze *et al*., 2005; Ferracane, 2006).

3.8.5 Rationale for including different bonding agents

The need to provide improved bonding agents for dental composites (Shajii and Santerre, 1999; Yoshida *et al*., 2002; Zandinejad *et al*., 2006) was investigated by incorporating Silane, phosphate and feldspar bonding agents into the alumina/feldspar composite design.

Silane and SR Link® adhesive bonding agents (Ivoclar Lichtenstein) were used to determine their influence on the wear of alumina/feldspar composite specimens. It must be noted that SR Link® was not developed for ceramic to resin adhesion, or for the purpose that it was used for in this study but was developed to improve resin composite adhesion to a metal surface. Biological safety of its use for the purpose of this study has not been established. SR Link® was used because silane bonding agents should be improved on since they deteriorate with time, but no other bonding agent has been introduced as a silane replacement for composites. Although SR Link® would be used outside of its intended purpose, if used to replace silane in dental composite materials, it may with the restrictive use of silane be worth testing as a more stable composite coupling agent simply because there are no alternatives. Monobond S® and SR Link® adhesive coupling was further included so that adhesive bonding benefit could be compared to benefit from feldspar chemical bonding.

3.8.6 Rationale for using the Instron® 44 testing machine

The need to consider flexibility and flexural strength in order to prevent marginal leakage (Ferracane and Condon, 1999; Lawn *et al*., 2004; Kahler *et al*., 2006) resulted in the inclusion of flexibility and flexural strength tests using the universal ISO 10477 Instron® 44 testing machine.

3.8.7 Rationale for choice of Vitadur® porcelain

Vitadur® porcelain (Nova Dental, Johannesburg) was used to incorporate feldspar bonding and vary the degree of chemical bond between alumina particles. The Coefficient of thermal expansion (CTE) of Vitadur feldspathic porcelain was compatible with alumina. Finding a material with compatible CTE was a major obstacle.

3.8.8 Rationale for choice of urethane dimethacrylate resin (UDM)

The urethane dimethacrylate resin (UDM) (Ivoclar Lichtenstein) was used because it was in liquid form that enabled infiltration through the porous alumina/felspar material. UDM is more desirable than bis GMA because bis-GMA polymers possess higher leachable amounts of unreacted monomer, while UDMA mixtures result in more crosslinking than the bis-GMA mixtures (Floyd and Dickens, 2006).

3.8.9 Rationale for using the Rosaduna®, granite opposing surface

The specimens were subjected to a polished Rosaduna®, granite surface (Stone of age, Durban) for wear analysis. Rosaduna® was chosen since it is extremely wear resistant and maintained a smooth consistent surface that typified a smooth ceramic surface. Producing an opposing surface of the required size (for Minimet® polishing) with dental porcelain was not possible with the standard laboratory equipment available as a result of feldspar shrinkage.

3.9 STATISTICAL METHOD

The statistical method applied for amount of wear, flexural strength and flexure was the univariate analysis of variance (One-way ANOVA analysis comparing each group with SR ADORO®). Post hoc tests were conducted to evaluate the results between groups. The univariate analysis of variance allowed comparisons of each group with the standard set by SR ADORO®.

The following statistical criteria were included:

- Those specimen groups that had mean wear values less than the SR ADORO® group met the required wear resistance sought in this study
- Those specimen groups that had mean flexural strength values higher than the SR ADORO® group met the required flexural strength sought in this study
- Those specimen groups that had mean flexibility values lower than the SR ADORO® group met the required flexibility sought in this study

The level of significance for all test was $\propto$ = 0.05. The p – values were used to establish significant difference in values in order to make the decision (Thomas, 2001).

According to Leedy (1985:195-196) the coefficient of variation (CV) indicates the relative magnitude of the Standard Deviation (SD) as compared with the mean of the distribution of measurements as a percentage. Thus the formula is: CV= SD x 100/Mean. The coefficient of variation is useful to compare variability of two data sets relative to the general level of values (and thus relative to the mean in each set). These values help establish the standard of manufacture and test performance of specimens.

CHAPTER FOUR

RESULTS AND DISCUSSION

How you live today, will influence the way you live for the rest of your life

4.1 WEAR TEST RESULTS

The wear test results for all 7 sample groups and the control group are summarized in Appendix A. Table 4 depicts the mean wear value (mm) standard deviation (SD) and coefficient of variance (CV) of the control sample group and the 7 alumina feldspar sample groups (overall ANOVA p value < 0.01).

	Group A	Group B	Group C	Group D	Group E	Group F	Group H	Group I
Mean	**3.06**	**2.43**	**1.11**	**2.55**	**2.31**	**1.38**	**1.02**	**1.37**
SD	**0.35**	**0.20**	**0.16**	**0.29**	**0.17**	**0.15**	**0.05**	**0.11**
CV	**11.43**	**8.23**	**14.41**	**11.37**	**7.35**	**10.86**	**4.90**	**8.02**

Table 4 Mean wear value, standard deviation (SD) coefficient of variance (CV) and *p* value of alumina/feldspar and SR ADORO® composites

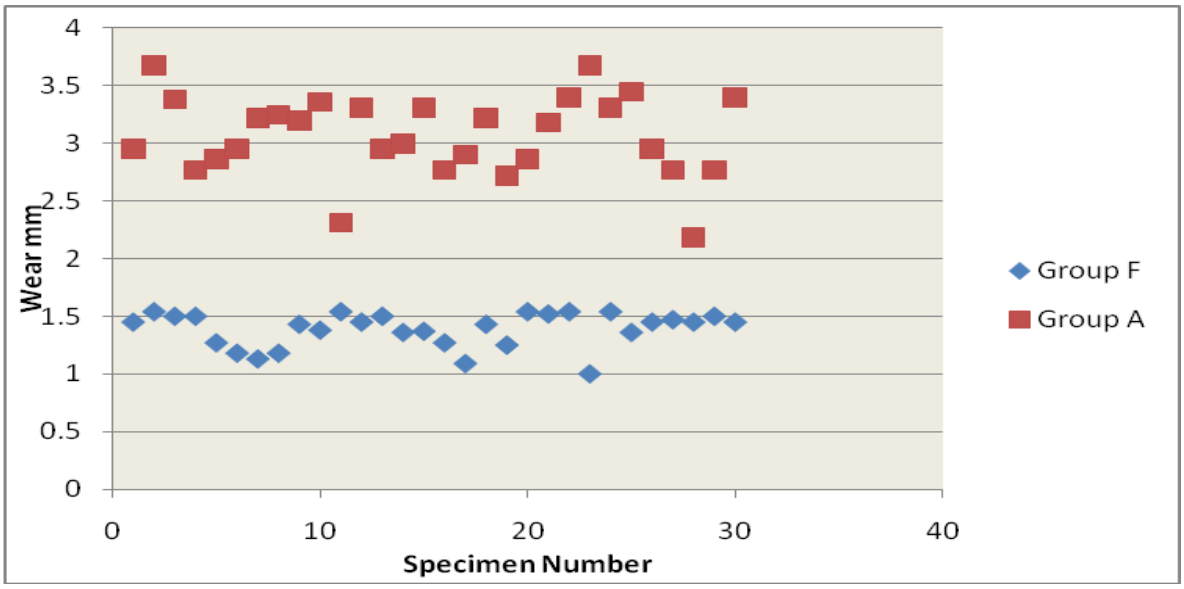
Comparison of post hoc *p* values for wear of all 7 sample groups and the control group are given in Table 5.

(I) Group	(J) Group	*p* value
F	**A**	0.00
F	**B**	0.00
F	**C**	0.00
F	**H**	0.00
F	**I**	0.73
A	**C**	0.00
A	**D**	0.00
B	**C**	0.00
B	**D**	0.03
B	**E**	0.03
C	**D**	0.00
C	**H**	0.15
C	**I**	0.00
D	**E**	0.00
H	I	0.00

Table 5 Comparing post hoc *p* values for wear

4.1.1 Wear values of the sintered alumina composites (Group A) as compared to SR ADORO® (Group F)

Graph 2 depicts the wear values (mm) of sintered alumina resin infiltrated composite material specimens with silane bonding (Group A) with SR ADORO® composite specimens (Group F).



Graph 2 Wear (mm) of Sintered alumina (Group A) and SR ADORO® (Group F) specimens

Group A provided data on the wear (mm) of the sintered alumina composite specimens when a silane bonding agent was incorporated with the sintered alumina resin infiltrated composite material. The wear values of Group A specimens were compared to wear values of SR ADORO® composite specimens. Success could only be attributed to the alumina/feldspar material design if lower mean wear values than the SR ADORO® composite specimens were obtained.

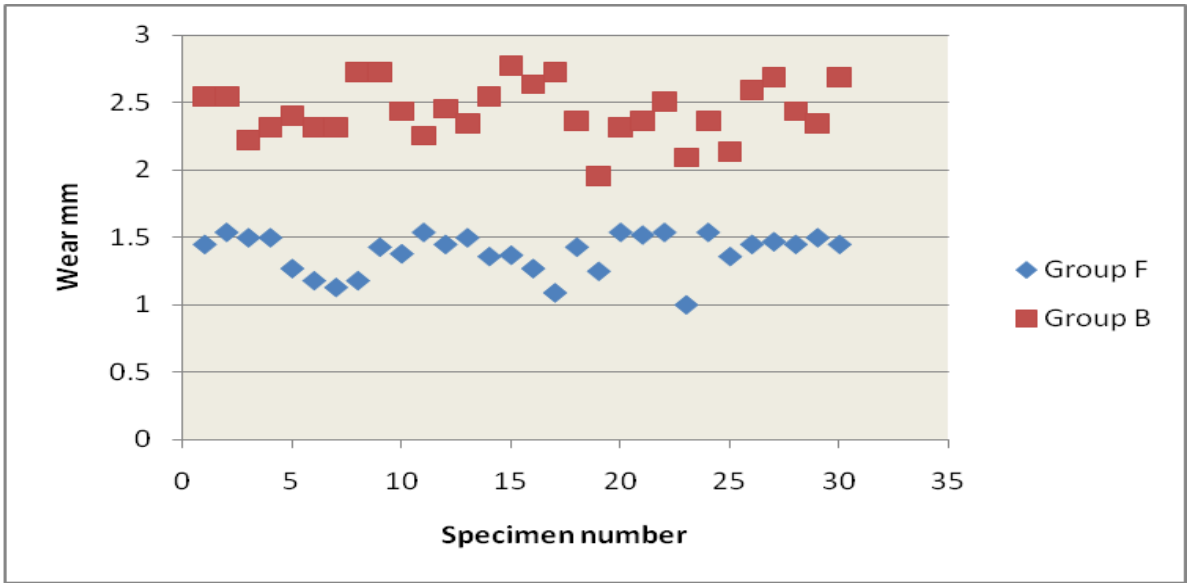
The SR ADORO® group gave a mean result of 1.38 mm. This value was much lower than the mean result of 3.06 mm for the sintered alumina, silanized specimen group (p=0.00). From Graph 2 can be seen that the sintered alumina, silanized specimens had more than double the amount of wear than the SR ADORO® group. The Sintered alumina, silanized specimens (Group A) did not provide lower wear values than the Commercial SR ADORO® specimen group (Table 4 and Table 5). Group A therefore did not achieve the success desired.

The coefficient of variation (CV) of Group A (11.43%) was close to the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens was therefore possible with the Minimet® polishing machine.

Since the aim to increase wear resistance of SR ADORO® was not achieved this material was not subjected to further evaluation. The wear (Appendix, Plate 13) of Group A wear can be visualized in comparison to that of SR ADORO® (Appendix, Plate 14). The high wear of Group A can be understood through literature that reports a poor silane bonding mechanism between resins to non etchable filler materials such as zirconia and alumina (Matinlinna *et al*., 2006).

4.1.2 Wear of alumina/feldspar resin infiltrated composite with no adhesive bonding agent and 30% feldspar (Group B) as compared to SR ADORO® (Group F)

Graph 3 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with no adhesive bonding agent and 30% feldspar (Group B) composite specimens with SR ADORO® (Group F) composite specimens.



Graph 3 Wear (mm) of alumina/feldspar with no adhesive bonding agent and 30% feldspar (Group B) and SR ADORO® (Group F) specimens

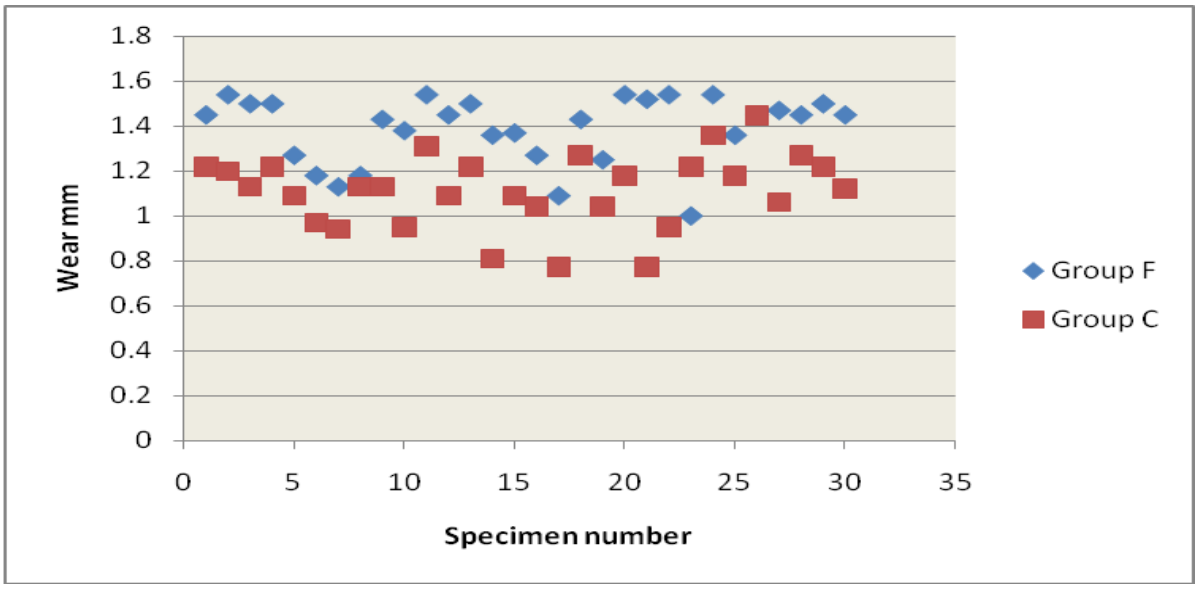
The SR ADORO® (Group F) gave a mean result of 1.38 mm. This value was lower than the mean result of 2.43 mm for the Group B specimens (p=0.00). From Graph 3 can be seen that the Group B specimens wear values were all below that of the SR ADORO® specimen group. Group B did not achieve the success desired.

The coefficient of variation (CV) of Group B (8.23%) was lower than the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens

were possible with the Minimet® polishing machine. The aim to improve on wear resistance of SR ADORO® was not achieved (Appendix, Plate 15).

4.1.3 Wear of alumina/feldspar resin infiltrated composite with silane and 60% feldspar (Group C) as compared to SR ADORO® (Group F)

Graph 4 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with silane and 60% feldspar **(**Group C) composite specimens with SR ADORO® composite specimens (Group F).



Graph 4 Wear (mm) of alumina/feldspar with silane and 60% feldspar (Group C) and SR ADORO® (Group F) specimens

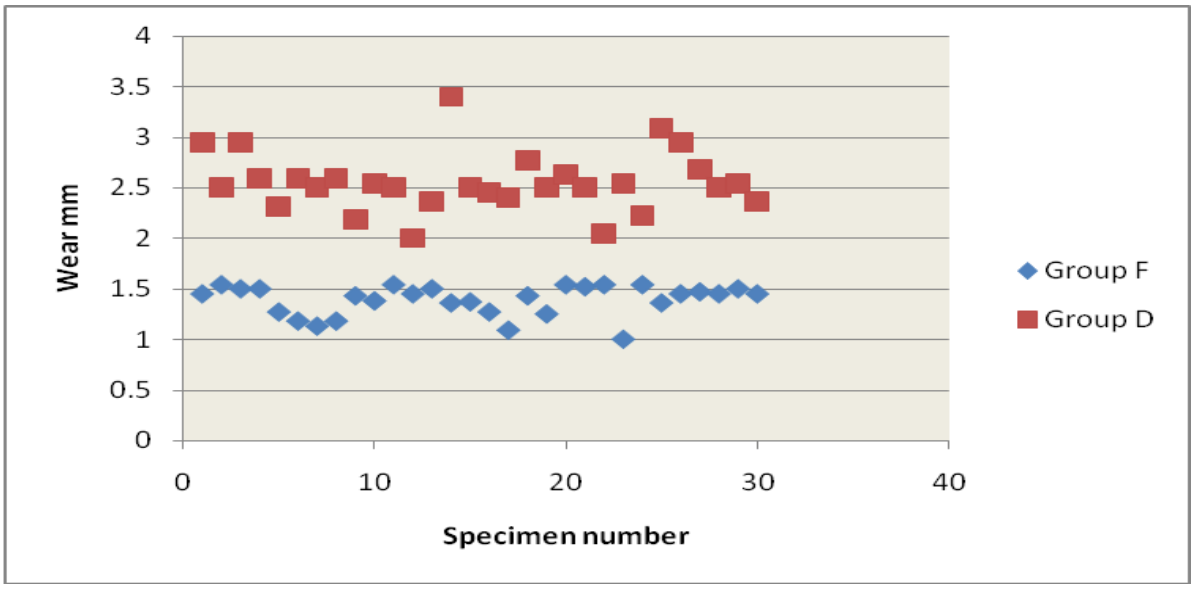
SR ADORO® (Group F) gave a mean result of 1.38 mm. This value was higher than the mean result of 1.11 mm for the Group C specimens (p=0.00). From Graph 4 can be seen that the majority of Group C specimens wear values were lower than that of the SR ADORO® (Group F) specimens. Group C therefore did achieve the success desired.

The coefficient of variation (CV) of Group C (14.41%) was higher than the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens was possible with the Minimet® polishing machine.

The aim to increase wear resistance as compared to SR ADORO® by incorporating 60% feldspar mass and silane with the alumina/feldspar resin infiltrated composite material was reached (Appendix, Plate 16). As a result this material was subjected to further evaluation of flexural strength and flexibility after which recommendations of this material could be made for commercial production.

4.1.4 Wear of alumina/feldspar resin infiltrated composite with silane and 30% feldspar (Group D) as compared to SR ADORO® (Group F)

Graph 5 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with silane and 30% feldspar **(**Group D) composite specimens with SR ADORO® (Group F) composite specimens.



Graph 5 Wear (mm) of alumina/feldspar with silane and 30% feldspar (Group D) and SR ADORO® (Group F) specimens

The SR ADORO® group gave a mean result of 1.38 mm. This value was much lower than the mean result of 2.55 mm for the Group D specimens (p=0.00). From the Graph 5 can be seen that the Group D specimens wear values were all higher than that of the SR ADORO® specimen group. Group D did not achieve the success desired.

The coefficient of variation (CV) of Group D (11.23%) was close to the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens were possible with the Minimet® polishing machine.

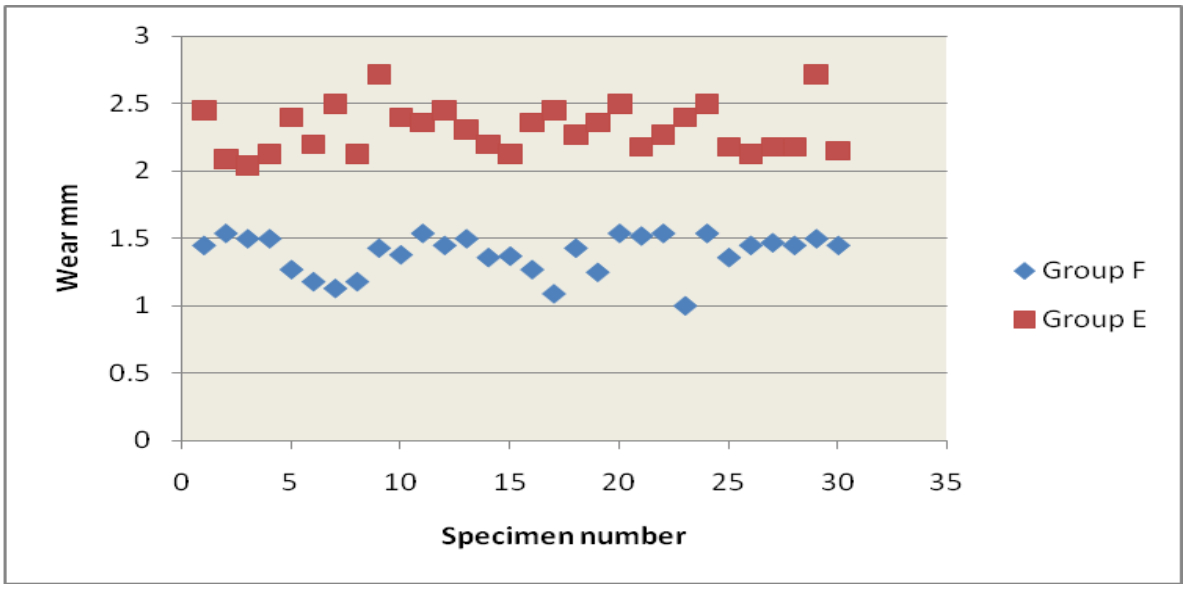
The aim to increase the wear resistance as compared to SR ADORO® by incorporating 30% feldspar mass and silane with alumina/feldspar resin infiltrated composite material was not reached. The mean wear value of Group D was higher

than the mean wear value of SR ADORO® and commercial benefit is not expected from this material.

Some wear resistance was derived from the 30% feldspar mass since the mean wear value was lower than the sintered alumina resin infiltrated material with silane bonding (Group A) when compared to SR ADORO®, but no benefit was gained by including silane. This reinforced the earlier conclusion that alternative adhesive bonding agents to silane might be required for bonding resin to alumina (Matinlinna *et al*., 2006). The wear results of these specimens were similar to that of Group B with 30% feldspar mass and no silane (Appendix, Plate 17).

4.1.5 Wear of alumina/feldspar resin infiltrated composite with SR link® and 30% feldspar (Group E) as compared to SR ADORO® (Group F)

Graph 6 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with SR link® and 30% feldspar (Group E) composite specimens with SR ADORO® (Group F) composite specimens.



Graph 6 Wear (mm) of alumina/feldspar with SR Link® and 30% feldspar (Group E) and SR ADORO® (Group F) specimens

The SR ADORO® group gave a mean result of 1.38 mm. This value was much lower than the mean result of 2.31 mm for the Group E specimen group (p=0.00). From Graph 6 can be seen that the Group E specimens wear values were all higher than that of the SR ADORO® group. Group E therefore did not achieve the success desired.

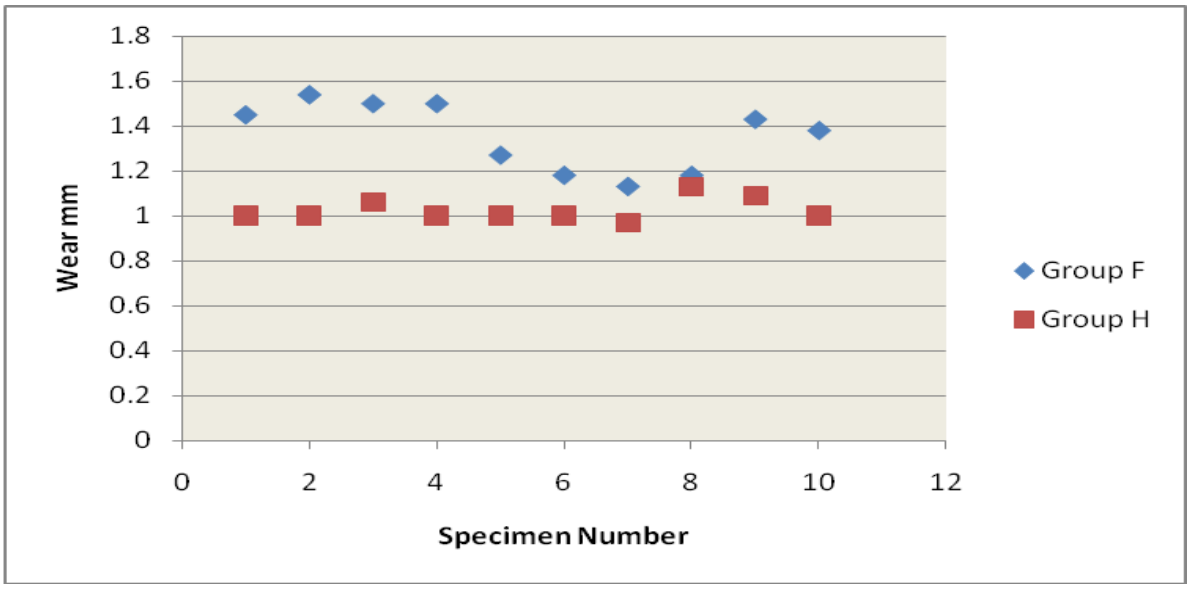
The coefficient of variation (CV) of Group E (7.35%) was lower than the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens were possible with the Minimet® polishing machine.

Although SR Link® was developed for a purpose other than was to be used within a dental composite material, few other adhesive bonding agents other than silane have been used for dental composites. SR Link® is a more stable bonding agent

than silane. Replacing SR ADORO® with SR Link® was done because the literature provided did not appear to report on the potential performance of SR Link® as an adhesive bonding agent within dental composite materials. The aim to increase wear resistance as compared to SR ADORO® by incorporating 30% feldspar mass and a phosphate bonding agent with the alumina/feldspar resin infiltrated composite was not reached and Group E was not subjected to further evaluation. The wear results of these specimens were similar to that of Group B and D with 30% feldspar mass indicating that the feldspar and not the adhesive bonding agent was the main contributing factor to wear resistance of these materials (Appendix, Plate 20).

4.1.6 Wear of alumina/feldspar resin infiltrated composite with silane and 50% feldspar (Group H) as compared to SR ADORO® (Group F)

Graph 7 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with silane and 50% feldspar (Group H) composite specimens with SR ADORO® (Group F) composite specimens.



Graph 7 Wear (mm) of alumina/feldspar with silane and 50% feldspar (Group H) and SR ADORO® (Group F) specimens

The SR ADORO® group gave a mean result of 1.38mm. This value was higher than the mean result of 1.02mm for Group H (p=0.00). From Graph 7 can be seen that all of the Group H specimens wear values were lower than that of the SR ADORO® specimen group. Group H therefore did achieve the success desired.

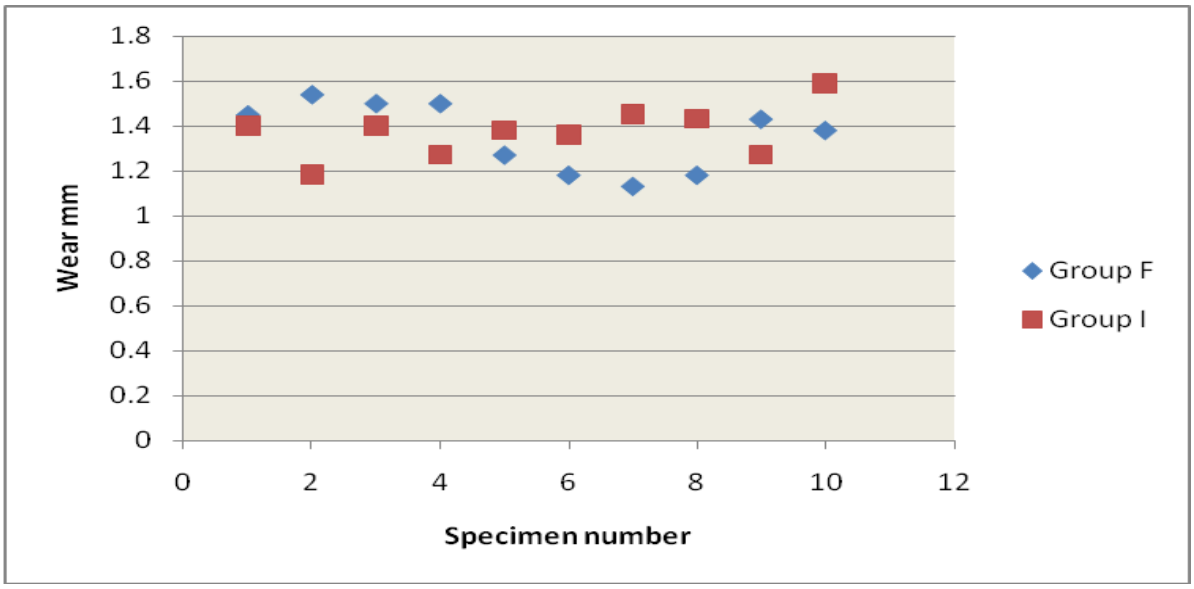
The coefficient of variation (CV) of Group H (4.90%) was lower than the CV of Group F (10.86%). Consistent manufacture and wear performance of specimens were possible with the Minimet® polishing machine. The low CV may be attributed to equal feldspar to alumina mass distribution that would maintain even bond strength throughout the specimen thereby reducing weak bonded areas that would increase the CV.

The aim to increase wear resistance as compared to SR ADORO® commercial composite material by incorporating 50% feldspar mass and silane with the

alumina/feldspar resin infiltrated composite material was reached. As a result this material was subjected to further evaluation of flexural strength and flexibility. The specimens with 50% feldspar and 50% alumina provided a good pore size for infiltration and a smooth worn surface without extrusion of resin or ceramic particles. For this reason manufacture techniques of these specimens were more desirable than for the 60% feldspar specimens (Group C). The ceramic and resin contribution of these specimens were therefore optimized and commercial use of this material is expected to be of benefit as a dental restorative material. The low of wear of Group H can be observed in Plate 18 of the Appendix.

4.1.7 Wear of alumina/feldspar resin infiltrated composite with silane and 40% feldspar (Group I) as compared to SR ADORO® (Group F)

Graph 8 depicts the wear values (mm) of alumina/feldspar resin infiltrated composite with silane and 40% feldspar (Group I) composite specimens with SR ADORO® (Group F) composite specimens.



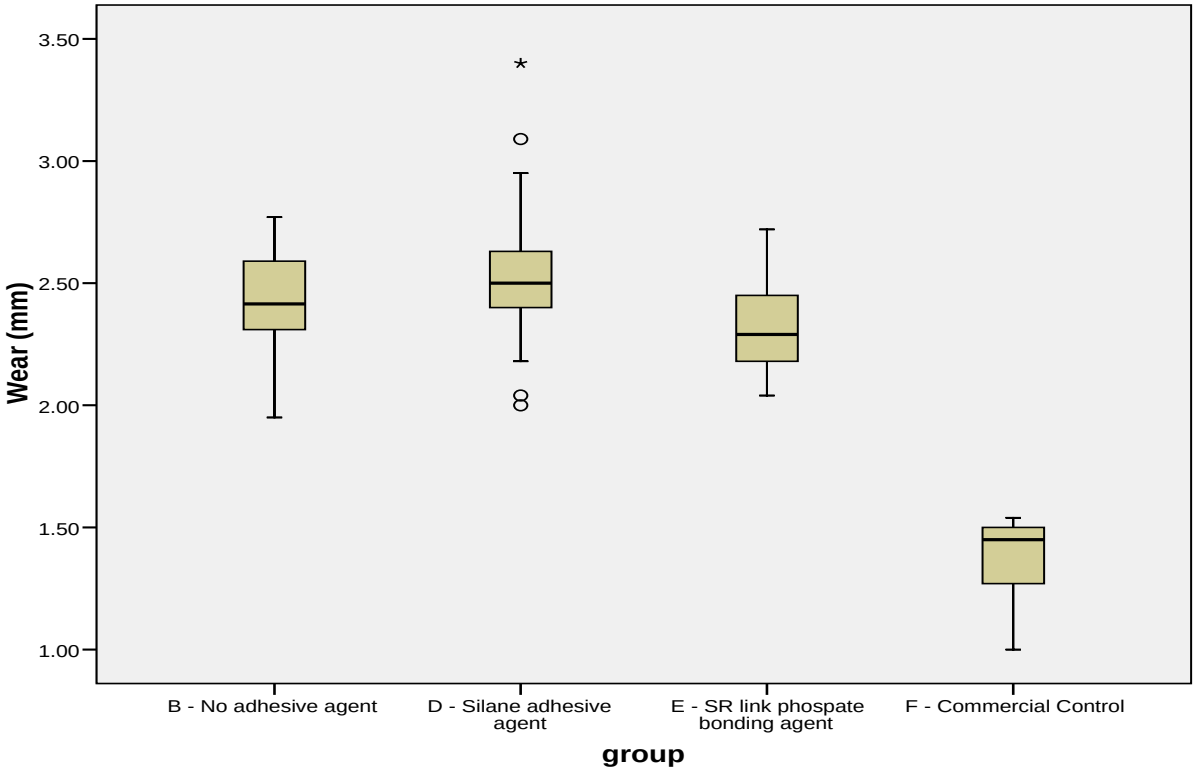
Graph 8 Wear (mm) of alumina/feldspar with silane and 40% feldspar (Group I) and SR ADORO® (Group F) specimens

The SR ADORO® group gave a mean result of 1.38mm. This value was similar to the mean result of 1.37mm for Group I (p=0.73). From Graph 8 can be seen that Group I had half the number of specimens with lower wear values than Group F. Group I did not achieve the success desired since a significantly lower mean wear value was not obtained (Appendix, Plate 19).

The aim to increase wear resistance as compared to SR ADORO® by incorporating 40% feldspar mass and silane with the alumina/feldspar resin infiltrated composite material was not reached. Since half the wear values of Group I were higher than Group F this material was subjected to further evaluation. It was hoped that if this material possessed equal or greater flexural strength and lower flexibility than SR ADORO® that commercial benefit could be considered.

4.1.8 Wear of 30% feldspar mass (Group B), 30% feldspar mass with silane (Group D), and 30% feldspar mass with SR Link® (Group E) resin infiltrated composites as compared to SR ADORO® (Group F)

Graph 9 depicts the median (50th percentile), 25th and 75th percentiles (interquartile range) as well as the range of values for Group B, Group D, Group E and Group F (SR ADORO®) composite specimens respectively.



Graph 9 Wear of no adhesive agent (Group B) , silane adhesive agent (Group D) and SR link phosphate bonding agent (Group E) as compared to the SR ADORO® commercial control (Group F)

The SR ADORO® (Group F) gave a mean wear result of 1.38mm (Table 4) This value was lower ($p=0.00$) than the alumina sintered/silanized and adhesive bonded Groups B (2.43mm), Group D (2.55mm) and Group E (2.31mm). The alumina/feldspar material with phosphate bonding (Group E) did not significantly improve on the wear resistance properties of the SR ADORO® commercial control (Group F).

Significant wear difference ($p<0.05$) were observed between the sintered alumina (Group B), silane adhesive bonding (Group D) and the phosphate adhesive bonding (Group E). Group E gave the lowest mean wear value showing increased wear resistance compared to Group B with no adhesive bonding, while Group D had the highest mean wear value.

From comparisons of the mean values, SR Link® does seem to afford some benefit to improve wear resistance, however the wear benefit is relatively small (Graph 9). Although the aim to increase wear resistance by including SR Link® might be realized it is too early to make recommendations before additional data on the alumina/ SR Link® bond is provided. There appears to be no literature to support the use of SR Link® to replace silane when used with alumina and as an experimental material further testing would be required before SR Link® may safely be used for this purpose.

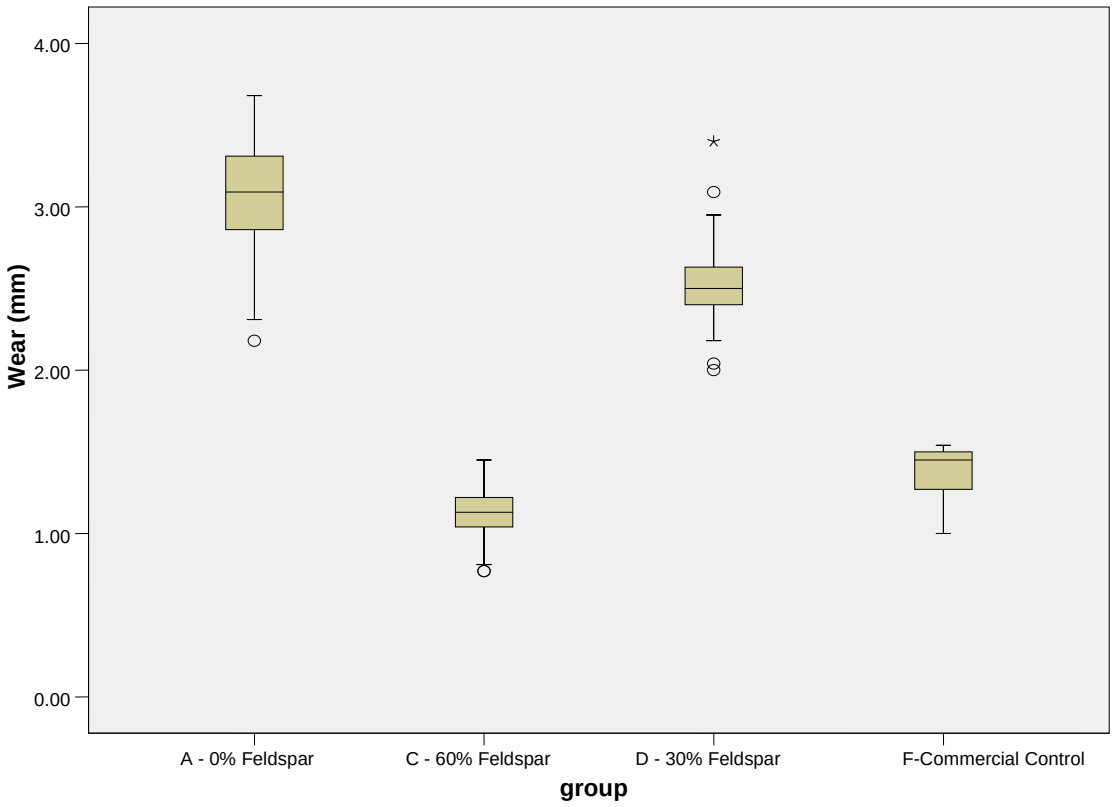
The addition of silane with 30% feldspar appears to have reduced the bond to alumina filler since Group B provided more wear resistance than Group D. There is therefore no benefit in using silane with alumina feldspar composites that include

30% feldspar but there is rather a concern that it may reduce the wear resistance of this material. This result supports findings in literature that the silane alumina bond is not well understood and does not contribute to bond strength in the way that silane bonding to glass does (Matinlinna *et al*., 2006).

Wear differences between groups when adhesive bonding agents were tested ($p<0.05$) were small when compared to the wear difference between groups when feldspar content was varied. Adhesive bonding agents therefore are expected to have a small influence in increasing wear resistance in alumina/feldspar dental composites where 30% to 60% feldspar is included.

4.1.9 Wear of feldspar mass variations with silane (Group A, Group C, Group D) as compared to SR ADORO® (Group F)

Graph 10 depicts the median (50^{th} percentile), 25^{th} and 75^{th} percentiles (interquartile range) as well as the range of values for Group A, Group C, Group D and Group F (SR ADORO® commercial control) composite specimens respectively.



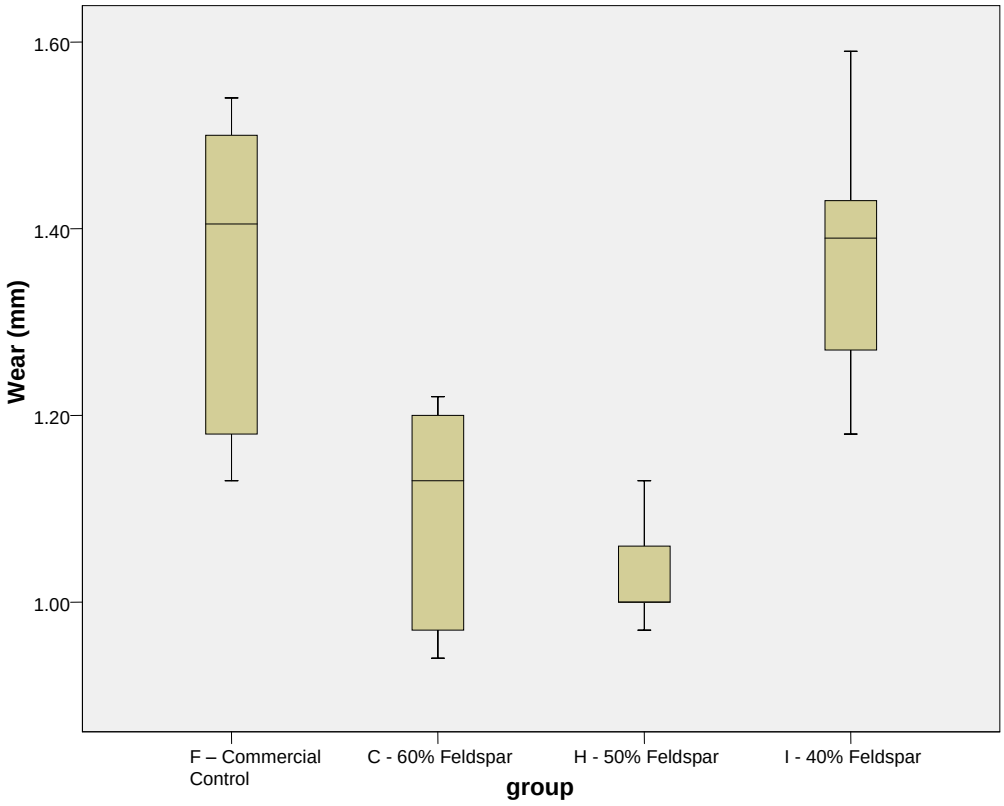
Graph 10 Wear of 0% feldspar (Group A), 60% feldspar (Group C) and 30% feldspar (Group D) as compared to the SR ADORO® commercial control (Group F)

The SR ADORO® group gave a mean wear result of 1.45mm. This value was lower than the alumina/feldspar and adhesive bonded specimen Group A (3.06mm) and Group D (2.55mm) but higher than the wear value for Group C (1.12mm). From Graph 10 and Graph 11, it can be seen that the alumina/feldspar material with 60% feldspar mass (Group C) did improve on the wear resistance properties of the commercial SR ADORO® (Group F) ($p < 0.001$), especially in the lower wear range. The lower wear range improvement of Group C may warrant investigation, to see if the wear of alumina/feldspar composites can be reduced further.

The aim to increase the wear resistance of alumina/feldspar resin infiltrated composite materials as feldspar mass increments were included was reached. The wear resistance did increase significantly (P<0.001) from 0% to 30% and 60% feldspar mass. Although no reports on alumina/feldspar dental composites were obtained from literature sought, the results show that feldspar bonding has the potential to contribute to wear resistance of resin infiltrated dental composites.

4.1.10 Wear values of Group C, Group H, Group I and Group F

Graph 11 depict the median (50th percentile), 25th and 75th percentiles (interquartile range) as well as the range of values for Group C, Group H, Group I and Group F (SR ADORO® commercial control) respectively.



Graph 11 Wear of 60% feldspar (Group C), 50% feldspar (Group H) and 40% feldspar (Group I) as compared to the SR ADORO® commercial control (Group F)

The SR ADORO® commercial control (Group F) gave a mean result of 1.35mm. This value was higher ($p<0.001$) than the alumina/feldspar specimen Groups H (1.02mm) and C (1.09mm) and similar ($p=0.73$) to the value for Group I (1.37mm). Although Group H provided the greatest mean wear resistance, Graph 11 indicates that the wear range of Group H is within the Group C wear range. Both Group H and Group C appear to possess lower wear properties than what is commercially available (Group F).

A linear increase in wear resistance did not result. Group H gave a lower mean wear value than Group C. Groups C and Group H both provided lower wear values than the SR ADORO® (Group F) with Group H providing the lowest wear values consistently. The aim to increase wear resistance of the alumina/feldspar resin infiltrated composite materials as increments of 10% feldspar mass was increased was not reached since a linear increase in wear resistance did not result. The finding that Group H and not Group C gave the lowest mean wear value was not expected but appears to be as a result of the more desirable design of Group H. Unfortunately literature sought did not appear to elaborate on the reason for 50% alumina providing more consistent wear resistant benefit than 60% alumina except to state that, traditionally up to 50% alumina was added to feldspar to improve strength characteristics (Yamamoto, 1985).

4.1.11 Wear range of alumina/feldspar composites

Wear of alumina feldspar composite (from the results analyzed) can vary markedly between ceramic restoratives and resin restorative materials. The ability to manipulate the mechanical properties of a single restorative material to this extent

is noteworthy and indicative of future research potential for wear improvement and use as a dental restorative material.

The lower wear provided by alumina/feldspar specimens (Graph 4 and Graph 7) as compared to SR ADORO® indicated that the aim of this study was successfully achieved. Surprisingly, even with 30% feldspathic bonding and silane bonding (Group D) the wear values of SR ADORO® were approximately half that of Group D. The feldspar content was increased to 50% mass (Group H) before a significant mean wear value below that of the SR ADORO® was obtained (p-value <0.5). As a result of the significant improvements in wear resistance obtained with SR ADORO® longevity problems of SR ADORO® should be attributed to causes other than wear.

4.2 FLEXURAL STRENGTH TEST RESULTS

Flexural strength test results for 4 sample groups and the control group are summarized in Appendix B.

Table 6 depicts the mean value (N) standard deviation (SD), coefficient of variance (CV) and *p* value for flexural strength (N and MPa) of the control sample group and 3 alumina/feldspar sample groups (overall ANOVA p value < 0.08).

	F-Control (n=10)	**Group C (n=10)**	**Group H (n=10)**	**Group I (n=10)**
Mean	**115.5 (N) (86.6 MPa)**	**119.8 (N) (89.8 MPa)**	**126.7 (N) (95.0 MPa)**	**105.7 (N) (79.3 MPa)**
SD	**19.61**	**19.80**	**17.72**	**13.23**
CV	**16.97**	**16.52**	**13.98**	**12.52**

Table 6 Mean value, standard deviation (SD), coefficient of variance (CV) and *p* value for flexural strength (N and MPa)

The mean MPa results in table 6 are converted values from N values.

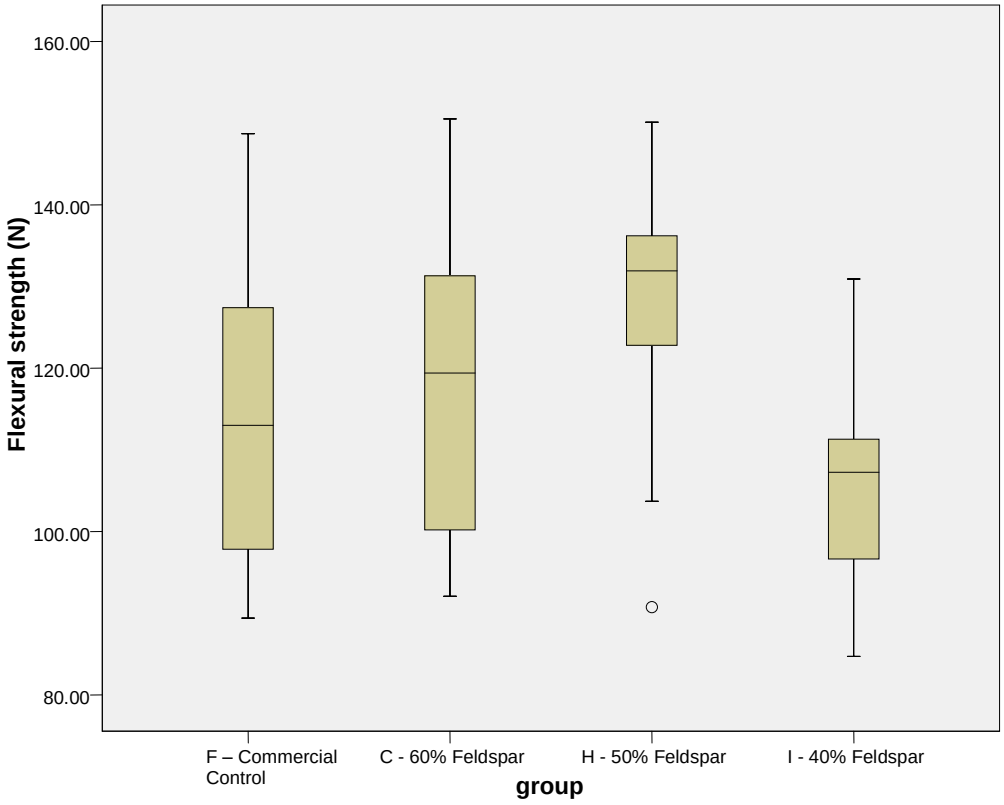
Comparison of post hoc *p* values for 3 sample groups and the control group are given in Table 7.

(I) Group	**(J) Group**	***p* value**
F	**C**	0.59
F	**H**	0.16
F	**I**	0.22
C	**H**	0.39
C	**I**	0.08
H	I	0.01

Table 7 Comparing post hoc *p* values for flexural strength

4.2.1 Flexural strength (N) of alumina/feldspar resin infiltrated composite materials (Group C, Group H and Group I) as compared to the SR ADORO® (Group F)

Graph 12 depict the median (50th percentile), 25th and 75th percentiles (interquartile range) as well as the range of values for Group C, Group H, Group I and Group F respectively.



Graph 12 Flexural strength (N) of alumina/feldspar (Group C, Group H and Group I) and SR ADORO® commercial control specimens (Group F)

The flexural strength of the SR ADORO® commercial control (Group F), was not significantly different (p=0.16) to Group H, Group C (p=0.59) and Group I (p=0.22).

Parametric ranges of sample groups (Graph 12) show that the flexural strength of Group C and Group H were not significantly higher than Group F ($p>0.05$), while the flexural strength of Group I was not significantly lower than Group F ($p>0.05$).

Significant difference in flexural strength ($p=0.01$) was observed between Group H and Group I (Graph 12) indicating that although the fracture perimeters were not significantly different from Group F, there would be some advantage in promoting strength characteristics of Group H rather than Group I (Table 6 and table 7). The mean stress (MPa) comparison provided lower values than (N) and were useful to allow clearer perception of the significant difference between Group H (95.0 MPa) and Group I (79.3 MPa).

The coefficient of variation (CV) of Group C (16.52), Group H (13.98) and Group I (12.52) were comparable to the CV of Group F (16.97). These values indicate consistent manufacture and flexural strength performance of specimens were possible with the Instron® 44 testing machine. The increase in flexural strength of alumina/feldspar composites as compared to Group F was not significant, or as high as was expected and recommendations to improve the strength of alumina/feldspar composites further are encouraged.

Although flexural strength improvements are desirable, the purpose of comparing the flexural strengths of alumina/feldspar composites against SR ADORO® was to establish whether strength values were acceptable when compared to a commercially successful material such as SR ADORO®. It was not the intention of this study to maximize the strength characteristic of this material as wear

properties was the main focus, and the influence of improved resins and curing methods on flexural strength of alumina/feldspar composites were not tested in this study. The inclusion of flexural strength and flexibility testing was to ensure that increased wear resistance of alumina/feldspar dental composites would not impact negatively on these properties should the wear be improved. The flexural strengths are considered further in section 4.9 (comparing design, wear resistance, flexibility and flexural strength of alumina/feldspar sample groups).

4.3 FLEXIBILITY TEST RESULTS

Flexibility test results for 4 sample groups and the control group are summarized in Appendix C.

Table 8 depicts the mean value (mm) standard deviation (SD), coefficient of variance (CV) and *p* value for flexibility of the control sample group and 3 alumina/feldspar sample groups (overall ANOVA p value < 0.01).

	F-Control (n=10)	**Group C (n=10)**	**Group H (n=10)**	**Group I (n=10)**
Mean	**0.38**	**0.19**	**0.17**	**0.22**
SD	**0.06**	**0.09**	**0.10**	**0.05**
CV	**15.78**	**47.36**	**58.82**	**22.72**

Table 8 Mean value, standard deviation (SD), coefficient of variance (CV) and *p* value between groups for flexibility of sample Group C, Sample Group H and sample Group I

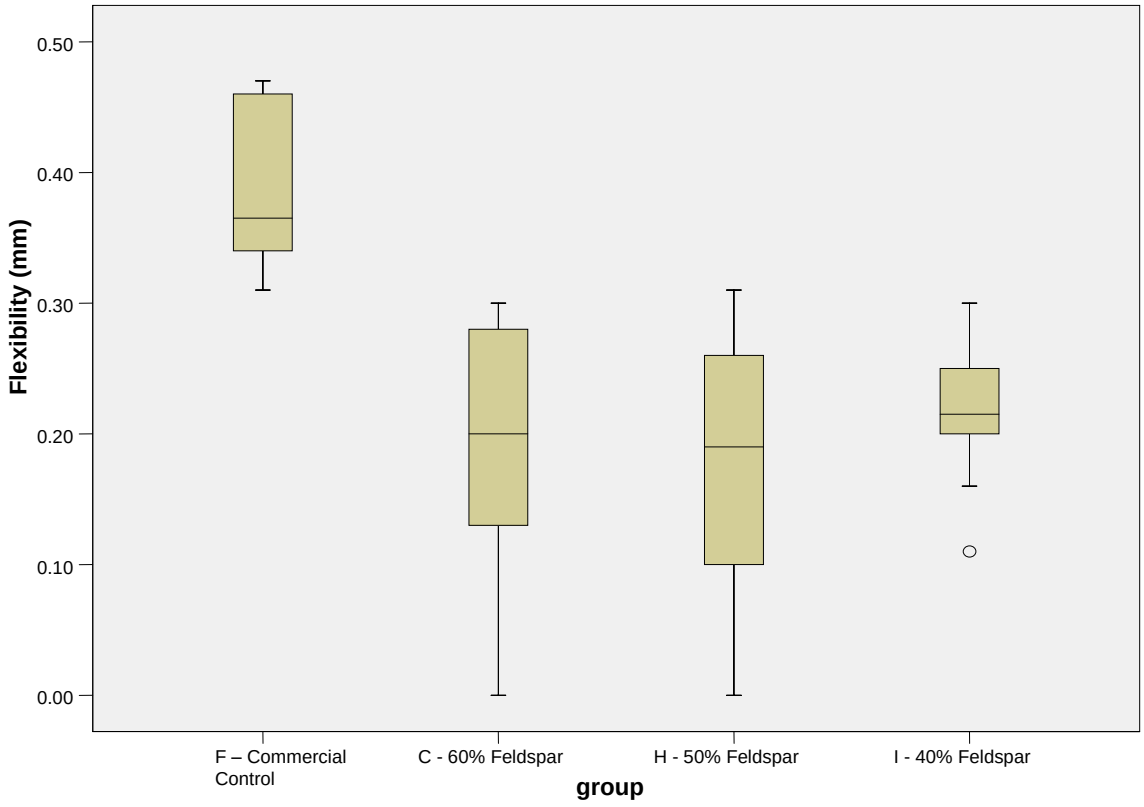
Comparison of post hoc *p* values for 3 sample groups and the control group are given in Table 9.

(I) Group	**(J) Group**	***p* value**
F	**C**	0.00
F	**H**	0.00
F	**I**	0.00
C	**H**	0.67
C	**I**	0.42
H	I	0.22

Table 9 Comparing post hoc *p* values for flexibility

4.3.1 Flexibility of alumina/feldspar resin infiltrated composite materials (Group C, Group H and Group I) as compared to the SR ADORO® (Group F)

Graph 13 depicts the median (50th percentile), 25th and 75th percentiles (interquartile range) as well as the range of values for Group C, Group H and Group I and Group F respectively.



Graph 13 Mean flexibility (mm) of alumina/feldspar and SR ADORO® (Group F) specimens

The SR ADORO® group gave a mean result of 0.38mm. This value was significantly higher (p=0.00) than Group H (0.17mm), Group C (0.19mm) and Group I (0.22). A significant increase in flexural strength did not result between

Group C, and Group H (p=0.39) or between Group C and Group I (p=0.08). Significant decrease in flexibility (Graph 13) did result between Group H and Group I (p=0.01), and can be seen in the lower flexibility rank of Group H. The mean flexibility of all three specimen groups (C, H and I) where virtually half as much as the SR ADORO® (Group F). Non parametric ranks (Graph 13), shows the lower flexibility of the alumina/feldspar specimens (Group C, Group H and Group I) as compared to the SR ADORO commercial control (Group F).

The difference in coefficient of variation (CV) of Group C (47.36), Group H (58.82), Group I (22.72) and Group F (15.78) has been dealt with in the introduction to the discussion. Consistent manufacture and flexibility performance of specimens were problematic with the Instron® 44 testing machine, specimen size and manufacture technique.

Lower flexibility of alumina/feldspar composites as compared to SR ADORO® is expected to result in reduced deformation of the alumina/feldspar composites at the margins of a bridge since bridge design conforms to the law of beams (Shillingburg,1997). Flexibility of each specimen was therefore dependent upon the force, width and distance between the beam support rather than the full length of the beam (Figure 29). The small variations in wear between specimen groups could therefore be ignored during flexural strength and flexibility testing because the worn surface was not positioned between beam supports and the test values were dependent on distance between beam support *(L*) rather than specimen length.

The stiffness of dental composite materials should ideally be higher than or at least match that of the tooth structure to which it is to be bonded in order to withstand compressive, flexural and shear stress (Katz *et al*., 2007). Marginal leakage may occur from flexural and shearing stresses as a result of wear that causes deformation to the bonded composite around the margins (Bonilla *et al*., 2001; Choi and Ferracane, 1999).

The problem with composite restorations is the occurrence of secondary caries as the composite material breaks down around the margins (Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Worm and Meiers, 1996; Behr *et al*., 2005). When a restoration fractures it is simply replaced to prevent further clinical problems. However, when the composite material breaks down around the margins, it is difficult to detect clinical problems such as secondary caries and the health of a tooth may be jeopardized as a result of delayed treatment (Splieth *et al*., 2003). As a result, factors that prevent flexure and increase flexural strength are important considerations for dental restorative materials.

The need to reduce flexibility is acknowledged by Visvanathan *et al*., (2007) who report that, improving flexural modulus and flexural strength will yield better marginal integrity**.** Bridge flexibility and influence on marginal seal can be visualized by viewing forces acting on a beam (representing a bridge) suspended between two stands (representing tooth structure support of the bridge) (Figure 10). Reduced flexibility combined with increased wear and fatigue resistance will

therefore reduce marginal leakage and provide better protection to the prepared tooth structure around the margins. This is important when considering that composite wear reduces the functional efficiency and protective mechanism of adjacent tooth stricture at the margins (Figure 9).

Flexure of composite materials will increase micro leakage around the margins of a poorly adapted composite material as a result of polymerization shrinkage and inadequate cementation (Figure 10). The longevity of flexible dental composites will be greatly reduced if they are not small and adequately protected from cusp concentrated occlusal forces that produce micro-crack fatigue.

Most of the *in vivo* longevity results of composites were found to be within an insufficient evaluation period of three years. Longevity evaluation of dental composites after three years showed a more rapid failure rate (Luo *et al*., 2000; Türkün *et al*., 2003 and Dijken *et al*., 2006). Flexibility of the material should not vary considerably from the opposing material as uneven loading and surface deterioration from wear may result (Figure 6). The problem with micro-filled composites as shown in Plate 6 is that even though the material is wear resistant, these composites crack up under high cuspal (point) loading. Chipping from micro-crack formation at the surface of SR ADORO® (a micro filled composite material) was observed (Appendix, Plate 21).

The success of commercial composites depends largely on placing small restorations in such a way that they are not exposed to high opposing cusp concentrated forces, as a result of adjacent tooth structure support. From *in vitro*

wear analysis the alumina/feldspar specimens are expected to resist cracking seen in micro-filled composites (Plate 21). From the literature received, no *in vitro* wear tests to date (2007) have been reported to simulate *in vivo* wear completely and the accuracy of the Minimet® polishing machine can only be completely verified if alumina/feldspar restorations perform similarly and do not result in micro cracks (Plate 21) after *in vivo* analysis. *In vitro* analysis has however, provided data that may increase confidence in further development of alumina/feldspar composites for use *in vivo*.

Advantages gained by the alumina/feldspar materials over the SR ADORO® material were: (i) Increased wear resistance and (ii) Reduced flexibility that is expected to improve longevity. Using 50% alumina/feldspar mix (by weight) resulted in improved wear resistance, flexibility, flexural strength and resin infiltration as compared to other alumina feldspar sample groups.

The stiffness of the alumina/feldspar composites were found to be similar to dental ceramic materials because the chemical bond between the alumina particles was formed by dental feldspar. Increased stiffness of alumina/feldspar materials is a desirable property that is expected to improve cementation and longevity of composite materials by increasing the ceramic contribution and reducing the undesirable resin influence (Anusavice, 2003).

4.4 DESIGN OF ALUMINA /FELDSPAR COMPOSITE MATERIALS

The design of alumina/feldspar resin infiltrated composite materials included a chemical bond (by incorporating feldspar or sintering alumina) in order to optimize wear resistance of dental composite materials. The alumina/feldspar design was evaluated by its ability to produce a porous alumina core material (Plate 1 and Figure 30) that could be resin infiltrated without resultant processing cracks occurring.

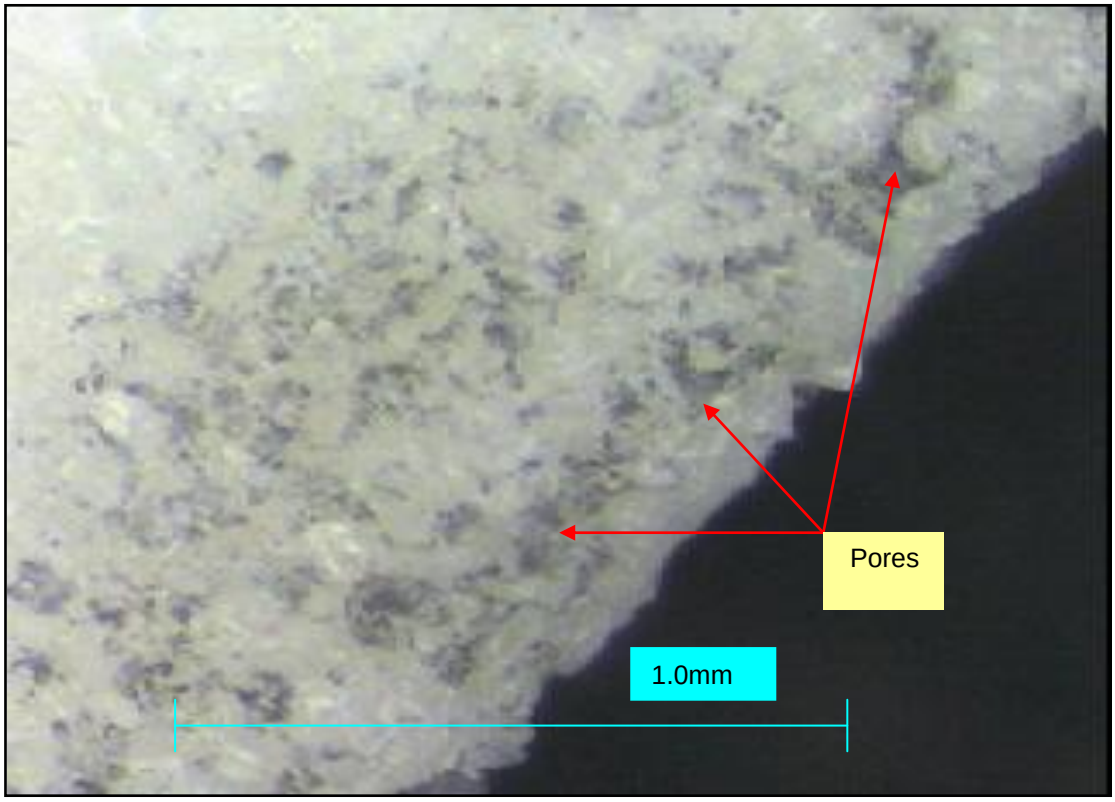


Plate 1 Magnified Image of fired porous alumina/feldspar material before resin infiltration (Group I)

The edge of a Group I specimen is orientated in order to view the porous structure before resin infiltration.

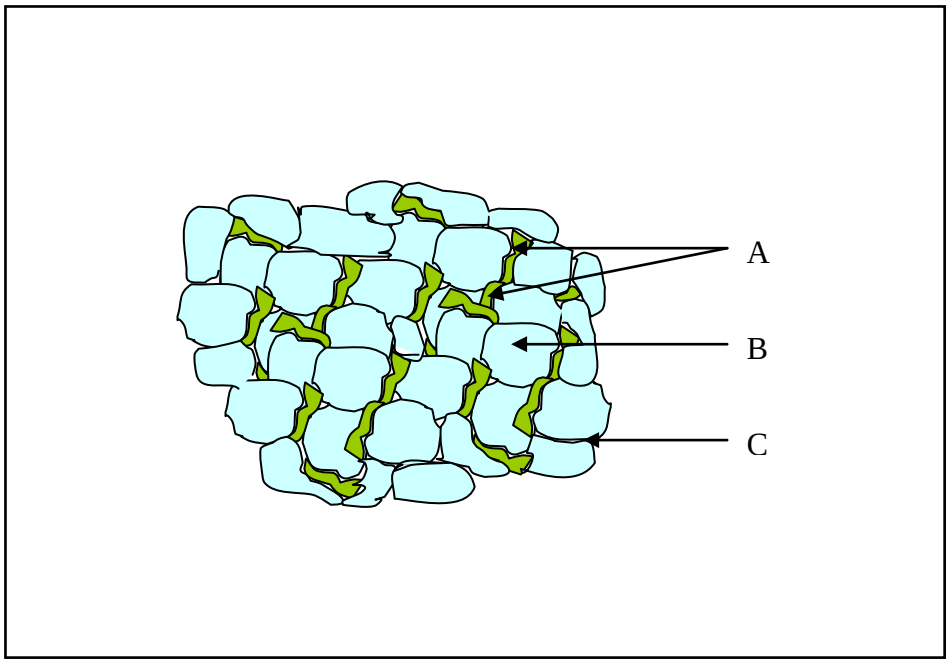


Figure 30 Schematic depiction of porous alumina/feldspar specimen before resin infiltration. *[A: Pores between alumina particles. B: Alumina particle. C: Grain boundary of alumina particle]*

4.4.1 Surface finish of alumina/feldspar composites

The resin infiltrated alumina/feldspar design allowed as smooth a surface texture after *in vitro* wear as before wear (Plate 2a and Plate 2b). *In vivo* longevity of surface structure still needs to be investigated for experimental alumina/feldspar dental composites.

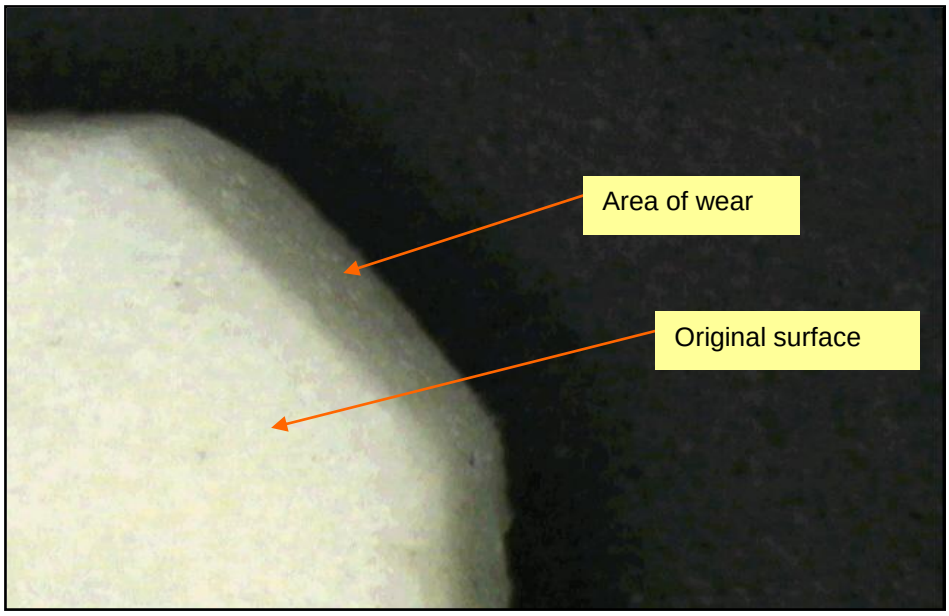


Plate 2(a) Magnified image (22x) depicting the surface wear of UDM infiltrated alumina particles

Surface of alumina/feldspar specimen orientated in such a way that the area of wear (of the 90^0 cusp) is visible (Note the surface remains as smooth after wear as before)

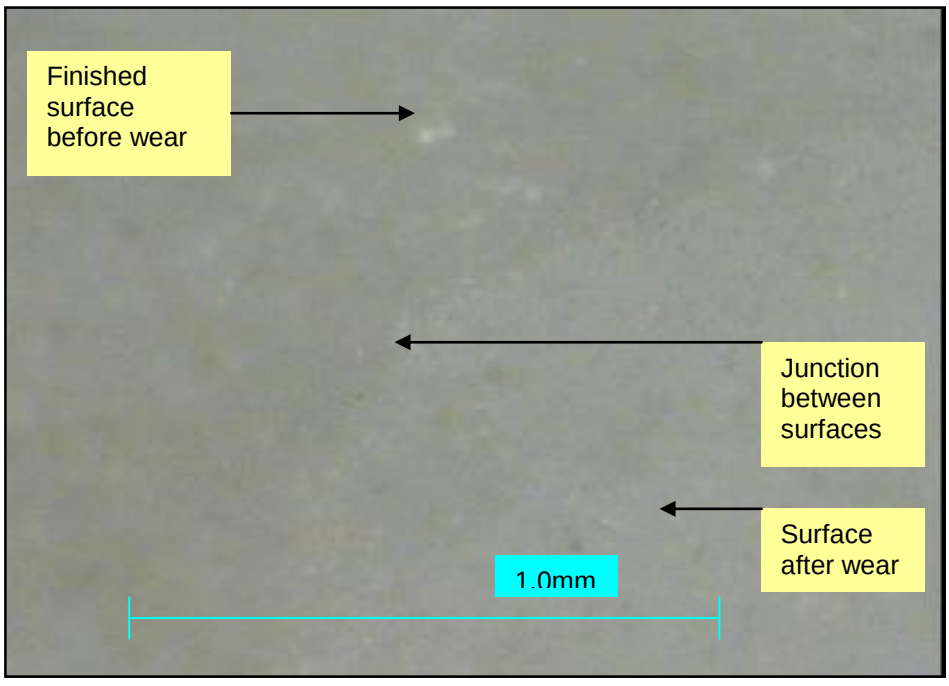


Plate 2(b) Finished surface compared to surface after wear

Note the similarity in smoothness between the two surfaces

The smooth surface finish resulted from alumina particles below 50µm (Plate 3a and Plate 3b). Larger alumina particles than 50µm are expected to result in a rougher surface due to increased gaps between the particles (Figure 31). Surface roughness when particle size is increased can be compared to the wear of fine or rough sandpaper.

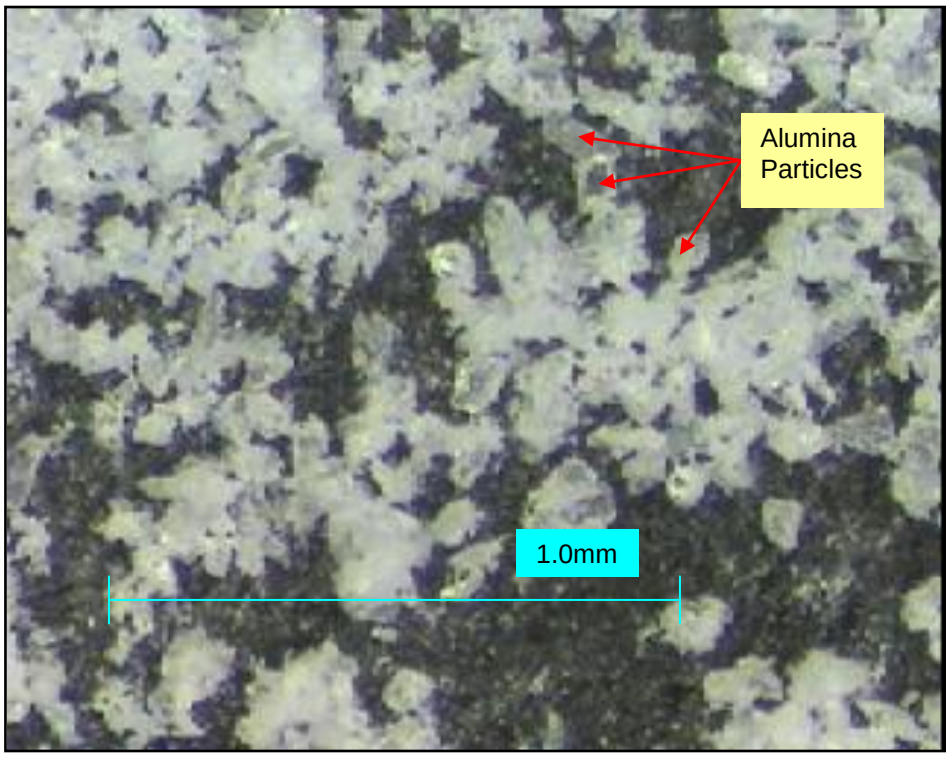


Plate 3(a) Magnified image (89x) depicting the distribution of alumina particles up to 50 µm

Ground 50 µm alumina particles are randomly dispersed over a black background allowing some individual and some groups of alumina particles to be viewed.

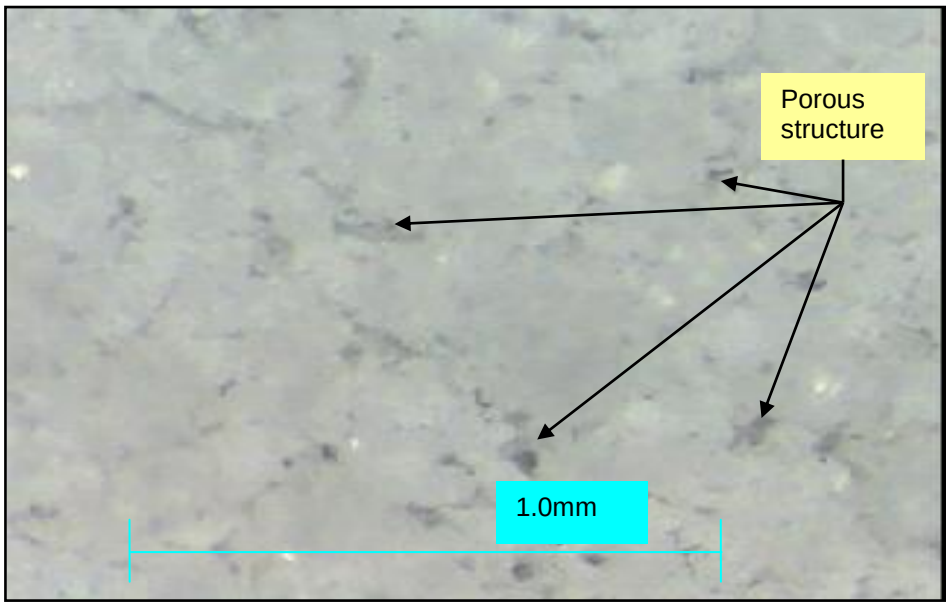


Plate 3(b) Magnified image of ground 50µm (unfired) alumina particles that provide a porous structure

A pile of ground 50 µm alumina particles are placed on a black background allowing the porous structure of compacted alumina particles to be viewed.

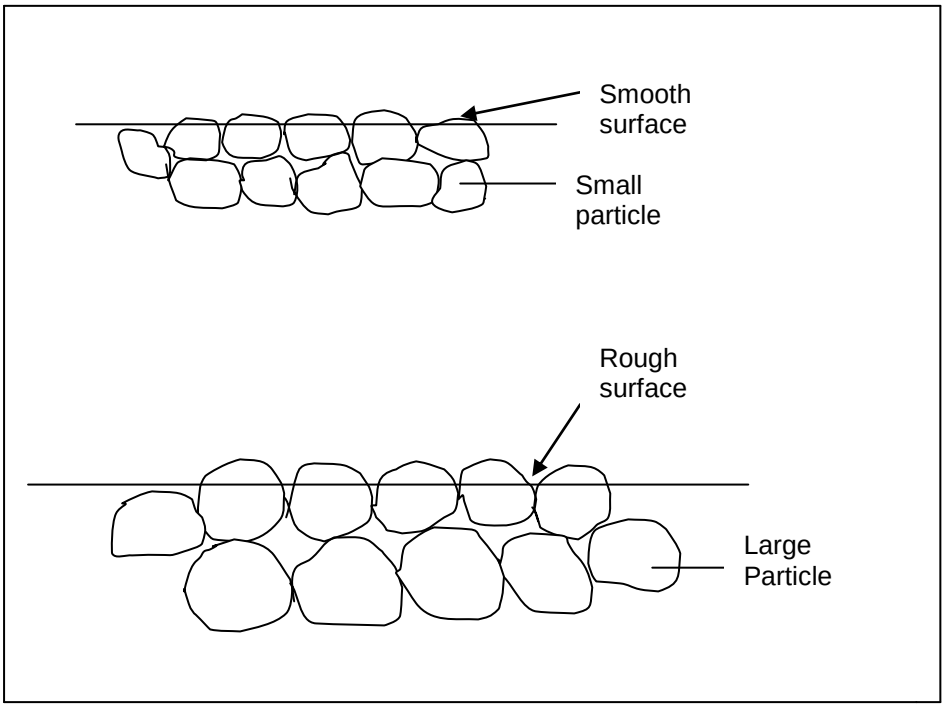


Figure 31 Schematic depicting relative surface roughness as the alumina particle size is increased

The purpose of the resin was to fill in the gaps between the particles and provide a surface that would remain smooth when subjected to continued wear. Filler particles above 50μm would not facilitate layering depth because of space constraints that are often experienced in conservative restorative dental practice (Kersten 1988). The ability to obtain fine anatomical detail would also be reduced as the alumina particle size was increased.

The surface of Group H specimens after resin infiltration (Plate 4) appeared to be similar to that of a ceramic surface before wear or polishing (Plate 5). The significant differences of alumina/feldspar composites are that the resin increases the wear while the alumina reduces the shrinkage. This has resulted in advantages sought because the wear resistance and shrinkage of feldspar porcelain is too high (Anusavice 2003).

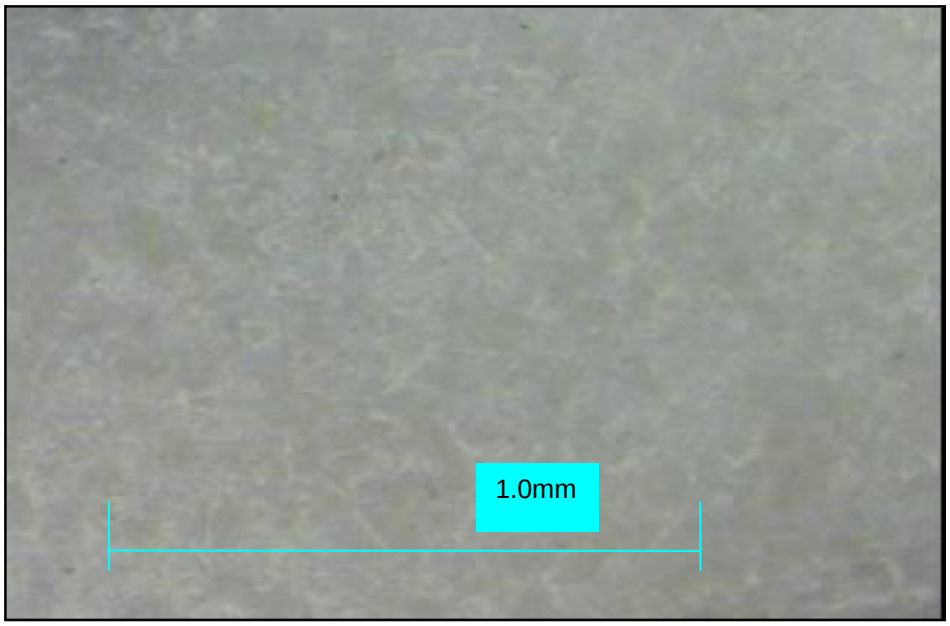


Plate 4 Magnified image of 50% feldspar mass alumina/feldspar specimen after resin infiltration and curing (Group H)

The colour contrast between white alumina and yellow resin depicts a relatively equal distribution of both materials after resin curing

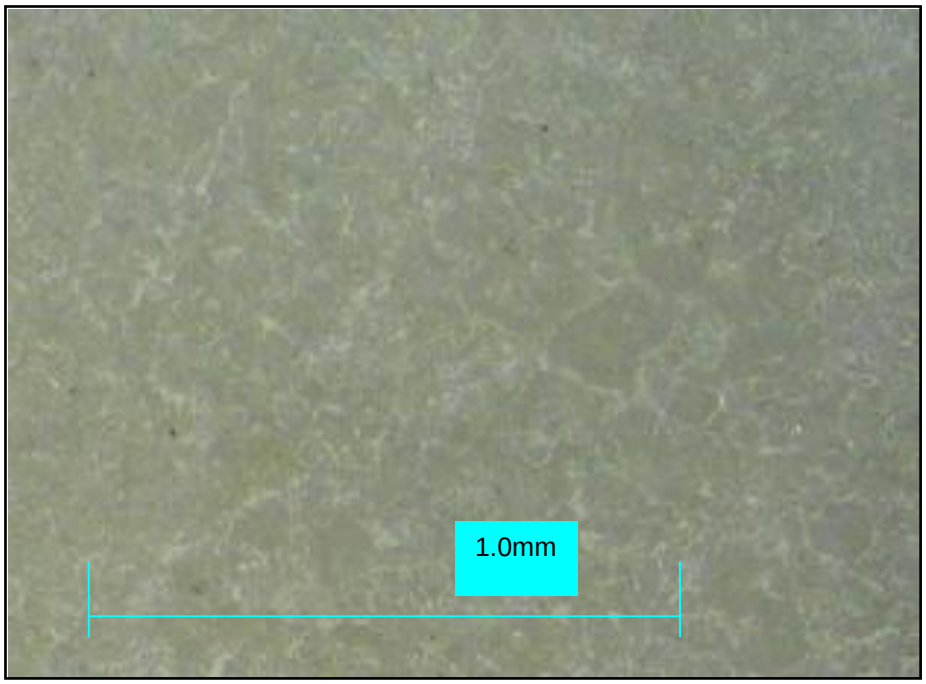


Plate 5 Magnified image of a porcelain surface

The alumina/feldspar composites with 50% feldspar mass (Plate 4) provides a similar surface structure to fired feldspar porcelain (Plate 5)

Surface finish is an important consideration when designing dental composite materials. Van Groeningen *et al*., (1986) observed surface degradation of dental composites placed in vivo that were not subjected to stressors and report that these findings are especially relevant for attrition of composites under stress bearing conditions in vivo. Micro-filler particles in the nanometer range (Figure 2) have been found to improve wear resistance and surface finish (Shi *et al*., 2004). The ability of resin infiltration to improve surface finish of macro filler composites does not appear to have been reported in the literature received.

The working characteristics and surface finish of alumina/feldspar composites suggest potential commercial success for alumina/feldspar experimental materials.

Surface structures of alumina/feldspar composite restorations can be smoothed with abrasives, pumice, polishing paste or a resin based sealant such as Palaseal® (Hereas Kulzer, Germany) (Plate 6). Palaseal® not only seals the surface but provides a smooth surface finish that simulates a glazed porcelain crown.

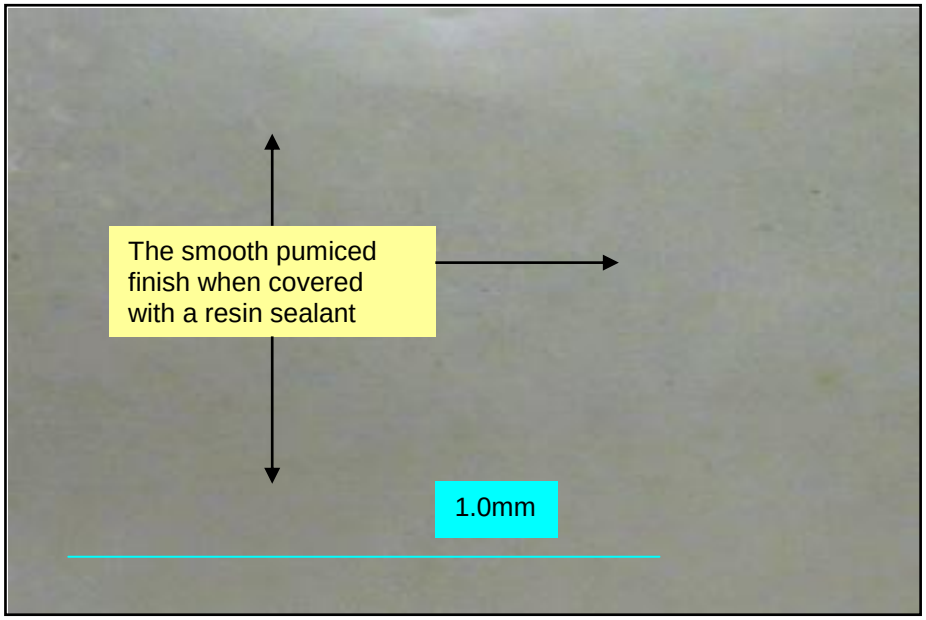


Plate 6 Magnified image of a 50% feldspar mass, alumina/feldspar surface that was pumiced and sealed with Palaseal®

4.4.2 Alumina/feldspar pore design

Desirable pore design for resin infiltration needs to be considered since literature received did not appear to report on pore size for resin infiltration. The maximum permissible feldspar mass added to the ground 50µm range alumina, that allowed resin infiltration, appeared to be 60% (Plate 7). Without adding feldspar, sintered alumina (Plate 8) could still be infiltrated with UDM resin without experiencing resultant cracks to the specimens.

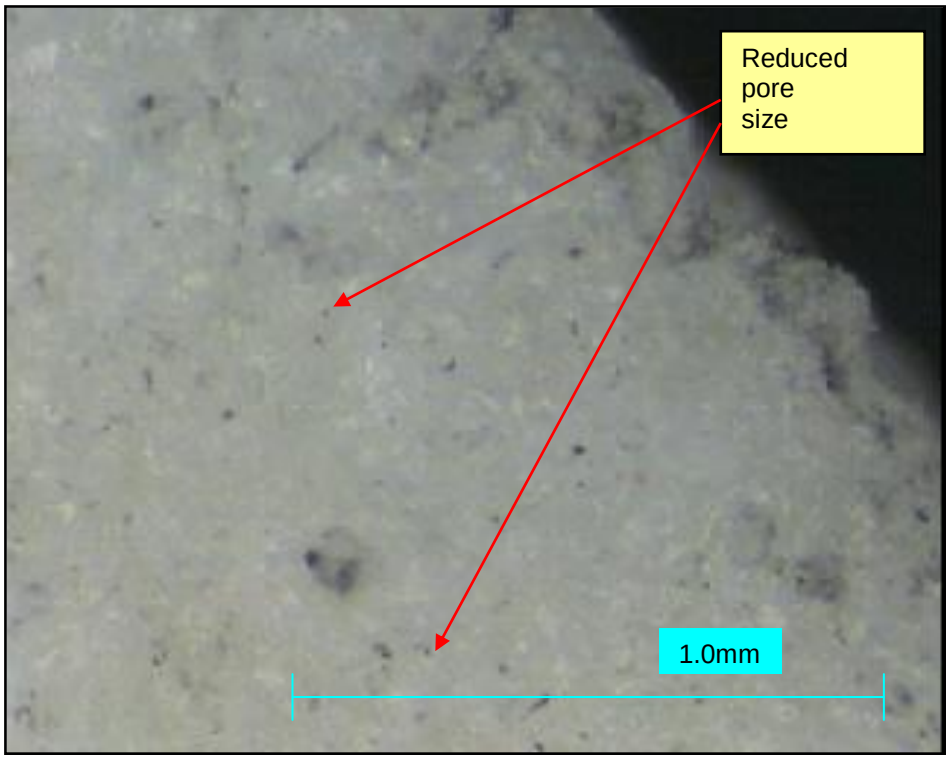


Plate 7 Magnified image of fired alumina with 60% feldspar mass added

The edge of a Group C specimen is orientated in order to view the porous structure before resin infiltration

Plate 8 Sintered alumina (Group A) before resin infiltration

The pores (Plate 8) are not as visible as those with specimens that contain feldspar, which appears to define the pore circumference. The alumina particles (after sinter firing) provide a chalky mass that breaks up with finger pressure (because of no feldspar bonding).

As the amount of feldspar decreased, infiltration time required decreased because the pore size increased (> 25μm). Eventually the pores were so large (Group A) since feldspar content was eliminated that the resin material tended to flow out of the alumina sintered core due to gravity. This resulted in problematic curing of the resin while trying to maintaining the desired specimen form. Another problem that arose was that as the pore size increased resin shrinkage resulted in small surface voids as the resin was cured (Plate 9).

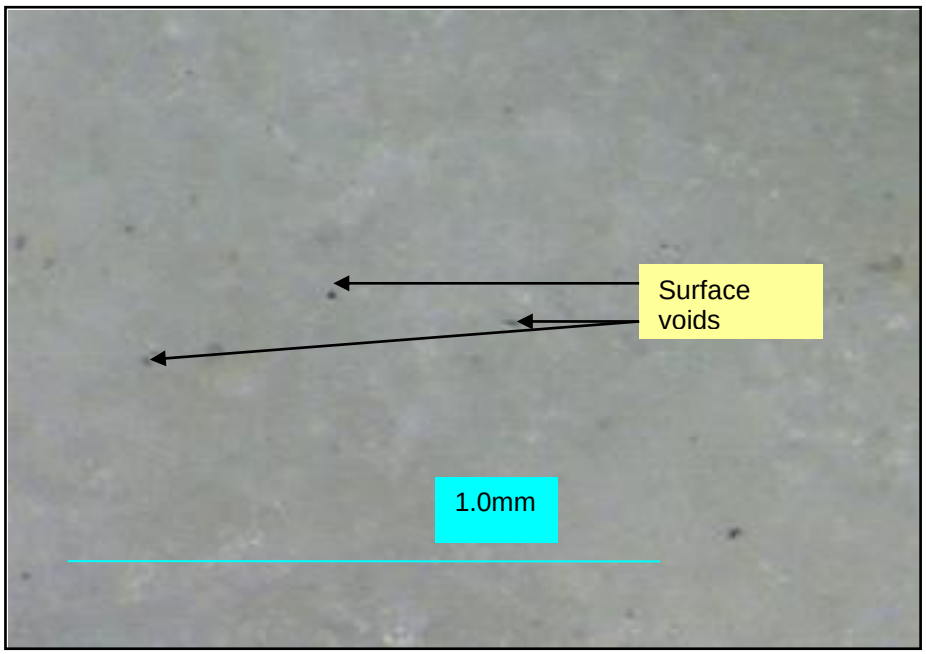


Plate 9 Magnified image of resin infiltrated specimen that had a large pore size

Small black surface voids result from resin shrinkage

The 50% feldspar by weight (Group H) proved to be the most advantageous for resin infiltration and curing. This is probably because the feldspar chemical bond was proportionate to the alumina filler particles thus improving on physical properties such as wear resistance of the material. Lowering the feldspar mass below 50% resulted in increased pits after the specimens were subjected to wear testing. This may be due to either extruded filler particles or resin extrusion from between the ceramic particles, or both (Plate 10 and Figure 32).

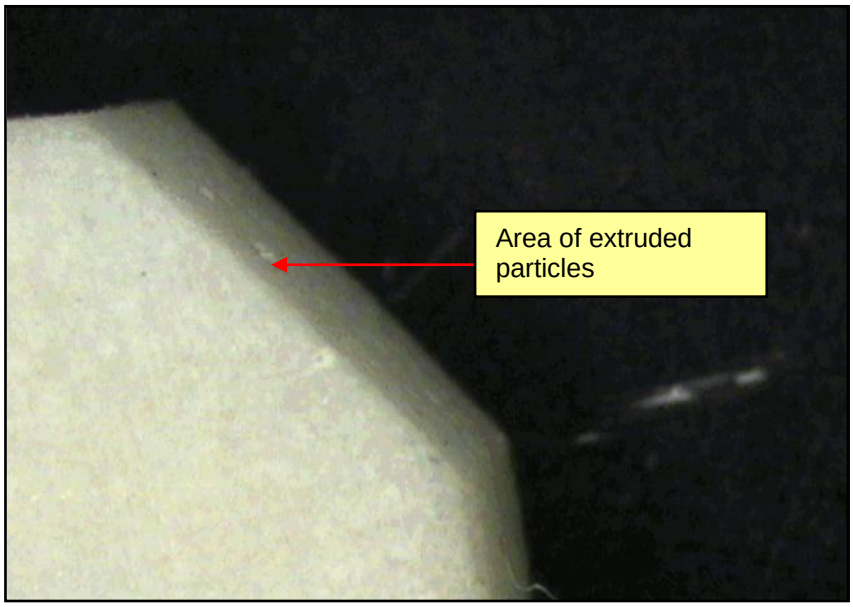


Plate 10 Magnified image (22x) depicting area of extrusion of particles on the surface of a sintered alumina resin infiltrated specimen as a result of wear (Group A).[1]

Surface of alumina/feldspar specimen orientated in such a way that the area of wear (of the 90^0 cusp) is visible

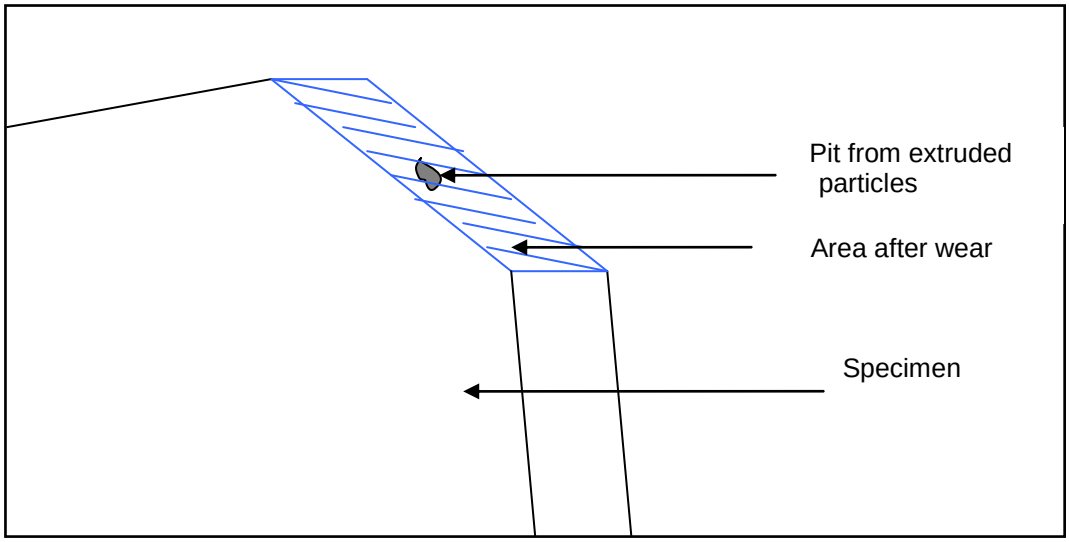


Figure 32 Schematic depicting surface pit from extruded particles after specimen wear

[1] Group A (which did not contain feldspar) showed the largest wear and proved to be the weakest of all specimen groups.

Where 50% alumina and 50% feldspar was mixed by weight extrusion of the material was reduced markedly, probably because desired pore size (25μm) and feldspar bonding of alumina particles supplied adequate strength to the filler and resin to prevent extrusion of either material. Although literature did not appear to report on alumina/feldspar resin infiltrated composites it stands to reason that if two materials form lattice structures around each other (to form a composite), that the thinnest or weakest material structure may break away with wear. Resistance to extrusion may to a lesser extent rely on the adhesive bonding mechanism (the weakest link) between the two insoluble (resin and ceramic) materials.

Equal volume of resin and alumina resulted in an average pore size of 25 μm as an average of alumina particle size. The results suggested that small pore size (<25 μm) was desirable to prevent resin shrinkage and alumina particle extrusion, while a large pore size (>0.25 μm) provided bulk to the resin material and as a result a compromise in pore size for resin infiltration was beneficial. The results showed that the 50% feldspar material provided the most desirable pore size (Plate 11 and Plate 5) to prevent excessive resin shrinkage while allowing sufficient volume for resin.

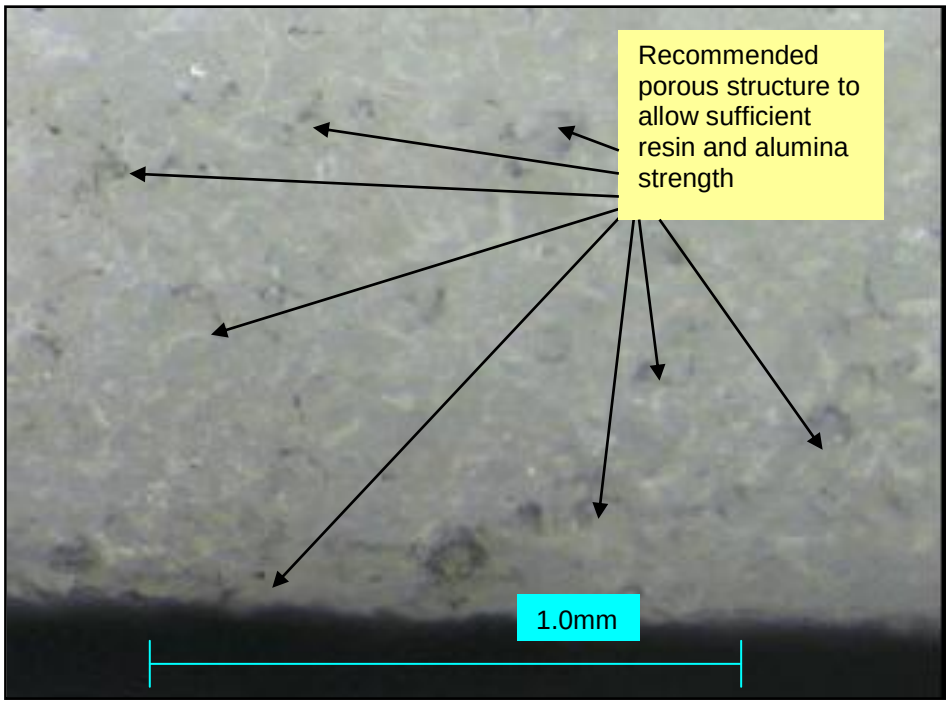


Plate 11 Magnified image of 50% feldspar mass alumina/feldspar specimen before resin infiltration.

The edge of a Group C is orientated in order to view the porous structure that results with 50% feldspar mass before resin infiltration.

The aim was to design, and construct alumina/feldspar resin infiltrated composites that could have the chemical bond strength varied (by sinter firing or incorporating different amounts of feldspar). Wear values varied between specimen groups as feldspar mass varied. Since resin infiltration was possible with sintered alumina as well as a range of feldspar mass (between 30-60%) this study was successful having achieved a design and construction of alumina/feldspar resin infiltrated composites that has the ability to vary the chemical bond strength by incorporating different amounts of feldspar. The alumina/feldspar materials can therefore be manipulated to obtain the desired wear that ranges between wear of resin materials and ceramic materials.

4.5 DESIGN CRITERIA

4.5.1 Design of composite restorations

Anusavice (2003) states that the laws of beams, applies to fixed prosthodontic bridges. Since the specimen design conforms to the specifications that comply with those of a beam appropriate design should consider the law of beams. Strength characteristics will improve if the design is corrugated or rounded at the connectors (Shillingburg 1997). Thin areas such as the connectors and margins are more likely to fracture with dental composite restorations and these areas must therefore be maximized (Shillingburg 1997). The flexure strength of alumina/feldspar composites may be improved by thickening the connectors to the maximum when used for posterior bridges (Plate 12).

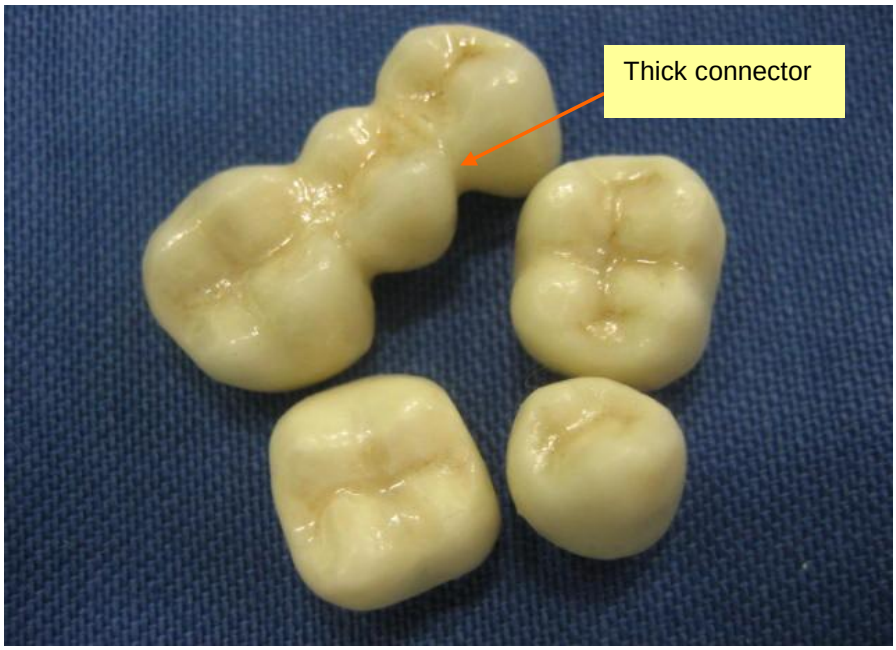


Plate 12 Crowns and a bridge produced from alumina/feldspar composite material

The recommended thick connector design increases the strength of the bridge

4.5.2 Design of alumina/feldspar materials

Since both ceramic and resin contribution to mechanical properties must be maximized the ratio of feldspar to gap size for resin infiltration and strength within itself to resist extrusion on function must be optimized. Bonding of both materials within themselves, together with a lattice design structure that will support each other when subjected to wear appears to be beneficial in reducing the reliance on the silane bonding agent. The resin material is infiltrated through the porous alumina/feldspar material and is connected together throughout the specimen once cured, while the alumina particles are bonded together by feldspar throughout.

The feldspar bond of alumina particles supplied the greatest contribution to the mechanical properties of the material but resin adhesion within itself is important to prevent small particles of resin from breaking off and extruding during function. The resin component is weaker than the alumina/feldspar material but is important to fill in the gaps, to seal the material and improve the surface smoothness and finish.

4.6 ADHESIVE VS CHEMICAL BONDING

4.6.1 Introduction

For the purpose of this study the silane bond strength was assumed to be consistent for all alumina/feldspar specimens, since the silane was applied consistently to all specimens that were silane bonded. There may in reality be slight variation where feldspar contact was increased and hence alumina contact with silane decreased.

Since there was little wear difference (0.12 mm) in performance of adhesive bonding agents (Group D and Group E), when compared to feldspar mass influence (Group B) any variation in adhesive bond strength between specimens would be small in comparison to contribution of the feldspar bond. Wear values when variation of 30% feldspar was tested (Group C and Group D), had a far greater influence (1.44 mm). These results indicate that feldspar bonding appears to have the potential to completely replace silane bonding in alumina/feldspar dental composites. This is an important finding since silane deteriorates with time and has a detrimental influence on longevity of dental composite materials (Zandinejad *et al*., 2006; Shajii and Santerre, 1999).

4.6.2 Benefits of the feldspar chemical bond

The main reason for introducing a feldspar chemical bond was to increase the wear resistance of dental composite materials. Since other physical properties such as strength and flexibility may also be improved alumina/feldspar materials may be used with the latest CAD/CAM milling technology.

Manufacture problems and production time have been improved with CAD/CAM milling technology that has resulted in the quest for improved materials that can be accurately milled. As no additional expensive equipment other than exists in a ceramic dental laboratory is required alumina/feldspar restorations may also be produced according to the materials and methods of this study. These production methods may be beneficial for cost effective use in countries where advancing (CAD/CAM) technology in dentistry is not affordable.

4.6.3 Disadvantages of the feldspar chemical bond

Chemical bonding would seem at first to be advantageous in all respects as a replacement for adhesive bonding agents such as silane but the following disadvantages to this design may be experienced:

(i) In order to obtain a chemical bond between the alumina particles the alumina must be fired resulting in possible increase in production time if CAD/CAM technology is not employed

(ii) The restoration must be manufactured indirectly and then cemented. Light cure dental composites that can be directly manufactured in the mouth are preferred because of time and cost saving (Zandinejad *et al*., 2006).

4.6.4 Advantages of alumina in dental composites

The following benefits of alumina make it advantageous for restorative materials (Anusavice 2003):

(i) Alumina is white and provides more natural aesthetics than metal alloy restorations especially for posterior restorations

(ii) Alumina is radio-opaque and provides good x-ray imaging

(iii) Alumina is strong and resists wear very well

(iv) Alumina is desired because of its excellent biocompatible properties

(v) Alumina can be fired above feldspar thereby allowing a chemical bond without changing its shape due to its excellent sag and creep resistance

(vi) As a ceramic material its coefficient of thermal expansion can closely be matched to that of feldspar

(vii) Alumina is inexpensive compared to metal alloys.

4.6.5 Advantages of resins

Resins are desirable as dental restoratives because of the following properties (Anusavice 2003):

(i) They can be manipulated and cured at low temperatures

(ii) They are easy to polish and provide smooth surface texture

(iii) Their processing techniques are easy and do not require expensive equipment

(iv) Processing time is reduced when compared to other dental restorative materials

(v) Biocompatibility is acceptable

(vi) They are not as brittle as porcelain and provide adequate strength

(vii) They are inexpensive[2].

[2] A major problem with dental resins is however the limited shelf life before they must be used, that may adversely influence costing of this material.

4.6.6 Microwave curing

Microwave curing took place in moderate temperature ranges that did not adversely affect the aesthetics of the UDM resin and was a therefore a safe curing method for the purpose of this study. The reason microwave curing was used was because the liquid resin could not be cured in boiling water (due to the material design and liquid resin form) and a alternative heat curing method had to be found. Microwave curing allowed reliance of the material on the bonding agents since resin curing was consistent. Research beyond the time constraints of this study would be required to develop and test other more complicated curing methods that might involve light, heat and pressure curing.

4.7 WEAR OF ALUMINA/FELDSPAR COMPOSITES

Controversy surrounding the acceptance of dental composites for posterior application has continued for over a decade (Ferracane, 2006). Considerable interest in dental composite wear performance for posterior restorations has continued among wear scientists who have attempted to define the problems that persist (Anusavice 2003; Ferracane, 2006). Although wear of small restorations were no longer seen to be of clinical concern, the wear of dental composites is still of clinical concern for large stress bearing restorations (Ferracane, 2006).

4.7.1 Wear of feldspar porcelain in relation to wear of alumina/feldspar composites

Lendenmann (2003) reports on the need to reduce wear of dental ceramic materials. This is an important consideration since composite wear is higher than wear of opposing porcelain surfaces (Appendix D).

The alumina/feldspar composite materials possess the ability to vary the wear resistance and provide lower wear than SR ADORO®. Alumina/feldspar dental composites may potentially fill the wear gap between resin and ceramic restoratives. Statistical analysis between wear of SR ADORO® and the alumina resin infiltrated composite specimens with silane bonding (Group A) was possible. This is manifest by the *p*-value <0.5, which "*in hypothesis testing is the exact probability of obtaining, when the null hypothesis is true, a value of the test statistic as extreme as or more extreme (in the appropriate direction) than the one actually computed*" (Thomas, 2001).

4.8 THE SAMPLE SIZE AND EXPERIMENTAL OVERVIEW

The sample size (Graph 1), for Group A, Group B, Group C, Group D and Group E was statistically viable (n=30) to permit confidence in eliminating the specimens with significant wear values above that of Group F. The level of significance for all test was $\propto$ = 0.05. The p – values were useful in establishing significant difference in values in order to make the decision. Only the first 10 specimens of Group C and Group F were retained for wear, flexural strength and flexibility comparison with Group H (n=10) and Group I (n=10). This was because Group A, Group B, Group D and Group E provided higher mean wear values than Group F (p-value <0.5)

The mean wear value of Group C changed from 1.11mm (n=30) to 1.10mm (n=10). The mean wear value of Group F changed from 1. 38mm (n=30) to 1.36mm (n=10). These changes did not alter the mean values significantly and were made to obtain the same sample size for Group C, Group F, Group H and Group I. Confidence in wear data as a result of the change is confirmed in the consistent wear performance and the similar coefficient of variation (CV) values of Group F, Group C, Group H and Group I.

Similar CV values for flexural strength of Group F, Group C, Group H, and Group I allowed confident comparison. The high CV for flexibility of Group C and Group H would be of concern if not considered in relation to the purpose of flexibility evaluation of this study.

Similar CV values between Group's F, C, H, and I for flexibility might have been obtained if the speed of the Instron® 44 testing machine was reduced (<10 mm per

minute) and the length of the specimens increased to 25 mm. Testing for modulus of elasticity is problematic for dental composites because the ISO specifications require a length of 25 mm which requires the material curing to be overlapped because of restricted curing areas with specimen construction that is larger than tooth size (Palin *et al*., 2003; Palin *et al*., 2005; Hussain *et al*., 2005; Al-Turki *et al*., 2007; Walker *et al*., 2006; Tanimoto *et al.,* 2006). The performance of these long specimens may as a result not be the best comparison for dental composite restorative materials and the specimen size was reduced in support of the notion that flexibility standards of smaller specimens be examined as a more practical comparative tool to test composite rigidity (Palin *et al*., 2003; Palin *et al*., 2005; Hussain *et al*., 2005; Al-Turki *et al*., 2007; Walker *et al*., 2006; Tanimoto *et al.,* 2006).

Reducing the speed and increasing the length of specimens would not simulate *in vivo* function accurately by *in vitro* methods. Ceramic materials distort cataclysmically beyond 0.1% deformation, resulting in what is referred to as a "*modulus of rupture*" (Preston, 1984). This increases the CV in practice resulting in 7% of unexplained fractures (Yamamoto 1985).

The small differences in measurement (0.01mm) measured at 95% confidence with the Instron® 44 machine may have contributed to minor CV differences. Larger CV difference observed may be as a result of the small specimen size, unexplained modulus of rupture performance as a result of "*Griffith's flaws*" in the feldspar (Yamamoto 1985). The grinding procedure (during specimen preparation) of Group C, Group H and Group I (Figure 21) and *in vitro* wear testing may have

contributed further to the higher CV values. The specimen preparation included standard laboratory techniques with regard to restoration size and manufacture methods.

For the purpose of this study the results were beneficial in showing higher flexibility values for Group F than for all the alumina/ feldspar composite groups tested. The CV of Group F and Group I are comparable since they are lower than Group C and Group H. Flexibility of Group F (mean value = 0.38mm) was higher than Group I (mean value =0.22mm). Higher flexibility of Group F was expected since improvements in reducing the modulus of elasticity of dental composites are advised (Visvanathan *et al*., 2007).

The literature received did not report on the modulus of elasticity of alumina/feldspar dental composites as it appears to be the first time that these experimental materials have been tested. The feldspar bond however appears to be responsible for the reduced flexibility of the alumina/feldspar materials due to the increased ceramic bonding contribution to mechanical performance (Anusavice, 2003).

4.9 COMPARING DESIGN, WEAR RESISTANCE, FLEXIBILITY AND FLEXURAL STRENGTH OF ALUMINA/FELDSPAR SAMPLE GROUPS

The final aim of this study was to compare all the results in order to recommend which alumina/feldspar composite material provided optimum improvements when compared to Group F.

SR ADORO® (Group F) gave a mean wear result of 1.36 mm. This value was higher than the alumina/feldspar silanized specimen Group H (1.02 mm) and Group C (1.10mm) and similar to the value for Group I (1.37 mm). Comparison of the non parametric wear ranks (Graph 11) confirm that the alumina/feldspar material Group C (60 % feldspar) and Group H (50% feldspar) provided the overall improvement on the wear properties sought when compared to the commercial SR ADORO® material (Group F) Group I (40%feldspar) and Group C (60% feldspar).

Comparisons (Graph 12), between the alumina/feldspar materials (Group C, Group H and Group I) and SR ADORO® revealed that similar flexural strengths to SR ADORO® were obtained with Group C and Group H.

The SR ADORO® group gave a mean flexibility result of 0.38 mm. This value was higher than the alumina/feldspar specimen Group H (0.17 mm), Group C (0.19 mm) and Group I (0.22 mm). All the alumina/feldspar specimen groups significantly reduced the flexibility as compared to the commercial SR ADORO® group. The alumina/feldspar material Group H (50% feldspar) provided the overall improvement on the flexibility properties sought when compared to the commercial

SR ADORO® material (Group F), Group I (40% feldspar) and Group C (60% feldspar).

The alumina/feldspar composites with 50% feldspar mass (Group H) provided the most desirable pore size for UDM resin infiltration. The alumina/feldspar composites with 50% feldspar mass (Group H) also provided the greatest resistance to particle extrusion during the wear tests.

Evaluation of physical properties of wear resistant characteristics, strength and flexibility indicated that Group H with 50% feldspar mass provided the optimal mechanical properties sought. Design considerations also indicate that Group H possessed the most desirable design properties. Group H was considered to be the optimum alumina/feldspar experimental material within the sample groups tested.

Recommendations regarding design, performance and manufacture of alumina/feldspar materials are needed to solve the main problems of wear resistance and marginal leakage of dental composites that persist (Ferracane and Condon, 1999; Bonilla *et al*., 2001; Yap *et al*., 2002;. Applequest and Meiers, 1996; Goracci *et al*., 1996; Luo *et al*., 2000; Peris *et al*., 2003; Shinohara *et al*., 2001; Splieth *et al*., 2003; Thordrup *et al*., 2001; Tung *et al*., 2000; Ulukapi *et al*., 2003; Worm and Meiers, 1996; Behr *et al*., 2005; Fennis *et al*., 2005; Ferracane, 2006). The process of specimen evaluation allowed the alumina/feldspar material group that had the lowest wear, the highest flexural strength, the lowest flexibility and the most advantageous design to be selected.

Group H with 50% feldspar mass, was selected because this group provided the most sought after results. Group H had the lowest consistent wear, comparable flexural strength to SR ADORO®, and the lowest flexibility of all the material groups tested. Group H also had the most advantageous design of all the alumina feldspar specimen groups. These characteristics are desirable in order to increase the longevity of dental composites.

Although the performance of posterior composite restorations have improved during the last decade the mean age of dental composites are reported to be between 7and 8 years (Burke-2001; Thordrup 2001). Studies report between 32.6% - 62.5% failure rate of dental composites ranging from small non stress related inlays to full posterior crowns (Anusavice 2003, Bolsen 2003). The mean life expectancy of dental composites is much lower than that of metal ceramic restorations (25% failure after 15 years) and amalgam restorations (10% failure after 10 years) (Anusavice 2003, Bolsen 2003).

The success of dental composites have been jeopardized as a result of marginal or bulk wear and fracture due to fatigue especially in cusp and stress bearing posterior regions (Thordrup 2001). Small to moderate restorations were reported to be more successful within a one to three year period (Luo 2000; Türkün 2003). Preventing marginal stresses in dental composites as a result of shrinkage, undesirable stiffness and wear are important considerations for preventing fatigue and increasing longevity.

The development of stresses at the restoration-tooth interface was shown to be detrimental to longevity of dental composite restorations. Stiffness of the composite

material needs to be higher than or more closely match the tooth structure otherwise stresses at the restoration tooth interface occurs (Ensaff, 2001). When combined with the desirable properties of dental composites such as the ability to manipulate wear resistance within a larger range, providing processing techniques that reduce shrinkage and potential reduction in construction time group H composite material should be of commercial interest to solve the problems associated with wear and flexibility. Group H material, may after *in vivo* testing prove to increase longevity of dental composites.

4.10 PRACTICAL SIGNIFICANCE OF ALUMINA FELDSPAR COMPOSITES

When compared to the average of 1.02 mm for Group H with a lowest wear value of 0.97 mm the pure feldspar (ceramic) specimens all had a lower wear value than all the alumina/ feldspar specimens. When compared to the mean wear value of SR ADORO® (1.35 mm) it appeared that Group H alumina/feldspar specimens showed wear values half way between that of pure feldspar and SR ADORO®.

Trying to maximize the desired properties of alumina and resin in a dental composite material is not easy because the two materials are not soluble in each other. Desirable properties of one may negatively influence other desirable properties of the other composite material. Mechanical, chemical and adhesive bonding mechanisms are available to assist with mechanical properties but the design concepts are largely contribute to the results as well.

Dental feldspar as restorative materials are brittle and weak on their own and must be reinforced whereas dental composite materials show improved mechanical properties that allows them more readily to be used without a reinforcing substructure. Unfortunately dental composites show higher functional deformation and fatigue that reduces their lifespan considerably when compared to dental ceramic materials (Anusavice 2003). Since the alumina/feldspar materials have reduced flexibility compared to SR ADORO® they should be investigated *in vivo* to determine their longevity.

The alumina/feldspar resin infiltrated material could potentially make up the whole restoration since the resin, unlike glass does not etch in the oral environment. For this reason resin infiltration will be much more suitable to improve wear of alumina strengthened dental restorative materials as long as mechanical properties of strength and resistance to flexure or functional deformation are not adversely affected.

Although glass infiltration of much finer sintered alumina particles has been successful as a ceramic core for porcelain jacket crowns the glass will etch and form a rough surface in the oral environment if not completely covered with feldspar. This prevents the use of the glass infiltrate on the outer surface of the restoration.

4.10.1 Coefficient of thermal expansion (CTE) variation

Wear of the specimen groups are presented in the Appendix (Plates13 to 21). With regards to the CTE no cracks were observed (in any of the specimen groups) using the Nikon® SMZ800 Microscope.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

There is nothing like returning to a place that remains unchanged, to find the ways in which you yourself have altered.

Nelson Mandela

5.1 CONCLUSIONS

5.1.1 Wear results

From the wear results of alumina/feldspar specimens the following can be concluded:

1. Introducing a feldspar chemical bond between the alumina particles in order to improve on the wear resistance of SR ADORO® was successful with 60%, and 50% feldspar mass added to alumina/feldspar specimens. With 50% feldspar mass added to alumina, the wear of the specimens was significantly improved when compared to the 60% feldspar mass addition and the SR ADORO® specimens. Similar wear to SR ADORO® ($p>0.05$) was obtained with 40% feldspar mass added to alumina/feldspar composite specimens.

2. The wear resistance of alumina/feldspar composites was increased by including a chemical feldspar bond rather than using silane or phosphate bonding agents. Little information was obtained with regard to the silane-alumina bond mechanism.

3. Phosphate bonding with SR Link® provided significantly ($p<0.05$) increased wear resistance to silane bonding in alumina/feldspar composite materials. SR Link® should however not be used out of its intended purpose without further investigation.

4. The alumina/feldspar materials can be manipulated to obtain the desired wear that ranges between wear of resin composite materials and ceramic materials.

5.1.2 Flexural strength results

From the flexural strength results of alumina/feldspar specimens the following can be concluded:

1. The flexural strength of 50% feldspar specimens was slightly higher than SR ADORO®, the mean difference was not statistically significant (p-value>0.05)

2. The flexural strength of 50% feldspar mass specimens was significantly higher than 40% feldspar mass specimens (p-value<0.05). Improving wear resistance of alumina/feldspar composites by a 10% feldspar mass addition therefore increased the flexural strength significantly in this instance

3. Feldspar mass addition from 50% to 60% resulted in a small insignificant reduction in flexural strength (p-value>0.05).

5.1.3 Flexibility results

From the flexibility results of alumina/feldspar specimens the following can be concluded:

1. The flexibility of all alumina/feldspar specimens were reduced significantly when compared to SR ADORO® specimens (p-value<0.05) and improved the longevity of these materials is expected as a result of potential marginal stability.

2. The flexibility of alumina/feldspar composites were similar (p-value>0.05) for sample groups with feldspar mass between 40% and 60%.

5.1.4 Design results

From the design results of alumina/feldspar specimens the following can be concluded:

1. The pores for resin infiltration are not completely blocked by incorporating feldspar, unless too much feldspar is added (>60%). The pore size decreases as feldspar mass increases resulting in the need for increased resin infiltration time

2. The CTE of alumina and feldspar can be matched successfully with the material design used in this study

3. The alumina/feldspar material with 50% feldspar mass appeared to provide the most desirable pore size to facilitate ease of resin infiltration.

5.2 RECOMMENDATIONS

It is recommended that the alumina/feldspar composite material with 50% feldspar mass be investigated for use as an experimental dental composite material due to its potential advantages in wear resistance, flexural strength, flexibility and design. Further investigations to improve the wear resistance and flexural strength of this material by using different infiltration resins, curing techniques and adhesive bonding agents are advised.

The benefits of alumina/feldspar composites as dental restorative material with CAD/CAM technology should be investigated. *In vivo* experimental use of alumina/feldspar dental composites should be compared to *in vitro* results of this study.

5.3 PRACTICAL SIGNIFICANCE

1. Alumina/feldspar resin infiltrated dental composites may need to be refined to improve on the desirable benefits of both ceramic and dental composite properties for *in vivo* use. Consistent *in vitro* wear results with the Minimet® polishing machine indicated that the wear method employed to obtain data was accurate and beneficial.

2. Materials and equipment required are cost effective since conventional use does not require additional expensive equipment other than those in a dental metal ceramic laboratory.

3. Manufacture techniques as with any experimental material are expected to improve with use over time.

4. Feldspar bonding may be a forerunner in eliminating silane bonding in dental composites.

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Appendix A

Wear test results of alumina/feldspar sample groups and the SR ADORO® group F.

	Group A	Group B	Group C	Group D	Group E	Group F	Group H	Group I
1	2.95	2.54	1.22	2.95	2.45	1.45	1.00	1.40
2	3.68	2.54	1.20	2.50	2.09	1.54	1.00	1.18
3	3.38	2.22	1.13	2.95	2.04	1.50	1.06	1.40
4	2.77	2.31	1.22	2.59	2.13	1.50	1.00	1.27
5	2.86	2.40	1.09	2.31	2.40	1.27	1.00	1.38
6	2.95	2.31	0.97	2.59	2.20	1.18	1.00	1.36
7	3.22	2.31	0.94	2.50	2.50	1.13	0.97	1.45
8	3.25	2.72	1.13	2.59	2.13	1.18	1.13	1.43
9	3.20	2.72	1.13	2.18	2.72	1.43	1.09	1.27
10	3.36	2.43	0.95	2.54	2.40	1.38	1.00	1.59
11	2.31	2.25	1.31	2.50	2.36	1.54		
12	3.31	2.45	1.09	2.00	2.45	1.45		
13	2.95	2.34	1.22	2.36	2.31	1.50		
14	3.00	2.54	0.81	3.40	2.20	1.36		
15	3.31	2.77	1.09	2.50	2.13	1.36		
16	2.77	2.63	1.04	2.45	2.36	1.27		
17	2.90	2.72	0.77	2.40	2.45	1.09		
18	3.22	2.36	1.27	2.77	2.27	1.43		
19	2.72	1.95	1.04	2.50	2.36	1.25		
20	2.86	2.31	1.18	2.63	2.50	1.54		
21	3.18	2.36	0.77	2.50	2.18	1.52		
22	3.40	2.50	0.95	2.04	2.27	1.54		
23	3.68	2.09	1.22	2.54	2.40	1.00		
24	3.31	2.36	1.36	2.22	2.50	1.54		
25	3.45	2.13	1.18	3.09	2.18	1.36		
26	2.95	2.59	1.45	2.95	2.13	1.45		
27	2.77	2.68	1.06	2.68	2.18	1.47		
28	2.18	2.43	1.27	2.50	2.18	1.45		
29	2.77	2.34	1.22	2.54	2.72	1.50		
30	3.40	2.68	1.12	2.36	2.15	1.45		

Appendix B

Flexural strength test results for sample groups C, H, I and F

	F-Control	Group C	Group H	Group I
1	127.40	120.90	122.80	96.64
2	148.70	150.50	103.70	111.10
3	139.20	92.08	142.90	106.00
4	114.00	131.30	132.10	103.30
5	89.40	117.00	124.40	130.90
6	111.70	97.61	132.30	84.72
7	91.49	117.90	90.74	89.53
8	112.0	100.20	150.10	108.50
9	97.83	147.90	136.20	111.30
10	123.20	122.60	131.70	114.80

Appendix C

Flexibility test results for sample groups C, H, I and F

	F-Control	Group C	Group H	Group I
1	0.37	0.28	0.07	0.21
2	0.32	0.21	0.21	0.30
3	0.46	0.00	0.31	0.21
4	0.34	0.30	0.17	0.20
5	0.31	0.15	0.10	0.30
6	0.37	0.19	0.26	0.25
7	0.47	0.13	0.31	0.22
8	0.34	0.09	0.21	0.16
9	0.36	0.25	0.10	0.11
10	0.46	0.30	0.00	0.24

Appendix D

Although not part of the methodology of this study results were desired as to comparative wear performance of feldspar ceramic specimens to alumina/feldspar composites. In order to get an idea of wear of feldspar porcelain in relation to wear of alumina/feldspar composites five feldspar specimens were subjected to the same wear tests using the Minimet® polishing machine. Statistical methods were not applied to the feldspar specimen group which provided the following data:

(i) Feldspar specimen gave a wear value of 0.54mm
(ii) Feldspar specimen gave a wear value of 0.54mm
(iii) Feldspar specimen gave a wear value of 0.77mm
(iv) Feldspar specimen gave a wear value of 0.72mm
(v) Feldspar specimen gave a wear value of 0.72mm

Feldspar specimen wear values were in the 0.5mm to 0.7mm range. From this data wear trends can be recognized to be lower than Group H. Group H gave a mean wear value of 1.02mm and a lowest value of 0.97mm while the highest wear value of the feldspar specimens was 0.77. These results indicate that Group H has reduced the gap between the feldspar specimens and SR ADORO® specimens (Group F) which gave a lowest value of 1.13mm and a mean value of 1.35mm.

Appendix E

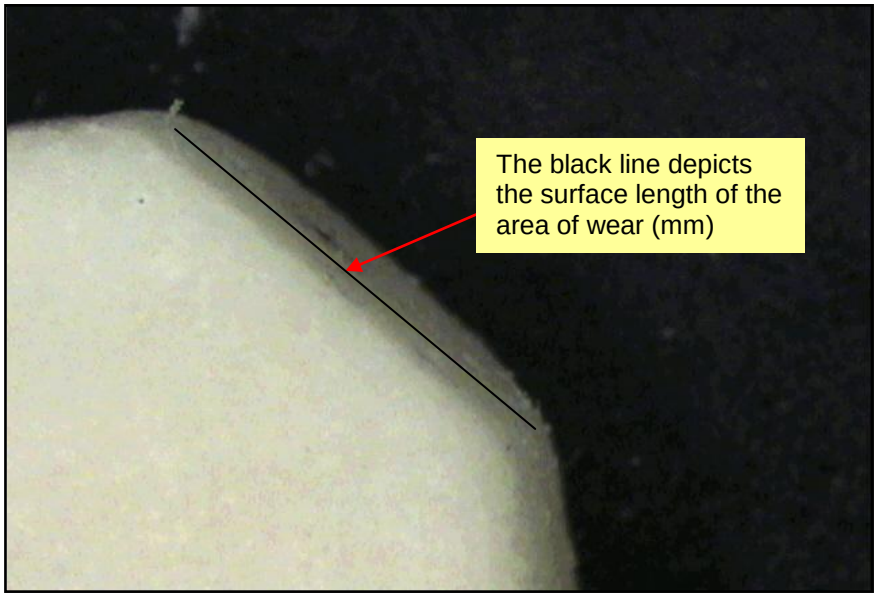

Plate 13 Magnified image (22x) of worn surface of sintered alumina resin infiltrated composite specimen with silane bonding (Group A)

Surface of alumina/feldspar specimen orientated in such a way that the area of wear (of the 90^0 cusp) is visible

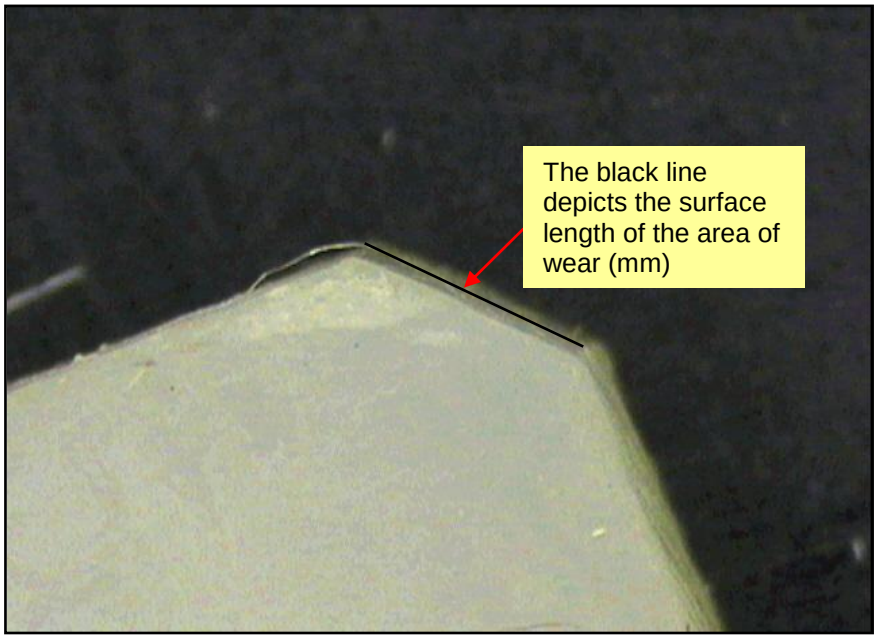

Plate 14 Magnified image (22x) of surface wear of SR ADORO® specimen with silane bonding (Group F)

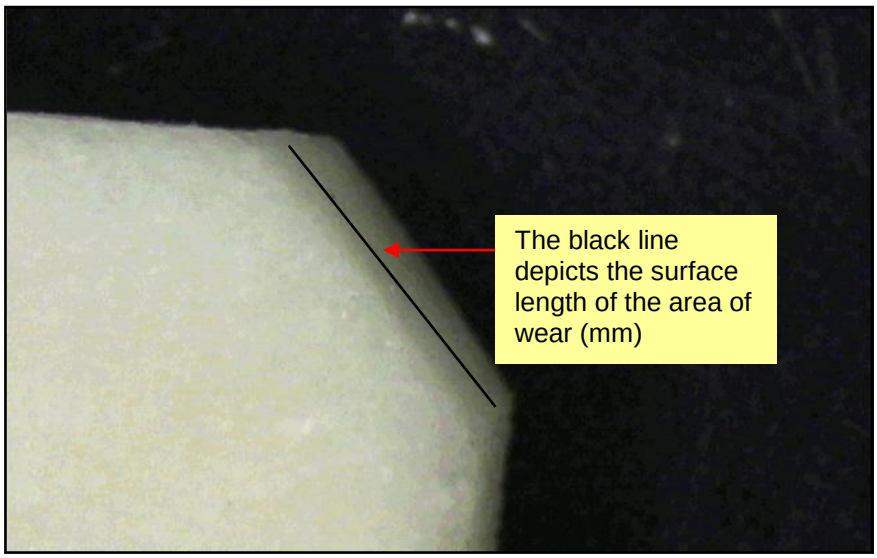

Plate 15 Magnified image (22x) of worn surface of 30% feldspar mass added to alumina composite specimen (Group B). The mean wear of Group B specimens was higher than the mean wear of SR ADORO® (Group F) specimens.

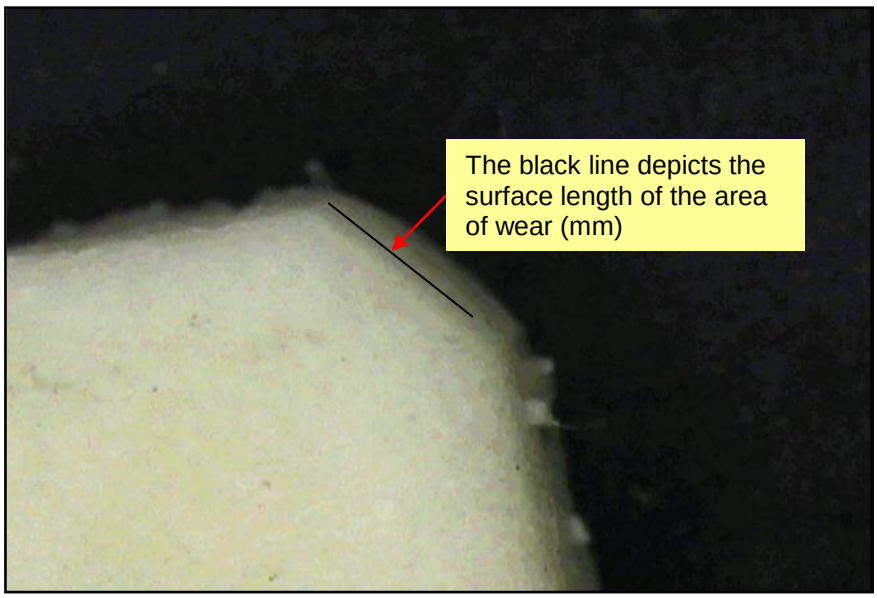

Plate 16 Magnified image (22x) of worn surface of a 60% feldspar mass added to alumina and silane bonded specimen (Group C)

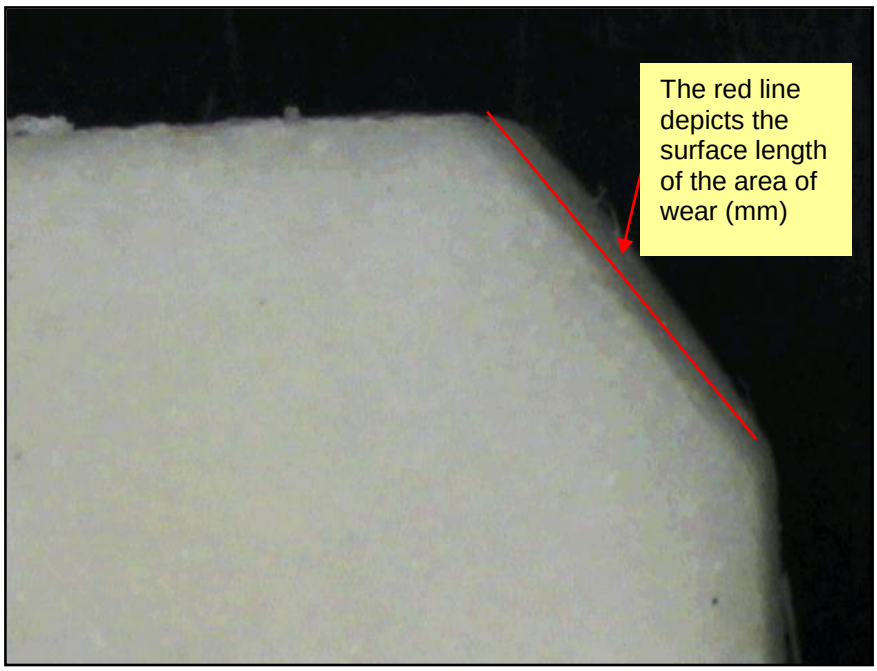

Plate 17 Magnified image (22x)of worn surface of 30% feldspar mass added to alumina and silane bonded composite specimen (Group D)

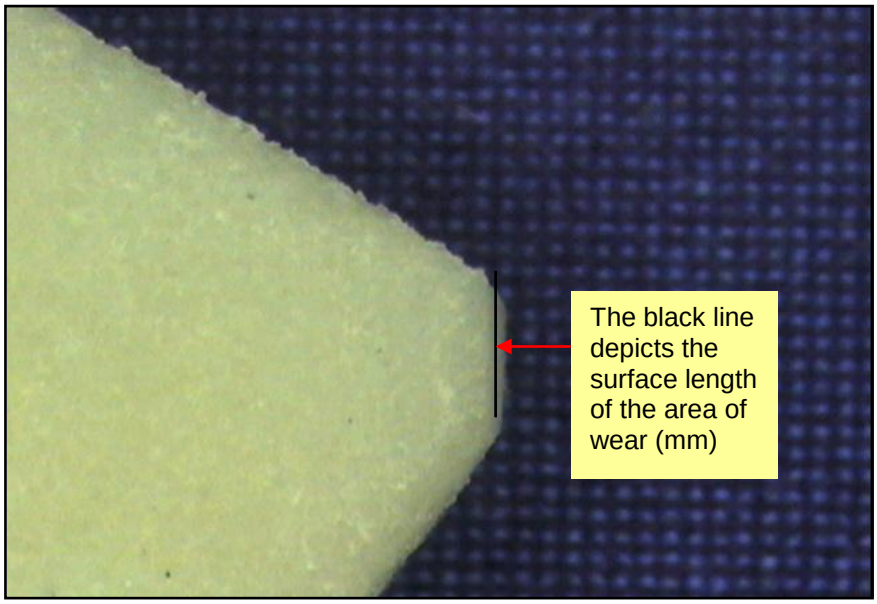

Plate 18 Magnified image (22x) of worn surface of 50% feldspar mass added to alumina and silane bonded composite specimen (Group H)

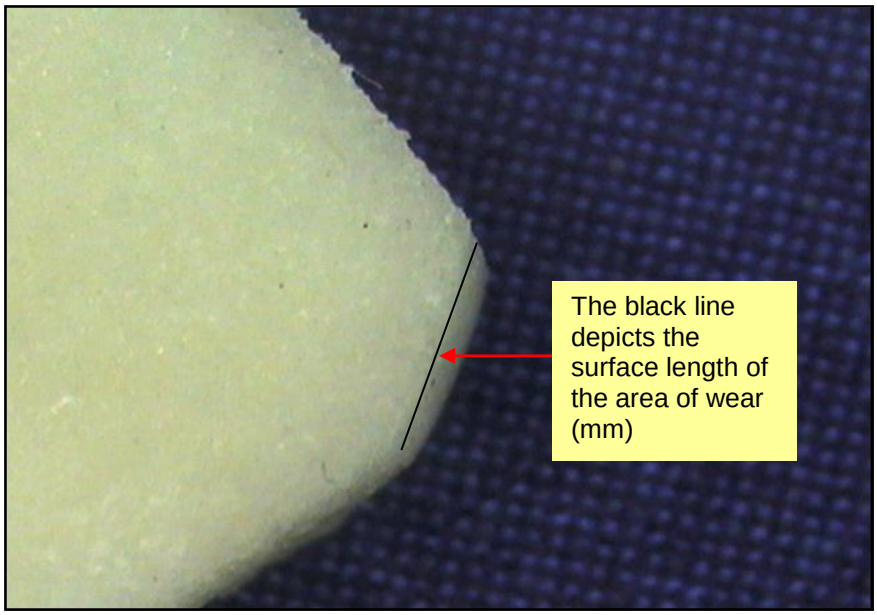

Plate 19 Magnified image (22x) of worn surface of 40% feldspar mass added to alumina and silane bonded composite specimen (Group I)

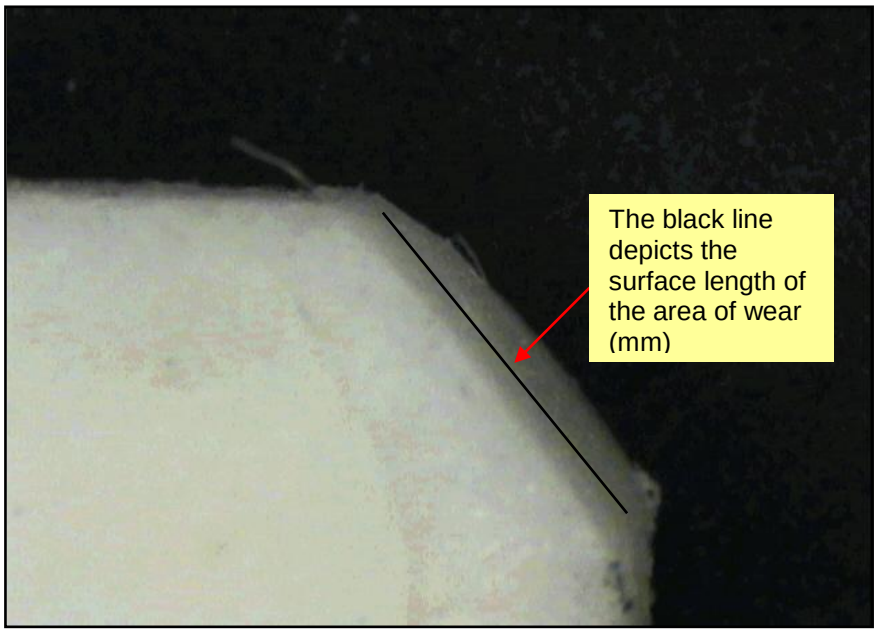

Plate 20 Magnified image (22x) of worn surface of 30% feldspar mass added to alumina and SR Link® phosphate bonded composite specimen (Group E)

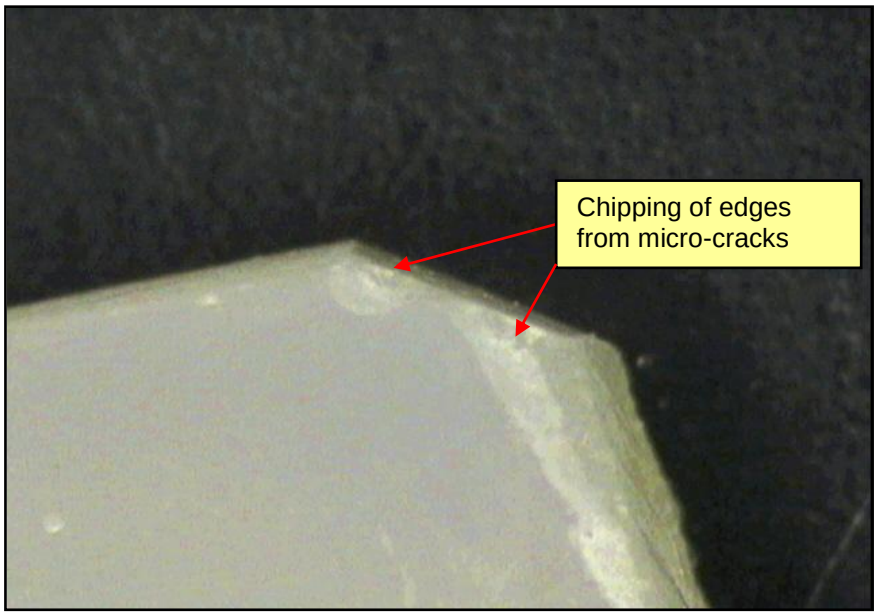

Plate 21 Magnified image (22x) of chipped edges from micro-crack formation on the surface edge (Group F)