Fiber-Reinforced Composites: A Breakthrough in Practical Clinical Applications with Advanced Wear Resistance for Dental Materials

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Abstract

Newer dental fiber-reinforced composites can provide service with less wear than enamel. Further, fibers in bulk molding form pack oriented parallel to the occlusal-dentinal floor planes and wear by uniform thinning into micrometer-sized fiber remnants and subsequent plate-like particulate with a more flat bottom surface that bond by compression back into the polymer matrix. The fiber wear-in process is accomplished by creating fine crystalline chemically resistant nanoparticulates that become an exceptional polishing agent. Resulting consolidation by the underlying fiber network squeezes plasticized polymer and partially hydrolyzed polymer chains along with residual monomer, pendant methacrylate groups and nano-sized particulate to the surface that surround larger exposed micrometer-sized particulate and smallest fiber remnants. Eventually consolidation of the polymer matrix squeezes up and engulfs the top particulate or fiber remnants forming a smooth polished hard polymer-matrix composite wear surface probably filled with small nanoparticulate. The final hardened polymer wear surface may show particulate from worn fibers, but displays no signs of the original fibers after a 3-year in vitro wear simulator test. The fibers that have broken down into nanoparticulate generally re-consolidate back in to the top wear surface for a polished toughened polymer surface or behave as a polishing agent. The underlying fiber-reinforced composite network supports wear loads that greatly reduce wear especially as fibers extend well beyond a critical length that prevents fiber debonding from the matrix. Further, fiber-reinforced composite consolidation can aid in cavity molding placement by applied pressure to squeeze monomer, resin and particulates from the fiber network toward collapsing or filling in voids and removing entrapped air.

Keywords: Wear; Fiber-Reinforced Composite; Particulate-Filled Composite; Critical Length; Consolidation; Voids

Abbreviations

FRC: Fiber-Reinforced Composite; PFC: Particulate-Filled Composite; Lc: Critical Length; AFM: Atomic Force Microscopy; SEM: Scanning Electron Microscopy; Vf: Fiber Volume Percentage; Vm: Matrix Volume Percentage; Ra: Surface Roughness

Introduction

Polymer matrix fiber-reinforced composites (FRCs) have design capability to significantly increase dental filling composite wear resistance. The dominant composite constituent as fibers are well-known to have great potential for exceptionally high strength and modulus that can transfer such enhanced mechanical properties through a composite when combined into a strong-bonded polymer matrix. FRCs have demonstrated various heightened statistically significant increases in mechanical properties for strength, modulus, fracture

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toughness and Izod impact toughness compared to standard dental particulate-filled composites (PFCs) and even amalgam for strength and fracture toughness [1-10]. Further, FRCs can significantly statistically pack better than amalgam [6,9], eliminate voids in the class II proximal box [6,9] and can wear less than enamel [6,10]. FRCs with continuous lengths are one of the most important material developments in the world today key to many diverse applications including use in various types of aerotech structures, aircraft, marine manufacturing, commercial/military cars and trucks, ballistic material, pipelines, water resistant surface protection and repairs. The micromechanics for critical fiber length ($L_c$) are defined by a required minimum length of a given fiber diameter and strength and fiber-matrix bond strength to attain maximum stress by preventing entire fiber debonding from the matrix within a unidirectional FRC during a properly applied tensile-type loading force [2,3,11,12]. FRCs are termed continuous when fibers are at least about 15 times $L_c$ [11]. On the other hand, FRCs are termed discontinuous when fibers are greater than $L_c$ but less than $15L_c$ [11]. Unfortunately, fibers less than $L_c$ provide no reinforcement as the matrix debonds entirely before the fiber can attain maximum strength and break [11]. Subsequent discontinuous FRCs satisfying $L_c$ are utilized in similar material applications to continuous use but where some material flow is needed for easy fiber distribution during molding to reduce production costs [13]. Effective discontinuous lengths for a dental FRC now in development start at approximately 1.0 mm or 2X $L_c$ [3]. Further, discontinuous FRC molding compounds are acknowledged as industrial materials utilized at length in electrical, automotive and general customer merchandise manufacturing [14]. As such, discontinuous FRCs meeting $L_c$ requirements have found important high-tech service for example in primary structures as in large engine strut fairings and secondary structural window frames for the Boeing 787 Dreamliner [13]. Another favorable consideration for FRC wear resistance at the susceptible cavity margin bondline related to common secondary caries is use of discontinuous fibers in supersonic aircraft adhesives with a glass fiber carrier for support as a means to provide pressure and minimize voids [15].

FRC Basics

Molding and Air Entrapment

Discontinuous FRC placement into a relatively complex internal dental cavity design requires molding capability with material deformation during placement. Due to the importance of molding fabrication, manufacture for resin pre-impregnated FRC complex curved parts into a preferred shape has been routinely studied. Individual fibers or sheet plys of fibers in discontinuous mat or in woven form must reorient or slip to slide past one another during the forming layup placement process and conform within the internal mold cavity surface that is accomplished by bending and stretching of the uncured fibers or fiber plys [16-19]. Resin pre-impregnation of fibers or a prepreg is the most common technique for processing FRCs [14] that is somewhat similar to resin pre-impregnation of particulate filler by reactive photocure resin into a paste form for dental composites. FRC prepreg supplied with moldable properties can produce complex formed parts during the application of pressure by a compression process [14,16,17,19-29]. In addition, resin pre-impregnated FRCs after forming into complex intricate shapes must also have sufficient tack to stick and generate a bond between adjacent fibers or plied sheets to stay in place before curing is initiated [16,17,23,27,30]. Trapping air pockets between each layer of material inserted into a cavity is an unavoidable and critical issue to control [14,15,20,25,26,28,29,31,32]. Before curing FRC prepregs must be consolidated with applied pressure to squeeze out entrapped air between prepreg layers, collapse voids and form the material through deformation to the exact complex internal cavity mold surface geometries by dispersing fibers uniformly [14,15,19-22,25,27-29,31-33]. As the FRC prepreg is compressed air is forced out of the cavity at the mold surfaces [14,15,19-22,25,27-29,31-33]. The fiber network provides sufficient permeability so that pressure carried initially by resin-rich areas forces excess resin to migrate into void space and toward escape vents at the surface where resin squeezes out of the prepreg FRC [14,15,19-22,25,27-29,31-33]. Pressurized resin flow with vents allows gas to escape that has been trapped between fibers, fiber plys and matrix during layering placement or mixed in with the resin before fiber resin pre-impregnation so that applied pressure can reduce subsequent possible voids and porosity in the final FRC polymerized hardened product [15,20,25,26,28,29,32].

Consolidation

Consolidation is a process that occurs in almost every FRC manufacturing process [14,31] particularly used for polymer matrix composites when making a part where pressure is applied to squeeze air and resin out of the prepreg to change the final material dimen-

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sions before reactive curing [14,15,19-22,25,27-29,31-33]. Usually the FRC prepreg resin content is relatively high [14,15,20,22,24-27,29,31,33]. However, compaction mold pressure supplies the means to provide a consolidation process where the fiber network also squeezes the matrix resin, monomer and particulates toward escape vents and out at the free-boundary surfaces so that the fiber bed ultimately carries most of the load and the uncured resin pre-impregnated FRC is compacted for a higher fiber volume percentage \(V_f\), lower matrix volume percentage \(V_m\) and minimum thickness [14,15,20,22,24-27,29,31,33]. During consolidation in the mold forming process resin can flow parallel to the fibers, transverse to the fibers or by shearing flow due to fiber redistribution movements from high pressure [14]. As the prepreg is compressed through-the-thickness fibers and resin will spread laterally and with fiber bending if necessary to eventually conform by the constraints from the internal dimensions of the cavity mold that may be complex curved geometry [14]. Fiber bed permeability relative to the resin flow is a new issue where resin flow or permeability increases with lower \(V_f\) [14,31]. During consolidation as the \(V_f\) increases with increasing pressure and time the fiber bed starts to carry more of the applied transverse compressive load [14,31]. Consequently at maximum consolidation and \(V_f\) spacing between fibers decreases producing significant fiber-fiber contact so that fibers carry the entire load and no force is carried by the resin also with no resin flow occurring [14,31]. Consolidation with forming of the FRC prepreg by pressure into the internal mold cavity for reactive polymerization of the resin is the final processing step. In turn, by appropriate interfacial shear bond strength with the polymer matrix, the \(V_f\) is important for enhancing FRC performance and mechanical properties with higher modulus, strength and fracture toughness [4-7,11,12,24,26,29,33].

Fiber Wash and Tack

Of some concern that causes detrimental reductions in mechanical properties, increasing pressure can possibly add to fiber wash as resin bypasses some fibers with unsatisfactory resin impregnation of fibers [33]. However, mechanical action has been shown capable of creating high local pressures that can assist in fiber coating, uniform fiber impregnation and resin movement that can be forcibly squeezed onto the fibers [33]. Lower resin viscosity with poor tack can also produce fiber wash even under lower pressure molding conditions with unsatisfactory resin impregnation of fibers. As a solution to prevent fiber wash, resin stickiness provided by tack provides a slight adhesion between fibers to reduce resin permeability through the fibers resulting in an increased more controlled resin pressure with even fiber distribution and low-resin-rich areas during compaction. For large parts FRCs are often made with resin pre-impregnated fibers or prepregs that are protected on both sides with easy-to-remove plastic release film. Often included as part of a prepreg matrix resin is an adhesive tack to assist in lay-up by adhering to adjacent plies during ply stacking [34,35]. Low viscosity resin and resin rich areas that create detrimental fiber-depleted areas and deviate from ideal micromechanics during stress transfer are important considerations in FRCs [14]. Tacky polymeric materials can provide substantial secondary bonding forces to hold resin chains better together and thicken the overall resin with increased viscosity. Further, tacky polymeric materials allow the higher viscosity resin to still moderately flow by breaking the weak secondary bonds linking resin chains together under an applied pressure and so reduce the formation of large detrimental low viscosity resin pools especially along all fibers and also at the cavity mold substrate surfaces. Nanoparticulate in the 50 nm diameter size provide tack presumable by weak secondary bonds since sintering that increases particle size to 270 nm diameters “switches” off tackiness [36]. Subsequent 50 nm diameter nanoparticulate can increase viscosity by holding resin chains better together to restrict resin flow. In addition, because of an active high surface area, nanofibers at small concentrations will also thicken the resin to increase viscosity and eventually produce a gel tackifier.

Dental FRC Basics

Fiber Alignment, Pressure Consolidation and Particulate Distributions

50 nm diameter size particulate will pack through a cavity mold to nest around and between larger particles for example in the 270 nm diameter size or larger [36,37] so that small diameter nanoparticulate continue to squeeze upward and accumulate at the surface. In fact in an FRC filling, particulate of all sizes along with monomer and resin will squeeze upward from the fiber bed to form a type of PFC top surface layer with fibers lying underneath [2,6,8,9]. Fibers well above \(L_c\) align parallel to the surfaces [1,6]. So, when fibers press flat

to the filling surfaces corresponding to the occlusal surface from the cavity floor toward the top, the FRC matrix with particulate, resin and monomer squeezes away from the fiber network to produce a relatively smooth top surface depending on the surface of the compaction tool. Even with high fiber damage during rough hand mixing and packing processes, because the fiber is well above $L_c$ requirements, the fiber is still tightly bonded in the FRC matrix shown by atomic force microscopy (AFM), figure 1. The FRC matrix is composed of resin, monomer and particulate that squeeze away from the FRC network within the cavity mold to conform around and over a relatively immobile damaged fiber retained at the surface. The AFM sample for figure 1 was compacted with a somewhat smooth compaction tool using a glass slide as the final mold surface for a low surface roughness (Ra) without any surface finishing or polish. The AFM image was taken from the bottom photocure sample surface performed through interrelated polymerization shrinkage tests.

![Figure 1](image.png)

**Figure 1:** A single fiber in an FRC with PFC-type matrix compresses flat onto a cavity mold surface. The fiber network squeezes PFC matrix toward the surface with removal of any excess at the margin borderline vents to expose a damaged fiber that has been bent to about a 90 degree angle with many surface defects that still provides an Ra of only 16.019 nm.

As previously explained, during the average compaction process for a discontinuous FRC the material flow toward the surface removes excess matrix that squeezes out of the fiber network at the border surface escape vents. Therefore, before any finishing or polishing, the result is a surface composed entirely of matrix with the fiber network generally entirely beneath the surface. In addition, flowable molding pressure helps to impregnate fibers better by a high viscosity matrix with sufficiently high tack properties that holds fibers together before cure and fills in void space or surface porosity especially alongside internal surfaces of the cavity mold. FRC finishing with polish practically eliminates the top matrix surface layer so fiber wear thinning with smoothing of the surface fibers that shelter the PFC matrix

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with resin, monomer and particulate becomes the eventual final surface finish [10]. In fact, fibers wear thin into flat plate-like particulate that continue to break down into nanoparticulate conceivably capable of creating a polish for the final wear surface [10]. AFMs show the top PFC-type matrix surface layer of a discontinuous dental FRC that can compact even better with higher packing forces than an amalgam [6,9], figures 2A and 2B. Further, chips in two PFC-type matrix surfaces are shown blocked by underlying fibers beneath, figures 2C and 2D. The compaction tool surface for a top cover with the samples in figure 2 was machined non-polished from ultra high molecular weight polyethylene during related polymerization shrinkage tests. The fiber has a surface area a couple orders of magnitude larger than a particle of similar diameter. Subsequent fibers beneath the surface then act as major barriers to crack propagation in addition to adding high fracture toughness properties for the PFC-type matrix and virtually stop crack propagation near the material surface. Further, the Ra of the fiber chip defect surface appears considerably smoother than the PFC-type matrix top surface Ra.
Figure 2: AFM for FRC with PFC matrix compressed flat by a non-polished machined tooled cover into a cavity mold squeezes PFC matrix toward the surface with removal at the margin borderline escape vents. No subsequent cutting, finishing or polish: (A and B) Without surface chip. (C and D) With surface chip exposing fiber underneath. Average Ra for 2 samples: PFC-type matrix surface 202 nm; PFC-type matrix with surface chip exposing a fiber surface underneath 354 nm. Ra not statistically significantly different between PFC surface and PFC surface with a chip.

Scanning electron microscopy (SEM) imaging contrasts fracture surfaces between a polymer matrix PFC and the same polymer PFC matrix with 35 wt% pure quartz fibers well above L, from Izod impact fracture toughness tests [1,2], figure 3. The PFC filler was composed

of multimodal spherical zirconia silicate ranging in sizes from 10 nm to 3.5 µm, figure 3A. Following fracture with fiber debonding, figure 3B reveals how the fiber network compresses the PFC matrix with sufficient force to coat the fiber surfaces with an exceedingly smooth layer of polymer and probable small nanoparticulate. In addition, the polymer matrix surrounding the fibers demonstrates a highly consolidated multimodal particulate.

**Figure 3:** SEMs Izod impact toughness fracture surfaces. Magnification 5000x, Scale Bars 5µm. (A) PFC. (B) PFC with 35 wt% quartz fibers well above Lc.

**FRC Advancements in Mechanical and Clinical Properties**

FRC resin pre-impregnation or fiber wetting by a reactive resin can eventually produce a slightly tacky material that may be incrementally layered before initiating cure or co-cured in separate incremental layers [14] so that dental photocure applications seem ideal.
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For clinical use. Because of the molding capability for discontinuous FRCs, a breakthrough in dental filling materials with fibers notably greater than \( L_c \) has become the most important development in dentistry since the amalgam. FRCs complying with \( L_c \) requirements for enhanced FRC properties have demonstrated great statistically significant improvements in various tests over 2 dental PFCs, a dental PFC with fibers below \( L_c \), and a popular Tytin® amalgam in mechanical properties for flexural and yield strengths in addition to fracture toughness properties for resilience, work of fracture, critical strain energy release and critical stress intensity factor also with Izod impact toughness tests over two PFCs [1-10]. Although FRCs demonstrate statistically significant increases for the mechanical property of modulus over 2 dental PFCs and a dental PFC with fibers not attaining \( L_c \) [1-10] the amalgam modulus tested higher than the FRC and much higher than two dental PFCs or a dental PFC with minimal microfiber lengths [1,2,8]. Of common clinical importance, higher mechanical properties translate into less gross fracture with increased strengths, less microcracking with higher modulus, and reduced marginal chipping with higher fracture toughness properties. Further, FRCs at just 9.2 wt% fibers have demonstrated statistically significant better ability to reestablish the interproximal contact than a dental PFC and two high consistency amalgams [6,9]. Also, FRC filling materials show far-reaching statistically significant improvement for void reduction compared to dental PFCs [6,9].

Clinical Concerns

Amalgams vs. Dental PFCs

The main barrier for both dental PFCs and amalgam is secondary caries, but PFCs have higher decay-related failures when measured against the amalgam [38-63]. However, the fracture of filling material was a second common failure often seen [44,49,54,55,60,6,1]. Overall, the PFC failure rates are summarized as 2-3X greater than the amalgam at 3 - 7 years in well-controlled randomized clinical trials [53,54,59,62] with expected increased PFC failure rates over longer periods of clinical service. Failure due to secondary decay was 3.5X more frequent with PFCs than amalgam [54]. Interproximal failure rates for PFCs increase even more to 5.4X greater than amalgams for secondary decay [52]. Repairs were 7 - 8 times more frequent for PFCs than amalgams [53]. Also, significantly higher marginal ditching from occlusal wear of PFCs compared to lower amalgam wear rates should be a key related clinical problem that appears to greatly contribute to secondary decay. In terms of wear, PFCs wear considerably more than enamel [64,65] while amalgam wears only slightly more than enamel [64].

Modulus Resistance to Debonding at the Margin

Contributing toward marginal leakage with secondary decay, the PFC must distort more with increased shear debonding at the margins due to a very low PFC modulus [8,66] of approximately 6 to 19.5 GPa [1-4,6,8-10,66-71] when compared to the high modulus of enamel at approximately 48-94GPa [72,73]. However, an accurate modulus for amalgam at 44 GPa is closer to the modulus of enamel that should greatly reduce problems with alloy deformation and shear debonding along the margins [8]. On the other hand, the dentin modulus of about 13.7 - 18.6 GPa [68,72,74-77] appears to limit enamel shear debonding loads to a degree by communicating with pain receptors of the pulp from occlusal forces directed at the pulpal floor. Further, water is a solvent that produces plasticization of a polymer evaluated as decreased hardness or increased softening of the top surface layer [78-82] that would reflect lower PFC modulus at the surface and accentuate shear deformation along the margins. Also, the PFC appears to debond a bit more at the margin from occlusal forces due to relief of internal residual stresses created during polymerization cure shrinkage [65,83] although there is only limited clinical evidence [66]. In addition, leachable monomers of dental PFCs [84,85] have been found capable of supporting bacterial growth [45,84], whereas silver antimicrobial properties [86-91] are an important advantage for amalgam.

For another advantage, the FRC molding compound can have a modulus of 31 - 34 GPa that is closer to amalgam and should reduce interfacial shear debonding between the filling and the tooth substrate under occlusal loading [8]. Most importantly, extremely high modulus quartz fibers at around 72 - 78 GPa [92,93] that are more similar in stiffness to the enamel modulus of about 48 - 94 GPa may bond to tooth substrate for an even greater reduction in the problems associated with deflections and shear debonding along filling cavity margins. FRCs produce nil polymerization shrinkage stress along the cavity mold surfaces because of the extremely high modulus fibers that pack parallel to a plane with almost full complete cure shrinkage occurring transversely toward the substrate for improved adhesive...
bonding [94]. In fact, adhesive fiber films have been shown to support bonding pressure for increased resin flow with good substrate coverage contact and to maintain resin in the bond plane that minimizes voids or porosity [15]. Further, chemically resistant fibers act as barriers to moisture that should reduce leakage at a bond interface [95]. In addition, due to high viscosity FRCs can successfully incorporate efficacious broad-spectrum triclosan antimicrobial without producing an unmanageable material as the dental PFCs that lose all consistency and turn into a gluey state when triclosan is added [96-98].

**Technique Sensitivity of PFC Placement**

Amalgam is widely accepted as the clinical standard for direct filling placement. On the other hand, dental PFCs are considered technique sensitive in terms of placement for posterior teeth [99] that appears to be a chief cause of failure during the first five years, but secondary caries becomes the main failure after five years [100]. The amalgam filling is considered to be much easier to use than the dental PFC [101-107]. Dental PFCs have been estimated to take twice as long to complete as a similar amalgam [102]. As an aesthetic replacement, the FRC produces a material with highly increased excellent packing consistency for easy handling [8,9]. Many problems that occur with PFCs include poor interproximal contacts [101,103,105-120], voids in the proximal box [108,121-125] and overhangs [8,9,101,126] that can be eliminated with FRCs [8,9]. Due to low viscosity with poor consistency, dental PFCs do not pack during placement with condensation forces of sufficient degree, comparable to the amalgam that frequently prevents reestablishment of the interproximal contact in a class II filling. The addition of even small amounts of 3 mm length quartz fiber at only 9.2 wt% average to a Z100® PFC could produce statistically significantly increased interproximal contact circumferences over a 3M Corp. Z100® PFC and also greater contact circumferences than 2 high viscosity amalgams (p < 0.0001) [9,94]. Incorporation of 3 mm length quartz fiber at just a 9.2 wt% average to the Z100® PFC could produce statistically significantly greater condensation forces than both the 3M Corp. Z100® PFC and a high viscosity amalgam (p < 0.001) [9,94]. Because incorporation of a standard 30 - 35 wt% 3 mm length fiber into a similar 3M Corp. Z100® PFC can be accomplished for molding even higher condensation forces are expected than the PFCs at 9.2wt% fiber. Additional results show that PFCs with a small amount of quartz fibers significantly statistically reduce voids compared to similar identical PFCs without fibers p < 0.00001 [9,94].

**Wear Problems with PFCs**

Concerns about material wear are reflected by moderately high rates with a ditched trench at the filling margins that have been much deeper for dental PFCs than the amalgam [64]. Also, wear for dental PFCs increases with wider cavity preparations with less protection from enamel that "shelters" the dental PFC [37]. Early PFC wear problems were corrected with nanoparticulate that were found to fit between and shelter larger particulate for subsequent reductions in PFC wear rates in the new microhybrids [37,127]. Although PFCs as 100% nanoparticulate or microfills wear better than microhybrid composites, nanocomposites are weak and fail more frequently by marginal fracture compared to common microhybrid dental PFCs [127]. Nevertheless, microfill nanoparticulate PFCs still create a marginal trench during wear [64,127]. Today even the best dental PFCs wear 10 to 20 μm more per year than enamel that is approximately 100 to 200 μm over ten years [65]. FRC wear studies have shown material strength reduces wear rates because fibers support loading conditions and are particularly wear resistant as fiber lengths become longer than the wearing plowing grooves [128,129]. Further, FRCs with high modulus fibers decrease strain related microcracking and as a result moisture adsorption [95,130] that should help to reduce polymer plasticization, hydrolysis and chain scission [130-132]. Of great clinical significance, higher mechanical properties convert into less bacterial plaque accumulation or marginal leakage due to fewer defects from gross fracture, chipping of the margins or marginal shear debonding. Also, PFC marginal wear ditch trenches appear to be a significant defect problem after only 3 years of clinical service [64]. The American Dental Association has recommended on several occasions not to use PFCs in load bearing areas [133-135]. As an example, deeper larger more extensive fillings with more surfaces are considered grounds for using amalgam instead of a PFC [51,136]. In addition to loss of the sheltering effect by tooth enamel, as fillings increase in size a greater amount of filling material is subjected to direct occlusal loading while more margins are susceptible to larger stresses that might deflect a low modulus PFC and cause distortion with shear failure to break an adhesive marginal bond [8].
In Vitro Wear Tests

The University of Alabama at Birmingham (UAB) in vitro wear device was developed to predict long-term clinical use of PFCs by three-body wear with a 44 µm diameter unplasticized polymethacrylate bead slurry as a stable artificial food bolus [64]. Molar occlusal surfaces are ground down flat to a 600 grit silicone carbide metallographic paper and still entirely in enamel so that a cavity preparation can be placed at 4.0 mm diameter and 3.0 mm deep [64]. Subsequent filling materials can be placed for wear with a flat polyacetal 8.0 mm diameter stylus vertically loaded at 17 pounds or 75 Newtons for evaluation after 400,000-cycles at 92 hours corresponding to 3 years of clinical service [64]. At maximum load the stylus rotates 30° and then counter rotates back before lifting up. Wear patterns for the UAB in vitro wear device correspond to clinical service that show ditched trenches below the enamel margins for all PFC dental materials including a smaller marginal defect for amalgam, figure 4. No enamel sputter coating desiccation occurs because all SEM samples are duplicate replicas after a debridement with subsequent impressions and stone models for accurate representations without artifacts.

Figure 4: Scanning electron micrographs for a series of materials after 400,000 cycles at 500X magnification. The materials are (A) Dispersaloy amalgam (L.C. Caulk), (B) Heliomolar (Ivoclar North America), (C) Herculite (Kerr Manufacturing Co.), (D) P-50 (3M Dental Products Co.), (E) P-30 (3M Dental Products Co.), (F) Prisma APH (L.D. Caulk), (G) and Ful-Fil (L.D. Caulk). (H) represents P-30 after wear testing at a higher magnification-scale bar 24 um. The arrows in H indicate a dislodged particulate space and two extruded quartz particles. [Reprinted from the Journal of the American Dental Association, Volume 130 (9), Karl F. Leinfelder and Shiro Suzuki, In Vitro Wear Device for Determining Posterior Composite Wear, Pages 1347-1353, 1999, with permission from Elsevier].

FRC Wear with Fibers Well Above \( L_c \), Compared to a PFC with Microfibers Well Below \( L_c \)

From identical UAB 3-body wear tests at 400,000 cycles the FRC molding compound material with fibers over \( L_c \) have tested to wear far better than a dental PFC with fibers less than \( L_c \) [10,137] and even wear better than enamel shown by profilometer tracing in figure 5 [10,137]. Consequently, because amalgam or all dental composites wear more than enamel [64], FRCs can thus be developed to provide possible wear better than both amalgams and dental PFCs. The FRC has high-strength quartz fibers above the \( L_c \) that do not debond from the filling material for subsequent effective sheltering protection of the PFC matrix. Conversely, the PFC with microfibers below \( L_c \) also in profilometer tracing by figure 5 wears by forming a ditched trench at the margins that is found with all of the other dental PFC filling materials tested by UAB in figure 4 showing well-defined cavity filling wear depressions. So, the FRC molding compound satisfying \( L_c \) wears above enamel with a smooth transition to the enamel margin without the common dental PFC material detrimental ditching trenches that can pool fluids, accumulate bacterial plaque and accelerate wear. Blending equipment should rectify a small problem with a quartz fiber/
yarn pull-out during wear testing as the result of weaker and insufficient hand mixing the FRC composite necessary for an extremely thickened and easily packable molding compound. Fibers are known to be the strongest form of material and produce some of the strongest materials known to mankind. Consequently, when comparing silica-based ceramics as similar types of examples that are well known for wear resistance with low rates only slightly more than enamel [64,138] 99.9% silica pure quartz fibers well above $L_c$ at some minimum FRC material percent are correspondingly expected to produce low wear. Fortunately, fibers pack parallel to the pulpal-occlusal plane to evenly wear thin and smooth [10] whereas ceramics especially with larger crystalline particulate and higher surface hardness values are associated with increased abrasion of opposing enamel [138-141].

**Figure 5:** Profilometer tracings (A) FRC with fibers greater than $L_c$ shows low wear above the enamel margin and a relatively smoother surface. (B) PFC with microfibers much less than $L_c$ produces deeper wear below the enamel margin with a relatively rougher surface.

**SEM Imaging for FRCs with Fibers Well Above $L_c$ and PFCs with Microfibers Well Below $L_c$**

SEM imaging in figure 6A from the UAB FRC wear tests demonstrates a smoother appearance for the wear surfaces of the FRC with fibers attaining lengths greater than $L_c$ compared to the PFC with microfibers well below $L_c$. In fact, the general overall FRC image is a wear-in polish seen in figure 6A that is almost similar to smooth polished enamel. The rougher FRC surface in figure 6B is an exception rather than the common typical smooth polished appearance and could be the result of lower rotational and counter-rotational loading force patterns by the UAB wear device that can limit the breakdown rate of surface fibers into smaller particulate. Further, the obvious signs of microfiber debonding from the PFC matrix are predicted by micromechanics for fibers that do not satisfy the requirements for $L_c$. The differences in surface roughness between the FRC and PFC with microfibers below $L_c$ are somehow similar in magnitude when comparing the profilometer tracings and SEM imaging. Quartz fibers at 9 µm diameters in the FRC after compaction placement, finishing, polishing and 400,000 wear cycles are not visible at 200X magnification and apparently completely covered with a polymer-style matrix material. However, the microfibers with approximate similar 10 µm diameters in the PFC are easily seen in isotropic orientation with figure 6C with some planar rudimentary unidirectional orientation in figure 6D after identical wear testing. Also, the PFC microfibers do not demonstrate exceptional sheltering properties for the matrix such that the 3-body 44 µm diameter PMMA bead slurry is capable of wearing the matrix between the more wear-resistant fibers.

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Figure 6: SEMs Magnification 200X and Scale Bars 100 μm: (A) Typical FRC smooth and even surface with no fibers or particulate evident. (B) FRC imaging of an occasionally rougher area with particulate bumps spread across the level surface, but no fibers present. (C) PFC with much rougher microfiber surface. (D) PFC with extremely much rougher microfiber surface.

SEM at higher magnification show the atypical FRC surfaces with a rougher finish to better understand and visualize fiber wear with fiber thinning into more-flat plate-like particulate. Flatter particulate distribute wear loads to a plasticized polymer matrix more evenly than spherical particulate that transfer loads by shear forces through the weaker hydrolyzed polymer matrix surface. Flat plate-like particulate compress wear loads by an even stress transfer to form adhesive bonds with the surface polymer matrix. Conversely, unsheltered irregularly-shaped spherical-like particulate concentrate wear stresses into the polymer matrix by shearing forces that can then dislodge the more sphere-shaped particulate. Resultant flatter plate-like particulate then create a more even polymer surface with few hills or valleys. Subsequent flatter particulate apparently compress back into the FRC polymer matrix with particulate bonding or recementing as a manner of wear resistance, figures 7A and 7B. Relatively flat particulate are spread uniformly over the entire surface in figure 7A. Larger

remnants of a fiber not yet broken down into particulate still visible and identified with arrows are bonded into the polymer matrix, figure 7B. However, the wear-in process is incomplete for the rougher FRC surfaces imaged at 5000X. Most importantly, over time fibers apparently appear to continually wear thin and possibly produce a smooth surface similar to figure 6A.

Figure 7: SEMs Magnification 5000X and Scale Bars 5 μm showing a more uncommon rougher portion of an FRC (A) FRC following fiber wear thinning more-level and even flat-like particulate are spread uniformly over the entire surface and compressed into the polymer matrix. (B) Larger FRC fiber remnants are identified by arrows and still visible before breaking down completely into small flatter particulate with a more level plate-like bottom surface.

The chief common wear property for the FRC with fibers longer than $L_c$ is a smooth hard polymer wear-in surface that is similar to enamel with no apparent features for fibers or particulate, figure 8A. A rougher FRC surface is imaged before the wear smoothing is complete with figure 8B. As an important point in micromechanics, the PFC with microfibers that do not attain $L_c$ can apparently produce one of the rougher surfaces with fiber ends extending upward from the PFC matrix and small particulate visible, figures 8C and 8D. Two PMMA beads in figure 8C and upper right corner appear to have been trapped, hydrolyzed by water adsorption, torn apart, and then retained by the sharp fiber ends to be compacted back into the PFC. Also, FRC polishing may be created in part with nanoparticulate by wearing and breakdown from hard crystalline fiber material. Further, the FRC compressive consolidation properties for a fiber network underneath the polymer surface appear to provide recompaction of particulate as fibers are broken down and then compressed back into a plasticized polymer matrix under wear loading forces. A P30 dental composite from figure 4E is shown for comparison to emphasize the wear ditching trench that is approximately 44 μm deep, figure 8E. Also, note the moderately rough enamel wear surface at the top of the SEM in figure 8E. For further evaluation, the P30 dental PFC from figure 4H has a relatively rough surface with rapidly undulating high and low areas covering the surface, figure 8F.

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Figure 8: SEMs A-D Magnification 500X and Scale Bars 50 μm (A) FRC has worn sufficiently to produce a smooth polymer surface that is extremely well polished. (B) FRC with rougher surface and particulates before the wear-in process is complete. (C) PFC with isotropic microfiber orientation has an extremely rough surface. (D) PFC even with some planar and unidirectional microfiber orientations has an even rougher surface.

Discussions
Fiber Crystalline Properties

The properties for strength and modulus are higher with fibrous structure than in any other material form most notably due to a high crystallinity with low defects [142]. When fibers are extruded alignment occurs in the filament and with more orientation at the surface due to interaction at surfaces of the spinneret holes [142]. Further, the spun fiber is stretched that increases alignment along the fiber axis with increased strength and modulus [142]. Increased orientation increases fiber crystallinity that reduces moisture adsorption and increases chemical resistance [142]. Conversely, inorganic silica-based (SiO₂) glasses are amorphous with non-crystalline structure due to the addition of some metal oxides that break the silicon-oxygen bonds and produce isotropic properties with low crystallographic orientations [142]. Subsequent amorphous glasses produce softer, weaker particulate with less crystalline structures and no long-range orientation similar to a fibrous material [142]. Also, moisture decreases amorphous glass strength that increases with constant loading forces [142]. As a result crystalline fibrous material breaks down into harder particulate at a reduced rate compared to amorphous particulate.

Crystalline Nanoparticle Polishing

The dental FRC molding compound was reinforced with a pure quartz 99.9% silica fiber that is considered the most crystalline of silica-based fibers. On the other hand, the PFCs with microfibers much less than Lc were made with a weaker less crystalline fiber that might wear down faster to then eventually degrade at a higher rate. Subsequent high crystalline nanoparticulates then apparently produce a smaller longer-lasting particulate for a more polished wear surface than amorphous nanoparticulate with defects that can accelerate wear and increase the subsequent silica dissolution rate. SEMS for figure 9A-9D show enamel surrounding the filling materials that are somewhat identical with all enamel surfaces polished to a high shine. In addition, the SEMs were completed with desiccated enamel from the gold sputter coating that formed numerous cracks.

Wear of fibers into more crystalline chemically resistant nanoparticulate may produce a high-quality polishing agent that is a possible reason for the highly polished enamel on all samples. The overall wear for the FRC samples is for a smooth polished surface whereas the general wear for the PFC with microfibers well below Lc is rough. With fibers well above Lc, fibers do not debond and support wear loads with orientations parallel to the occlusal plane for fiber wear thinning and a smooth surface. However, with isotropic fibers well below Lc, fibers debond from the matrix and also extend vertically to orient away from the occlusal plane in some manner. Subsequent exposed isotropic fiber ends increase stress on the fiber that appears to heighten wear for the PFC microfiber material with extremely rough surfaces.

Sample margins are imaged by SEM at 400X in figure 10A-10D. But, gold sputter coating has created margins with enamel desiccation. The typical FRC is sufficiently smooth so that no fibers or particulate are visible. On the other hand, the PFC with microfibers well below \( L_c \) is roughened extensively by the isotropic orientations of the disoriented fiber arrangements. However, the enamel surrounding all samples is polished smoothly to a high shine. But, the FRCs may be polished slightly better with a somewhat higher shine than the PFCs with the small microfibers less than \( L_c \). By close examination of the SEM at 500X the enamel surface above the wear ditch in figure 8E from a dental PFC without fibers has a rougher surface with the same identical UAB wear simulator methods.

![Figure 10: SEMs Magnification 400X and Scale Bars 50 um.](image)

**Figure 10:** SEMs Magnification 400X and Scale Bars 50 um. (A) FRC with fibers well above \( L_c \) shows a smooth surface and polished enamel. (B) FRC with fibers well above \( L_c \) shows a smooth surface and polished enamel. (C) PFC with microfibers well below \( L_c \) shows a rough surface but with a polished enamel. (D) PFC with microfibers well below \( L_c \) shows a rough surface and with less smooth enamel.

SEM imaging provides an FRC sample with one of the rougher margins. FRCs with planar oriented fibers wear thin into more flat-like particulate that compress back into the polymer matrix, figure 11, also see figure 7 at higher magnification. Regardless of exposing FRC particulate in the matrix, the enamel is still well polished to a high shine. Fibers oriented parallel with the occlusal-pulpal plane during the packing pressure for clinical insertion appear to wear thin and even grind up into the crystalline more-even flatter wear particles or possibly some broken down fiber remnants. Subsequent more-flat particulate and small fiber remnants can then press back into the polymer surface through compression with consolidation from the underlying fiber network. Alternatively, fiber wear-thinning can eventually produce a fine crystalline nano-polishing agent. Also, other common amorphous matrix nanoparticulate appear to have the capability to squeeze under wear pressure away from the fibers and rise to the surface through consolidation properties of the fiber network underneath to densify and harden the polymer matrix surface. PFCs with more spherical matrix particulate without underlying fiber network consolidation on the other hand create rougher surfaces due to particulate that dislodge from the matrix and increase wear rates.

Figure 11: SEM Magnification 400X and Scale Bar 50 um. A rougher margin of an FRC with fibers above Lc, but no fiber lengths are seen on the surface. The enamel to the right appears highly polished when compared to the ditched enamel margin on the top in Figure 8E for the PFC P30 from Figure 4E. Enamel fracture lines are due to gold sputter coating desiccation.

Polymer Matrix Plasticization and Hydrolysis

The polymer matrices for all composite materials studied were subjected to moisture during the 3-body wear tests through a 45 µm diameter PMMA bead water slurry under combined rotational and counter-rotational 75N loading conditions that would produce extra moisture adsorption of the polymer matrix at the composite filling surface. High loading can increase strain related microcracking that increases the rate of moisture adsorption [95,130,143]. A molecule is considered polar when a permanent dipole exists because of unbalanced electron distributions with positive and negative charges on separate functional group areas [144,145]. Water is a polar solvent that allows electron conduction [145] so that increasing polarity of the resin chain can increase moisture adsorption [146,147]. As water molecules diffuse into the polymer matrix and occupy positions between the polymer chains, interchain distance is increased while secondary intermolecular bonding is reduced [78,146,147]. Consequently, polymer chains become less entangled and can become mobile to slip past one another under higher loading conditions [78,146,147]. As a result, water causes the polymer to soften by plasticization and polymer degradation from bond cleavage through water hydrolysis with an apparent overall loss of modulus [81,130-132,146-148]. Subsequent moisture adsorption of a polymer can reduce all of the mechanical properties like strength, modulus, or fracture toughness.

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[130-132, 146, 147, 149-151]. In addition, water or solvent plasticization of a polymer is measured by reduced hardness or increased softening of the top surface layer [78-82]. Hydrophilic or polar polymers readily adsorb water to increase degradation rates [146,151]. As a primary example, triethylene glycol dimethacrylate (TEGDMA) is a hydrophilic dental monomer [152] that is highly soluble in water [45,84,85,152,153] and been shown to produce high water plasticization or reduced surface hardness [78]. Further, a standard dental polymer of vinyl ester resin (BisGMA) and TEGDMA will accelerate degradation by enzymatic action to produce a softened surface with increased wear that will break down even more easily by mechanical forces [154].

FRC Flat-Particulate Bonds and Wear-in Consolidation

As well as water adsorption degrading polymers by hydrolysis [81,130-132,146-148], some residual unreacted monomer [153,155] and remaining unreacted pendent resin groups [155,156] have high potential to eventually interact at the polymer surface. As polymer chain scission occurs, dangling free radicals may combine with unreacted residual surface monomers and unreacted pendent methacrylate double bonds to help retain broken fiber remnants and more flat-like particulate by free-radical adhesive bonding. Also, secondary bonding with the polymer matrix will help maintain hardened silica inclusions and particulate that smear by compression into the softened polymer surface layer. Particulate with flattened lower surfaces will distribute forces more evenly to better bond by a compaction process into the softened polymer surface compared to spherical particulate with possible concentrated forces that shear into the composite and increase the breakdown of the polymer matrix through surface fracture. As a general appearance for the FRC wear surface, newly formed more-flat particulate appear to compress back with consolidation into the composite. Concurrently, unreacted monomer or resin, hydrolyzed or plasticized polymer and smallest particulate squeeze upward away from the highly immobile fiber network underneath to move toward the surface and surround the new plate-like more-flattened particulate. Although nanoparticulate tend to pack around larger particulate [36,37], smallest particulate still tend to flow during FRC consolidation and accumulate in excess at the surface to then possibly harden and densify the resultant polymeric surface. The subsequent average FRC wear loaded surface has a rather unremarkable glossy hardened polymer facade with virtually no visibly appearing particulate or fibers evident. Consequently, crystalline nanoparticulate must also compress into the polymer matrix as a portion of the final wear surface. In fact, although nanoparticulate are impossible to see in SEM imaging [37], theoretical calculations have considered the possibility that no more than 6 wt% non-agglomerated evenly dispersed nanoparticulate at 20 nm radius with less than 100 nm spacing are needed to protect the polymer surface during wear [37]. The mechanical wear action of producing highly crystalline nanoparticulate from wear-thinning of pure quartz fibers combined with rotational wear loading forces to compress nanoparticulate back into the polymer surface would appear able to also laterally smear the top surface layer and provide a form of densified uniform dispersion. The dissimilar smoother polymeric FRC surfaces compared to the rougher flattened-particulate-filled FRC surfaces have differences possibly related to constant different artificial rotational loading smearing forces that either create higher compacting forces for greater consolidation or lower forces for less consolidation. Resultant higher compaction wear rotational loading forces then appear to squeeze hydrolyzed and plasticized polymer with amorphous nanoparticulate upward while simultaneously entirely wearing flat particulate down into fine crystalline nanoparticulate. Then with lateral smearing compression nanoparticulate are possibly then spread back into the polymer surface to create the typical hard and wear-resistant smooth surface. Conversely, the less common lower consolidation can retain exposed broken surface fiber more-even flattened particulate that could possibly eventually continue to break down into small nanoparticulate and then better compress back at a later time by fiber-network consolidation into a smooth polished composite polymer surface.

Wear Defects Related to Plaque Accumulation

Secondary decay with PFCs is related to marginal defects that reduce longevity for lower filling material lifespan and increase bacterial leakage with greater plaque accumulation [157]. Subsequent bacterial leakage is thought to produce pulpal damage in dental PFCs [157]. However, amalgams seal filling margins with corrosion by-products [157]. Of alternate concern, after the initial marginal shear debonding between low modulus PFCs and the enamel [8], wear ditch trenches must be considered a contributing factor toward marginal defects that accumulate. By some relevance in comparison to marginal defects, initiating primary pit and fissure decay occurs with high incidence before smooth surface decay and is the most common type of decay [158-165]. Pit and fissures are on the order of about 0.1 mm wide with deep invaginations that prevent removal of plaque by proper toothbrush cleaning with bristles on the order of 0.2 mm diameter [163]. But, regarding minimum sizes for material imperfections with moderately good natural cleaning by shearing forces of the tongue, on lingual material surfaces defects of approximately just a few micrometers were initiating points for bacterial adherence [160]. Further,
rougher dental composite surfaces have more bacterial adherence than smoother surfaces [166]. Narrow pit and fissure decay influences caries occurrence 8 times more often than a smooth surface and account for 80 - 90% of caries in permanent teeth [165]. Although the 1st and 2nd molars account for only 13% of all tooth surfaces, they represent 88% of decay [164]. In fact, Buonocore introduced pit and fissure sealants in 1971 as a protective layer to reduce microbial colonization into pits and fissures [167,168]. Also, *Streptococcus mutans* is considered an important part in the development of caries and found with significant quantities in carious fissures when compared to non-caries fissures [158]. Consequently in terms of secondary decay, wear ditch trenches increasingly reduce toothbrush access and normal salivary cleaning over time when compared to initial smooth filling transitions with the enamel when a filling material is first inserted. Combined relationships between marginal leakage at the susceptible cavity bondline for recurrent decay with wear ditch trenches are examined in further details by studies investigating interactions between material surface free energy, Ra and bacterial adhesion. Reduced surface free energy or hydrophobic and nonpolar surfaces [169-173] and lower Ra or smoother surfaces [166,173] will both reduce bacterial or plaque accumulation. Consequently, the importance of the initiating primary pit and fissure decay as surface invaginations is a key relationship observed in a large assortment of oral Ra bacterial adhesion studies [173] and a relevant concern with marginal wear ditch trenches in all posterior fillings.

**FRC Adhesives and Hydrophobic Resin Systems**

In addition to FRC reduced wear less than enamel to protect the cavity margin, FRCs have potential to seal the margins with insoluble fibers. The use of fibers in aerotech adhesives with a glass fiber carrier for support as a means to provide pressure and minimize voids [15] can also be applied to FRC hydrophobic resin adhesive and bonding systems for infiltrating a substrate surface. Surfaces are not perfectly flat and contain roughness with high and low points surrounding air pockets that require pressure from a deformable adhesive like a carrier FRC film to fill in the low saddle points for uniform contact and reduce entrapped air [174,175]. Trapped air reduces adhesion while resin flow increases substrate contact and displaces entrapped air [175]. As a result, detrimental hydrophilic monomers or acids that are incorporated for surface capillary infiltration in dental adhesive bonding agents [176-178] can be eliminated by FRC adhesive pressures [8,9]. With high FRC packing forces, hydrophobic resin systems, nanoparticle and nanofibers can be squeezed from adhesives and bonding agents by operator pressure into mechanical retention sites from the core fiber network. Higher FRC viscosity with increased packing consolidation pressure provides for incorporation of the low-viscosity vinyl ester oligomer ethoxylated bis phenol A dimethacrylate (BisEMA) resin [9,179]. BisEMA increases the hydrophobicity with lower polarity of the molecular chain for improved moisture resistance [180]. But, low viscosity BisEMA requires thickeners for adequate clinical packing placement pressures that are supplied most effectively with fibers above Lc and some nanoparticulate [9]. As a result, along the filling margins particulates, resin and monomer can then be compressed out of the FRC adhesive system onto a surface [2,8,9], figures 1-3, to secure the bond with high percentages of chemically resistant quartz fibers, nanofibers and particulate. Subsequent high concentrations of insoluble fibers and particulate with an adequate surface resin coating are projected to help complete a lasting seal as a fine moisture barrier at the margins [94,181]. After the filling margins are sealed effectively with insoluble fibers consolidated by nested particulate, the remaining cavity can be incrementally cured with FRC molding compound.

**Conclusions**

FRCs with fibers well above Lc can provide multiple advantages to offset numerous problems encountered with PFCs to reduce secondary decay and extend the lifetime of a dental composite filling. Common wear problems identified with dental PFCs most notably with ditched trenches well below the enamel surface can be eliminated by FRCs with fibers well above Lc that have shown smooth wear surfaces under identical conditions to be less than enamel. Consequently, ditched PFC wear trenches that would accumulate plaque and pool fluids with bacteria can be replaced by FRCs that wear less than enamel and produce a crystalline nanoparticulate polish. As a result, FRCs that wear smooth above the enamel margin can then provide a self-cleansing type of transition with the enamel margin and polished filling surfaces.

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Conflicts of Interest
Authors declare no conflicts of interest.

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