EFFECT OF COMPOSITE TYPE AND PLACEMENT TECHNIQUE ON POLYMERIZATION SHRINKAGE STRESS

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ABSTRACT

Vilhelm G. Ólafsson: Effect of Composite Type and Placement Technique on Polymerization Shrinkage Stress
(Under the direction of Terence E. Donovan)

Objective: To compare the polymerization shrinkage stress exerted on tooth structure by bulk-fill and conventional composite resins, via measuring cuspal strain. **Materials and Methods:** Fifty extracted maxillary premolars were mounted into phenolic rings and divided into 5 groups of 10. Strain gages were bonded to the buccal and lingual cusps. Proximal matrices were made with polyvinyl siloxane. Specimens received standardized MOD cavity preparations and strain gages were connected to a data acquisition unit. A 2-step self-etch adhesive (OptiBond XTR) was applied and the preparations were restored with materials placed and light-cured as follows: Filtek Supreme Ultra in 2mm increments (FSUI); Filtek Supreme Ultra in bulk (FSUB); SonicFill in bulk (SF); SureFil SDR flow in bulk, covered with a 2mm occlusal layer of Filtek Supreme Ultra (SDR/FSU); Tetric EvoCeram Bulk Fill in bulk (TEBF). A maximum strain value (in micro-strain, με) was obtained from each specimen. Data were subjected to one-way ANOVA and pairwise comparisons using LSMeans. **Results:** Mean maximum strain values and standard deviations were: FSUI: 730.6±104.8 με, FSUB: 1264.2±1418.8 με, SF: 539±75.9 με, SDR/FSU: 506.3±69.3 με, TEBF: 624.1±147.4 με. A significant difference was found between group FSUI and groups SF, SDR/FSU and TEBF ($p=0.0002$, $p<0.0001$ and $p=0.0280$, respectively), as well as between groups SDR/FSU and TEBF ($p=0.0158$). The FSUB group was excluded from the statistical analysis due to the high mean and standard deviation of the group, both the result

of cuspal fractures due to shrinkage stress. **Conclusions:** The tested bulk-fill composite resins exerted less shrinkage stress on tooth structure than the incrementally placed conventional composite resin. Shrinkage stress generated by bulk-fill materials seems to be product-dependent. Bulk-filling with conventional composite resins is unpredictable and contraindicated.

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LIST OF ABREVIATIONS

µm: Micrometer

µε: Microstrain

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate

4-META: 4-methacryloxyethyl trimellitate anhydrate

ADA: American Dental Association

APS: Average particle size

BHT: Butylated hydroxytoluene

Bis-GMA: Bisphenol A glycidyl methacrylate

BPDM: Biphenyl dimethacrylate

BPW: Buccopalatal width

CEJ: Cemento-enamel junction

DEJ: Dentin-enamel junction

GPa: GigaPascal

HEMA: 2-hydroxyethyl methacrylate

MDW: Mesiodistal width

mm: Millimeter(s)

MPa: MegaPascal

PENTA: Penta acrylate monophosphate

PVS: Polyvinyl siloxane

SDR: Stress Decreasing Resin, Smart Dentin Replacement

TEGDMA: Triethylene glycol dimethacrylate

UDMA: Urethane dimethacrylate

vol.: Volume

wt.: Weight

CHAPTER 1: INTRODUCTION

The ability to attach restorative materials to tooth structure, generally referred to as "Adhesive Dentistry", has evolved remarkably over the last four decades. Adhesive attachment of composite resin restorative materials, or simply *composite resins*, to enamel and dentin has revolutionized modern dentistry. Composite resins were initially only considered substitutes for small anterior silicate or polymethyl methacrylate restorations. Currently they have been developed into a broad category of direct and indirect restorative materials and cements that are used in virtually every aspect of dentistry. Reasons for preferential use include ease of handling, ability to polymerize on demand, relative insolubility in oral fluids, good physical properties, a wide range of colors and translucencies for restorative materials, initial esthetics that rival those of ceramics and ability to adhesively attach to tooth structure and many restorative materials.

Composite resins, as a class of restorative materials, require strict attention to protocol and thus are technique-sensitive. Adhesive techniques depend on optimal moisture control and, therefore, effective isolation of the area to be restored is critical. Furthermore, composite resin placement is time-consuming because adhesion and material placement usually require multiple steps. Composite resins have a strong and reliable bond to enamel that resists microleakage. However, the bond of these materials to dentin has been shown to deteriorate over time secondary to resin hydrolysis and proteolytic degradation of the collagen matrix. Structural reinforcement of tooth structure, initially gained when adhesively

bonding an intracoronal restoration in place, has not been proven to last. The initial esthetic beauty of composite resins is diminished overtime due to increases in surface roughness and staining through water sorption processes. Of greater concern is the observation that composite resin restorations do not survive as well as amalgam restorations in clinical trials. Clinical complications include post-operative sensitivity, microleakage, marginal breakdown and staining, fractures of both the tooth and restorative materials and secondary caries. Most of these complications are associated with the volumetric shrinkage that takes place as the resin matrix of the material polymerizes. During polymerization shrinkage, stress is imposed on the adhesive interface, and, by extension, on the adjacent tooth structure. Despite numerous improvements in material composition over the last few decades, adverse effects of shrinkage stress remain inevitable. Clinical methods proposed to counteract it depend on careful attention to application technique details, and may even be of limited value.

Recently a new class of composite resin materials has been developed that are referred to as "bulk-fill" composite resins. Manufacturers claim these materials have properties that allow them to be placed and polymerized in larger volumes of material, a process generally referred to as "bulk-filling." This has the potential to significantly reduce composite resin placement time, technique sensitivity and, perhaps most importantly, polymerization shrinkage stress. Little is known about the levels of polymerization stress that occur when these materials create when they are placed in bulk. Therefore, the aim of this study was to compare the polymerization shrinkage stress exerted on tooth structure by three types of bulk fill composite resins, with that of a nanofilled composite resin inserted in increments or bulk, by measuring cuspal strain.

LITERATURE REVIEW

1.1. Enamel

Enamel covers the anatomic crown of the tooth. Its thickness varies based on location on the tooth and on the tooth type. It is thickest at the incisal edges of incisors (2 mm) and cusp tips of molars (2.5-3 mm). From the tips of cusps the enamel thins out towards junctions of lobes occlusally, and towards the cementoenamel junction (CEJ) cervically. Enamel is a highly mineralized crystalline structure, having hydroxyapatite (90-92% by volume) as the main constituent in the form of a crystalline lattice. Other constituents are water (4-12% by volume), organic matrix proteins (1-2% by volume) and other minerals in trace amounts.[1]

Structurally, enamel is composed of millions of rods or prisms, which typically run perpendicular to the dentinoenamel junction (DEJ) to the external surface of the tooth in a wave-like manner, with the exception of the cervical area of permanent teeth where they run from the DEJ externally in a slightly apical direction. The rods are described as keyhole-shaped in cross section, with a head 4-5 μm in diameter. Each rod runs the full thickness of the enamel, with the exception of a narrow, highly mineralized zone without rod structure both at the surface and at the DEJ. Each row of rods is offset, so the core of each rod is surrounded by rod sheaths and a cementing inter-rod substance.[2]

Enamel is the hardest substance of the human body. The hardness of the enamel varies across the external tooth surface.[3] It is generally greatest at the surface, and decreases closer to the DEJ. It is rigid and brittle in nature, with a high elastic modulus of about 30-80 GPa, depending on its location and thickness,[4,5] high compressive strength of 384 MPa but low tensile strength of 10 MPa.[4]

Enamel is relatively translucent in nature and the degree of its translucency depends on the degree of mineralization. Its color is mainly a function of its thickness and that of the underlying dentin, hence the color of teeth usually increases in chroma and decreases in value towards the cervical part as the enamel thickness decreases.[6] Enamel is a non-vital and non-sensitive tissue. It has no reparative function.[2] The incredible, long-term durability enamel demonstrates in the oral environment is due to its connection to its supporting dentin. Tooth preparation strategies are designed so as to maintain enamel rod connection with supporting dentin, especially in areas that are subject to masticatory forces. Unsupported rods are prone to fracture which can compromise the tooth-restorative interface.

1.2. Dentin

Dentin is the external, mineralized component of the pulp-dentin complex and, because it is able to respond thermal, chemical and tactile external stimuli, is considered a vital tissue. Coronal dentin provides both an elastic foundation and color for the enamel, and with the root dentin, which is covered with cementum, forms a protective encasement for the pulp and the bulk of the tooth. It is pale yellow and slightly harder than bone and becomes harder with age.[1,6] _ENREF_5 Although dentin is mineralized, it is relatively flexible, with an elastic modulus of approximately 18 GPa.[4] This flexibility helps it to support the more brittle, non-resilient enamel. The tensile strength of dentin is approximately 98 MPa, almost 10-fold that of enamel, and its compressive strength is approximately 297 MPa.[4]

Dentin is less mineralized than enamel. It contains about 45-50% inorganic material by volume, about 30% of it is inorganic material and about 10% are water.[6] The organic phase is approximately 90% Type 1 collagen and 10% non-collagenous proteins.[1] The inorganic

mineral phase consists of hydroxyapatite crystals, randomly embedded in a cross-linked organic matrix of collagen fibrils. Odontoblasts, through a cytoplasmic extension referred to as the odontoblastic process (or *Tomes fiber)*, form dentin through a system whereby collagen molecules are secreted and organized into a pre-dentin matrix. The surrounding, extracellular pre-dentin matrix is further modified through the binding of odontoblastically secreted non-collagenous proteins. The collagen binding of these proteins affect a net negative charge on the collagen such that mineralization into dentin begins. Mineral ions necessary for mineralization are also secreted by the odontoblastic processes. The odontoblast receives nutrients and mineral ions for dentinogenesis from the pulp tissue and its blood supply.

Primary dentinogenesis continues until the root apex closes. Secondary dentinogenesis then continues throughout the life of the odontoblast which results in gradual thickening of the dentin over the life of the odontoblast. The ~ 200-300 micrometer (µm) long cellular process of each odontoblast ultimately is completely surrounded by mineralized matrix (dentin) through the continual transformation of a ~ 10 µm wide zone of pre-dentin into dentin. The end result of this sophisticated system is that dentin contains a myriad of dentinal tubules which extend through the entire width of the dentin from the DEJ to the pulp. The tubules are filled with a protein- laden dentinal fluid similar to blood plasma. The tubular fluid is under a slight, but constant outward pressure from the pulp. The intra-pulpal pressure is estimated to be 25-30 mm Hg.[7]

Once dentinogenesis is complete the wall of each tubule has been identified to be hypermineralized and is referred to as *peritubular dentin.* Tubular fluid is able to pass between tubules, through the less mineralized *intertubular dentin* via small lateral canals

termed *canaliculi*. The canaliculi formed when lateral processes from the odontoblastic processes communicated with each other, thus forming a connecting network. This network is particularly dense closest to the DEJ. Dentinal tubules are widest near the pulp, averaging about 2.5-3.0 µm, and they taper towards the DEJ with an average terminal diameter of 0.8 µm. The average density of tubules near the DEJ is about 20,000 per mm^2, and it increases to about 45,000 per mm^2 near the pulp.[8] The tubules thus occupy about 4% of the superficial dentin area, and 28% of the deep dentin area.[9] The intertubular dentin occupies most of the remaining area. Due to different tubule densities the dentin permeability changes regionally, being greater in areas of greater tubule density and diameter.[10] Furthermore, the fluid-filled nature of the tubules means that dentin water content tends to be greater in deeper parts of a tooth preparation.

Each odontoblastic process has a sensory nerve fiber wrapped around it. Dentinal sensitivity is believed to derive from rapid movement of dentinal fluid in the lumen of the dentinal tubules. This rapid movement of fluid physically elongates the odontoblastic processes and their associated nerve fiber resulting a depolarization and nerve conduction. Rapid fluid movement can derive from thermal, mechanical, chemical, physical, bacterial or traumatic stimuli.[9] This theory of pain transmission has been termed the Hydrodynamic Theory, and was developed by Brännström in 1972.[11] Cutting procedures or other moderate stimuli to dentin such as caries or attrition have the potential to cause the death of regionally affected odontoblasts. That triggers odontoblast-like cells of mesenchymal origin to differentiate from the pulp tissue. These cells begin to form reparative dentin (also termed *tertiary dentin*) on the pulpal side of the affected area in an effort to protect the pulp from

further insult. Reparative dentin is highly atubular and structurally different from primary and secondary dentin.

1.3. Adhesion to Tooth Structure

Dr. Michael Buonocore´s introduction of the use of acid to etch (superficially demineralize) enamel, so as to improve adhesion of acrylic materials to tooth structure, marked the beginning of adhesive dentistry.[12] In the early days, acrylic restorative materials were only bonded to the enamel, the smear layer covering the dentin was not removed and the underlying dentin was not etched. It was Fusayama[13] who introduced the concept of acid-etching of dentin, another true revolution in adhesive dentistry which eventually led to the development of the first truly successful dentin bonding agents.[14]

1.3.1. Adhesion to Enamel

The acid-etched enamel bond is the most important bond in adhesive dentistry. The shear bond strengths of composite resin to acid-etched enamel generally exceed 20 MPa, and can range up to over 50 MPa depending on the test method used.[15-18] The bond to enamel is strong enough to resists polymerization shrinkage forces and therefore limits the potential for microleakage along the restoration margins.[19,20] Clinical success with adhesive restorations generally depends on achieving a good seal to sound enamel margins.

Buonocore's initial idea was based on a common industrial method of using phosphoric acid to improve the adhesion of acrylic paints to metallic surfaces.[12] He found that a 30-second etching time on enamel with 85% phosphoric acid promoted bonding of acrylic restorative materials. Being a true visionary, he proposed several restorative applications for this new method such as Class III and V restorations. He also envisioned the use of this

method for prevention, proposing its use for pit and fissure sealants, thus paving the way for adhesive and additive dentistry.

Acid-etching of enamel selectively removes about 10 µm of the enamel surface, leaving a microporous layer ranging from 5 to 50 µm deep. The etched surface has high surface free energy (about 72 dynes/cm), which is more than twice that of unetched enamel.[21] Three patterns of enamel etching have been described.[22] In *Type 1* etching, the enamel prism cores are preferentially dissolved. In *Type 2* etching, the prism peripheries are preferentially dissolved. In *Type 3* etching, the pattern is not as specific and involves both areas that resemble each of the other patterns and regions where the etching pattern seems unrelated to prism morphology.

Successful wetting of the etched high-energy enamel surface depends on application of a resin that is hydrophilic and fluid enough to flow, via capillary action, into the surface irregularities (microporosities). Polymerization of the resin inside the microporosities creates an intimately interlocked pattern of resin tags and enamel, fundamental for enamel adhesion.[23] Two types of resin tags have been identified that form in these etching patterns.[24] *Macrotags* are formed circularly between the prism peripheries and *microtags* are formed at the cores of enamel prisms where the resin monomers polymerize (cure) into a multitude of individual crypts formed where the enamel hydroxyapatite has dissolved. As a result of their greater number and surface area, microtags are believed to contribute more to enamel bond strength. [25]

Various concentrations of phosphoric acid have been investigated to etch enamel since Buonocore initially proposed a concentration of 85%. An ideal concentration should remove

only the amount of hydroxyapatite necessary to create a strong bond to the enamel. Silverstone reported that phosphoric acid concentrations between 30% and 40% provided the most retentive enamel surfaces.[26] Solutions that are much stronger or much weaker can produce precipitates that can physically block the microporosities and interfere with resin infiltration and thus the bonding process.[27,28] As a result, most phosphoric acid gels today have a concentration between 30% and 40%, with 37% being the most common concentration. Etching times have also been reduced from the traditional 60 second application. Several studies have shown equal bonding effectiveness with etching times from 15 to 60 seconds.[29,30]

Alternative acids for etching enamel have been studied, reporting a significant decrease in bond strength when weaker etchants are used.[31] Regardless of the type and concentration of acid used, thorough rinsing is an essential step in creating an adequate bond. A 10-second rinsing time is generally recommended for cavity preparations of complex geometry.

Enamel bonding agents are commonly based on bisphenol A diglycidyl methacrylate (Bis-GMA), which was developed by Bowen in 1962,[32] or urethane dimethacrylate (UDMA). Both monomers are viscous and hydrophobic as a result of their molecular size and composition. Improved handling and functional characteristics are obtained by dilution with monomers of higher hydrophilicity and lower viscosity such as triethylene glycol dimethacrylate (TEG-DMA) and 2-hydroxyethyl methacrylate (HEMA). The bond between the enamel and the restorative materials is formed by polymerization of the resin in the enamel macro- and micropores, and copolymerization of the available carbon-carbon double bonds with the monomers in the composite resin matrix.

1.3.2. Adhesion to Dentin

While successful bonding to enamel was achieved with relative ease, the development of predictable bonding to dentin has been a more challenging task. Much of that difficulty is related to the heterogeneous structure of dentin, which is intrinsically moist (hydrophilic) as a result of the dense fluid-filled tubular network extending between the DEJ and the pulp. In addition dentin is only about half as mineralized as enamel, with the hydroxyapatite crystals are irregularly arranged around the collagen matrix. The collagen matrix has also been termed a scaffold. Moreover, the volume of dentin that is occupied by tubules increases from 4% in the superficial dentin to 28% in deep dentin.[9] The clinical implication of this anatomic difference is that the deeper the defect progresses toward the pulp, the less intertubular dentin there is available for bonding.

The process of developing an adhesive attachment to dentin involves dissolution of the superficial inorganic crystalline phase so as to expose the collagen network and then replacing the crystalline phase with resin that is subsequently polymerized. The bond thus relies on resin infiltration of the collagen network if retention, which is considered micromechanical in nature, of the restoration is to be successful. In some cases, chemical adhesion of functional monomers to hydroxyapatite is of additional benefit.[33]

Similar to enamel, the cutting of dentin produces a surface layer of debris termed the *smear layer*, which is a 0.5-5.0µm thick layer of residual organic and inorganic components, mainly ground hydroxyapatite and denatured collagen matrix. The composition and thickness of the smear layer varies according to the cutting technique.[34,35] The smear layer is essentially a diffusion barrier as it occludes the orifices of the dentinal tubules. Tubular

occlusion reduces dentin permeability by nearly 90%.[36] The reduction of permeability limits rapid fluid movement in the tubules and, therefore, limits the potential for post-operative sensitivity. However, the smear layer must be altered or removed when bonding restorative materials to the dentin, since it is only loosely bound to the underlying substrate. Most classifications of adhesives relate to how they affect the smear layer. The following is a brief chronological classification and outline of current available classes.

The first adhesive was developed by Buonocore *et al.*,[37] who reported that a resin containing glycerophosphoric acid dimethacrylate could bond to hydrochloric acid-etched dentin. The bond strengths of this early adhesion technique were severely diminished by water immersion. Attempts at overcoming this problem were made by Bowen,[38] who synthesized N-phenylglycine glycidyl methacrylate (NPG-GMA), a "surface-active comonomer", that theoretically could bond to enamel and dentin by chelation with calcium and had improved water resistance. This chemical was the basis of the first commercially available dentin bonding agent, Cervident (SS White, Lakewood, NJ, USA). The clinical results of this adhesive were uniformly poor.

A second generation of adhesives was developed in the early 1980s. These adhesives were based on phosphorous esters of methacrylate derivatives. Their adhesive mechanism involved enhanced surface wetting as well as ionic interaction between negatively charged phosphate groups and positively charged calcium.[39] These adhesives had modest shear bond strengths, usually reported to be between 1 and 10MPa,[39-41] which is too weak to counteract polymerization shrinkage stress of composite resins.[19] Poor clinical results were reported for these dentin adhesives.[42-45] This was in part was due to poor hydrolytic stability in oral

fluids,[46] and in part because the primary bond was to the smear layer rather than to the underlying dentin.[47]

Third generation adhesives were developed in the late 1980s. These adhesives were developed after Fusayama's revolutionary introduction of acid-etching of dentin[13] had gained popular acceptance in America and Europe. The proposed adhesive mechanism was that the etched dentin would provide micro-mechanical retention for the restorative composite resin by allowing penetration of the bonding resin into the opened dentinal tubules. However, the early resins were too hydrophobic for this mechanism to work properly. The pressure of dentinal fluid and its abundant presence in the area of the bonding site hindered resin infiltration and the resultant micromechanical attachment.[25] Several promising adhesives emerged on the market, such as Clearfil New Bond (Tokyo, Japan), which contained 2-hydroxyethyl methacrylate (HEMA), a small bifunctional hydrophilic resin, and 10-methacryloyloxy decyl dihydrogenphosphate (10-MDP), a molecule with long hydrophobic and short hydrophilic active components, and systems containing 4-methacryloyloxyethyl trimellitate anhydride (4-META), another bifunctional (i.e. a hydrophobic region and a hydrophilic region) molecule. These adhesives later gave rise to very successful contemporary adhesives. Nakabayashi *et al*.[48] used a 4-META system to demonstrate the micromechanical bonding mechanism that is still used by current adhesive systems: *hybridization*, or the forming of a *hybrid layer*. Hybridization involves initial demineralization of dentin via acidic conditions, exposing the collagen network and the interfibrillar microporosities, subsequently infiltrating them with low-viscosity monomers. Thus, the *hybrid* that is created is a mixture of collagen and resin. Collectively, the thirdgeneration systems were not very successful clinically and did not completely eliminate

microleakage, although Scotchbond 2 was the first dentin bonding system to receive "provisional" and "full" acceptance from the American Dental Association (ADA).[49,50] The third generation adhesives either modified or removed the smear layer entirely, thus paving the way for the two broad classes of contemporary adhesives: etch-and-rinse and self-etch adhesives.

Etch-and-rinse adhesives are divided in to three-step etch-and-rinse adhesives (fourth-generation adhesives) and two-step etch-and-rinse (fifth-generation adhesives). These adhesives were based on the notion that the smear layer is an obstacle that must be removed to permit resin infiltration of the demineralized dentin surface. The etch-and-rinse method, as the name implies, involved a brief (15-30 second) exposure of enamel and dentin to the etching (demineralizing) effects of phosphoric acid followed by a thorough water rinse. Acid application to dentin partially or completely removes the smear layer and demineralizes dentin to about a depth of 7.5µm, depending on the concentration and application time of the phosphoric acid. The acid demineralizes the intertubular and peritubular areas thereby exposing the dense collagen matrix and increasing the microporosity of the affected area. Demineralization of the peritubular dentin widens the entrance to the dentinal tubules.[51]

All etch-and-rinse adhesive systems contain three essential components: (1) a gel of phosphoric acid that is rinsed off, (2) a *primer*, containing reactive hydrophilic monomers in a solvent of ethanol, acetone or water (or a mixture thereof) and (3) an *adhesive* which is a filled or unfilled resin bonding agent. The primers usually contain bifunctional hydrophilic monomers such as HEMA, biphenyl dimethacrylate (BPDM), dipentaerythritol penta acrylate monophosphate (PENTA) and 4-META, to facilitate penetration of the demineralized and intrinsically hydrophilic intertubular dentin. Some resin diffusion into the

dentinal tubules inevitably will occur at the same time. Removal of mineral leaves the collagen matrix unsupported and collapse of the matrix can occur with desiccation following acid removal. Introduction of the primers serves to "wet" and penetrate the demineralized, unsupported collagen meshwork, raising it to almost its original, un-collapsed level.[52] The priming process also converts the hydrophilic dentinal surface to a hydrophobic surface secondary to the presence of the hydrophobic end of the bifunctional resin molecules. This completes the priming of the demineralized dentin surface so that the hydrophobic adhesive resin molecules are able to infiltrate the surface. The adhesive usually contains Bis-GMA, frequently combined with more hydrophilic monomers such as HEMA to facilitate fusion of the primer and adhesive.[49] Filled adhesives have performed better than unfilled adhesives in clinical trials.[14,53]

Clinically and *in vitro*, the three-step etch-and-rinse adhesives have been very successful, and, by many authors, they are still believed to be the gold standard in dentin adhesion.[14,54-57]

Two-step etch-and-rinse adhesives combine the priming and bonding step into one bottle. These adhesives are believed to be less technique-sensitive than three-step adhesives. However, less consistent results have been obtained *in vitro* and *in vivo* with them.[14,56] Combining the primer and adhesive into one bottle makes the adhesive more hydrophilic and more subject to hydrolysis.[14] Hybridization can be compromised since both primer and adhesive are applied at once, potentially limiting access of the former to the dentinal surface. Furthermore, having the hydrophilic priming molecules dispersed throughout the adhesive layer makes it more susceptible to fluid ingression (of external or pulpal origin), compromising the integrity of the adhesive interface and polymerization of the resin restorative material.[58]

Self-etch adhesives can be divided into two-step self-etch adhesives (sixth-generation adhesives) and one-step self-etch adhesives (seventh-generation adhesives, also currently called all-in-one adhesives). Self-etch adhesives do not remove the smear layer, rather, dissolve it and incorporate it into the hybrid layer.

Two-step self-etch adhesives can be further subdivided into mild (pH>1.5) and strong (pH<1). Two-step self-etch adhesives have self-etching primers – i.e. they combine the etching and priming step into one. Most self-etching primers have acidic phosphate monomers that simultaneously etch and prime the dentin and they are not rinsed off. The elimination of rinsing and drying steps simplify the technique and reduces the possibility of over-wetting or over-drying the dentin substrate, both of which can have adverse effects on adhesion.[59,60] Another advantage of simultaneous etching and priming is that the primer may have increased ability to reach the full demineralization depth, which is much less likely to happen with etch-and-rinse adhesive systems due to the counter-flow of dentinal fluid after the separate etching step.

One common disadvantage of self-etching primers is that they do not etch uninstrumented enamel well enough due to their relatively low pH. Several *in vivo* studies have shown evidence of marginal staining and leakage in enamel margins when self-etching primers are used, which likely stems from inadequate etching of the enamel margins by mildly acidic primers.[61-63] To overcome this problem, a separate phosphoric acid etching step limited to enamel margins has been proposed prior to applying the self-etching primer to the whole cavity preparation, which indeed has resulted in improved clinical performance.[56] Most mild two-step self-etch adhesives have performed excellently in clinical trials, especially those that contain the bifunctional phosphate monomer 10-MDP, which promotes added strength

and stability to the dentin bond via a chemical bond to hydroxyapatite.[56,64] That coupled with low technique sensitivity has made them very popular. Strong two-step self-etch adhesives have not performed as well as mild self-etch systems.[56]

One-step self-etch adhesives combine the etching, priming and bonding steps all into one. Like two-step self-etch adhesives, they are subdivided into mild (pH>1.5) and strong (pH<1.5).[56] Initial versions of those adhesives showed poor clinical performance.[55] They were criticized for being too hydrophilic, functioning as semi-permeable membranes that freely allow passage of water molecules, leading to rapid hydrolysis of the adhesive interface and compromising resin polymerization.[58,65,66] These adhesives have improved, however, and in a recent review the mild one-step self-etch adhesives performed quite well, with an annual failure rate of 3.6%, while the strong one-step self-etch adhesives had an annual failure rate of 5.4%.[56] Still, these results are mostly from short-term (18 months to 3 years) clinical trials and it remains to be seen how well these adhesives will perform in the long-term.

1.4. Composite resins

The Glossary of Prosthodontic Terms defines "composite resin" as "a highly cross-linked polymeric material reinforced by a dispersion of amorphous silica, glass, crystalline, or organic resin filler particles and/or short fibers bonded to the matrix by a coupling agent."[67] The invention of composite resins is credited to Rafael Bowen, who introduced initial work on composite resins a year after Buonocore introduced etching of enamel.[68] Six years later Bowen patented a formula for composite resin containing Bis-GMA, the major backbone monomer of modern resin matrices.[32] Apart from developing Bis-GMA, Bowen's major contribution was also a method of bonding filler particles to the highly polymerized resin

matrix via organic silanes, or *coupling agents*, which greatly improved the physical properties of the material.[69]

Composite resins were a welcome addition to the market since previous direct anterior restorative materials had several shortcomings. Silicate cements were soluble in oral fluids which resulted in restoration surface roughness and porosity. PMMA restorations were stronger, less soluble and very polishable. However, PMMA based restorations had increased water sorption, poor color stability, excessive thermal expansion and contraction, poor wear resistance and polymerization shrinkage., Open margins, microleakage and secondary caries limited their successful use as restorative materials.[70]

Composite resins have overcome many of these problems, but some of them, at least in part, still persist, as will be discussed in detail later in this text. Current composite resin materials contain an activator-initiator system required to initiate and complete polymerization of the matrix components, pigments to alter color and opacity, ultraviolet absorbers and other additives to improve color stability. They also contain polymerization inhibitors which are needed to prevent spontaneous polymerization. The presence of these inhibitors helps to increase shelf-life and working time. The three main constituents of composite resins are: 1) a highly cross-linked polymeric resin matrix, 2) glass, mineral or resin filler particles and 3) coupling agents, bonding the filler to the resin matrix.

1.4.1. Resin Matrix

The matrix in composite resins is an organic polymer based on a blend of aromatic and/or aliphatic dimethacrylate monomers, which form a highly cross-linked, strong, rigid and durable polymer structure. The predominant monomer used in commercial dental composites

is Bis-GMA, with UDMA being another common monomer. These monomers are large, of high molecular weight (512g/mol and 470g/mol, respectively) and highly viscous, necessitating dilution with other monomers of lower molecular weight to obtain a workable consistency. Triethylene glycol dimethacrylate, or TEGDMA (molecular weight of 286g/mol), is a very common diluent monomer.[71] However, the addition of lower molecular weight monomers considerably increases the polymerization shrinkage that occurs upon polymerization. Bis-GMA and UDMA are of such high molecular weight that volumetric shrinkage can be as low as 0.9% (average of 1.5%) when they are combined with inorganic filler particles at levels of up to 88% by volume.[70] Unfortunately, smaller diluent monomers undergo greater polymerization shrinkage, increasing the overall volumetric shrinkage of the material.

1.4.2. Filler Particles

Fillers can be organic or inorganic. Organic filler particles are pre-cured, finely ground particles of resin matrix. Inorganic filler particles are usually transparent glass minerals. These include quartz, silicates, ytterbium fluoride, barium, strontium, zirconium and zinc glasses. They can be spherical or irregular and range in sizes from 0.005 to 100µm. The most common way of classifying contemporary composite resins is by filler particle size, or specifically, average particle size (APS) and size distribution. A classification of contemporary composite resins is in Table 1.

Filler particles are added to the resin matrix mainly to improve physical properties and provide a range of benefits.[70] These include: 1) increased compressive strength, tensile strength, modulus of elasticity, toughness and resistance to abrasion and fracture, 2)

reduction of volumetric shrinkage, by reducing the amount of resin that polymerizes , 3) reduction of thermal expansion and contraction, since glass and ceramics expand and contract less than polymers do, 4) improved handling properties, filler addition makes pastes more workable and reduces slumping, 5) decreased water sorption, by lowering the matrix ratio and 6) impartation of radiopacity, since heavy metal fillers are radiopaque. Generally, the more filler, the better the physical properties and, the smaller the filler particles, the better the wear resistance, polishability and esthetics. However, there is an upper volumetric limit to how much filler can be added. Filler load above 80% results in composite pastes that are unworkable.[70] Therefore, a tradeoff always exists among the requirements for workability, durability and esthetics. Regardless of the filler volume, the filler cannot contribute to improved clinical performance unless it is well bonded to the resin matrix, hence the importance of resin-filler coupling agents.

1.4.3. Coupling Agent

Organosilanes, such as γ-methacryloxypropyl trimethoxysilane, are the most commonly used coupling agents. These are difunctional surface-active compounds that form covalent bonds with the resin matrix and siloxane bonds with filler particles, allowing stress transfer from the resin matrix to the more rigid filler particles.

1.4.4. Activation/Initiation System

Composite resins polymerize via an addition polymerization reaction initiated by free radicals**.** Free radicals can be generated by chemical activation or by activation from external light energy of an appropriate wavelength. Current composite resin systems are designed to be photo-activated by visible blue light in the clinical setting.

Chemically activated (also known as chemically *cured*, or auto-cured) composite resins consist of two pastes. One contains a tertiary amine activator and the other a benzoyl peroxide initiator. Upon mixing, the tertiary amine activator reacts with the benzoyl peroxide to form free radicals that initiate addition polymerization. A disadvantage of chemically cured composite is the frequent incorporation of air in the mix. Air voids in the mix compromise the integrity and strength of the restoration. Also, exposure to air forms an oxygen-inhibited layer (unpolymerized layer) on the surface of the composite since the reactivity of oxygen to a radical is much higher than that of a monomer. Normally, an oxygen-inhibited layer is to be expected on the surface of all composite resin restorations that were cured in ambient air, but the incorporation of oxygen into the mix lowers the overall *degree of conversion* (percentage of carbon-carbon double bonds converted to single bonds during curing) due to air voids being trapped in the mix. Another disadvantage of chemically cured composite resins is that the operator has no control over the working time, since once mixed, the reaction will proceed at its own pace.[70]

Light-cured composite resins overcome many of the disadvantages of chemically-cured composite resins. They do not require mixing, have a controllable working time and have better color-stability than chemically cured composite resins. Since they are light-sensitive they have to be delivered in lightproof syringes. The first light-cured composites were formulated so that ultraviolet light was able to initiate free radicals. These now have been substituted by systems that are sensitive to visible blue light, usually of a wavelength about 468nm. These systems have a photoinitiator, usually camphoroquinone (CQ) and an amine which acts as an activator. Light irradiation produces an excited state of the photoinitiator, which then reacts with the amine activator to form free radicals that initiate

addition polymerization. Only small amounts of photoinitiator (about 0.2% by weight or less) are required.[70] Typically, it is possible to polymerize a 2-mm thick layer of composite resin with 20 seconds of light irradiation from modern curing lights. Most modern composite resins do not allow light penetration deeper than 2mm, forcing the operator to place deeper restorations in multiple increments to ensure adequate polymerization and associated physical properties. The polymerization reaction in light-cured composite resins occurs much more rapidly than in chemically-cured composite resins. The paste solidifies in a matter of seconds as opposed to several minutes.

Light-cured composite resins also need to contain inhibitors. These are added to prevent spontaneous or accidental polymerization during storage and limit polymerization when the composite resins are exposed to room lighting. In this way shelf-life and working time are prolonged. A typical inhibitor is butylated hydroxytoluene (BHT), which is commonly added in small concentrations of 0.01% by weight. It has strong reactivity potential with free radicals and reacts with them faster than the radicals can react with monomers. Chain propagation can thus not begin until all the inhibitor has been used up. This phenomenon can be quite useful in controlling the rate of the polymerization reaction of light-cured composites,[72] which is desirable since the polymerization kinetics are closely related to the increasing stress that occurs as a bonded composite resin restoration shrinks during polymerization. Indeed, polymerization shrinkage stress is one of the most complicated and significant problems with composite resins, and will be discussed here in greater detail.

1.5. Polymerization Shrinkage Stress

Composite resins polymerizing in unrestrained conditions develop minimal internal stresses. This is because the resin material is able to flow and deform during the inevitable volumetric reduction that occurs during polymerization. Restraining the material, by bonding it to cavity walls, results in polymerization shrinkage stress since the flow of the material has been restricted via the bond. This stress, first described by Bowen in 1967,[73] is imposed on the bond, also known as the adhesive interface, and, by its extension, on the surrounding tooth structure, and can lead to a host of clinical complications. These complications include cusp deflection,[74-77] fracture of enamel margins,[78-81] ENREF_79 debonding,[19,81-83] micro-cracking of the shrinking composite,[84] microleakage,[85-88] post-operative sensitivity[87-89] ENREF_90 and pulpal irritation.[90]

Even though some of the shrinkage stress will be relieved over time due to hygroscopic expansion of the resin (as it absorbs water), damage done to the adhesive interface or tooth structure due to original shrinkage stress will not be repaired. In addition, the process of hygroscopic expansion is slow and un-uniform throughout the restoration since not all surfaces are equally exposed to the oral fluids.[91,92] If the adhesive interface and tooth structure hold up against the forces of polymerization shrinkage, the remaining stress is maintained by elastic deformation of all materials involved. As a result, a restored tooth remains under stress even when not loaded, which theoretically can increase the risk of failure when under function.[93,94] Despite that, no clinical evidence currently exists to directly link shrinkage stress with clinical failure.[95,96] ENREF_95 However, the primary reason for failure of composite resin restorations has always been secondary caries,[97-103] for which

leakage of restorative margins is a suspected culprit. Polymerization stress may result in premature failure of the adhesive interface at the restorative margins.

The forces of polymerization shrinkage stress vary based on the material, its boundary conditions and clinical methods used in placement. The most important material factors determining shrinkage stress are the volumetric shrinkage and elastic modulus. Assessing the specific role of volumetric shrinkage and elastic modulus on stress development is a difficult task because both properties are affected by the same variables

The volumetric shrinkage of contemporary composite resin materials can range from as little as 1% to as much as 6% depending on material -specific formulation, although most fall into the range of 2.4-2.8%.[104-106] Resin monomers are held together by Van der Waals forces, with an average inter-molecular distance of about 0.4nm, prior to polymerization. The formation of covalent bonds during polymerization changes the inter-molecular distance to about 0.15nm, resulting in volumetric reduction.[107,108] The pure monomers Bis-GMA and TEGDMA have volumetric shrinkages of 5.2% and 12.5%, respectively, but the actual shrinkage in modern composite resins is less since approximately 60% of their volume is occupied by non-shrinking filler particles.[109] Thus, the extent of the shrinkage is determined by the volume fraction of the non-shrinking filler particles, the specific composition of resin monomers (i.e., their relative molecular weights), and the extent of the polymerization reaction (i.e., the ultimate degree of conversion).[108] Smaller molecular weight monomers form more double bonds per given volume. Therefore, an increased relative concentration of smaller molecular weight monomers will result in an increase in both volumetric shrinkage and shrinkage stress.[107] This complicates the issue for more highly filled composite resins, which require their addition to ensure proper handling. Consequently, more highly filled

materials typically exhibit low volumetric shrinkage, but high shrinkage stress.[93] One study has shown that even though the addition of diluent monomers reduces both the viscosity and elastic modulus of the material, both of which are beneficial for shrinkage stress reduction, their addition still causes an increase in shrinkage stress due to the great increase in volumetric shrinkage.[110] This finding suggests that the volumetric shrinkage and the associated degree of conversion are the most important factors in shrinkage stress development that occurs in composite resins.

A low elastic modulus allows the material to deform slightly when physically loaded, lowering the polymerization shrinkage stress.[104,109,111] Reducing the filler load of a composite resin lowers the elastic modulus, meaning that an inverse relationship exists between the elastic modulus and volumetric shrinkage of composite resins.[104,112] Increasing the inorganic filler load will decrease the volumetric shrinkage but also increase the stiffness, reducing the materials capacity to yield to shrinkage stress.[111] A trade-off therefore has to exist between good physical properties and shrinkage stress. Accordingly, flowable composite resins typically exhibit high volumetric shrinkage, a low elastic modulus and poorer physical properties. This is in contrast to hybrid composite resins, which typically exhibit low volumetric shrinkage, a high elastic modulus and better physical properties, although great intra- and inter-class variations exist.[104,111]

The degree of conversion affects the elastic modulus and volumetric shrinkage simultaneously, increasing both as the reaction approaches completion.[113,114] Furthermore, the *rate* of the reaction (i.e. *polymerization kinetics*) can significantly influence shrinkage stress.[72,115] This fact, coupled with trade-off issues of physical properties and shrinkage stress in composite resin composition, has led to significant research on polymerization kinetics.

Composite resins are solids with complex viscoelastic behavior. As they polymerize the viscosity and elastic modulus rapidly increase.[114,116] Early in the polymerization, chain growth and primary cyclization prevail over cross-linking. The relative lack of stiffness in this initially poorly cross-linked network allows monomers and short chains to rearrange and slip into new positions, which alleviates stress in the rapidly stiffening mass. This stress-relieving process is commonly referred to as *internal flow*. The polymerization then reaches a moment, referred to as the *gel-point* (which typically occurs between 5% and 10% conversion),[117] after which the material becomes too stiff to allow any further stress-relief via internal flow of the increasingly viscous material. Beyond the gel-point cross-linking of chains prevails, and small increases in conversion lead to significant increases in stress due to a rapidly increasing elastic modulus.[118] Consequently, the post-gel shrinkage is the main contributor of shrinkage stress.[119] Thus, it is of clinical interest to reduce the polymerization rate of composite resins to prolong the pre-gel phase as much as possible to maximize stress relief via internal flow. Increasing the amount of inhibitor in light-cured composite resins is one way to accomplish this, without sacrificing physical properties.[72] Self-cured composite resins generally exhibit less polymerization shrinkage stress than light-cured since the polymerization occurs over the course of several minutes as opposed to a few seconds. Consequentially, more pre-gel-point shrinkage can take place. A thicker oxygen-inhibited layer, a lower degree of conversion and air voids also contribute to lower shrinkage stress with self-cure composite resins.[115,120]

Composite resins also exhibit *external flow*, which can alleviate shrinkage stress. Unbonded composite resin is free to deform as a result of volumetric reduction during polymerization, resulting in less shrinkage stress.[121] Composite resins tend to shrink towards

the area of the strongest bond during polymerization.[122] Constricting composite resins from multiple sides allows for very limited stress relaxation by external flow and stress build-up can lead to debonding in any areas where the forces of the stress exceed the bond strength.[19] Feilzer and colleagues highlighted the clinical relevance of this phenomenon and by developing a ratio known as "the configuration factor" or *C-factor*, back in 1987.[123] This is defined as the ratio of bonded vs. unbonded surface regions in a composite resin restoration. The higher the C-factor, the less external flow can occur during polymerization. Thus, restorations, or composite increments, with higher C-factors are subject to higher shrinkage stress and greater chances of complications related to areas of bond failure. In fact, several investigators have reported a progressive decrease in bond strength as the C-factor increases, and a potential detrimental effect on marginal integrity and gap formation.[124-128] Clinical alternation of composite resin insertion techniques have been designed in the hope of reducing C-factors (i.e., reducing polymerization stress by increasing external flow).

1.6 Clinical Methods Employed to Reduce Polymerization Shrinkage Stress

Clinical methods have been developed in an attempt to reduce polymerization stress. These methods are based on the potential for stress relaxation (from internal flow) to occur when the pre-gel phase is prolonged during polymerization. In addition, other methods are based on composite resin manipulation techniques that lower the C-factor and allow for external flow to occur during polymerization.

1.6.1. Photoactivation Methods to Prolong the Pre-gel Phase

Several authors have advocated modified photoactivation methods so as to extend the pre-gel phase. These are, collectively referred to as "soft-start" methods While they differ in

their specific application, the rationale behind them is the same – lower light intensity for a specific period of time to lower the rate of polymerization, followed by a higher light intensity to ensure adequate polymerization. In theory, this prolongs the pre-gel phase of the polymerization reaction (since the conversion rate is proportional to the square root of the light density in mW/cm^2),[129] allowing more stress relaxation to take place via internal flow of monomers prior to reaching the gel-point. Some investigators have reported reductions in shrinkage stress with these methods when compared to continuous high-intensity photoactivation, but to a different degree.[130-132] The effectiveness of these methods depends on the composition of the composite resin and the initial light intensity level, since these dictate the rate of initial reaction and may limit any chances for internal flow to reduce stress.[132,133] Two of these methods have been compared with conventional photoactivation in a seven-year prospective clinical trial of composite resin restorations in non-carious cervical lesions, where they were not found to be superior.[134]

As mentioned before, dimethacrylate resins reach the gel-point at relatively low conversion levels between 5% and 10%.[117] It may therefore be that applications of very low light intensity for prolonged periods would result the slow curing rates that have sufficient internal flow to significantly reduce shrinkage stress. This method would be clinically impractical. Another concern that has been raised regarding soft-cure photoactivation methods is that low intensity irradiation can be associated with relatively few polymer growth centers. Limited polymer growth centers result in a more linear polymer structure with fewer cross-links. This would lead to a weaker composite resin restoration that would be more the composite resin weaker susceptible to softening by chemicals in foods and beverages.[135]

1.6.2. Incremental Placement and Curing

The incremental placement technique aims to maximize the free surface in each increment of composite that is placed to allow for more stress relief by external flow.[136] While horizontal, vertical and oblique insertion techniques have all been recommended, the rationale is always the same in that the C-factor of each increment should be as low as possible. An added benefit of this placement technique is that it can improve physical properties by enabling thorough light curing of each increment since the depth of cure in modern composite resin seldom exceeds 2mm.[106] Versluis *et al.*[137], using a finite element analysis model, reported that forces during bulk-filling are more evenly distributed throughout the cavity preparation and result in less tooth deformation. However, Abbas *et al.*[138] measured tooth deformation while comparing bulk and incremental filling with hybrid composite resins in MOD cavities in extracted premolars. They confirmed the findings of Versluis *et al.*, but found that reduced tooth deformation during bulk-filling was a result of inadequate polymerization of the resin, due to poor light penetration into the bulk increment. Poor polymerization of conventional composite resins placed and cured bulk in large MOD cavity preparations has been confirmed by other authors as well,[77] so bulk-filling of large cavities with hybrid composite is generally thought to be contraindicated. Several in vitro studies using standardized methods with aluminum blocks, or extracted teeth, have confirmed the superiority of the incremental method in reducing shrinkage stress,[139-141] and optimizing bond strengths.[142] Incremental placement has also resulted in superior cavity floor adaptation compared with bulk-filling *in vivo*.[87]

1.6.3. Use of Stress-Breaking Liners

Numerous investigators have evaluated and promoted the use of flexible or deformable liners, techniques collectively known as “the elastic cavity wall concept”. It is theorized that, by nature of a lower elastic modulus, the liner materials can deform and absorb some of the shrinkage stress that builds up as the overlying composite resin polymerizes.

Glass-ionomers have successfully been used for this purpose in numerous studies.[143-145] Two techniques have classically been advocated, the so called open-sandwich technique, where the glass-ionomer material contacts the external surface, and the closed-sandwich technique, where only the capping composite resin contacts the external surface. The advantages of glass-ionomer materials are numerous. They provide stable, chemical bonding to cavity walls,[14] they release fluoride and can be recharged with fluoride,[146] they reduce shrinkage stress by allowing elastic deformation through a low elastic modulus and they also reduce shrinkage stress by reducing the volume of composite inserted.[144] However, skepticism has arisen regarding the alleged benefits of the sandwich technique. A long-term retrospective clinical study[147] found a significantly higher fracture rate for restorations made with a closed-sandwich technique vs. composite-resin only restorations, suggesting that resin-modified glass-ionomer materials might not be strong enough to reliably support composite resin materials in the long-term.

Flowable composites have also been evaluated as stress-absorbing liners but with conflicting results, presumably due to the fact that flowable composite resins have a wide range of filler loads and elastic moduli, and therefore vary greatly in their stress-relieving

potential.[111] Some studies have verified the effectiveness of flowable composite resins with a low elastic modulus to reduce shrinkage stress,[148,149] but the opposite has also been found.[140,148] The stress-reduction potential of flowable composite resins therefore remains debatable. The use of flowable liners has been evaluated in two long-term clinical trials, and their use did not result in better performance of the composite resin restorations.[95,150]

1.7. Methods to Measure Polymerization Shrinkage Stress

Several methods have been employed to measure polymerization shrinkage stress, including linear variable differential transformers (LVDTs), strain gages, photoelastic models and finite element analyses. The most common ones involve measuring the generation of force with a transducer (load cell or strain gage, or both) attached directly to the polymerizing composite or to a surface to which it is bonded.[108] Several variations of these methods have been used, but the most important difference is relative to the compliance, or yield, of the system employed. The compliance of the system can be described as the deformation of all surfaces and/or measurement devices involved that takes place as the composite resin polymerizes. This deformation will result in proportional lowering of the recorded forces, and therefore makes comparison of shrinkage/shrinkage stress values between studies impractical. Consequently, polymerization shrinkage stress values for typical composite resins have ranged from less than 1MPa to over 15MPa.[108] These measurement systems are therefore most meaningful when comparing differences between materials or conditions within a given system, rather than to predict absolute magnitudes of shrinkage stress identified by various separate research efforts.

Electrical resistance strain gages, as the name implies, measure strain. When a body is stressed (subjected to a load) it deforms. *Strain* is a measurement of the deformation that takes place. The change in length of the body, compared to its original length, is the definition of *strain*. Strain gages, as components in electrical circuits, exhibit some opposition (resistance) to the flow of the electrical current. Levels of resistance within the gages can be measured and interpreted with data acquisition conditioners. When the gages are stretched (or *strained*), along with the underlying surface to which they are bonded, the resistance to the electric current changes in direct proportion to the strain. Therefore, measurement of the change of resistance allows the strain to be known. Positive linear change of a substrate to which a strain gage is attached will result in a positive strain output and vice versa. Strain gages bonded to the outer surfaces of tooth cusps will give a positive output when the cusps flex towards the middle of the occlusal surface. Occlusal forces that displace the cusps away from the midline, causing outward flexure and compression of the outer surfaces of the tooth, would result in a negative output from the strain gages.

Electrical resistance strain gages are practical in several ways to measure shrinkage stress of composite resins. Strain gages been used successfully in experiments to monitor polymerization kinetics and measure post-gel shrinkage, where the gages were bonded directly to disc specimens of polymerizing composite resin.[151-153] By bonding them to external surfaces of extracted teeth they can also be used to measure shrinkage stress as a result of cuspal strain, when cavity preparations are restored *in vitro*.[77,143,154] Several studies have confirmed that there is a direct correlation between polymerization shrinkage stress and cuspal deflection when composite resins are used to restore MOD cavity preparations *in vitro*.[74-76]

1.8. Bulk-Fill Composite Resins

The main disadvantage of the incremental placement technique is that it is time-consuming and technique sensitive. The restorative materials market is driven by a consumer demand for faster and easier procedures. Table 2 lists contemporary bulk-fill composite resins, their recommended placement methods and depth of cure. Bulk-fill composite resins have been introduced to the market in an effort to meet this demand. As the name implies, they are indicated to be placed and cured in large (bulk) increments, 4mm thick or more, which can greatly reduce technique sensitivity and chair time required for composite resin restorations. In order to allow bulk placement without compromising marginal integrity and physical properties these materials have allegedly been radically reformulated to allow a greater depth of cure and reduced shrinkage stress. Bulk-fill materials challenge all current theories regarding shrinkage stress and recommended methods for its reduction. These materials must demonstrate adequate depth of cure to the proposed depth, physical properties consistent with the demands of the oral environment and actual reduction of polymerization shrinkage stress. Most laboratory studies that purely compare material properties agree that the ability of bulk-fill materials to reduce shrinkage stress is material-dependent.

Steps taken by manufacturers to increase the depth of cure of bulk-fill materials include addition of photoactive monomers to the resin matrix,[155] new and more potent photoinitiator systems and increased translucency of the materials.[156] Due to a higher translucency, most of these materials are generally not indicated for anterior restorations. Conflicting findings are in the literature on whether some of these materials have the ability to polymerize to the claimed depth, which in part relate to the testing methods used. A recent study found that the standard method to investigate depth of cure, the ISO 4049 method, is unsuitable for bulk-fill

composite resins, resulting in overestimated curing depths.[157] Other methods, such as the acetone-shake method[158] (a method of removing unpolymerized monomers with acetone) and hardness measurements of opposite ends of polymerized composite resin cylinders have been proposed as substitutes. In the latter, a ratio of 80% from bottom/top hardness values has been found to represent a clinically acceptable depth of cure.[159]

Concerns over physical properties of bulk-fill composite resins are quite understandable since a trade-off has classically existed between low shrinkage stress and good physical properties with composite resin formulations.[104,109,111] The two groups of bulk-fill materials have different clinical indications, and as such there are considerable inter-and intra-class differences with physical properties just as with conventional composite resins. These materials differ greatly in composition and use, but can be broadly classified into two categories: low-viscosity bulk-fill materials and high-viscosity bulk-fill materials.

1.8.1 Low Viscosity Bulk-Fill Composite Resins

Low viscosity bulk-fill composite resins generally have a lower filler load and consistencies comparable to flowable composite resins. They are intended to be used as bases, or "dentin replacement materials", and rely on capping (occlusal coverage) with a conventional composite material for improved strength, wear resistance and esthetics. *In vitro* studies have consistently shown that l*ow*-viscosity bulk-fill materials polymerize to the proposed curing depths, regardless of the testing method used.[156,158,160-165] Low-viscosity materials are indicated for dentin replacement only. Leprince *et al.* and Tiba *et al.*[160,166] investigated the physio-mechanical characteristics of several bulk-fill materials. They found the elastic modulus of low-viscosity materials to be similar or lower than that reported on

average for conventional flowable composite resins. Furthermore, Leprince *et al.* found that the low-viscosity materials were very susceptible to ethanol softening. They questioned the rationale from switching from conventional hybrid composite resins which, at best, reach the lower values of elastic modulus reported for dentin, to materials of an even lower modulus to replace the majority of the lost tissue, and stressed the absolute need for capping. All low-viscosity materials fulfil the ISO 4049/2009 requirement for flexural strength, which requires a value of at least 80MPa.[158,160]

In general, low-viscosity bulk-fill materials exhibit higher volumetric shrinkage than conventional composite resins, and do not always exhibit lower polymerization shrinkage stress, mirroring their flowable counterparts.[160,162,164] While little is known about the specific formulations used to lower the polymerization shrinkage stress, the formulations generally aim at controlling the polymerization kinetics, resulting in delayed reaching of the gel-point. SureFil SDR (Stress Decreasing Resin, Smart Dentin Replacement) flow contains modified UDMA, which has a polymerization modulator chemically embedded into the resin backbone. This makes for a high molecular weight photoactive group that has the ability to optimize flexibility and network structure of the SDR resin. The activated group shows a relatively slow radical polymerization rate, resulting in delayed gelation of the material and more stress-relief via internal flow.[155]

Experiments with SureFil SDR flow have shown reduced gap formation and higher bond strengths at the bottom of high C-factor cavities compared with conventional composites, both a result of lower polymerization shrinkage stress.[82,142] Use of SureFil SDR flow has not been found to negatively influence marginal integrity, either before or after thermo-mechanical loading.[167,168] _ENREF_169 Moorthy *et al.* restored large MOD cavity preparations

wither with conventional composite resins applied incrementally, or with low-viscosity bulk-fill bases (SureFil SDR flow and X-tra base) capped with conventional composite resins. Both bulk-fill materials resulted in markedly reduced cuspal flexure.[169] SureFil SDR flow is also the only material that has been used in a published clinical study. After three years of observation, Class I and Class II restorations placed using SureFil SDR flow capped with a conventional composite resin had a 0% annual failure rate, vs. a 1.3% annual failure rate for the composite resin-only group.[170]

1.8.2 High Viscosity Bulk-Fill Composite Resins

The high-viscosity materials generally exhibit similar or lower volumetric shrinkage than their conventional composite resin counterparts. Laboratory studies that purely compare material properties have exhibited reduced shrinkage stress for these materials, but to different extents.[160,162,164] As with low-viscosity bulk-fill materials, little is known about their specific stress-reducing formulations.

High-viscosity bulk-fill composite resins have a higher filler load and have physical properties and consistencies closer to those of conventional hybrid composite resins. They can be used for the entire restoration and do not require a capping layer.[166] Conflicting results have been obtained regarding the abilities of the high-viscosity materials to do the same, even when substitute methods to the ISO 4049 are used.[156,158,160-162,164,165] Thus, controversy still exists regarding the ability of high-viscosity bulk-fill materials to polymerize to the proposed depths. Most high-viscosity bulk-fill materials are intended for both dentin and enamel replacement, in which case a capping layer of conventional composite resin is not indicated. Most laboratory studies are in agreement that the physical properties of high-

viscosity bulk-fill materials are quite comparable to those of their high-viscosity counterparts, making them equally suitable for posterior restorations in that regard.[158,160,164,166] However, wear of these materials has not been evaluated. Since some of these materials have larger filler particles than most contemporary micro/nano hybrid materials,[156] wear might again become a clinical problem with composite resins with some of these bulk-fill materials, and should be investigated.[164] All high-viscosity materials also fulfil the ISO 4049/2009 requirement for flexural strength.[158,160]

Tetric EvoCeram® Bulk Fill contains pre-polymerized filler particles functionalized with silane. This inorganic filler has a relatively low elastic modulus (~10GPa), causing it to act like a microscopic spring, attenuating the forces of shrinkage stress.[171] ENREF 165 SonicFill™, a highly-filled (83.5% wt/83% vol.) rheologically modified high-viscosity bulk-fill composite resin requires a special handpiece for application. The handpiece applies sonic energy to the material. That activates rheological modifiers in the material's matrix, causing a viscosity drop of 87%, resulting in flowable-like consistency upon application which gradually increases to a sculptable consistency.[172] The viscosity drop increases particle mobility in the early stages of polymerization. This increased mobility delays gelation of the material, and also enables greater stress-relief via internal flow prior to gelation (personal communication: Dr. Weijie Huang, research Scientist, Kerr Corporation). *In vitro* studies on gap-formation in low-compliance cavity preparations have yielded more favorable results for SonicFill™ than for Tetric EvoCeram® Bulk Fill,[82,165] which mirrors the results from laboratory studies on shrinkage stress.[160] Currently, no clinical trials investigating the performance of high-viscosity bulk-fill composite resins have been published.

1.9 Summary

In summary, there is great intra- and inter-class variation in the depth of cure, physical properties and shrinkage stress values of bulk-fill composite resins. Considerable research has been accomplished for the purpose of assessing the ability of bulk fill composite resin materials to resolve the clinical problems associated with conventional systems. However, their ability to lower polymerization shrinkage stress in a clinically significant manner has yet to be proven.

Table 1.1: Classification of Composite Resins According to Average Particle Size (APS)

Modified from: Anusavice KJ, Phillips RW, Shen C, Rawls HR. *Phillips' science of dental materials*. 12th ed. St. Louis, Mo.: Elsevier/Saunders; 2013.

Composite Resin Type	APS (µm)	Filler Load (%wt)	Indications
Macrofilled	10-100	70-80	n/a
Small Particle Hybrid	0.5-3	80-90	n/a
All-Purpose Hybrid	0.4-10	75-80	General purpose
Microfilled	0.04-0.4	40-70	Low stress areas, class V, esthetic areas
Nanofilled	0.004-0.02 (0.6-20 clusters)	72.5-78.5	General purpose
Nanohybrid	0.005-1	75-80	General purpose
Packable Hybrid	15-80	Fibrous	Class I and II
Flowable	0.6-3	40-81	Class II (difficult areas), repairs, stress breakers
Silorane	0.4-0.7	76	Posterior restorations

Table 1.2: Contemporary Bulk-Fill Composite Resins

Name	Manufacturer	Depth of Cure (mm)	Filler load (%wt)	Type
Alert Condensable Composite	Pentron	5	84	High Viscosity
Quixx Posterior Restorative	Dentsply Caulk	4	86	High Viscosity
SonicFill	Kerr Corporation	5	83.5	High Viscosity
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent AG	4	79-81	High Viscosity
x-tra fill	VOCO	4	86	High Viscosity
everX Posterior (Xenius)	GC America	4-5	74.2	Fiber-reinforced base
Hyperfil	Parkell, Inc.	Unlimited (Dual Cure)	-	High Viscosity Dual Cure
Filtek Bulk Fill Posterior Restorative	3M ESPE	5	76.5	High Viscosity
Filtek Bulk Fill Flowable Restorative	3M ESPE	4	64.5	Low Viscosity
SureFil SDR flow	Dentsply Caulk	4	68	Low Viscosity
Venus Bulk Fill	Heraeus Kulzer	4	65	Low Viscosity
x-tra base	VOCO	4	75	Low Viscosity

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CHAPTER 2: MANUSCRIPT

Effect of Composite Type and Placement Technique on Polymerization Shrinkage Stress

2.1 Introduction

Composite resin materials shrink volumetrically during polymerization. The amount of shrinkage is dependent on the filler load and matrix composition of the material.[1] If the material polymerizes in unstrained conditions, minimal internal stress builds up because the material can flow and deform externally to compensate for the volumetric shrinkage.[2] Confining the composite resin by bonding it to cavity walls results in polymerization shrinkage stress because the flow of the material has been restricted.[3] The greater the bonded surface area vs. the unbonded surface area of the composite increment, the greater the restriction and the greater the shrinkage stress upon polymerization.[4] The ratio of bonded to unbonded surface area in a tooth preparation has been termed the *Configuration Factor*, or *C-factor*. Polymerization shrinkage stress is inflicted upon the adhesive interface and by its extension onto the surrounding tooth structure. These forces can be damaging to the tooth, adhesive interface, and restorative material itself if they exceed the cohesive or adhesive strengths of each. Shrinkage stress can thus lead to a host of clinical complications such as cusp deflection,[5-8] fracture of enamel margins,[9-12] _ENREF_79 debonding,[3,12-14] micro-cracking of the shrinking composite,[15] microleakage,[16-19] post-operative sensitivity[18-20] _ENREF_90 and

pulpal irritation.[21] Although no direct clinical evidence currently exists linking shrinkage stress with clinical failure,[22,23] ENREF_95 clinical trials reveal that the primary reasons for failure of composite resin restorations are secondary caries and material fractures.[24-30] Thus, it is desirable for clinicians to minimize polymerization shrinkage stress which might predispose restorations to such failure.

The incremental placement technique is one of several proposed clinical methods used to reduce polymerization shrinkage stress. While horizontal, vertical and oblique increments have been proposed, they all have the same goal of reducing the C-factor in each increment of composite resin, thus maximizing the potential for stress relaxation via external flow.[31] Reduced polymerization shrinkage stress via incremental placement and light-curing has been confirmed in several *in vitro* studies.[32-34] Other benefits of the technique include optimal physical properties due to adequate light polymerization of each increment and optimal bond strengths due to controlled shrinkage stress.[8,35] ENREF_35 Researchers have also shown improved cavity floor adaptation with the incremental technique vs. bulk-filling with conventional composite resins *in vivo.*[18]

In spite of this evidence, the incremental placement method has not been universally accepted. Some authors have argued that incremental placement results in more shrinkage stress being inflicted on the tooth structure, and recommended bulk-filling with conventional composite resins.[36] While some researchers have verified a reduction in shrinkage stress *in vitro* while bulk-filling with conventional composite resins, further investigation revealed that the reduction in stress was due to poor polymerization due to limited light penetration into the composite resin.[8,37] While the incremental placement technique may have numerous advantages, it is quite technique-sensitive and time-consuming. The market for restorative

dental materials is driven by a demand for faster and easier procedures. In an effort to meet this demand, manufacturers have introduced several bulk-fill composite resins to the market.

Bulk-fill composite resins are intended to be placed and cured in bulk increments 4-6 mm thick. Manufacturers claim an increased depth of cure and lower shrinkage stress with these materials, compared with conventional composite resins, while eliminating the need for a sophisticated layering technique. Mirroring their conventional counterparts, the bulk-fill materials can be classified into high-viscosity and low-viscosity groups. The low-viscosity materials are intended as bases, or dentin-replacement materials, which need to be covered with conventional composite resins for improved strength, wear resistance and esthetics while the high-viscosity materials can make up the entire restoration.

Several reformulations have been made in attempts to lower the shrinkage stress associated with bulk-fill composite resins. These include photoactive monomers, or "polymerization modulators" that prolong gelation time of the resin matrix, leading to greater stress relief via internal flow during the pre-gel phase.[38] Other reformulations include prepolymerized filler particles which lower the elastic modulus of the material.[39] Yet other reformulations include handpieces that apply sonic energy to the material, lowering the viscosity temporarily which allows for increased particle motion in the pre-gel phase of the polymerization, leading to increased stress-relief via internal flow.[40]

Laboratory studies on low-viscosity bulk-fill materials have shown that they exhibit higher volumetric shrinkage than conventional high-viscosity composite resins, and do not always exhibit lower polymerization shrinkage stress, mirroring their "flowable" composite resin counterparts.[41-44] The high-viscosity bulk-fill materials however, generally exhibit

similar or lower volumetric shrinkage than their conventional composite resin counterparts and have been found to reduce shrinkage stress, but to different extents.[1,41-43] Thus it appears that the overall ability of bulk-fill composite resins to reduce shrinkage stress is product-dependent.

Stress applied to a body (e.g., cusp) deforms it. Strain is a measurement of the deformation that takes place, being defined as the change in length over its original length of the body. Currently published *in-vitro* studies, seeking to compare the polymerization shrinkage stress of bulk-fill composite resins to conventional composite resins, have limited similarity to clinical conditions. Efforts to improve this have resulted in the use of electrical resistance strain gauges. It is possible to measure polymerization shrinkage stress by bonding electrical resistance strain gages to the buccal and lingual surfaces of extracted teeth. Polymerization shrinkage stress may be calculated from measurement of the cuspal strain that occurs when intra-coronal cavity preparations are restored *in vitro*.[8,45,46] Several studies have confirmed a direct correlation between polymerization shrinkage stress and cuspal deflection when composite resins are used to restore MOD cavity preparations *in vitro*.[5-7] Strain gages bonded to the underlying surface deform with the cusp. The change in resistance to the electrical current flowing through the gage as it deforms is directly proportional to the strain. They are therefore a convenient way of recording cuspal strain,[47] which is proportional to the shrinkage stress imposed on the tooth structure.[5-7]

The purpose of the present study was to compare the polymerization shrinkage stress exerted on tooth structure by two high-viscosity, one low-viscosity bulk-fill, and a standard nanofilled composite resin inserted in increments or bulk, by measuring cuspal strain. The

null hypothesis was that there is no significant difference in the polymerization shrinkage stress exerted on tooth structure between bulk-fill and conventional composite resins.

2.2. Materials and Methods

2.2.1. Specimen Preparation

The use of human teeth for this experiment was reviewed and approved as exempt by the Institutional Review Board of the University of North Carolina at Chapel Hill. Fifty freshly extracted maxillary first and second premolars extracted for orthodontic purposes, were collected and stored in 0.5% thymol at 5°C until ready for use. Prior to storage they were cleaned and carefully examined with 2.7x magnification and illumination for cracks, caries, wear facets or other defects. The specimens were mounted with the long axis vertical into 1" phenolic rings (Buehler, Lake Bluff, IL, USA) using clear epoxy resin (Buehler). Based on buccolingual measurements, the specimens were grouped into small (8.5-9.24 mm), medium (9.25-9.99 mm) and large (10.0-10.74 mm) groups. The mesiodistal dimension was also recorded and used as a reference for the cavity preparation later. They were then randomly assigned to 5 groups of 10 with equal size distribution. Strain gages (model EA-06-062AP-120/LE, Micro-Measurements, Raleigh, NC, USA) were trimmed with a scalpel blade and bonded to the buccal and lingual cusps using the acid-etching enamel method and cyanoacrylate adhesive (M-Bond 200, Micro-Measurements) according to the manufacturer‘s instructions. The gages were aligned with the long axis of the tooth, with the grid of the gages centered 3.5 mm below the palatal cusp tip, in height of the pulpal floor of the MOD cavity preparation that would be performed (Figure 1). The gages and lead wires were then coated with self-curing silicone rubber (M-Coat C, Micro-Measurements), which was

allowed to cure in a humid environment for at least 24 hours. Impressions were then made of the proximal surfaces with rigid polyvinyl siloxane (PVS) material (Regisil 2x, Dentsply Caulk, Milford, DE, USA), to be used as proximal matrices during the restorative phase. Care was taken to prevent dehydration of the specimens at all times.

2.2.2. Cavity Preparation

Large, standardized MOD cavity preparation (Figures 2 and 3) were made in all specimens using a parallel-sided, round-ended diamond rotary instrument (model #835KR.31.008, Brasseler USA, Savannah, GA, USA), mounted in an air turbine, using copious water spray. The cavity dimensions were as follows: the width of the occlusal isthmus was 40% of the maximum buccopalatal width (BPW). The pulpal floor was 3.5 mm below the palatal cusp tip. The axial wall was 2 mm high and the axial wall depth was 25% of the maximum mesiodistal width of the specimen. All internal line angles were rounded, and all proximal cavosurface angles were oriented so as to achieve a 90° cavosurface angle. All dimensions were confirmed with a digital caliper with 0.01 mm accuracy during cavity preparation. This allowed for a cavity preparation of fixed depth, with other dimensions being based on the dimensions of the tooth. This cavity preparation design weakens the specimens in a relatively similar manner, allowing for comparison between specimens despite inevitable variations in anatomy. It also created cantilever beams out of the buccal and lingual cusps, allowing them to deform around the pulpal floor over which the strain gages were centered.

2.2.3. Specimen Restoration

After cavity preparation the specimens were mounted on a polyvinyl siloxane base for stabilization and the strain gages were connected to a data acquisition unit (Model D4 Data Acquisition Conditioner, Micro-Measurements). In addition to the specimen being restored, another prepared but unrestored specimen was always connected to the data acquisition unit during the restorative procedures to control for temperature and humidity fluctuation (control specimen). The primary investigator performed all tooth preparations and restorations in this study. Prior to restoration, the PVS proximal matrices were placed tightly up against the proximal surfaces. If needed, they were connected to one another with additional PVS material to increase stability throughout the restorative procedure. A 2-step self-etch adhesive (OptiBond XTR, Kerr Corporation, Orange, CA, USA) was applied and light polymerized according to the manufacturer‘s instructions.

The specimens were restored in the following manner: Filtek Supreme Ultra (3M ESPE, St. Paul, MN, USA) in ≤2 mm increments (positive control, FSUI); Filtek Supreme Ultra in bulk (negative control, FSUB); SonicFill (Kerr Corporation) in bulk (SF); SureFil SDR flow (Dentsply Caulk) in bulk, covered with a 2 mm occlusal layer of Filtek Supreme Ultra (SDR/FSU); Tetric EvoCeram Bulk Fill (Ivoclar Vivadent AG, Schaan, Principality of Liechtenstein) in bulk (TEBF). In group FSUI, the proximal boxes were first restored with 2 cuboidal increments each and the occlusal part was restored with 4 oblique increments, 2 against each cusp (Figure 4). For the SF and TEBF groups, the proximal boxes were first restored with one increments each, and then the occlusal part with a bulk increment to avoid violating the manufacturer‘s recommended maximum depth of cure. SDR was applied according to the manufacturer‘s instructions, leaving 2 mm of occlusal space for a

conventional composite resin (Filtek Supreme Ultra, 3M ESPE). The proximal matrices provided a guide to the volume of composite resin that should be inserted, since each one was a recording of the specimen's pre-operative shape. They also provided the operator with a location of the marginal ridge, giving an indication of height and shape of the occlusal table (Figure 5). The occlusal increments were placed in continuation of the existing cuspal inclines and primary occlusal anatomy was created (Figure 6). All increments were photopolymerized for 20 seconds with a high energy output LED curing light (Bluephase G2, Ivoclar Vivadent) on "high" setting, with the tip as close to the specimen as possible without touching it. After the final occlusal layer had been photo polymerized, the investigators waited for 4 minutes to let the polymerization reaction progress. After the 4 minutes, the proximal matrices were removed and an additional 20-second photopolymerization was performed directly onto the buccal and lingual surfaces, after which the polymerization was allowed to progress for another 4 minutes prior to terminating the recording. The energy output of the curing light was measured prior to every restorative sequence with a radiometer (Bluephase Meter, Ivoclar Vivadent). The average output throughout the study was 1262 mW/cm^2. Table 1 lists all restorative materials used in this study.

2.2.4. Data Manipulation and Statistical Analysis

Two investigators (GRJ and SNA) recorded all of the data in this study, making the primary investigator blinded to the live feed of strain output as the specimens were being restored.

All data collected during proximal matrix removal and additional buccal and lingual light- curing was eliminated from the analysis due to physical and thermal disturbance. The

maximum output values (in microstrain, με) were identified for the buccal and lingual cusps for each specimen, after having adjusted all values according to the output from the control specimen. The data was analyzed according to maximum buccal and lingual strain separately, and also according to the maximum buccal and lingual strain combined for a maximum total strain value per specimen. The data were subjected to a one-way ANOVA and pairwise comparisons using LSMeans. Group FSUB (negative control) was excluded from the statistical analysis due to the excessively high mean and standard deviation of the group.

2.3. Results

The values for maximum buccal and lingual strain per group can be seen in Table 2. The values for combined buccal and lingual strain per group can be seen in Table 3 and graphically in Figure 7. All bulk-fill materials resulted in lowered cuspal strain, on average, compared to conventional composite resin placed incrementally. For the buccal strain only, group FSUI was statistically significantly greater than groups SF ($p=0.0133$), SDR/FSU ($p=0.0011$) and TEBF ($p=0.0425$). For lingual strain, group FSUI was statistically significantly greater than groups SF ($p=0.0003$) and SDR/FSU ($p=0.0002$), but not group TEBF ($p=0.1962$). Group TEBF was statistically significantly greater than groups SF ($p=0.0097$) and SDR/FSU ($p=0.0090$).

For maximum combined strain, group FSUI was significantly different from groups SF ($p=0.0002$), SDR/FSU ($p<0.0001$) and TEBF ($p=0.0280$). Group SDR/FSU was also significantly different from group TEBF ($p=0.0158$).

2.4. Discussion

The null hypothesis of this study was rejected since all tested bulk-fill materials resulted in less shrinkage stress than incrementally placed composite resin, as measured via cuspal strain. The magnitude of the cuspal strain was product-dependent however.

The decision to not include group FSUB in the statistical analysis warrants discussion. The high mean value of the group is a result of excessive strain that resulted in cuspal fractures occurring in a few of the specimens. A fracture was characterized by a very sudden and dramatic change in strain output, several times higher than any output received as a result of shrinkage stress. Invariably, those fractures occurred in a semilunar fashion in line with the pulpal floor, more often in the lingual cusp than in the buccal cusp. The fracture lines could be clearly seen in the enamel. Other reasons contributing to the wide distribution of strain values in group FSUB might be debonding of the adhesive interface, in the event of the forces of polymerization shrinkage stress exceeding the bond strengths, and poor polymerization of the composite resin in deeper parts. Both these phenomena might explain very low strain values in a few of the specimens. Better cavity floor adaptation has been found for incremental placement than bulk-filling with conventional composite resins *in vivo*, indicating debonding when bulk-filling is applied.[18] Low cuspal deflection values while bulk filling with conventional composite resins have been obtained in another *in vitro* experiment, in which the investigators found the reason to be poor polymerization of the composite resin.[37] Other investigators have also confirmed poor polymerization of conventional composite resins placed and cured bulk in large MOD cavity preparations.[8] Furthermore, poor dentin bond strengths in deeper areas of cavity preparations when bulk-filling with conventional composite resins have been reported.[35] Poor polymerization and debonding

coupled with fractures may have contributed to the wide range of values in group FSUB. Future research designed to evaluate this potential is warranted.

The decision to prepare the occlusal isthmus to 40% of the maximum BPW was a result of trial-and-error during a pilot study. Initially, the isthmus was prepared to 50% of the maximum BPW. The wider isthmus resulted in numerous cuspal fractures due to shrinkage stress in the restorative phase, both in the positive and negative control groups, prompting the examiners to reduce the isthmus width. Interestingly, all the specimens restored with bulk-fill composite resins in the group prepared with the isthmus width to 50% of the maximum BPW survived, further supporting the hypothesis that the bulk-fill composite resins investigated in the study may decrease shrinkage stress compared to conventional composite resins placed incrementally.

The results of this study are in agreement with the findings of Moorthy *et al*,[48] who compared cuspal flexure of maxillary premolars when large MOD cavities were restored with two low-viscosity bulk-fill composite resins (SDR, Dentsply, and x-tra base, Voco GmbH, Cuxhaven, Germany) veneered with conventional composite (GrandioSO, Voco GmbH), versus the conventional composite placed incrementally. Both low-viscosity bulk-fill composite resins veneered with the conventional composite were associated with significantly less cuspal flexure than the conventional composite placed in increments.

When the results of this study are compared to other laboratory studies in which shrinkage stress was investigated, without investigating its effect on tooth structure, the results are in partial agreement. Tiba *et al* found the shrinkage stress of SDR and Sonic Fill to be lower than that of Filtek Supreme Ultra, while they found the shrinkage stress of Tetric

EvoCeram Bulk-Fill to be slightly higher.[41] Jang *et al*[42] found the shrinkage stress of Tetric N-Ceram Bulk-Fill to be lower than that of Filtek Supreme Ultra, while they found the shrinkage stress of SDR to be higher, contradicting the findings of Tiba *et al*. These discrepancies between the studies may be explained by the fact that different testing systems of different compliances were used. Furthermore, the polymerization shrinkage stress of SDR in the present study cannot be compared directly to the other studies. In the present study it was veneered with a conventional composite resin based on manufacturer's recommendations.

A further investigation into the actual strain values from the buccal and lingual strain gages prior to polymerizing the veneering increments in the SDR/FSU group revealed very low strain values averaging at 47.75 με between both cusps. If those values are compared to the mean total values for the group, it is clearly seen that about 80% of the strain generated in that group was generated by the veneering composite material. However, it must be recognized that the cavity geometry is likely to mask a part of the strain generated by the SDR material since the bottom 2 mm of the cusps were supported by the axial wall, thus not fully allowing them to flex freely as cantilever beams in the part restored by the SDR material.

To enable a greater depth of cure, bulk-fill materials in general have greater translucency and more potent photoinitiator systems than conventional composite resins.[39,40,49] There are conflicting findings are in the literature on whether some of these materials have the ability to polymerize to the claimed depth, which in part relate to the testing methods used. A recent study found that the standard method to investigate depth of cure, the ISO 4049 method, is unsuitable for bulk-fill composite resins, as it resulted in overestimated curing depths.[50]

Other methods, such as the acetone-shake method[51] and hardness tests on opposing ends of polymerized composite resin cylinders have been proposed as substitutes. In the latter, a ratio of 80% from bottom/top hardness values has been found to represent a clinically acceptable depth of cure.[52] Regardless of testing methods, the low-viscosity bulk-fill materials have consistently been shown in *in-vitro* studies to polymerize to the claimed curing depths.[41-43,51,53-56] However, conflicting results have been obtained regarding the abilities of the high-viscosity materials to do the same, even when substitute methods to the ISO 4049 method are used.[41-43,51,53,54,56] Thus, controversy still exists regarding the ability of high-viscosity bulk-fill materials to polymerize to the proposed depths. Inadequate polymerization would indeed result in lower cuspal strain values, which might lead to overestimation of the reduced shrinkage stress these materials may be producing. In the present study, no attempt was made to confirm adequate polymerization at the bottom of the cavity preparation. According to the manufacturer, the allowable increment thicknesses of SonicFill, SureFil SDR flow and Tetric EvoCeram Bulk Fill are 4 mm, 5 mm and 4 mm, respectively. To avoid violating the recommended maximum depth of cure for these materials, the proximal boxes (2 mm deep from the pulpal floor) were first restored, followed by the occlusal portion which was 3.5 mm deep from the palatal cusp. According to this, the incremental thicknesses were well below the maximum recommended limit.

One of the alleged benefits of bulk-fill composite resins is the reduced placement time. In the present study the groups SF, SDR/FSU and TEBF resulted on average in a reduced placement time of 7, 3 and 5 minutes respectively, as compared with the incremental placement technique. The average time spent to place a restoration in the FSUI group was 25 minutes, including the 8-minute waiting time. It does not come as a surprise that the

application time of SonicFill is faster than that of Tetric EvoCeram Bulk Fill since the latter is a paste-like composite resin which has to be manually applied, condensed and shaped, while the former is in a syringe, which upon application of shear stress by a designated handpiece, drops in viscosity by 85% to a flowable-like consistency which can be injected directly into the cavity preparation.[40] Regardless, with both high-viscosity bulk-fill materials, some of the time saved with bulk-filling is offset by a greater amount of time required to shape the entire occlusal surface to correct anatomy. The time saved with SureFil SDR flow was less than of the two high-viscosity materials since it required 2 separate veneering increments, one from each cusp, which both had to be shaped and polymerized.

The present study is not without limitations. *In vitro* studies are generally not good predictors of clinical performance, and should therefore be interpreted with caution. Using extracted teeth for *in vitro* research introduces a host of different variables that are impossible to standardize. In addition, due to difficulties with obtaining enough specimens both first and second maxillary premolars were used, further adding to the heterogeneity of the specimens. Cavity dimensions inevitably also vary to some minor extent since they are prepared by hand.

Weighing increments is a useful way to standardize the volume of inserted composite resin for each increment.[32] While this method can be used in laboratory studies dealing with standardized equipment, it was not considered a practical method since all specimens, and hence all cavity preparations in this study differed. Small differences in increment size may have contributed to the higher standard deviation in the FSUI group than in groups SF and SDR/FSU. Without performing tests to confirm adequate polymerization one can only speculate on the higher standard deviation in group TEBF than that of the other bulk-fill

groups. The material did however have a notably thicker, more viscous consistency than other composite resins evaluated in this study. Since the proximal PVS matrixes, although made of a relatively rigid material, were not very rigidly attached a placement of high-consistency material into the cavity preparation may, on occasion, have displaced them. Any displacement could result in slight overfilling of some cavities although this was not noted upon inspection of the specimens created in this research study. The other materials were of considerably lower thinner consistency so matrix distortion and overfilling would not have been as likely in other groups. Increasing the flow of thicker materials via warming techniques may help to eliminate this potential but would likely introduce other variables.

2.5. Conclusions

Based on the limitations of this in vitro study, the following conclusions may be made:

- Restoration with bulk-fill composite resins resulted in less shrinkage stress than restoration with incrementally placed composite resins, as measured via cuspal strain.
- The shrinkage stress of bulk-fill composite resins appears to be material-dependent.
- The bulk-fill approach is less time-consuming than the incremental placement method, although the time saved varies with the type of bulk-fill material chosen.
- Bulk-filling with conventional composite resins is unpredictable and contraindicated, and can be detrimental to the tooth and adhesive interface, as well as the physical properties of the composite resin itself.

While the materials tested in this study may reduce shrinkage stress, further studies on other factors such as depth of cure and degree of conversion, physical properties and wear are needed before these materials can be recommended for routine use. Ultimately, long-term

clinical trials will be needed to assess the relative predictability of bulk-fill composite resin systems.

Table 2.1: Restorative Materials Used in This Study

Product	Type	Manufacturer	Lot No.	Matrix composition	Filler Type	Filler Size (µm)	Filler Load (bw %)
OptiBond XTR	2-step Self-etch Adhesive	Kerr Corporation, Orange, CA, USA	4925697 4989266	Primer: acetone, ethyl alcohol, HEMA. Adhesive: ethyl alcohol, alkyl dimethacrylate resins, filler particles	Barium aluminoborosilicate glass, fumed silica (silicon dioxide), sodium hexafluorosilicate	-	15
Filtek Supreme Ultra	Nanofilled composite resin	3M ESPE, St. Paul, MN, USA	N529636	Bis-GMA, UDMA, TEGDMA, bis-EMA(6),	Ytterbium trifluoride, Non-aggregated silica, non-aggregated zirconia, Zirconia/silica clusters	0.004-0.02 (0.6-20 clusters)	78.5
SonicFill	High viscosity bulk-fill composite resin	Kerr Corporation, Orange, CA, USA	4948133 5021080	TMSPMA, EBPADMA, bisphenol-A-bis-(2-hydroxy-3-mehacryloxypropyl) ether, TEGDMA	Glass, oxide, chemicals, silicon dioxide	0.05-4	83.5
SureFil SDR flow	Low-viscosity bulk-fill composite resin	Dentsply Caulk, Milford, DE, USA	131022	Modified UDMA, EBPADMA, TEGDMA	Ba-Al-F-B-Si glass, SR-F-Si glass	1-10 (4.2 average)	68
Tetric EvoCeram Bulk Fill	High viscosity bulk-fill composite resin	Ivoclar Vivadent AG, Schaan, Liechtenstein	S21840	Bis-GMA, Bis-EMA, UDMA	Ba-glass, YbF3, mixed oxide, pre-polymerized filler	0.16-0.7	79-81

HEMA: 2-hydroxyethyl methacrylate, Bis-GMA: bisphenol A glycidyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, Bis-EMA: ethoxylated bisphenol A dimethacrylate, TMSPMA: 3-trimethoxysilylpropylmethacrylate, EBPADMA: ethoxylated bisphenol-A-dimethacrylate

Table 2.2: Mean Buccal and Lingual Strain per Group

		Buccal Strain (με)		Lingual Strain (με)	
Group/material	**Shade**	**Mean**	**SD**	**Mean**	**SD**
1 – FSUI	**A2B**	357.2 [B]	74.8	379.4 [B]	57.9
2 – FSUB	**A2B**	359.5	119.9	911.9	1405.1
3 – SF	**A2**	275.6 [A]	41.9	268.1 [A]	49.5
4 – SDR/FSU	**U/A2B**	246.4 [A]	55.7	267.3 [A]	30.5
5 – TEBF	**IVA**	291.3 [A]	95.8	343.2 [B]	91.6

Groups marked with the same letter are not significantly different.

Buccal: Group 1 is significantly different from groups 3 (p= 0.0133), 4 (p= 0.0011) and 5: p= 0.0425).

Lingual: Group 1 is significantly different from groups 3 (p=0.0003) and 4 (p=0.0002).

Group 5 is also significantly different from groups 3 (p=0.0097) and 4 (p=0.009).

Table 2.3: Mean Combined Strain per Group

Group/material	Mean (με)	SD
1 – FSUI	730.6 [D]	104.8
2 – FSUB	1264.2	1418.8
3 – SF	539.0 [A,B,C]	75.9
4 – SDR/FSU	506.3 [A,B]	69.3
5 – TEBF	624.1 [A,C]	147.4

Groups marked with the same letter are not significantly different.

Group 1 is significantly different from groups 3 (p=0.0002), 4 (p=0.0001) and 5 (p=0.0280).

Group 4 is also significantly different from group 5 (p=0.0158).

Figure 1: Location of Strain Gage in Relation to Pulpal Floor.

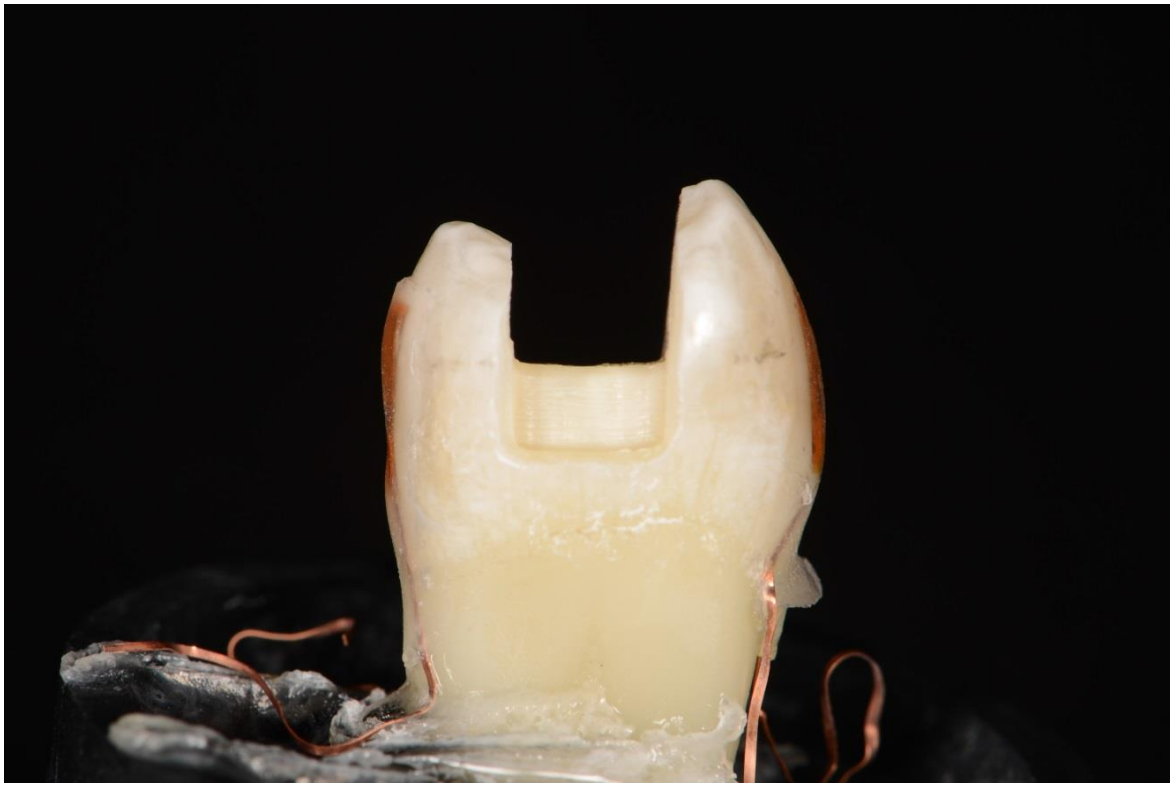

Figure 2: MOD Cavity Preparation - Proximal View.

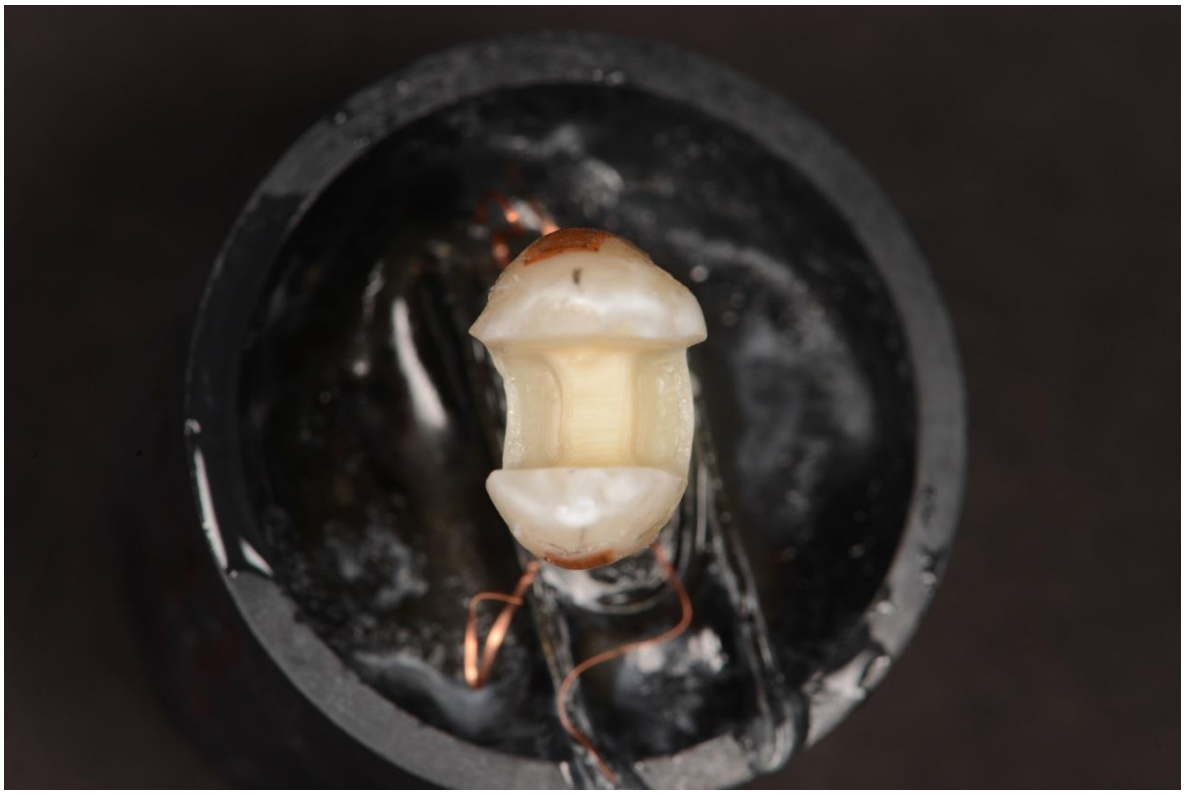

Figure 3: MOD Cavity Preparation - Occlusal View.

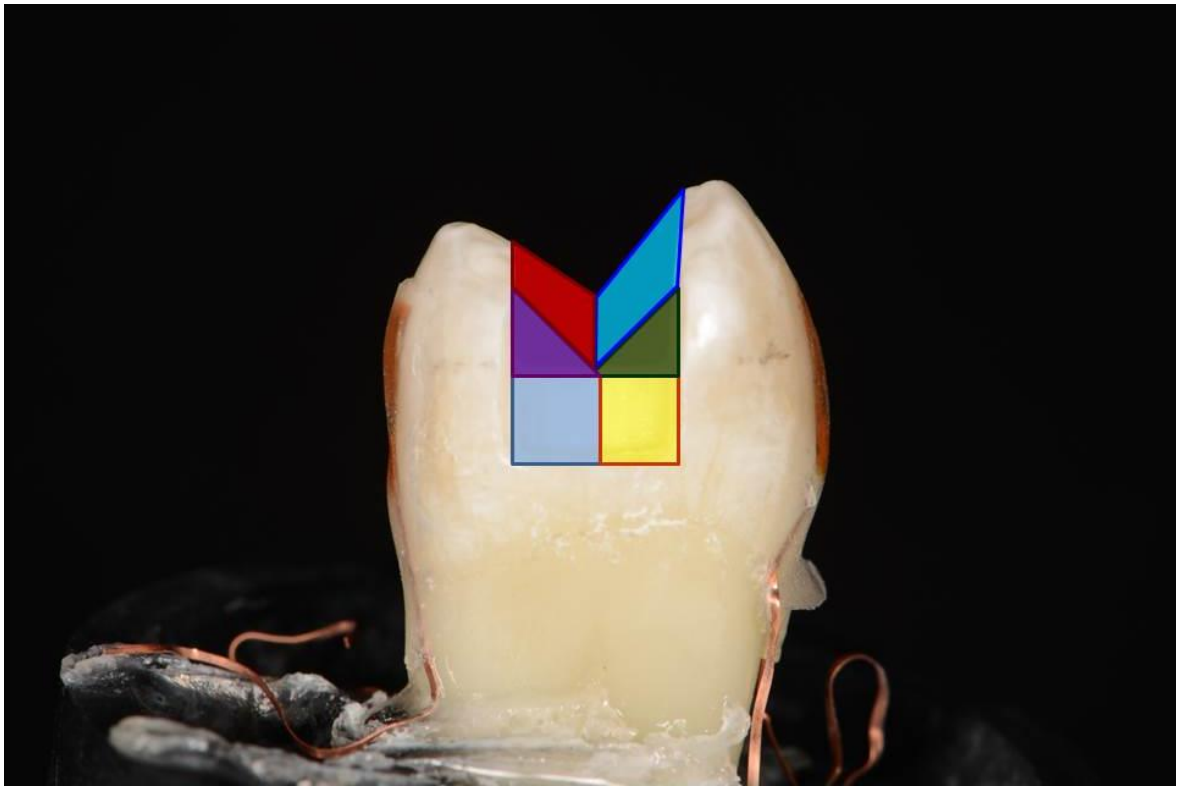

Figure 4: Incremental Placement Technique for Group FSUI.

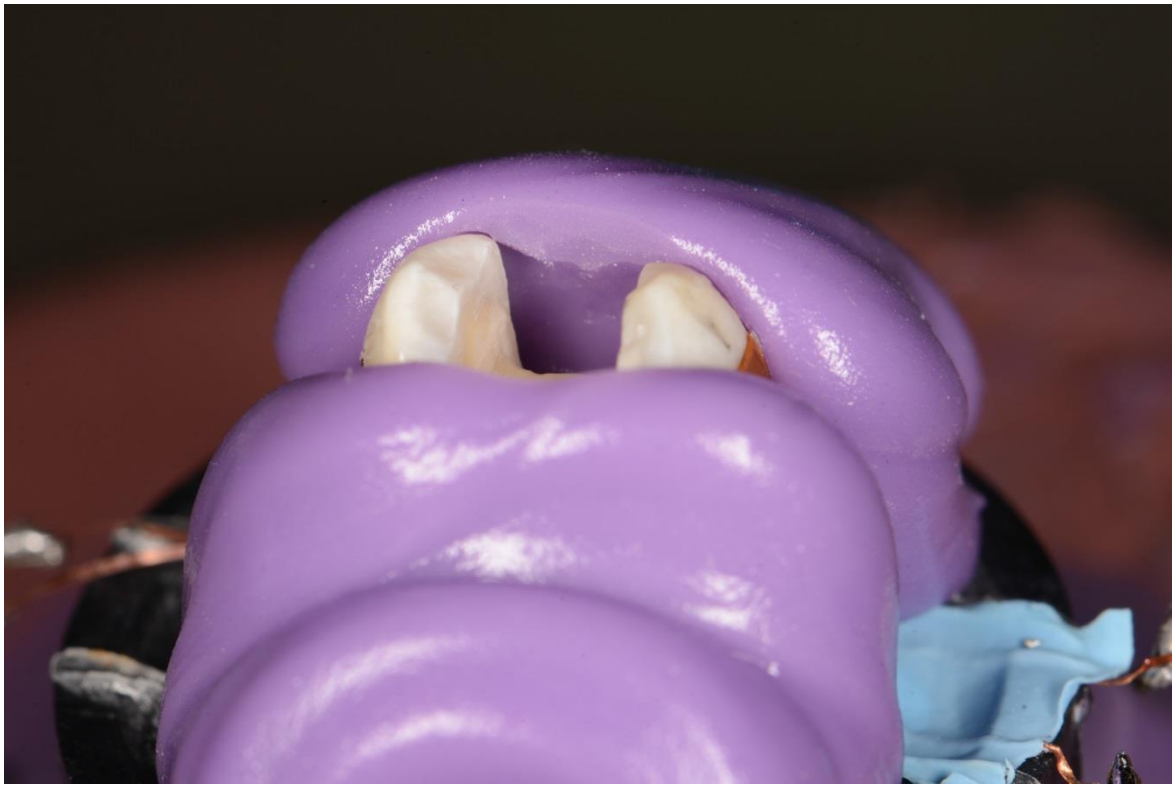

Figure 5: PVS Proximal Matrixes Guiding Composite Volume and Contour.

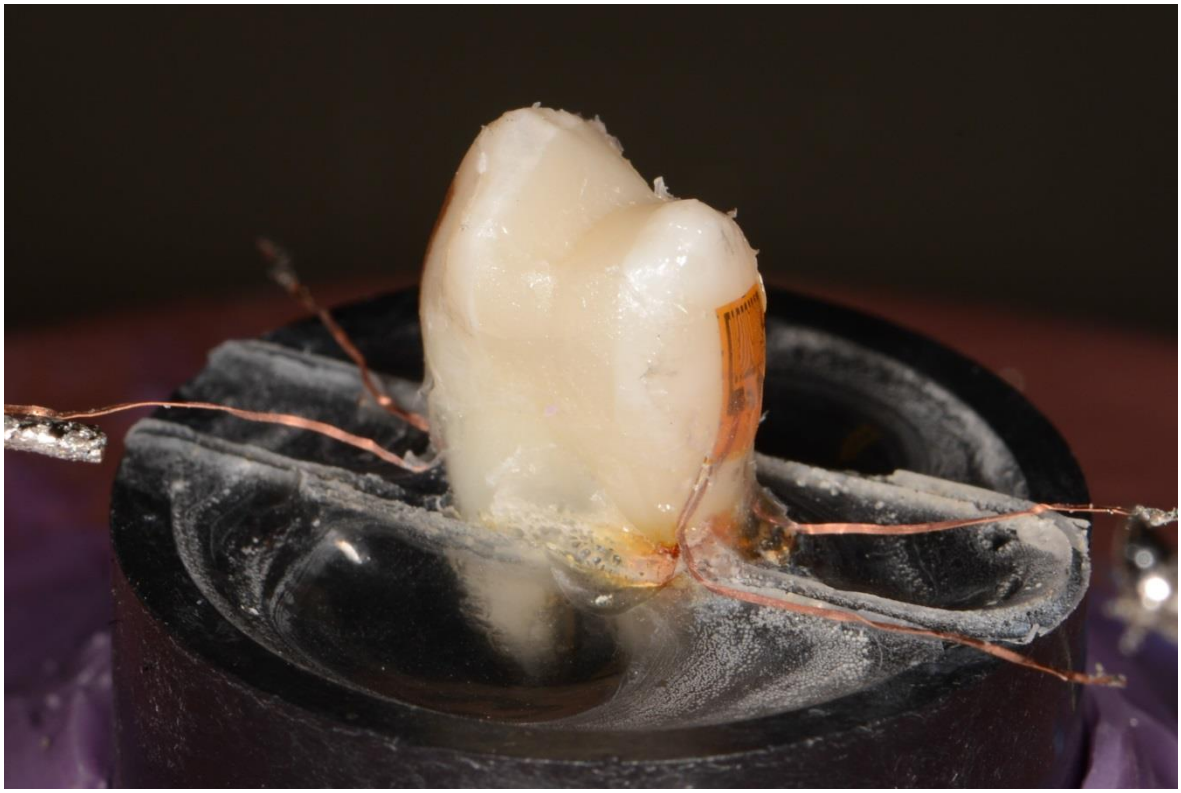

Figure 6: Restored Specimen.

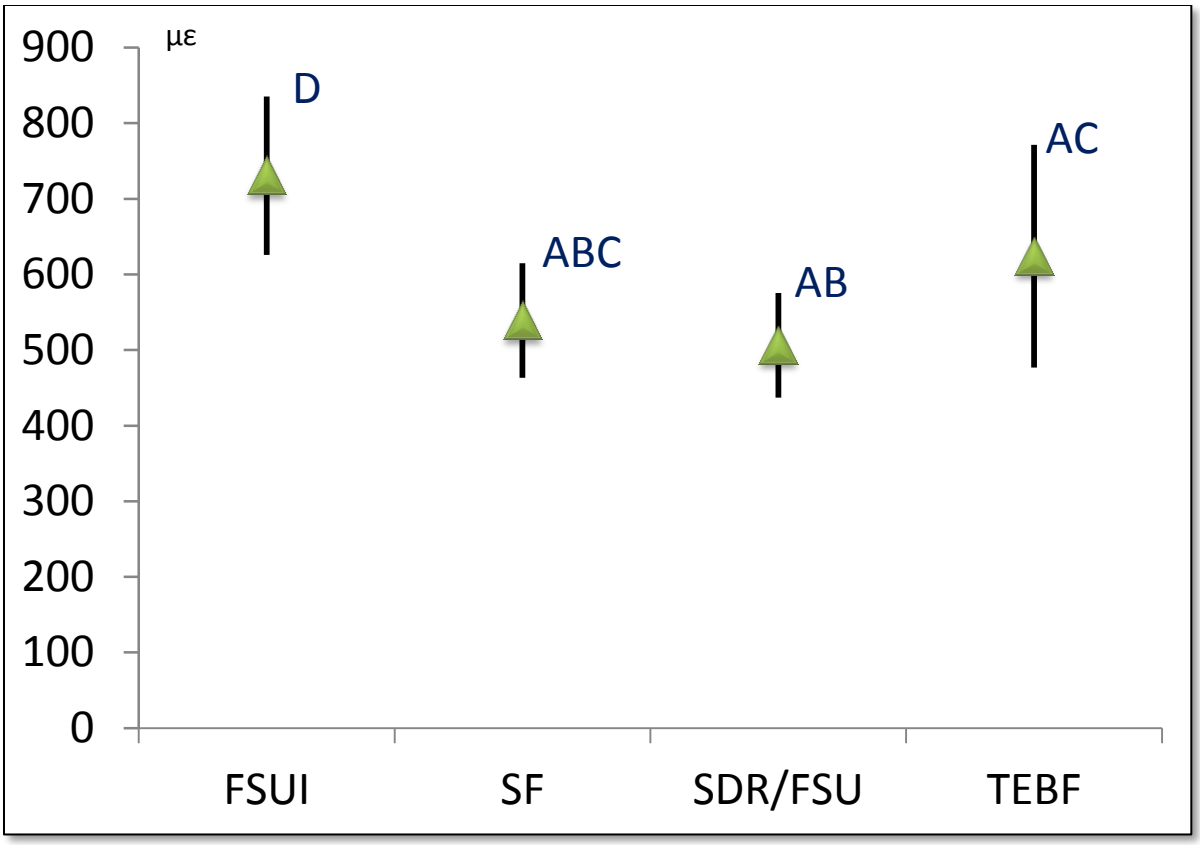

Figure 7: Mean and Standard Deviation for Maximum Combined Strain.

Groups marked with the same letter are not significantly different.

Group 1 is significantly different from groups 3 (p=0.0002), 4 (p=0.0001) and 5 (p=0.0280).

Group 4 is also significantly different from group 5 (p=0.0158).

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