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BI-LAYERED RESTORATIVE DENTAL COMPOSITE STRUCTURES: STRESS AND FRACTURE BEHAVIOR

Tarek A. Omran



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“In the name of God, the Most Gracious, the Most Merciful”

رَبِّ أَوْزِعْنِي أَنْ أَشْكُرَ نِعْمَتَكَ الَّتِي أَنْعَمْتَ عَلَيَّ وَعَلَىٰ وَالِدَيَّ
وَأَنْ أَعْمَلَ صَالِحًا تَرْضَاهُ وَأَدْخِلْنِي بِرَحْمَتِكَ فِي عِبَادِكَ الصَّالِحِينَ

“My Lord! Inspire me to ‘always’ be thankful for Your favours which You have blessed me and my parents with, and to do good deeds that please you. Admit me, by Your mercy, into ‘the company of’ Your righteous servants.”

To my beloved family, friends and mentors

UNIVERSITY OF TURKU

Faculty of Medicine

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Department of Biomaterials Science

TAREK OMRAN: Bi-layered restorative dental composite structures: Stress and fracture behavior

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ABSTRACT

Materials such as bulk-fill resin composites (BFC) and short fiber-reinforced resin composites (FRC) are gaining popularity in the clinics due to the reduced chair-time, ease of application and improved mechanical-and bulk-strengthening properties. However, these materials are often required to be capped occlusally with conventional particulate filler resin composite (PFC) which creates a bi-layered composite (BLC) restoration. Although much is known about the properties of the different composites themselves, less information is available on the outcomes of combining them in a BLC restoration.

This series of *in vitro* studies therefore, aimed at evaluating the mechanical and fracture behavior of such BLC structures using commercially available bulk-fill and fiber-reinforced materials. The purpose of the research was to optimize the use of BLCs in dentistry, using novel approach and methodologies to shed the light on optimizing the use of BLCs that are biomimetic to the naturally occurring bi-layered tooth structure (enamel and dentin).

Three different areas within BLC restorations were investigated. The first was the deepest layer that will be in direct contact with the tooth tissue, i.e. dentin-composite interface. Hence, in one study specimens were prepared to evaluate the influence of increment thickness on dentin bond strength and light transmission of BFCs, FRCs, and PFC (control). The second area investigated was the bulk and middle areas of the BLC restoration, i.e. the composite-composite interface. Thus, a pair of studies evaluated the effect of: (i) material type, individual material thickness and composite-composite interfacial adhesion. (ii) different 3D fabricated interface-designs, on the load-bearing capacity and fracture behavior of BLC structures. The third area investigated was the top layer of the BLC restoration that is vulnerable to direct mechanical deterioration. Therefore, a study investigated crack propagation and fracture behavior of BLC structures using a novel methodology to further predict their mechanical toughness.

The results showed that in the first area FRC can be applied safely in bulks of 4 mm increments same as other BFC. In the second area: (i) Loading tests demonstrated that thickness and adhesion had an effect on load-bearing capacity on the bulk-base materials investigated ($p < 0.05$). FRC groups demonstrated higher load-bearing capacity compared to BFC groups even with thin capping PFC layer. While, finite element modeling (FEM) showed less strain distribution for FRC groups compared to other BFC groups. (ii) Material and interface-designs had a significant effect on the load bearing capacity ($p < 0.05$). Fracture analysis of 3D fabricated designs showed that FRC groups demonstrated up to 100% partial bulk-fractures with pyramid interface-design, and no incidence of catastrophic bulk-fractures. Whereas BFC had no incidence of partial bulk-fracture and demonstrated up to 84.6% complete bulk-fractures with pyramid interface-design. Collectively, the 3D fabricated interface-designs studied enhanced the fracture behavior of BLCs.

In general, BLCs that utilized FRC demonstrated higher load-bearing capacities, as well as less catastrophic fracture behavior when compared with BLC groups. This highlights the possibility of conservative repairs in case of mechanical failure.

KEYWORDS: fracture behavior; fiber-reinforced composite; bulk-fill composites; biomimetic restoration; bi-layered composite restoration; interface surface design; 3D printing; dentin-enamel junction; composite-composite adhesion; oxygen inhibition layer, FEM

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TIIVISTELMÄ

Hammashoidossa käytetään enenevässä määrin yhdistelmämuoveja, kuten ns. bulk-fill muoveja (BFC) ja kuitulujitteisia muoveja (FRC) koska niiden käyttämisellä voidaan lyhentää hoitoon tarvittavaa aikaa ja lisätä korjatun hampaan mekaanista kestävyyttä. Yleensä sekä BFC että FRC -muovit peitetään hiukkaslujitetulla yhdistelmämuovilla (PFC) ja kokonaisuus on näin ollen monikerrosrakenteellinen (BLC). Monikerrosrakenteen muodostavista materiaaleista tiedetään paljon fysikaalisten ominaisuuksien osalta, mutta monikerrosrakenteen käyttäytymisestä purentaelimistön kuormitustilannetta jäljittelevässä tilanteessa tiedetään vähemmän.

Tässä väitöskirjatyön tutkimussarjassa selvitettiin BFC ja FRC -muoveista valmistettujen BLC-rakenteiden murtumista kuormitustilanteessa. Tutkimuksen päätavoite oli löytää paras mahdollinen BLC-rakenne, jolla saavutetaan valokoveteisen muovin hyvä kovettumisaste ja, että BLC -rakenne jäljittelisi mahdollisimman paljon hampaan kerrosrakennetta, joka koostuu hammasluusta ja kiilteestä, ja joiden välinen rajapinta on lujittunut kollageenikuiduilla.

Tutkimuksessa keskityttiin kolmeen BLC -rakenteeseen liittyvään kokonaisuuteen. Ensimmäisessä kokonaisuudessa tarkasteltiin yhdistelmämuovin ja hammasluun rajapintaa tilanteissa, joissa yhdistelmämuovin kerrospaksuus ja siten myös valokovetusvalon läpäisevyys BFC:n FRC:n ja PFC:n (kontrolli) läpi vaihteli. Toisessa osakokonaisuudessa tarkasteltiin BLC -rakenteen muovikerrosten rajapintaa. Tarkastelussa vaihdettiin BLC -rakenteen muodostavia muovimateriaaleja (i), rajapinnan pintarakenteen muotoa (ii) valmistamalla pintarakenne 3D -tulostusta apuna käyttäen. Rajapinnan vaikutus BLC-rakenteen kuormituksen kantokykyyn mitattiin sovelletulla puristuslujuustestillä. Kolmannessa tarkastelukokonaisuudessa perehdyttiin BLC -rakenteen rajapinnan kemiallisen liimautumisen vaikutukseen rakenteen kestävyys ja murtumiskäyttäytymiseen. Murtumiskäyttäytymisessä tutkittiin erityisesti murtuman etenemistä BLC -rakenteessa käyttämällä tutkimuksessa kehitettyä menetelmää rajapinnan murtositkeyden tutkimiseksi.

Tuloksina havaittiin, että FRC -muovi voidaan kerrostaa valokovetuksen näkökulmasta 4 mm kerroksina kuten aikaisemmin tiedetään voitavan tehdä kaupallisilla BFC -muoveilla. Kuormitustestit osoittivat, että sekä kerrospaksuudet että rajapinnan adheesio vaikuttivat tilastollisesti merkitsevästi BLC -rakenteen kuormituksen kantokykyyn ($p < 0.05$). FRC -muoviryhmissä kuormituksen kantokyky oli suurempi kuin BFC ja PFC -ryhmissä. FEM -mallinnus osoitti eroja kuormituksen aikaisen jännityksen jakautumisessa eri BLC -rakenteiden välillä. Materiaalikerrosten rajapinnan pintamuodolla oli myös tilastollisesti merkitsevä yhteys kuormituskestoon ($p < 0.05$). Murtumatyyppien tarkempi analysointi osoitti, että 3D -tulostusta apuna käyttäen valmistettu FRC muovin pyramidipintamuoto antoi parhaan lopputuloksen murtumatyyppien suhteen: kaikkien näytteiden murtuminen oli tyypiltään ”bulk fracture”, jolloin kerrosten välinen rajapinta ei pettänyt lainkaan. BFC -muovin tapauksessa kerrosten välinen rajapinta ei pettänyt 84,6 prosentissa näytteistä. 3D -tulostusavusteisesti valmistettu rajapinnan pintamuoto näytti parantavan kappaleen murtumiskäyttäytymistä.

Loppupäätelmänä todettiin, että BLC -rakenteissa FRC -muovin käyttäminen lisäsi kuormituksen kantokykyä sekä vähensi vaikeasti korjattavien murtumatyyppien määrää.

AVAINSANAT: Murtumatyyppi, kuitulujitteinen muovi, bulk-fill -muovi, biomimeettinen rakenne, raja kaksikerrosrakenne, rajapinta, 3D -mallinnus, kiille-dentiiniliitos, happi-inhibitio, FEM.

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Abbreviations

3D	Three-dimensional
ANOVA	Analysis of variance
BC	Before Christ
BFC	Bulk-fill resin composites
bis-GMA	Bisphenol-A-glycidyl dimethacrylate
BLC	Bi-layered composite
CQ	Camphorquinone
DA	Deteriorated-adhesion
DC	Degree of monomer conversion
DEJ	Dentin-enamel junction
EBADMA	Ethoxylated bisphenol-A-dimethacrylate
FBF	Flowable bulk fill
FE	Finite element
FEA	Finite Element analysis
FEPA	Federation of European Producers of Abrasives
FRC	Fiber-reinforced composite
LCU	Light curing unit
LED	Light emitting diode
MST	Modified notched strength test
OA	Optimal adhesion
PFC	Particulate filler composite
PMMA	Poly (methyl methacrylate)
PSE	Polyvinyl siloxane elastomer
SBS	Shear bond strength
SD	Standard deviation
SDR	Smart dentine replacement
SENB	Single-edge-notched-beam
SLA	Stereolithography
SPSS	Statistical Package for Social Science
TEGDMA	Triethylene glycol dimethacrylate
UDMA	Urethane dimethacrylate
VHN	Vickers hardness number

List of Original Publications

This thesis is based on the following original articles, which are referred to in the text by the Roman numerals I–IV.

- I. Omran Tarek A, Garoushi Sufyan, Abdulmajeed Aous A, Lassila Lippo V, Vallittu Pekka K. Influence of increment thickness on dentin bond strength and light transmission of composite base materials. *Clinical Oral Investigations*, 2017. <https://doi.org/10.1007/s00784-016-1953-6>
- II. Omran Tarek A, Garoushi Sufyan, Akikazu Shinya, Lassila Lippo V, Vallittu Pekka K. Bonding interface affects the load-bearing capacity of bi-layered composites. *Dental Materials Journal*, 2019, *In press*.
- III. Omran Tarek A, Garoushi Sufyan, Lassila Lippo V, Vallittu Pekka K. The effect of interface surface design on the fracture behavior of bi-layered composites. *European Journal of Oral Sciences*, 2019. <http://dx.doi.org/10.1111/eos.12617>
- IV. Omran Tarek A, Lassila Lippo V, Garoushi Sufyan, Vallittu Pekka K. Adhesive interface between layers of a bilayered composite influenced the fracture behavior and load. *Manuscript*.

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1 Introduction

The uniqueness of a dynamic biological structure that can last a lifetime of service, has made teeth and the field of dental sciences an area of interest for research and development for many centuries ago (Leek, 1967). In fact, one of the early records of dentistry as a profession was as early as 2600BC in Ancient Egypt (Weinberger, 1948). While the first documentation of a dentist not only in Egypt but in the world was Hesy-Ra who lived at about 2660 BC (Forshaw, 2009).

However, investigations of Egyptian mummies and the dental procedures they received shows that routine dental procedures such as prosthetic four unit bridges were made using human teeth to serve as pontics and gold wires to serve as connectors (Iskander et al., 1979). This archeological finding sheds the light on an important philosophy that seems to have existed thousands of years ago when choosing materials to restore teeth or their mechanical function and that is: biomimicry (bio = life and mimicry = imitation) (Deb & Chana, 2015). Thus, Ancient Egyptian Dentists used extracted teeth as a material to restore the lost masticatory functions (Iskander & Harris, 1977).

In comparison, 21st century dental procedures are more conservative and primarily utilize composite materials to restore aesthetics of teeth and their mechanical function (Özyürek et al., 2018). Direct restorative procedures are the most routine interventions in clinical dentistry and posterior composite restorations in particular, present the most challenging of these interventions (Ástvaldsdóttir et al., 2015; Schwendicke et al., 2016). Minute discrepancies in filling techniques may lead to premature failure of the restoration, making composite filling materials technique-sensitive (Nicola et al., 2016; Pfeifer, 2017). Recent systematic reviews and clinical trials have demonstrated that fracture of direct composite fillings is one of the most prevalent causes of failure in posterior restorations (Heintze & Rousson, 2012; Opdam et al., 2014). Hence, a closer look at the filling techniques and materials used for restorative treatment seems important due to the potential magnitude of their repercussions

Moreover, the constant development of the fundamental properties of the once known packable resin composite (Leinfelder et al., 1999). has given rise to new categories of composites, notably flowable and packable bulk-fill resin composites

as well as fiber-reinforced composite materials. Improved properties include improved mechanical toughness, modified viscosities to improve adaptability to cavity margins, and the ability to be placed in bulk increments of 4 mm to reduce the time needed to restore deep cavities (Bucuta & Ilie, 2014; Heintze et al., 2016; Leprince et al., 2014).

The introduction of novel fillers such as discontinuous E glass fibers that improve the mechanical properties (Nicola et al., 2016). have demonstrated a noteworthy behavior to fracture, where the glass fibers hinder crack propagation and lead to improved fracture toughness of the materials (Fonseca et al., 2016; Garoushi et al., 2018). Such fracture resistance properties are similarly observed in the natural dentition due to the presence of the collagen fibers in dentin as well as the dentin-enamel junction (Chan et al., 2011). This similarity highlights the potential of having a biomimetic approach to direct restorations, by also trying to mimic the DEJ in its gradient like characteristic as well as its intricate design.

Furthermore, another naturally occurring phenomenon that allows the teeth to have unprecedented longevity when compared to current resin composite materials, is the presence of a bi-layered structure consisting of two contrasting tooth tissues; enamel and dentin (White et al., 2005). This contrasting material properties causes the tooth to act as a dynamic heterogeneous structure when compared to the homogeneous structure of a single-material resin composite restoration (Zaytsev & Panfilov, 2014). This fundamental bi-layered structure of teeth has not been deliberately addressed when utilizing resin composite materials in direct restorations and hence this thesis project will investigate such a biomimetic approach.

Presently, flowable bulk fill composites and fiber-reinforced composite materials have gained popularity in being primarily utilized as dentin replacing materials that can replace tooth tissue in bulk increments (Do et al., 2014; Tanner et al., 2018). However, these materials are required to be finally capped with a conventional particulate filler composite (PFC) that has greater wear-resistance properties (Garoushi et al., 2017). Using two different materials in this situation gives rise to a bi-layered restoration that consists of two different materials with two different properties. Therefore, this creates a heterogeneous structure similar to that of a natural tooth. However, in contrast to the tooth, the differing properties of the materials that make up the bi-layered restorations influence the fracture behavior of the whole restoration (Garoushi et al., 2013).

Furthermore, light-curing two different increments individually raises the question of the quality of adhesion at the composite-composite interface due to the formation of an oxygen inhibition layer on the freshly cured resin composite (Truffier-Boutry et al., 2003). It is currently known that to ensure an optimal adhesion between the composite layers then optimal conditions should be verified, therefore, if anything disrupts the oxygen inhibition layer such as water, saliva, etc

then the mechanical behavior of the bi-layered structure can be drastically affected (Bijelic-Donova, Flett, et al., 2018; Bijelic-Donova et al., 2015; Chuang et al., 2001). However, the effect of adhesion quality at interface between surface PFC and bulk-base composites on the fracture resistance and behavior needs to be further investigated. Additionally, the different composite materials vary in filler content and type, and therefore exhibit a different micro-structure and mechanical properties. Similarly, the two materials used in bi-layered structures are required to be placed in varying increment thickness. It is likely that such difference in micro-structure and increment thickness can influence the mechanical behavior of the bi-layered composite structure in a similar manner to the concept of elasticity modulus mismatch present in the natural dentition.

The current literature is focused on evaluating the properties of materials used in single or bi-layered restorations individually. However, clinically, heterogenous bi-layered restorations serve as a combined dynamic structure (Van Dijken & Pallesen, 2017; Van Dijken & Pallesen, 2015). As a result, this thesis project has examined bi-layered structures as a single dynamic structure. Using various novel and conventional laboratory tests this project intended to preliminary evaluate and predict clinical fracture behavior of bi-layered structure by using in vitro models.

2 Review of the Literature

2.1 The Natural Tooth

Teeth are unique and complex organs formed from different embryonic layers. Mature tooth comprises enamel, dentin, cementum, and dental pulp. Through an intricate and highly synchronized complex processes, the tooth gets sculpted at the cellular level until it erupts into the oral cavity.

Teeth have various functions aside from their usual mechanical masticatory role. They are vital to aesthetics and beauty of the human face as well as assist in articulation of words and speech formation (Chen & Liu, 2014). However, their exceptional durability is due to the combination of various tissues with different properties and structure (Giacaman et al., 2016). Therefore, this present effort has focused on the structure of the loadbearing tissues of the tooth, i.e. the enamel, dentin and the dentin-enamel junction as a framework to follow for the development of novel approaches to emulate the tooth and enhance the performance of direct composite restorations.

2.1.1 Tooth Bi-Layered Structure

Interestingly, the most durable bi-layered biocomposite structure already exists in our teeth. The high wear-resistance of the hardest tissue in the human body is the dental enamel. It has hardness comparable to that of glass, and protects the tooth from the continuous chewing and from the oral environment (Chai et al., 2009). Enamel possesses a complicated hierarchical structure, where at least three levels could be identified. A microscopic scale made up of hydroxyapatite nanofibers, that are bundled together to form enamel rods that aggregate the mesoscopic scale. Whereas the third macroscopic level is an ordinate decussated structure of the calcified rods (Cui & Ge, 2007).

However, this highly mineralized tissue serves as a capping layer to the core-layer of the tooth—the dentin. Dentin is the less-mineralized tissue, softer than enamel; yet tough and forms the bulk of the tooth (Zaslansky et al., 2006). Due to its properties and microstructure, a human dentin can behave like some filled polymers (Liang, 2002).

Enamel and dentin can be described as a natural composite structure (He & Swain, 2009). Each having their own modulus of elasticity (enamel ~80 GPa, dentin ~20 GPa) and the unique aforementioned microstructures (Zhang et al., 2014). The mismatch in properties of these two layers, gives rise to a gradient like area in between them known as the dentin-enamel junction (Lin et al., 1993). This interphase-layer has been reported to enable efficient stress transfer between enamel and dentin and exhibit crack-arresting properties against cracks that arise in the brittle enamel (Sui et al., 2016). Thus, protecting the bulk of dentin from further crack propagation (Zaytsev & Panfilov, 2016).

2.1.2 Mechanical Behaviour

Teeth are the most important organ in the mechanical process of digestion (the chewing process of food) (Johnson, 2016). Incisors are sharp-edged allowing incisions and cuts into the food, while canines help tear the food. The premolars and molars are flat and well designed to grinding and crushing food. They can sustain high loads at different directions over a lifetime of service (Woelfel & Scheid, 2002). A recent study has shown that the bi-layered combination of enamel and dentin can withstand around 20MPa occlusion pressure 3000 times per day (Chen et al., 2014). Yet as clinicians we seldom witness complete fracture of teeth due to their normal masticatory function.

To help us better understand the mechanical behavior of teeth, a closer look at the micro and macro structure of the bi-layered structure is necessary (Sui et al., 2016). Nano-indentation profiling of this area showed that it is rather a zone with differing property and a scallop shaped design. Also, the enamel in proximity to the DEJ identified as the first enamel and is the hardest enamel and most mineralized (White et al., 2005). In a previous study it was shown that the crack progresses in length from the occlusal aspect, the fracture resistance of the enamel layer was exponentially increased (Bajaj & Arola, 2009). This is a unique mechanical property when observing the enamel on its own. This demonstrates that enamel is not having a uniform structure and rather is dynamic to resist fractures from different areas. The mechanical behavior of enamel largely owes to the enamel rods and bundling into a hierarchical configuration of key-hole. This forms a structure that is wear resistant due to the mineralization and hardness. Yet, enamel on its own is brittle and requires the support of the underlying softer dentin tissue. The elasticity and plasticity of dentin behaves in such a manner similar to polymer (Zaytsev et al., 2014). The modulus of elasticity mismatch allows the low-grade continuous loading of the structure to be withstood for a lifetime (Barani et al., 2011). The collagen fiber reinforcement present around and within the dentinal tubules also improves the toughness of dentin and thus if a fracture front arise in the bulk of the tooth then right

away it gets deflected along the collagen fibers and the energy of the crack front is dissipated (Ledogar et al., 2016).

A closer look at the DEJ between the two layers shows a more dynamic area as fracture fronts are always being deflected away from the junction and crack fronts are forced to parallel to the DEJ and not across it (Dong & Ruse, 2003). The configuration of the DEJ has been studied by many research groups as it serves as the one of the fundamental areas that allow the bi-layered structure to function effectively and efficiently (Brauer et al., 2011). Hence, many research groups have been trying to understand the design and how it affects the mechanical properties of the tooth. In a previous study, it was found that the DEJ's scalloped interface design allows enamel to bond strong to dentin and hence will not be affected by mechanical shear forces (Brauer et al., 2010).

The combination of all the aforementioned layers makes up a durable bi-layered biocomposite structure that serves in our bodies for a lifetime, unless associated with defects to the tooth structure, i.e. carious lesions or trauma that affects the natural dentition and causes tooth tissue loss (Feliciano & Caldas Jr, 2006). Modern dentistry and biomaterial science offer various treatment options and interventions to restore the mechanical function of the affected tooth.

2.1.3 Defects and Tissue Replacement Options

Defects to the tooth structure can be caused by abnormal development, chronic injury of hard tissue, traumatic injuries, and tooth diseases such as pulp disease, periapical disease, and dental caries. Dental caries is the most prevalent disease that results in local demineralization of tooth tissue due to active microbiologic destructive process that leads to the loss of tooth tissues and as a consequence cavity of the tooth (Tinanoff, 2019). If left untreated the disease progresses and extensive tooth tissue loss is induced. In extreme cases the disease can progress to the pulp of the tooth beyond the dentin jeopardizing the prognosis of the tooth (Özyürek et al., 2018).

However, regardless of the extent of the cavitation and healthy tooth tissue loss, conservative restorative and prosthetic solutions are available to most dental practices (Afrashtehfar et al., 2017). Ranging from the most extensive procedures like implants, to less extensive like removable prosthesis and fixed prosthesis such as crowns and bridges and then to the lesser extensive procedures such as indirect inlay and onlay restorations and finally to the most conservative, which is the focus of this thesis project - direct restorations that are predominantly resin composite restorations.

2.2 Direct Restorative Materials

2.2.1 Conventional Resin Composite

These materials were once known as packable composite, later on defined as resin-based composites or particulate filler composites PFC. (Leinfelder et al., 1999) These materials consist of a resin-based matrix that has filler incorporated within the matrix (Thomaidis et al., 2013). These restorative materials have been an area of study for many years. The first composite had quartz filler particles of 100 μm and had very poor wear resistance. Nowadays, PFCs contain finely ground glass and silica, with the addition of barium oxide (Sarrett, 2005). These finely ground glass particles give strength to the soft polymer matrix and hence form a composite material. Alongside the fillers in the resin matrix are photochemical components (camphorquinones (CQ) and ethyl 4-(dimethylamino) benzoate (EDAB)) that initiate a polymerization chain reaction converting the resin monomers into long chain polymers and the composite in turn hardens (Moszner et al., 2008). The polymerization reaction is initiated using a light source of utilizable wavelength 430-480 nm (Price et al., 2015).

Optimal polymerization is crucial for the longevity of the direct restorations due to the battery of factors that can jeopardize the restoration as a whole (Price et al., 2014). For instance, sufficient light penetration going through the materials from the occlusal aspect to the pulpal floor is detrimental to optimal bonding of the most bottom layer to the tooth tissue. Hence, for conventional resin composites 2 mm thick increments are advisable to ensure sufficient light curing to initiate in the composite material (Ilie et al., 2013). Also, a phenomenon studied thoroughly in the literature is known as polymerization shrinkage or polymerization stress which may also be a cause for marginal gap formation between the tooth and the composite filling material. As a consequence, such a gap can serve as an area of possible bacterial infiltration and stagnation which could lead to secondary marginal caries (Lee et al., 2007; Sarrett, 2005).

However, this work shall only lightly touch upon this broad topic of depth of cure and only look at the sufficiency of depth of cure when a new class of composites are being cured in thickness increment that exceeds that of the conventional 2 mm.

2.2.2 Bulk-Fill Resin Composite

As the name entails, bulk-fill resin composites are a new class of resin composite materials that have the added benefit to be cured in bulk increments of 4 mm (Van Ende et al., 2017). They are advantageous in saving time for the clinician as less light curing procedures are required per cavity (Yap et al., 2016).

Due to their improved depth of cure, they can safely be placed in bulk increment without jeopardizing effective polymerization and depth of cure (Ilie et al., 2013; Pfeifer, 2017). BFC materials can achieve such a depth of cure by either increasing the translucency, having less fillers, adding an extra photochemical initiator or by having fillers such as glass fillers that improves the scatter of the light further within the resin matrix and helps improve the degree of conversion (Ilie et al., 2013; Lassila et al., 2012; Miletic et al., 2017; *Vivadent Ivoclar. Research and Development Report no. 19. Schaan Liechtenstein*, 2013). Another related fundamental characteristic of BFCs is the degree of monomer conversion the material can attain during light-curing in bulk increments (Garoushi et al., 2015).

Some bulk-fill materials are considered to have an improved polymerization stress behavior in comparison to conventional PFC and therefore less stress or fractures will be liable to exist within such a restoration (Garoushi et al., 2008; Uctasli et al., 2005; Yamamoto et al., 2018). Some BFC materials, are require to be capped by layer of PFC as their properties do not allow them to sustain direct occlusal wear during mastication (Assis et al., 2016; Özyürek et al., 2018; Tomaszewska et al., 2015).

2.2.3 Fiber-Reinforced Resin composite

Fiber-reinforcement as a concept exists in many fields around us. In nature for instance, the tree stems can be considered fiber-reinforced composites. At the cellular level, they consist of fibers that are, embedded into matrix of parenchyma. They contain cellulose fibril aggregates that are aligned ‘fibers’, in a matrix of other polysaccharides. The matrix has isotropic material properties, while the fibers exhibit anisotropy (Shah et al., 2017). The orientation of the fibers in tree trunks however, can be appreciated when the wood is to be chopped, either in parallel or in perpendicular to the orientation of the fibers (Gibson, 2012). Similarly, such fibers with an intricate orientation can be observed in the tooth. The enamel fibrils and rods as well as the collage fibers present in detin, coagulate to form bundles of fibers oriented in multi-directions (Chan et al., 2011). The fibers provide fracture hinderance properties that hinder the propagation of fractures further through tooth tissues or at least deflect the crack front away from the bulk of the tooth (Kruzic & Ritchie, 2008).

In dentistry, fiber reinforcement has many utilities, including prosthodontics bridges and crown as well as composite fillings (Barreto et al., 2016; Perea-Lowery & Vallittu, 2018). According to previous thesis projects conducted at the University of Turku, fiber-reinforced composites were also investigated as oral implant abutments and root canal posts (Abdulmajeed, 2013; Bell-Rönnlöf, 2007; Mosquera, 2015).

The utility of discontinuous fiber-reinforcement in a novel resin composite material that can be used to restore deep cavities as a direct restorative material warrants further investigation. The fiber-reinforced composite is made up of a bisphenol-A-diglycidyl-dimethacrylate (bis-GMA), triethylene glycol dimethacrylate, and polymethylmethacrylate resin matrix. This matrix is known as having semi-interpenetrating polymer network (semi-IPN) and provides enhanced bonding properties for repairs and improves the toughness of the polymer matrix. Embedded in the matrix are short E-glass fibers ($\sim 1.5\mu\text{m}$) with random orientation. This gives the discontinuous-FRC an anisotropic property similar to that in nature (Vallittu, 2015). It has been demonstrated in previous studies that this property allows discontinuous FRC to be more resistance to fatigue and fracture in multi-directional masticatory forces, in a similar manner to the one experienced in the natural dentition, especially in the posterior cavity (Garoushi et al., 2006).

Additionally, FRC have demonstrated improved bond strength when compared to other composite materials, which has been hypothesized due to the fibers providing a micromechanical bond to the structure it is bonding to (Lastumäki et al., 2002). Similar to some of the aforementioned BFCs materials, FRC also need to be covered occlusally with a capping layer of PFC and hence would give rise to a bi-layered structure (Tanner et al., 2018). Due to the need to mimic the durability of the natural dentition, a thorough understanding of the mechanical behavior of composite materials needs to be established through a magnitude of both in vivo and in vitro studies. This can enhance our understanding of the composite materials we use and help in development of their mechanical properties.

2.2.4 Mechanical Properties

Due to the primary need for composite materials to restore the defected masticatory function in the affected teeth, several research projects have investigated the effect of the inherent mechanical properties of the materials (Abouelleil et al., 2015; Engelhardt et al., 2016).

By putting the focus on the how the materials behave under specific tests, more literature can start to form incrementally like pieces of a puzzle coming to completion. It is important to understand in a dynamic environment such as the oral cavity many factors play a role (Heintze et al., 2016). Nonetheless; to follow the scientific methodology of empirical testing, each mechanical property of the developed materials needs to be tested in an objective, standardized and reproduceable manner (Arola, 2017; Czasch & Ilie, 2013; Ferracane et al., 2016).

Therefore, from the many mechanical properties that the composite materials are expected to possess, this project has focused on a few properties such as, the load bearing capacity and fracture behavior (Bijelic-Donova et al., 2016; Nagata et al.,

2016; Schwendicke et al., 2016). Also, the adhesion to and within resin composite material has been shown to be very foundational and detrimental to the longevity of the composite restorations in previous studies (Bijelic-Donova et al., 2015; Bijelic-Donova, Uctasli, et al., 2018). As result understanding and optimizing these properties warrants a thorough evaluation.

2.2.5 Load Bearing Capacity

The ability for the composite restoration to withstand high loads specially in posterior region is vital for a material to be safely used in a patient's mouth. The maximum loads experienced by the posterior teeth in maximum intercuspal occlusion could range from 500-800 N (Ledogar et al., 2016). Thus, the minimum threshold a composite material needs to surpass must be within those limits. Load bearing capacity in general is a good preliminary indicator of how the materials are to behave under the mechanical forces in the mouth (Garoushi et al., 2018; Basaran et al., 2013). However, as the materials fatigue, they are more prone to cracks and fractures and hence a better understanding of fracture behaviors will be very useful to understand the fracture cascade that takes place in the different types of resin composite materials (Barreto et al., 2016; Batalha-Silva et al., 2013; Ferracane et al., 2016).

2.2.6 Fracture Behavior

The unprecedented behavior of the enamel, dentin and the DEJ when it comes to fracture behavior has shed the light on the importance of testing composite materials to similarly examine their behavior when they fracture (Chai et al., 2009; Zaytsev et al., 2016). The inherent filler types used in the composite materials have been shown to dictate the extensiveness of the fractures (Petersen, 2017). Particulate filler composites as well as bulk-fill composites are made from the micro-hybrid filler. These provide a solid structure that although can withstand high loads, unfortunately ends up catastrophically fracturing when the loads are too high (Bijelic-Donova et al., 2018).

On the contrary, the presence of fibers in the FRC influence the fracture behavior to be less extensive and render repairs more probable. Multiple studies have also demonstrated that in terms of fracture toughness as a material property, Fiber-reinforcement also improved the fracture toughness up to three fold when compared to conventional PFC (Barreto et al., 2016). According to Heintze, fracture toughness as a laboratory test has shown the closed association with fractures in the clinics (Heintze et al., 2016).

Therefore, testing for fracture toughness as well as examining the fracture behavior closely can inspire new materials that have fail-safe fractures mechanics (Malterud,

2014). Developing composites with predictable fracture outcomes, can help deliver more consistent and sustainable treatments to dental patients. Looking at the relationship of loadbearing capacity of the materials can be beneficial to further assess the correlation between load bearing capacity and fracture behavior (Hatta et al., 2011). However, a wide perspective is also needed to see the interaction of other properties to the restoration as a whole. Other fundamental elements such as adhesion within the resin composite layers or between the restoration and the tooth, it can also be a point of weakness to the fracture behavior and this warrants a closer look.

2.2.7 Quality of Adhesion and Design Between Interfaces

Resin composite restorations and today's concept of conservative dentistry are a possibility due to the adhesive chemistry that has developed substantially along the years (Dejak & Młotkowski, 2015; Truffier-Boutry et al., 2003). The introduction of aforementioned bulk-fill materials with proposed increased depth of cure raises the concern of the quality of adhesion to the most bottom layer in a restoration (Borgia et al., 2019). This deepest layer serves as the foundation of a composite restoration and hence special attention and care should be placed on it (Flury et al., 2014). Additionally, the requirement of composite materials to be placed in increments sheds the light on the adhesion quality between the two different and independently cured composite increments. Composite materials form an oxygen inhibition layer when light cured which has been shown to affect the composite structure if this layer is compromised (Aromaa & Vallittu, 2018; Bijelic-Donova et al., 2015). Therefore, examining the composite-composite interface and its effect of bi-layered composite restorations can be very useful.

In comparison to the bi-layered composite, the interface between the natural bi-layered structures enamel and dentine, involve a gradient-like area with an intricate scalloped interface known as the DEJ (Zaslansky et al., 2006). The DEJ is known to strongly influence the fracture behavior and stability of the bi-layered tooth structure. (Dong et al., 2003; White et al., 2005). This highlighted the possibility to fabricate a bioinspired gradient-like area with an interface design and to examine its effect on bi-layered composites.

2.3 Resin Composite Bi-Layered Structure

2.3.1 Mechanical Behavior of Bi-Layered Structures

Mechanical behavior of dental composite materials is of utmost importance, if they are to replace the missing tooth tissue and restore their daily mechanical function (Abdelmegid et al., 2016; Do et al., 2014). Therefore, mechanical and fracture

behaviors of bi-layered composite structures were the main area of focus in this thesis.

Filling materials such as the aforementioned fiber-reinforced and bulk-fill composite materials, are often required to be capped occlusally using a conventional particulate filler composite material (Garoushi et al., 2006; Van Dijken et al., 2017). Combining two different composite materials with differing properties gives rise to a bi-layered restoration that highlights the philosophy of biomimetics as it similarly follows the bi-layered structure naturally present in teeth .i.e. enamel and dentin. This is necessary to produce a final restoration with better wear properties (Garoushi et al., 2017) or to protect the fibers in the resin matrix from being exposed to the oral environment and acting as points of bacterial ingress, similar to that in the dentinal tubules (Garoushi et al., 2013).

Hence, investigating factors such as load-bearing capacity and fracture behavior will further help the dental material innovators to develop a biomimetic approach for bi-layered restorations.

2.3.2 Clinical Relevance & Application Guidelines

Generally, biomaterial sciences focus on individual materials properties. This renders research investigating the mechanical behavior of bi-layered composites highly necessary, if we are to draw up guidelines to be used by clinicians in their daily practice (Nagata et al., 2016). Using materials such as bulk-fill allows clinicians to substitute the placement of multiple increments into one single bulk-increment. However, this raises concerns over the efficacy of the bond of the bulk composite and its adhesion to the bottom layer of the tooth structure (Yamamoto et al., 2018). Following the same principle in bi-layered restorations, the composite-composite adhesion interface needs to be investigated to provide dentists with knowledge of what happens to the whole structure when the interface quality is compromised (Bicalho et al., 2014). Therefore, adhesion and bonding guidelines can be helpful for bi-layered restorations.

Furthermore, when combining two materials with differing properties such as modulus of elasticity and microstructure, the mechanical behavior is expected to vary. Hence, sufficient knowledge of the behavior of various material combinations can assist the dentist to predict their mechanical performance in the patient's mouth (Thomaidis et al., 2013).

Moreover, applying two different materials in different thicknesses sheds the light on the optimal thickness of each of the materials. In the case of the natural tooth, a thin (1.2-1.7 mm) enamel capping overlays the bulk of the tooth that is made of dentin (Mahoney, 2008). The thickness varies depending on the area of the tooth and the masticatory function it will experience (Grine, 2005). However, when

constructing bi-layered restorations, the exact and optimal thickness of the two different composites is not clearly outlined. It is expected to be dependent on the properties of the materials used in the bi-layered structure as well the area of the tooth tissue being replaced. Therefore, this thesis project also investigated the different thicknesses using an array of different materials. The data acquired can be useful in creating guidelines for increment thickness depending on not just location, but also the mechanical properties of the materials being used.

3 Aims

The research presented in this thesis was performed to investigate ways of optimizing fracture behavior of bi-layered composite restoration made of two different materials, by evaluating several parameters such as moduli of elasticity of each layer, adhesion and design of the composite-composite interface and thickness of layered increments.

Hence, the working hypothesis was that by changing either the materials used in the bi-layered restorations or modifying factors of the composite-composite interface and the dimensions of the overall bi-layered system, then the durability of such bi-layered restoration could be improved.

Correspondingly, the following aims were set to:

1. Investigate the influence of increment thickness on dentin bond strength and light transmission of different bulk-fill composites and a new discontinuous fiber-reinforced composite. (Study I)
2. Evaluate the effect of material type, adhesion and thickness on the load-bearing capacity of the bi-layered composite structures. (Study II)
3. Study the effect of 3D designed interfaces on the load-bearing capacity of bi-layered composite structures. (Study III)
4. Investigate the crack propagation and fracture behavior of bi-layered composite structures using a model that simulates clinical conditions where load is applied to a restoration with deep fissure. (Study IV)
5. Analyze how the presence of the O₂ inhibition layer at the composite-composite interface affects the fracture behavior and fracture toughness of bi-layered structures. (Studies II and IV)

4 Materials and Methods

The roman numbers after each heading specify the study in which the described methodologies were used. While, the materials used to fabricate the specimens in studies I-IV are listed in Table 1.

Table 1. Composite materials investigated in studies I-IV

Material (Shade)	Manufacturer	Type of composite	Matrix composition	Inorganic filler content	Study
G-ænial Anterior (A3)	GC Corp., Tokyo, Japan	Micro-hybrid conventional	UDMA, dimethacrylate comonomers (Bis-GMA free)	Prepolymerized fillers with silica filler content 73 wt% 63 vol%	I
Tetric EvoCeram® (IVA)	Ivoclar Vivadent AG, Liechtenstein	Packable bulk-fill	Dimethacrylate co-monomers	Barium glass filler 80 wt%, 60 vol%	I
G-ænial Posterior (A3)	GC Corp., Tokyo, Japan	Micro-hybrid conventional	UDMA, dimethacrylate comonomers (Bis-GMA free)	Prepolymerized fillers with silica filler content 81 wt% 65 vol%	II, III, IV
EverX Posterior™ (n/a)	GC Corp., Tokyo, Japan	Discontinuous fiber reinforced	Bis-GMA, PMMA, TEGDMA	Short E-glass fiber filler, barium glass 74.2 wt%, 53.6 vol%	I, II, III, IV
SDR™ (universal)	Dentsply Sirona, York, PA, USA	Flowable bulk-fill	TEGDMA, EBADMA	68 wt%, 44 vol%, barium borosilicate glass	I, II, III, IV

PMMA, polymethylmethacrylate; bis-GMA, bisphenol-A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; EBADMA, ethoxylated bisphenol-A-dimethacrylate; wt%, weight percentage; vol%, volume percentage.

4.1 Specimen Preparation

4.1.1 Preparation of Tooth Specimen (I)

One hundred eighty extracted, sound, and caries-free human teeth with comparable occlusal widths (molars and premolars) were used as bonding test substrates in

study I. The occlusal surface of each tooth was wet ground using a silicon carbide grinding paper (FEPA no. 180) and a grinding machine (Struers LaboPol 21, Struers A/S, Rodovre, Denmark) to initially create a flat enamel surface. Subsequently, the teeth were mounted individually into an acrylic block using cold cure auto-polymerized acrylic resin (Vertex-Dental B.V., Zeist, Netherlands). Following the setting of the acrylic resin, the blocks were then re-ground using a no. 1200 grit FEPA silicon carbide grinding paper until all enamel was ground away and the superficial dentin was exposed. Teeth that showed any visible pulp exposure or cracks were removed and excluded from study I, thus leading to a total of 180 specimens to be tested.

4.1.1.1 Bonding Procedures

The prepared teeth were divided into four groups according to the resin composite being used (Table 1). Each group was further divided into three subgroups (n = 15) according to the thickness of the added composite increments (i.e., 2, 4, and 6 mm) (Figure 1).



Figure 1 Shows the three subgroups of prepared specimen after composite build up. (6.0mm, 4.0mm and 2.0mm; left to right)

All teeth were then treated with Scotchbond Universal (3M Deutschland GmbH, Neuss, Germany) adhesive using a one-step, self-etch procedure. Application steps

followed were according to the manufacturer's instructions. Then, using a transparent polyethylene mold with an inner diameter of 3.6 mm, bulk increments of each composite were applied to the dentin substrates at different thicknesses. The composites were photopolymerized once and at full thickness from the occlusal aspect for 40 s using a hand-held light-curing unit (Elipar S10, 3M Espe, Seefeld, Germany) (utilizable wave-length range 430–480 nm with maximal intensity at 455 nm, light irradiance 1200 mW/cm²; according to the manufacturer's information).

The specimens were stored in water (room temperature) for 3 days prior to testing. During the entire preparation procedure, the specimens were maintained in a hydrated state

4.1.2 Bi-Layered Resin Composite Specimens

4.1.2.1 Composite-Composite Adhesion Interface (II)

In study II, a total of 180 cylindrical specimens having a bi-layered composite structure were prepared (diameter = 7 mm, height = 5 mm). Each specimen included a combination of two different materials i.e. a capping PFC material (top layer) and a base composite material (bottom layer). Gænial Posterior (GP) was used as the capping PFC material in all specimens, whilst the base composite material was either FRC (everX Posterior) or FBF (SDR). For each base composite material, 90 cylindrical specimens were fabricated in total.

In study II, the composite-composite adhesion interface within the bi-layered specimen was based on either: (1) a conventional O₂-inhibited layer to simulate optimal-adhesion, whereby the subsequent top surface layer was immediately placed after light-curing of the bottom layer; or (2) a polished (wet-ground) surface to simulate deteriorated-adhesion.

Three top surface thicknesses were prepared; 1.0 mm, 1.5 mm and 2.0 mm. However, the total height of bi-layered specimen was standardized at 5 mm. Additionally, the specimens at each thickness were subject to the two different preparation procedures to simulate different qualities of adhesion between the top and bottom layers. Study design and materials investigated in this study can be viewed in Figure 2 and Table 1.

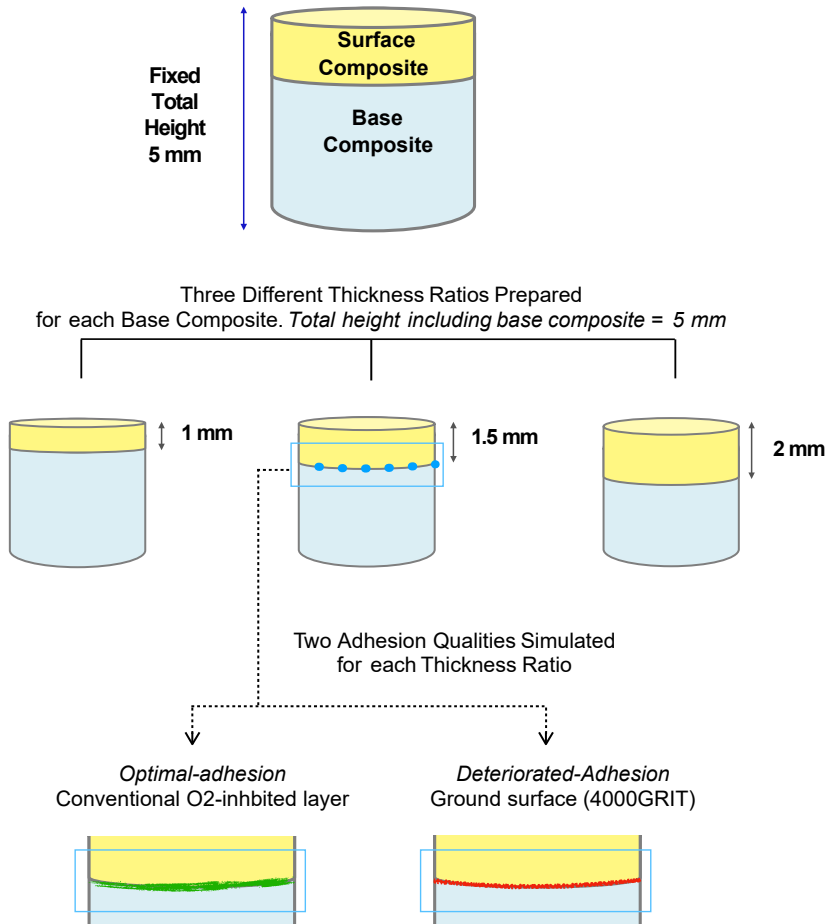


Figure 2 Schematic drawing of test specimen and study design. Adapted from original publication I.

In total, there were 12 groups ($n=15$) involving 3 top surface PFC thicknesses, 2 base materials and 2 adhesion interface conditions.

Moreover, the two layers were light-cured independently from the top aspect, perpendicular to the long axis of the specimen and at full thickness for 40 s. A LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) with a calibrated light intensity (MARC[®] Resin Calibrator (BlueLight analytics Inc., Halifax, Canada) of 1765.30 mW/cm² ($SD\pm 17.91$), was used throughout this study. Total energy measured was 70.61 J/cm² ($SD\pm 0.72$). Prior to testing, all specimens were stored for 48 hours in a dry environment at room temperature ($23 \pm 1^\circ\text{C}$).

4.1.2.2 Composite-Composite 3-D Interface Design (III)

In study III, four interfacial designs were prepared using 3D modeling computer software Rhinoceros 5 (Mc Neel Europe, Barcelona, Spain) (Figure 3). The finalized interfacial designs were then 3D printed using a Form2 3D printer (FormLabs, Somerville, Massachusetts). The printer utilized stereolithography (SLA) technology using methacrylate photopolymer resin.

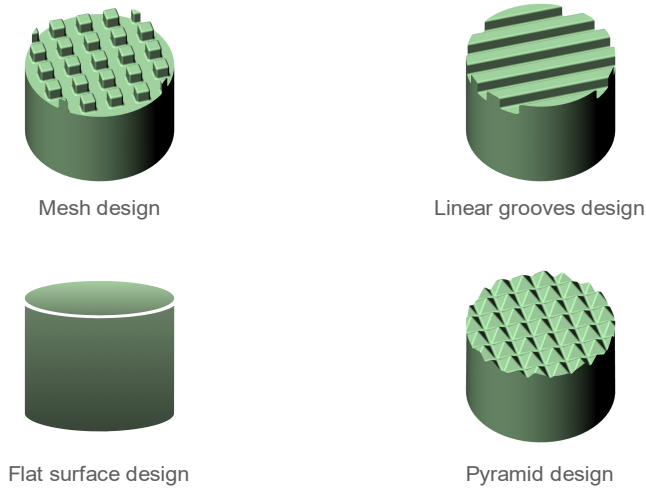


Figure 3 Computer-rendered three-dimensional (3D) models of the interface designs used in study III. The width and depth of the grooves in the mesh and linear designs are 0.5 mm. In the pyramid design, the width of the groove is 1 mm and the depth is 0.5 mm. Adapted from original publication III.

Subsequently, transparent polyvinyl siloxane elastomer (PSE) molds (Memosil®2 Kulzer, Mitsui Chemicals Group, Japan) were fabricated so they could be pressed onto the base composite to produce the following four designs: pyramidal, mesh, linear grooves and flat surface.

Eighty bi-layered cylindrical specimens were prepared using transparent cylindrical plastic molds (diameter: 7 mm; total height: 5 mm). Four interface-design test groups were fabricated using each of the base composites ($n = 10$ per group).

All bi-layered specimens comprised a bottom layer made of a base composite of either everX Posterior (GC, Tokyo, Japan), which is a short fiber-reinforced bulk-fill base composite, or SDR (Dentsply Sirona, York, PA, USA), which is a flowable bulk-fill composite. A top layer of G-aenial Posterior (GC, Tokyo, Japan), which is a conventional particulate filler composite (Table 1), was added. The exact thickness of each layer is presented in Figure 4.

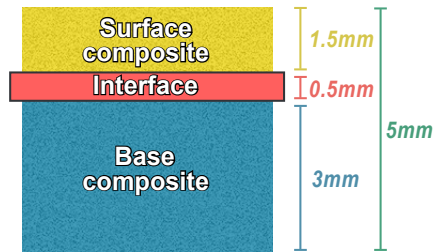


Figure 4 Two-dimensional schematic illustration of the prepared bi-layered specimen used in study III and the exact thickness of each layer. Adapted from original publication III.

Moreover, after the base composite materials were placed in the cylindrical plastic molds, a transparent PSE mold having an interface design was pressed onto the surface of the base composites to imprint the interface design onto the surface of the base composite before light curing (Figure 5).

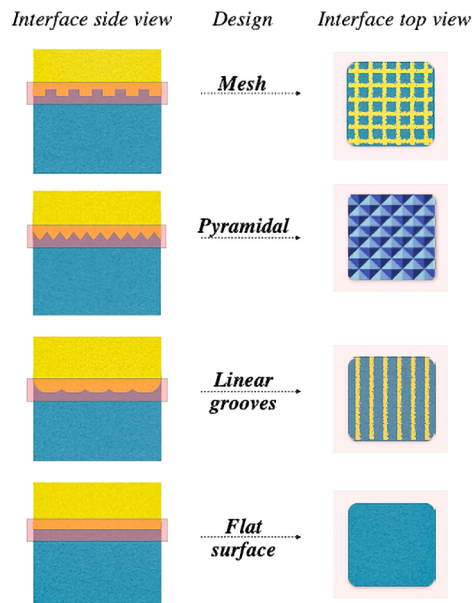


Figure 5 Two-dimensional schematic illustrations of the different interface designs fabricated in study III. Adapted from original publication III.

A LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) was calibrated using MARC[®] Resin Calibrator (BlueLight analytics Inc., Halifax, Canada). A calibrated average light intensity of 1765.30 mW/cm² (SD±17.91) was used throughout this study. The specimens were initially light cured from the top surface and through the transparent PSE mold for 5 s. After that, the molds were immediately

removed, and the light curing process was continued for 35 s. Finally, a top layer of particulate filler composite was used to cover the base composite and was light-cured for 40 s from the top surface.

The prepared specimens were stored for 48 h in a dry environment at room temperature ($23 \pm 1^\circ\text{C}$) before they were tested.

4.1.2.3 Bi-Layered Fracture Toughness Specimens (IV)

Forty bi-layered single-edge-notched-beam (SENB) specimens were prepared using a custom-made stainless-steel split mold ($2.5 \times 5 \times 25 \text{ mm}^3$) according to ISO 13586:2018 standard method to determine modified notched strength test (MST). The mold comprised of a centrally located pre-fabricated slot that extended in depth to half of its height (x). This allowed the production of bi-layered SENB specimen that had a precisely fabricated notch at the midline of the beams' length and a standardized crack depth of 2.5 mm.

Each bi-layered SENB specimen consisted of a base composite and a conventional capping PFC that incorporated a notch as a pre-fabricated crack (Figure 6). The quality of adhesion between the materials was also altered to test how the presence of the oxygen inhibition layer effected the fracture behavior.

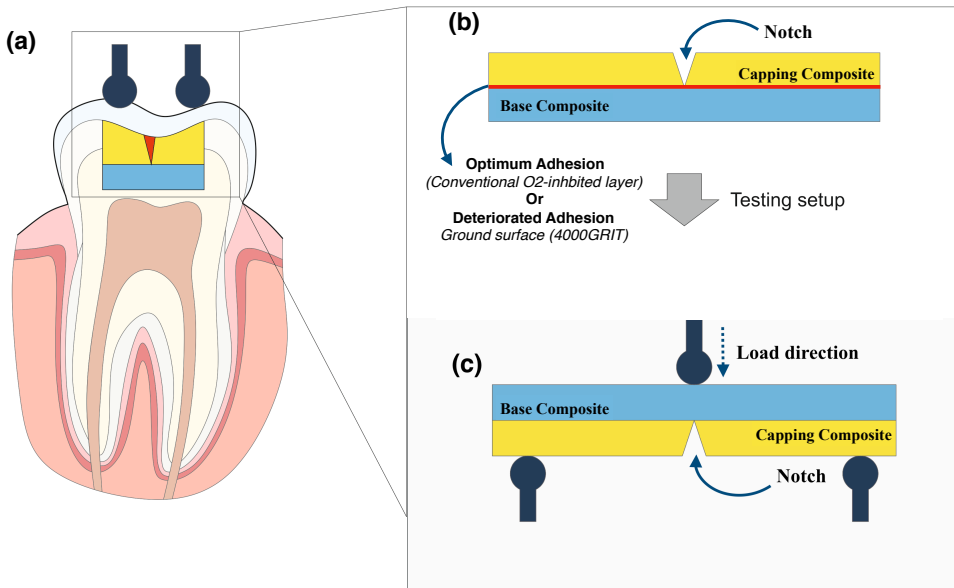


Figure 6 Two-dimensional schematic illustration of: (a) rationale of simplified model and simulation of clinical situation; (b) prepared single-edge-notched-beam (SENB) bi-layered specimen and the different interface designs investigated in this study; (c) 3-point bend loading setup. Adapted from original publication IV.

The custom-made mold was first placed on a Mylar-strip-covered glass slab. Then a base composite was placed to fill half the height of the mold in one increment before light-curing. The polymerization of all composites was performed from the top using five separate overlapping light-cures of 20 s each. An LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) with a calibrated using MARC[®] Resin Calibrator (BlueLight analytics Inc., Halifax, Canada) and the light intensity of 1765.30 mW/cm² (SD±17.91) was used throughout this study.

Materials used in study IV are summarized in Table 1. In each bi-layered SENB specimen the base composite used was either everX Posterior, which is a fiber-reinforced bulk-fill base composite (FRC), or SDR which is a flowable bulk-fill (FBF). While a conventional capping PFC made of Gænial Posterior was used to completely fill the remainder of the mold. Before polymerization of the capping PFC, a straight edged steel blade was inserted into the pre-fabricated slot to produce a sharp and centrally located crack. Then, Mylar-strip-covered glass slabs were pressed firmly on top of the mold's top surface on each side of the blade to ensure even distribution of the capping PFC material around the steel blade before polymerization took place using the aforementioned protocol. The split mold allowed specimens' removal without force, and the custom-made slot allowed the crack tip to be in precise and close proximity to the surface of the base composite.

4.2 Mechanical Tests

4.2.1 De-Bonding Test ("Shear Bond Strength Test") for Adhesion Interface (I,II,IV)

In Study I shear bond strength test was used to check the quality of adhesion between the dentin layer of the prepared teeth specimens and the investigated composites. The prepared specimens were mounted and secured in a mounting jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA) (Figure 7) and finally placed on the shear bond strength (SBS) test assembly.

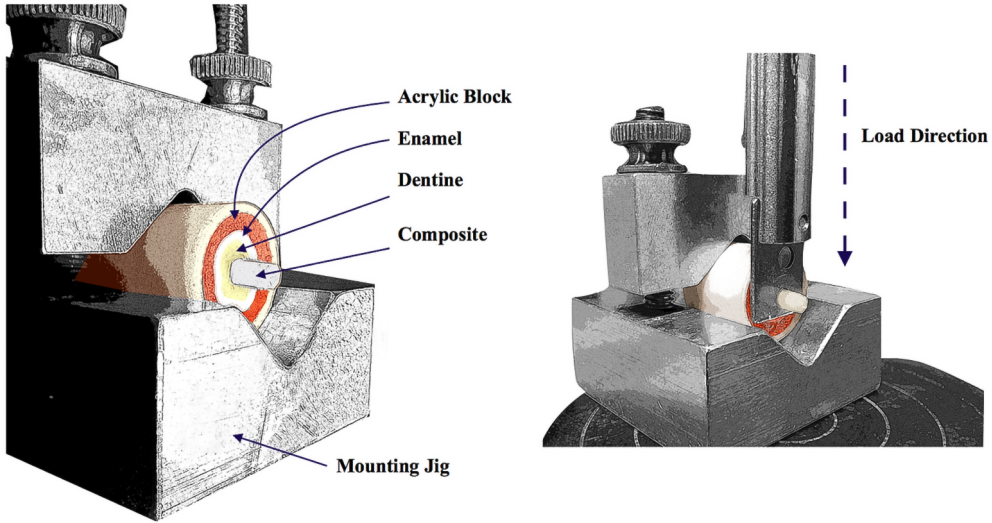


Figure 7 Debonding test (shear bond strength test) assembly. Specimen mounted and secured in a mounting jig. Adapted from original publication I.

The test was performed using a universal testing machine (Model LRX, Lloyd Instruments Ltd., Fareham, England) at room temperature (23 ± 1 °C), and the data was recorded using PC software (Nexygen, Lloyd Instruments Ltd., Fareham, England).

The shearing rod was placed against and parallel to the flat prepared bonding sites. A circular perforation of diameter 4.1 mm was made into the metal blade through which the composites (diameter = 3.6 mm) passed through until the metal blade was positioned at the composite-dentin interface, and then using a span length of 10 mm and crosshead speed of 1.0 mm/min, the specimens were loaded until fracture.

In studies II and IV the prepared bi-layered specimens were subject to two different preparation procedures to simulate different qualities of adhesion between the bulk-base composite and capping PFC layer. The adhesion between the composite layers was based on either: (1) a conventional O_2 -inhibited layer to simulate optimal-adhesion (OA), whereby the subsequent capping PFC layer was immediately placed after light-curing of the base composite layer; or (2) a polished (wet-ground) base composite surface to simulate deteriorated-adhesion (DA). The base composite surface was wet-ground after light-curing using a #4000 grit Federation of European Producers of Abrasives (FEPA) silicon carbide grinding paper. After that, the polished specimens were dried and placed back in a custom-made mold and the capping PFC was placed following the same protocol as the rest of the bi-layered specimens.

As a result, the two adhesion quality testing groups in studies II and IV were tested using a de-bonding test (“shear bonding strength test”) to check the bonding strength of the adhesion protocols, OA and DA (n=15 per material - per adhesion quality group).

A similar test setup to study I was used to determine the different adhesion protocols within the bi-layered specimens (Figure 8). Acrylic blocks were also made using cold cure auto-polymerized acrylic resin (Vertex-Dental B.V., Zeist, Netherlands). Standardized cylindrical cavities (diameter = 7 mm, height = 4 mm) were made into the blocks using a bench drill press machine (DP2000A, Rexon Industrial Corporation, Ltd., Taichung, Taiwan). Each of the base composite materials were then placed in a single bulk increment of 4 mm and light cured once for 40 s from the top aspect. Prior to testing, all specimens were stored for 48 hours in a dry environment at room temperature ($23 \pm 1^\circ\text{C}$).

Finally, a shearing rod was placed against and parallel to the interface between the surface PFC and base composite. Then using a universal testing machine (Model LRX, Lloyd Instruments Ltd., Fareham, England) at room temperature ($23 \pm 1^\circ\text{C}$), all specimens were loaded until failure. The crosshead speed was 1.0 mm/min. Data were recorded using PC software (Nexygen, Lloyd Instruments Ltd., Fareham, England). Bonding strength in studies I,II and IV was calculated by dividing the maximum load at failure (N) with the bonding area (mm^2) and recorded in megapascal (MPa).

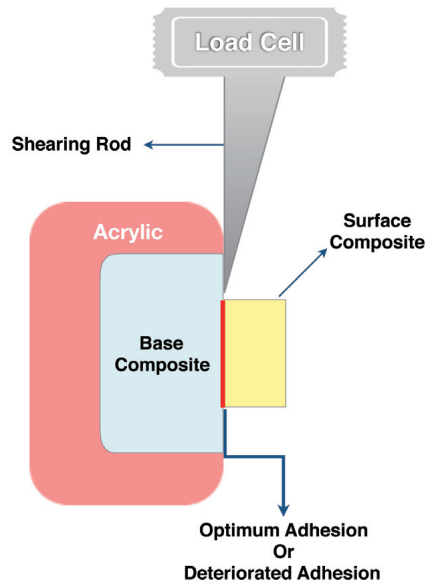


Figure 8 Schematic view of shear bond test setup. Adapted from original publication II.

4.2.2 Static Load Testing (Compression Test) (II, III)

Static loading was performed using a universal testing machine (Model LRX, Lloyd Instruments Ltd., Fareham, England) at room temperature ($23 \pm 1^\circ\text{C}$). Each bi-layered specimen was placed on the testing assembly and axially loaded to fracture using a stainless-steel sphere ($\varnothing 4 \text{ mm}$). The load was applied onto the surface PFC and parallel to the long axis of the specimen. The contact point of the loading sphere to the specimen was at the middle of the specimen and the extension rate was 1.0 mm/min . Loading data was computed using PC software (Nexygen, Lloyd Instruments Ltd., Fareham, England). (Figure 9)

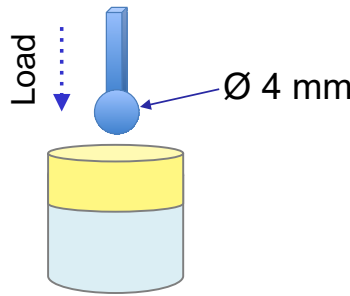


Figure 9 Two-dimensional schematic illustrations of the prepared bi-layered specimen and loading setup. Adapted from original publication II.

4.2.3 Surface Microhardness Test (III)

It is known that atmospheric oxygen forms a layer of unreacted monomer on the surface of freshly light-cured composite materials (Bijelic-Donova et al., 2015). Therefore, surface microhardness [Vickers hardness number (VHN)] measurements were carried out to analyze the interface design fabrication technique and light-curing protocol used in this study and its effect on the surface of the base composites fabricated. It has been reported that surface microhardness has a correlation with the degree of monomer conversion (DC%) (Ferracane, 1985; Le Bell et al., 2003). Therefore, this could affect the physical properties of the specimen.

The surface microhardness measurements (five points for each specimen) were carried out using a universal Vickers device (Struers Duramin, Struers, Ballerup, Denmark), with a load of 245.3 mN being applied for 15 s .

The time elapse between the point of indentation and the measurement of the deformation produced was standardized at 30 s to equate for possible elastic recovery of deformation after the point of indentation (Gayle & Cook, 2016; Yin et

al., 2004). Test indentations should be performed on a flat surface and therefore the flat interface design was chosen for surface microhardness testing.

The length of the diagonal of each indentation was measured directly using a graduated eye-lens. The VHN was obtained using the following equation:

$$H = \frac{1854.4 \times P}{d^2}$$

where H is Vickers hardness (kg/mm²), P is the load (g), and d is the length of the diagonals (μm).

Furthermore, three test protocols were prepared to test the surface microhardness of each of the base composite materials (n = 4 per group) (3.5-mm-thick cylinders with a diameter of 7 mm).

Test protocol 1 – study III light-cure protocol:

The flat polyvinyl siloxane elastomer mold was placed touching the base composite, and was light-cured for 5 s. Then, the polyvinyl siloxane elastomer mold was immediately removed and light curing was continued for 35s. Specimens were tested immediately after light polymerization.

Test protocol 2 – conventional light-cure used in clinics; exposure to atmospheric oxygen:

The base composite was light cured directly for 40 s without using the polyvinyl siloxane elastomer mold. Specimens were tested immediately after light polymerization.

Test protocol 3 – protected from atmospheric oxygen:

A microscope glass slab was placed on the base composite surface, after which the specimens were light polymerized. The glass slab was kept in contact with the base composite for an additional 360 min. Afterwards, the glass slab was removed, and the surface microhardness of the specimens was measured.

4.2.4 Modified Fracture Toughness Test (IV)

The prepared bi-layered specimens (n=40) in study IV were stored for 48 h in a dry environment at room temperature (23 ± 1°C) before they were tested. The bi-layered specimens were tested using 3-point bending set-up and loaded until complete fracture using a universal testing machine (Model LRX, Lloyd Instruments Ltd., Fareham, England) at room temperature (23 ± 1°C). The base composite was facing

the compression side; in contact with the cylindrical loading tip (2 mm diameter). The contact point of the loading tip to the bi-layered specimen was at the middle and parallel to the notch (crack) present on the tension side (Figure 10).

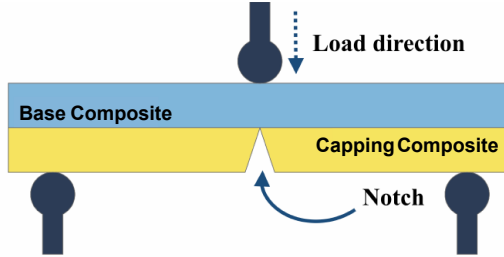


Figure 10 Two-dimensional schematic illustration of prepared single-edge-notched-beam (SENB) bi-layered specimen and 3-point bend loading setup. Adapted from original publication IV.

Loading data were computed using PC software (Nexygen, Lloyd Instruments Ltd., Fareham, England). The fracture toughness was calculated using the equation below:

$$K_{max} = f(x) \left[\frac{PL}{BW^{\frac{3}{2}}} \right] \sqrt{10} - 3$$

where: $f(x) = 3/2x^{1/2}[1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]/2(1+2x) (1-x)^{3/2}$ and $0 < x < 1$ with $x = a/W$. P is the maximum load in newtons (N), L is the span length (20 mm), B is the specimen thickness (mm), W is the specimen width (depth) in mm, x is a geometrical function dependent on a/W and a is the crack length in mm.

4.3 Analyses

4.3.1 Light Cure Irradiation and Transmittance

The specimens were evaluated to determine the irradiance power through the material (i.e., amount of light received by the bottom layer of the specimen) and total irradiant energy (defined as the mathematical product of the curing light irradiance (mW/cm^2) multiplied by the exposure duration in s through each thickness. Similar to the bonding procedure, the same material groups and increment height subgroups ($n = 3$) were used to fabricate new specimen. A LED light-curing unit (Elipar S10, 3M Espe, Seefeld, Germany) with a light intensity of $1765 \text{ mW}/\text{cm}^2$ and curing time 40 s was used. Total energy measured was $70.61 \text{ J}/\text{cm}^2$ ($\text{SD} \pm 0.72$). Light energy transmitted through each specimen was quantified by MARC® Resin Calibrator (BlueLight analytics Inc., Halifax, Canada), whereby each specimen was placed

directly on the surface of the resin calibrator's sensor and then light cured. A specially made jig was used to ensure the stability and proper placement of the light-curing unit. To determine the actual intensity of the light-curing unit, hollowed disks of the same thicknesses were made to compare the intensity of the light before and after specimen placement.

4.3.2 Fracture Analyses

In each of the different studies included in this thesis fracture analysis was conducted by three different examiners. This group evaluation was performed to objectively evaluate fracture patterns according to the defined criteria of each study.

In study I the failures were categorized into the following four categories: cohesive in composite failure, cohesive in dentin failure, adhesive failure, and mixed failure (involving any two aforementioned failure types).

In study II, the fracture patterns were categorized into four categories (Figure 12) (i) complete bulk fracture, (ii) partial bulk fracture, (iii) delamination without bulk fracture and (iv) delamination with bulk fracture.

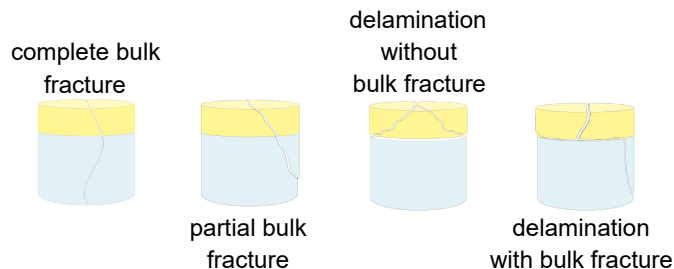


Figure 11 Schematic drawing showing bi-layered specimen and all types of reported fracture patterns in study II. Adapted from original publication II.

While in study III, the fracture patterns were categorized into four categories; (i) *Catastrophic fracture* being the most unfavorable type as the entire bi-layered specimen completely shattered in several pieces. (ii) *Complete bulk fracture* was when the fracture went through the bulk material and split the specimen in half, rendering a poor outcome for repair. (iii) *Partial bulk fracture* presented a more favorable fracture. This is because the fracture lines ran more superficially and away from the midline of the specimen, which preserved most of the bulk of the remaining material (Figure 12). Lastly, (iv) *adhesive fracture* was identified when the fracture line advanced along the interface without involvement of the base composite in the bi-layered structures.

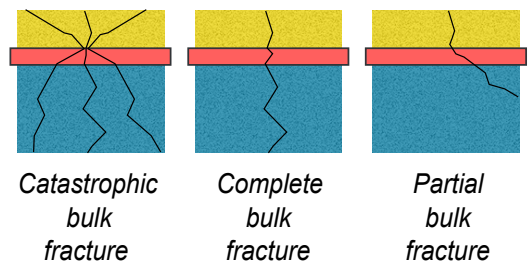


Figure 12 Schematic drawing showing bi-layered specimen and all types of reported fracture patterns in study III. Adapted from original publication III.

Finally, in study IV, the fractures were categorized into four types according to the bi-layered specimen's fracture position from the midline (Figure 13). *Type 1* fracture position at midline of specimen whereby the fracture went through and through the base composite. *Type 2* fracture position is deflected from midline, whereby the crack front advances up to 1 mm along the interface between the base composite and capping PFC materials before going through the base composite. *Type 3* demonstrates fracture position that has advanced more than 1 mm away from the midline and along the interface between the two materials. *Type 4* demonstrates fracture line that advanced away from the midline and along the entire length of the interface, causing a complete de-bonding between layers in the bi-layered specimen.

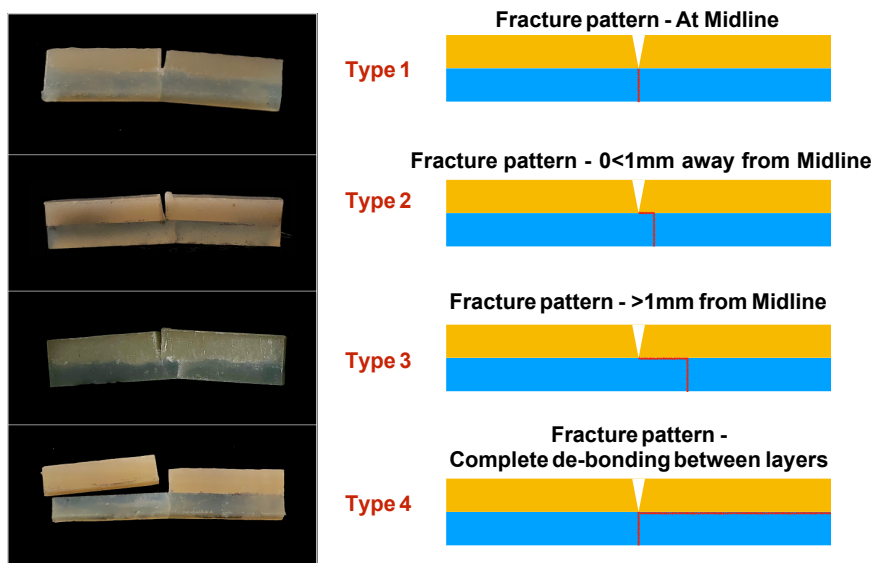


Figure 13 Showing pictures of how SENB bi-layered specimen fractured and a 2-D schematic representation of the extent of fracture. Fracture types represent the different fracture patterns through the bi-layered specimen. Adapted from original publication IV.

4.3.3 Finite Element Analysis (II)

Three-dimensional (3D) finite element (FE) models were constructed based on the specimen design. Based on the specimen in study II, the same three top surface thicknesses were prepared; 1.0 mm, 1.5 mm and 2.0 mm using same base materials, FRC and FBF. The total height of bi-layered specimen was also standardized at 5.0 mm. The materials were assumed to be isotropic, homogeneous, and linear elastic.

All solid models were derived from a single mapping mesh pattern that generated 2564 twenty-node brick elements (Solid 95 in ANSYS) and 4050 nodes. The force was applied to the center of top surface and load values were constant 600 N. The central point of the bottom surface was fixed in all directions. 3D FE analysis was presumed to be linear static and was performed on a personal work station (Z820 Work Station, Hewlett-Packard, Palo Alto, California, USA) using FE analysis software ANSYS 15 (ANSYS Inc.; Houston, TX, USA). The locations and magnitudes of stress distribution (MPa) were identified and used for evaluating the mechanical behavior.

4.3.4 3D Optical Profiling (III)

The four interface designs that were produced on the surface of base composites in study III were profiled with a 3D optical microscope (Bruker Nano, Berlin, Germany) using Vision64 software. The maximum interface design depth values (μm), representing the average of lowest or deepest points of all profile scans were calculated from different points.

4.4 Statistical Analysis

SPSS software (Statistical Package for Social Science, SPSS Inc., Chicago, IL) was used to perform all statistical analysis in each of the studies included in this thesis book.

In study I the means (standard deviations) were calculated for both, the SBS and light cure irradiation and transmittance values. Statistically significant differences between the acquired data were determined by using two-way analysis of variance (ANOVA) for the two factors: thicknesses and material, followed by Tukey's post hoc analysis at the $p < 0.05$ significance level. The differences in failure modes were analyzed by using the chi-square test at a significance level of 5 %.

In study II, statistically significant differences were analyzed using three-way analysis of variance (ANOVA) followed by Tukey's post hoc analysis at the ($p < 0.05$) significance level.

Pearson's correlation was utilized to identify the significance of trends between the specimen groups investigated. Chi-square test at a significance level of 5% was

used to analyze the differences between fracture types and their correlation to the different bi-layered configurations.

In study III, the maximum load values in the compression test were analyzed using two-way analysis of variance (ANOVA) followed by Tukey's post hoc analysis at the ($p < 0.05$) significance level.

In study IV, maximum load values in the fracture toughness test were analyzed using two-way analysis of variance (ANOVA) followed by Tukey's post hoc analysis at the ($p < 0.05$) significance level.

5 Results

5.1 Mechanical Tests

5.1.1 De-Bonding Test (“Shear Bond Strength Test”) for Adhesion Interface (I,II,IV)

The de-bonding test results from study I, acquired at different increment thicknesses, are summarized in Table 2. A comparative illustration of SBS mean values is presented in below in Figure 14.

Table 2 Shear bond strength test values of the investigated composites in relation to different thicknesses (2 vs 4 vs 6 mm)

Material (Shade)	Thickness of composite increment		
	2 mm	4 mm	6 mm
G-ænial Anterior (A3)	18.7 (3.8) ^{a;A}	17.6 (3.4) ^{a;A}	15.6 (3.7) ^{a;A}
EverX Posterior™	24.3 (6) ^{a;A}	20.0 (4.6) ^{a,b;A}	17.3 (5.2) ^{b;A}
Tetric EvoCeram® Bulk FILL (IVA)	20.6 (5.9) ^{a;A}	18.4 (5.3) ^{a;A}	17.4 (4.4) ^{a;A}
SDR™	22.7 (5.5) ^{a;A}	18.9 (3.6) ^{a,b;A}	18.1 (4.1) ^{b;A}

Values are presented as mean (\pm SD) in MPa (n=15).

Tukey's post hoc analysis is presented as superscript letters.

The same lower-case superscript letter in a row represents non-statistically significant differences ($p>0.05$). The same upper-case superscript letter in a column represents non-statistically significant differences ($p>0.05$). Adapted from original publication I.

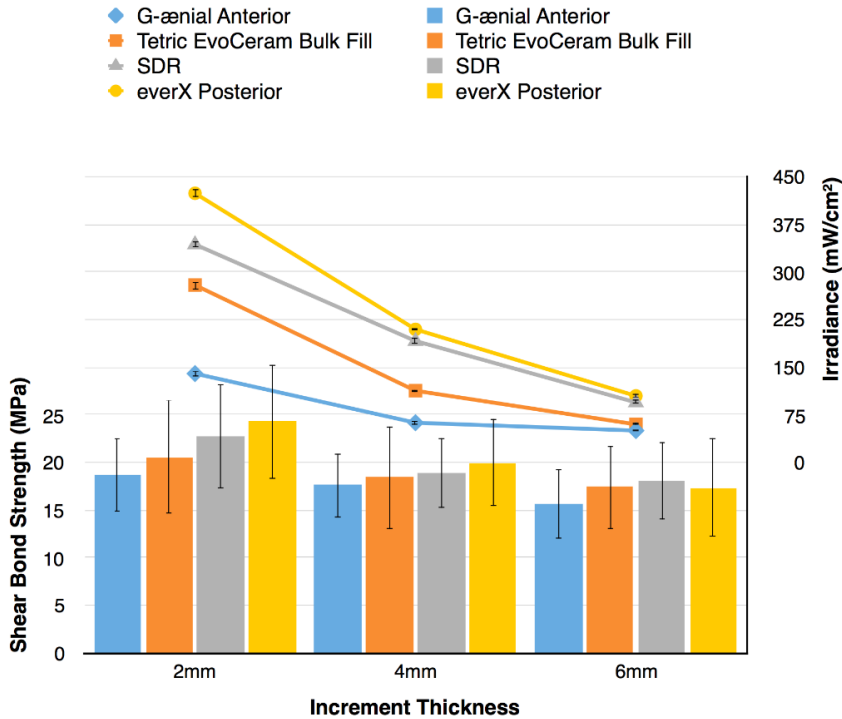


Figure 14 Shear bond strength (MPa) and Light Irradiance (mW/cm^2) of the investigated composites in relation to different thicknesses (2 vs 4 vs 6 mm). Data are presented as mean \pm standard deviation (SD). Adapted from original publication I.

Two-way ANOVA revealed statistically significant differences in SBS values acquired in both factors; thicknesses ($p < 0.05$) and material ($p < 0.05$). On Tukey's post-hoc analysis of each group, a significant ($p < 0.05$) difference in SBS values was present only between thicknesses 2mm and 6mm within everX Posterior (24.3 and 17.3 MPa) and SDR (22.7 and 18.9 MPa). In addition, there was no significant ($p > 0.05$) difference in SBS values between the different groups at all thicknesses. Furthermore, Pearson correlation analysis showed a significant negative correlation ($p < 0.05$) between thickness and SBS values in all groups except Tetric EvoCeram Bulk Fill.

In studies II & IV the bond strength test results collected from testing the different quality of adhesions are shown in Figure 15. Statistically significant differences in bond strength values ($p < 0.05$) were found between OA and DA in both FBF (14.7 and 6.1 MPa) and FRC (18.8 and 7.0 MPa).

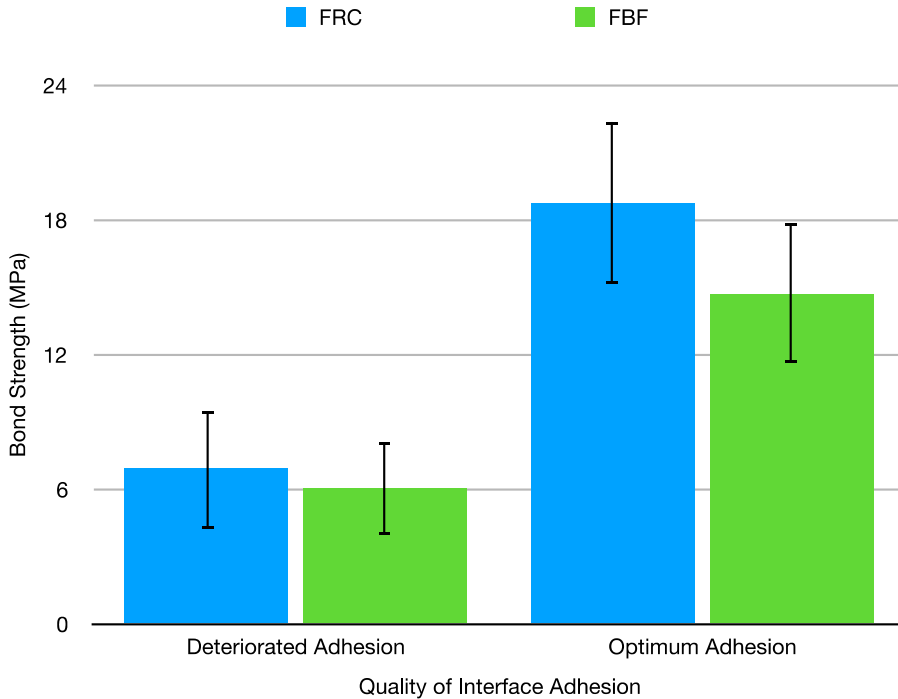


Figure 15 Bond strength test values (MPa \pm SD) between the surface and base composite layers at different qualities of adhesion. Adapted from original publication II.

5.1.2 Static Load Testing (Compression Test) (II, III)

The static load test results of study II, at different thicknesses and quality of adhesion between the surface PFC and base composites, are summarized in Figure 16. Tukey's post hoc test results are shown in Table 3.

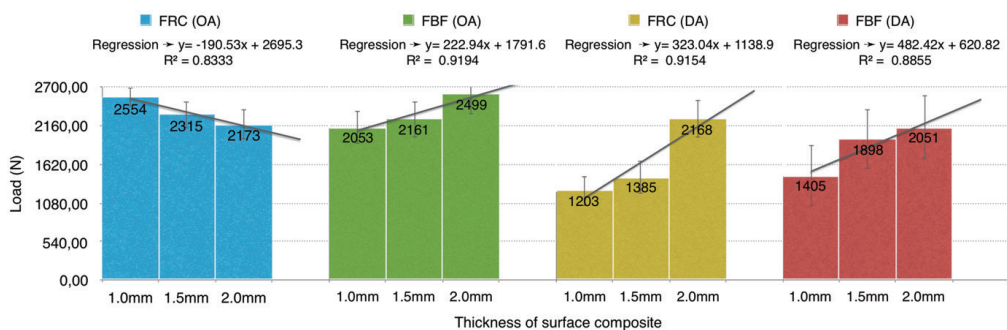


Figure 16 Load-bearing capacities (N \pm SD) of tested specimens at different thicknesses and quality of adhesion between the surface PFC and base composites. Adapted from original publication II.

Table 3 Static load values (N) of the investigated specimens with different adhesion interfaces in relation to different thicknesses (1.0 vs 1.5 vs 2.0 mm). Adapted from original publication II.

Adhesion interface	Base material		Surface-layer thickness	
		1.0 mm	1.5 mm	2.0 mm
Optimal	FBF	2052(±222) ^{Aa}	2161(±240) ^{Aab}	2499(±262) ^{Ba}
	FRC	2554(±130) ^{Ab}	2315 (±386) ^{ABa}	2173(±210) ^{Bab}
Deteriorated	FBF	1405(±403) ^{Ac}	1898(±404) ^{Bb}	2051(±431) ^{Bb}
	FRC	1203(±204) ^{Ac}	1385(±215) ^{Ac}	2168(±258) ^{Bab}

Tukey's post hoc analysis presented as letters.

Same superscript upper-case letter in a row represents non-statistically significant differences ($p>0.05$). same superscript lower-case letter in a column represents non-statistically significant differences ($p>0.05$).

In general, poor adhesion interface had a negative effect when using 1 mm thin surface PFC, but the effect of deteriorated adhesion decreased as the surface PFC thickness was increased to 2 mm.

Three-way ANOVA revealed statistically significant differences in static loading values acquired in factors; thickness ($p<0.05$), quality of adhesion ($p<0.05$), while the material type did not show a statistically significant difference ($p>0.05$). However, the fracture pattern between different base materials was significantly different ($p<0.05$).

Pearson correlation analysis showed a statistically significant negative correlation ($p<0.05$) between load and thickness, when using the FRC base material with optimal adhesion which can be seen in Figure 16. In contrast, all the other groups tested in this study demonstrated a statistically significant positive correlation ($p<0.05$), i.e. the increase of surface PFC thickness caused an increase in load bearing capacity. Typical load deflection graphs for all the tested groups are shown in Figure 17. In OA, FRC groups showed an initial failure point which was then followed by the final fracture at a higher load value. In contrast, FBF groups showed uninterrupted load-deflection curves before the fracture. In DA, the FRC and FBF groups followed the same trend, i.e. only specimens with 1 mm thick PFC layer showed an initial failure point.

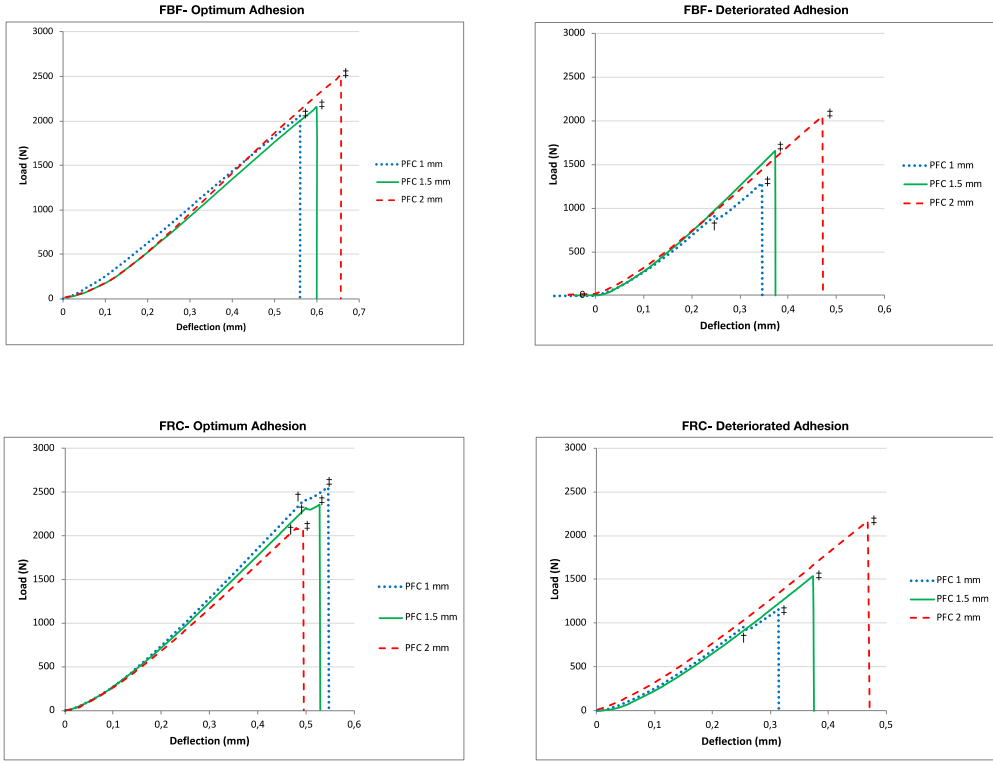


Figure 17 Load-deflection curves of tested specimens in study II showing the initial failure points [†] and final fracture points during loading [‡]. Adapted from original publication II.

In study III, two-way ANOVA revealed that the material and interface design had a significant effect on the load bearing capacity ($p < 0.05$). The mean loads values acquired at maximum compression are presented in Figure 18. The mean load values for flat interface designs is higher than that other interface designs for both base materials.

The FBF groups showed that a change in interface design significantly affected the loads acquired ($p < 0.05$). While in FRC, only mesh interface design showed statistically different load values than the other interface designs investigated ($p < 0.05$).

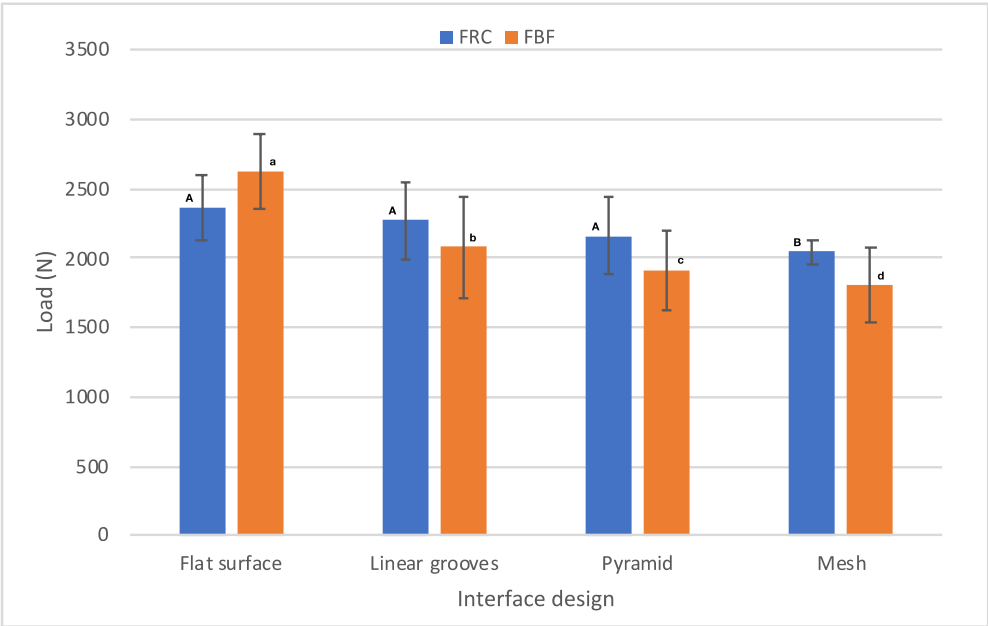


Figure 18 Load-bearing capacities (N \pm SD) of tested interface designs between the surface PFC and base composites. *Tukey's post hoc analysis between material and interface-design presented as letters. Same superscript upper-case letter represents non-statistically significant differences ($p>0.05$) in FRC groups. Same superscript lower-case letter in a column represents non- statistically significant differences ($p>0.05$) in FBF groups. Adapted from original publication III.

5.1.3 Surface Microhardness Test (III)

In study III, test Protocol 3 was the only protocol that yielded measurable indentations. FRC (VHN 61.8 ± 2.5) showed higher hardness values when compared to FBF (VHN 30.9 ± 1.7), while protocols 1 and 2 showed no visible indentations to be recorded.

5.1.4 Modified Fracture Toughness Test (IV)

Figure 19 summarizes the 3-point bending load and MST values of tested bi-layered specimens with different base composite and quality of adhesion between the capping PFC and base composites.

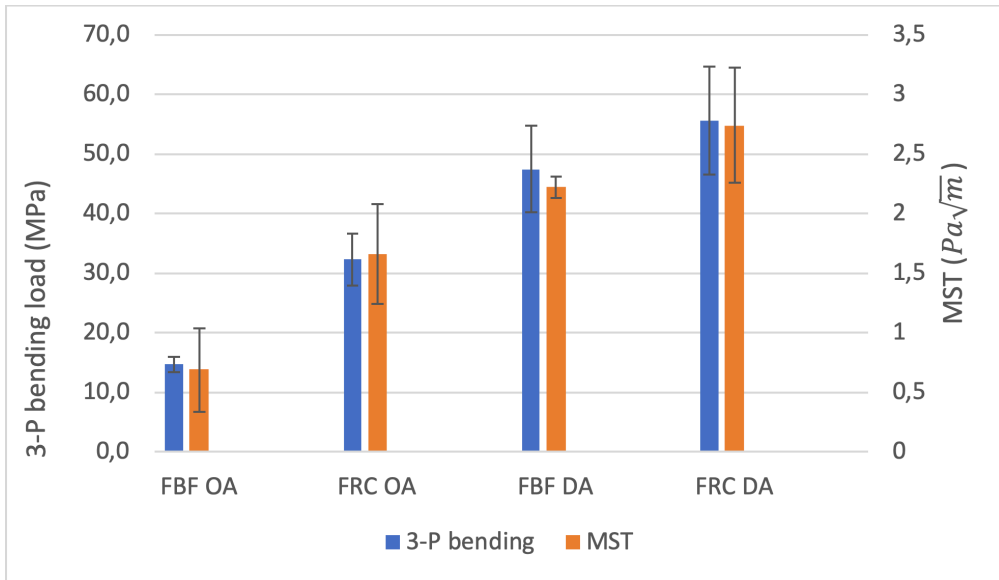


Figure 19 3-point bend load capacities (MPa \pm SD) & modified notched strength test (MST) ($Pa\sqrt{m} \pm SD$) values of tested bi-layered specimens at different quality of adhesion between the capping PFC and base composites. Adapted from original publication IV.

Tukey's post hoc test results are shown in Table 4. In general, bi-layered specimens with deteriorated adhesion interface had higher load and MST values than optimal adhesion specimen and this difference was statistically significant (ANOVA, $p < 0.05$).

Table 4 3-point bending load (N \pm SD) and modified notched strength test (MST) values of the investigated bi-layered specimens with different adhesion interfaces

Adhesion interface	Base material	Test values	
	3-P Bending	MST	
Optimal	FBF	14.7 \pm 1.2 ^a	0.69 \pm 0.09 ^a
	FRC	32.3 \pm 4.4 ^b	1.66 \pm 0.35 ^b
Deteriorated	FBF	47.4 \pm 7.2 ^c	2.22 \pm 0.48 ^c
	FRC	55.6 \pm 9.1 ^d	2.74 \pm 0.42 ^d

Tukey's post hoc analysis presented as letters.

Same superscript letter in a column represents non-statistically significant differences ($p > 0.05$).

Adapted from original publication IV.

FRC groups showed higher values of loads and MST than FBF specimens and this difference was statistically significant (ANOVA, $p < 0.05$). Typical load-deflection curves are shown in Figure 20.

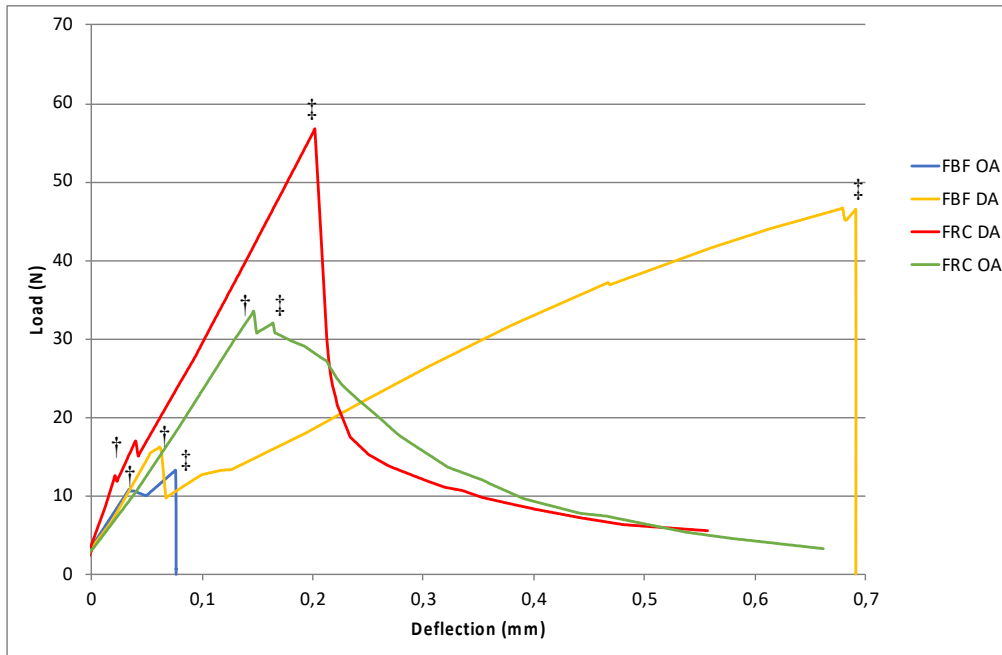


Figure 20 Showing the load (N) and deflection graphs of the different groups that were tested in study IV. In addition, it presents the primary failure points † and final fracture points ‡ during loading ‡. Adapted from original publication IV.

5.2 Analyses

5.2.1 Light Cure Irradiation and Transmittance (I)

The light irradiance test results, at different increment heights, are summarized in Table 5. A comparative illustration of light irradiance mean values is presented in Figure 15. Two-way ANOVA revealed statistically significant differences in light irradiance values acquired in both factors; thicknesses ($p < 0.05$) and material ($p < 0.05$). Tukey's post-hoc analysis revealed that increment height had a significant ($p < 0.05$) effect on light irradiance values within the different materials. Similarly, the investigated groups showed statistically ($p < 0.05$) different light irradiance values when compared to one another. Pearson correlation analysis showed a significant negative correlation ($p < 0.05$) between thickness and light irradiance values in all groups.

Table 5 Light irradiance test values of the investigated composites in relation to different thicknesses (2 vs 4 vs 6mm).

Material (Shade)	Thickness of composite increment		
	2mm	4mm	6mm
G-ænial Anterior (A3)	140.1 (3.2) ^{a:A}	62.7 (2.8) ^{b:A}	50.3 (0.6) ^{c:A}
EverX Posterior™	424.3 (6.2) ^{a:B}	209.8 (1.7) ^{b:B}	104.9 (1.8) ^{c:B}
Tetric EvoCeram® Bulk Fill (IVA)	280.0 (6.1) ^{a:C}	113.1 (1.3) ^{b:C}	60.1 (2.8) ^{c:C}
SDR™	343.7 (4.7) ^{a:D}	191.5 (4.8) ^{b:D}	94.7 (3.1) ^{c:D}

Values are presented as mean (\pm SD) in mW/cm² (n=3).

Tukey's post hoc analysis is presented as superscript letters.

The same lower-case superscript letter in a row represents non-statistically significant differences ($p>0.05$). The same upper-case superscript letter in a column represents non-statistically significant differences ($p>0.05$). Adapted from original publication I.

The total energy values (J/cm²) obtained from the investigated groups at 2mm, 4mm and 6mm were: G-ænial Anterior (5.60, 2.51 and 2.01), Tetric EvoCeram Bulk Fill (11.18, 4.52 and 2.40), SDR (13.74, 7.66 and 3.79) and everX Posterior (16.97, 8.39 and 4.20).

Generally everX Posterior and SDR showed the highest mean irradiance, however, everX Posterior had the highest mean irradiance values for both 2mm and 4mm in comparison to all the other tested groups.

5.2.2 Fracture Analyses (I,II, III, IV)

Fracture and failure analysis of the specimen after bonding test performed in study I are shown in Table 6.

Table 6 Results of failure mode assessment for all composite groups.

Group	Material (shade)	Thickness of composite increment	Percentage of different types of failure (%)			
			Adhesive	Cohesive in Composite	Cohesive in Dentin	Mixed
A	G-ænial Anterior (A3)	2mm	0	7	40	53
		4mm	7	7	66	20
		6mm	27	20	53	0
B	EverX Posterior™	2mm	7	33	27	33
		4mm	20	0	53	27
		6mm	13	7	53	27
C	Tetric EvoCeram® Bulk Fill (IVA)	2mm	13	7	40	40
		4mm	33	13	47	7
		6mm	19	7	47	27
D	SDR™	2mm	7	13	53	27
		4mm	20	0	60	20
		6mm	13	0	47	40

*Pearson's chi-square test revealed that increment thickness had a significant ($p<0.05$) association with failure type in only G-ænial Anterior. Adapted from original publication I.

The dominant mode of failure is cohesive failure in dentin followed by mixed failure. Adhesive failures were least at 2mm for all composite groups. Tetric EvoCeram Bulk Fill showed highest percentage of adhesive failure in both 2mm (13%) and 4mm increments (33%); while G-ænial Anterior showed the most adhesive failure at 6mm (27%). Both SDR and everX Posterior showed identical incidence of adhesive failure in all height increments (7% at 2mm, 20% at 4mm and 13% at 6mm). Pearson's chi-square test revealed that increment thickness had a significant ($p<0.05$) association with failure type in only G-ænial Anterior. In contrast, everX Posterior, Tetric EvoCeram Bulk Fill and SDR failure modes followed an unpredictable manner and thus chi-square test revealed no significant ($p>0.05$) association between failure type and increment thickness.

In study II, Chi square analysis showed that the base material itself had a significant effect on failure type ($p<0.05$). Failure modes of the specimen after static loading test are shown in Table 7.

Table 7 Results of failure pattern assessment for all composite groups investigated in study II

Adhesion interface	Base material	Thickness of surface composite	Percentage of different types of failure (%)			
			Complete Bulk Fracture	Partial Bulk Fracture	Delaminating Fracture	Delaminating with Partial Bulk Fracture
Optimum	FBF	1.0 mm	100	-	-	-
		1.5 mm	100	-	-	-
		2.0 mm	100	-	-	-
	FRC	1.0 mm	70	30	-	-
		1.5 mm	50	50	-	-
		2.0 mm	30	70	-	-
Deteriorated	FBF	1.0 mm	-	-	100	-
		1.5 mm	-	-	90	10
		2.0 mm	20	-	50	30
	FRC	1.0 mm	-	-	100	-
		1.5 mm	-	-	100	-
		2.0 mm	10	-	30	60

Adapted from original publication II.

For FBF groups: during OA, the type of failure was exclusively complete bulk fracture (100%), while during DA, the type of failure was predominantly following a delaminating manner, i.e. causing fracture only for the surface PFC. However, as the surface PFC thickness increased (1.5 mm & 2.0 mm) there was an increase incidence of base material partial fracture (10% & 30%, respectively). For FRC groups: during OA, the type of failure was mixed with some incidences of partial bulk fracture. As the surface PFC increment thickness increased (1.0 mm, 1.5 mm & 2.0 mm) there was an increase in the prevalence of partial bulk fracture (30%, 50% & 70% respectively), while during DA, the type of failure predominantly followed a delaminating manner, i.e. causing fracture only for the occlusal material. However, at surface PFC thickness 2.0 mm there was an increased incidence of base material partial fracture (60%). Pearson's chi-square test revealed that the failure type of FRC and FBF in the deteriorated group was changed by incremental thickness ($p < 0.05$).

Fracture types obtained from study III are summarized in Table 8 and shown in Figure 21. Adhesive and partial bulk fractures were nonexistent in the FBF groups. The two modes of fracture in the FBF groups were either catastrophic or complete bulk fractures. The interface design influenced the percentage of catastrophic and complete bulk fracture. The descending order of catastrophic fracture percentages with relation to interface design was as follows: Flat surface>Linear grooves>Mesh>Pyramid (85%, 54%, 50% and 40% respectively).

Adhesive and catastrophic fracture was nonexistent in the FRC groups. The two modes of fracture in the FRC groups were either partial or complete bulk fractures. The interface design influenced the percentage of complete and partial bulk fractures. The ascending order of partial bulk fracture percentages with relation to interface design was as follows: Flat surface>Lines>Mesh>Pyramid (64%, 70%, 80% and 100% respectively).

Table 8 Results of fracture pattern assessment for all bi-layered groups investigated in study III

Interface design	Base material	Percentage of different types of failure (%)			
		Catastrophic Bulk Fracture	Complete Bulk Fracture	Partial Bulk Fracture	Adhesive Fracture
Flat Surface	FBF	84.6	15.4	-	-
	FRC	-	36.4	63.6	-
Linear grooves	FBF	54.5	45.5	-	-
	FRC	-	30.0	70.0	-
Mesh	FBF	50.0	50.0	-	-
	FRC	-	20.0	80.0	-
Pyramid	FBF	40.0	60.0	-	-
	FRC	-	0.0	100.0	-

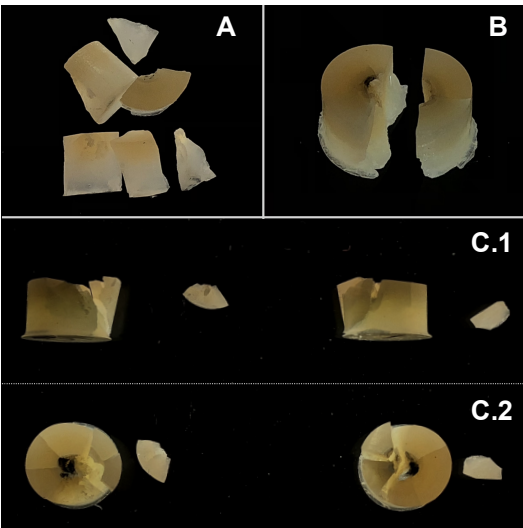


Figure 21 Showing sample photos of example fracture types - A: catastrophic bulk fracture, B: complete bulk failure, C.1: partial bulk fracture - side view & C.2: partial bulk fracture - top view. Adapted from original publication III.

In study IV, fracture analysis results and types of fracture patterns of the bi-layered specimen are shown in Figure 22. For FBF groups, during OA, the fracture was exclusively Type 1 fracture pattern (100%), while during DA, the fracture pattern was predominantly Type 4 and caused delamination of the surface PFC (50%). However, incidences of Type 1, 2 & 3 were also present among the same test group (20%, 20% & 10% respectively).

For FRC groups, during OA, the fracture pattern was predominantly Type 1 (50%) with some incidences of Type 2 and 3 (30% & 20%) and no incidence of Type 4. While during DA, the fracture pattern Types 1, 2, 3 & 4 had similar incidences.

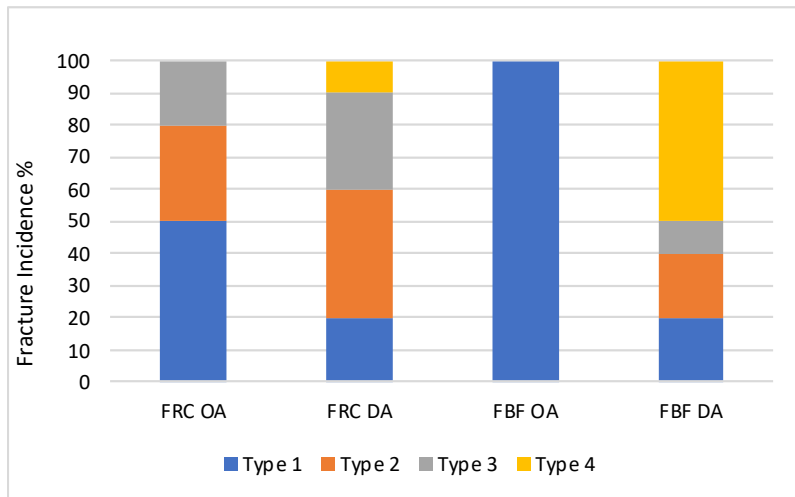


Figure 22 Showing the incidence of each grade of fracture within each of the test groups in study IV. Adapted from original publication IV.

5.2.3 Finite Element Analysis (II)

For all specimen models, differences in strain and stress distributions were observed using FE modeling. The highest stress was located under the loading point for all models. Strain distribution Figure 23 showed FBF (a & b) having a high strain area that reaches the interface when using 1.0 mm and 1.5 mm surface PFC thickness. However, at 2 mm surface PFC the high strain area was confined to the top surface layer away from the interface (c). FRC groups showed less strain distribution than FBF.

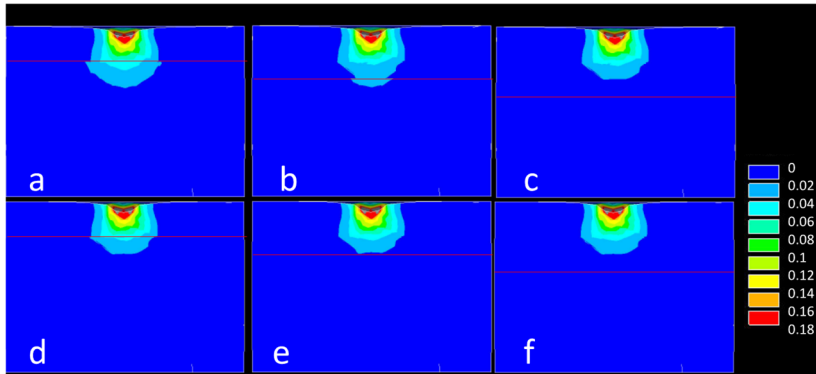


Figure 23 Finite element analysis of tested specimens showing the strain distribution in vertical cross section. a: FBF+1.0 mm PFC, b: FBF+1.5 mm PFC, c: FBF+2.0 mm PFC, d: FRC+1.0 mm PFC, e: FRC+1.5 mm PFC, f: FRC+2.0 mm PFC. Horizontal red line represents interface between surface PFC and base material. Adapted from original publication II.

Stress distributions Figure 24 revealed similar stress distribution irrespective of material and surface PFC thickness. High stress area is present at the interface when using 1 mm surface PFC. At 1.5 mm surface PFC, the high stress area was in close proximity to the interface line. At 2 mm surface PFC, high stress areas did not reach the interface line.

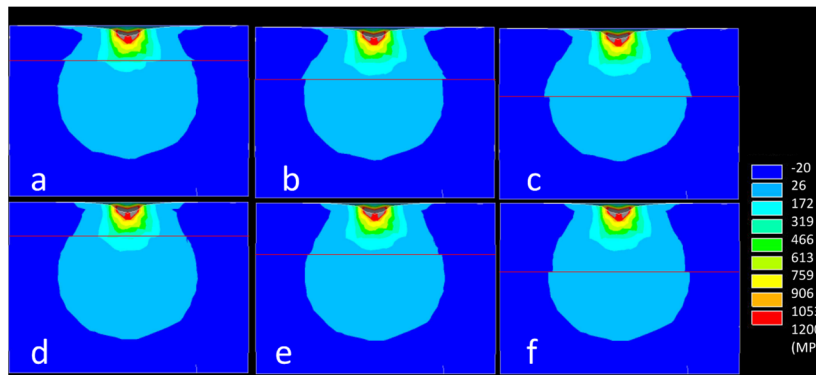


Figure 24 Finite element analysis of tested specimens showing the stress distribution in vertical cross section. a: FBF+1.0 mm PFC, b: FBF+1.5 mm PFC, c: FBF+2.0 mm PFC, d: FRC+1.0 mm PFC, e: FRC+1.5 mm PFC, f: FRC+2.0 mm PFC. Horizontal red line represents interface between surface PFC and base material. Adapted from original publication II.

5.2.4 3D Optical Profiling (III)

Optical profiling of the interface designs used in study iii and investigation of interface fabrication is shown in figure 25.

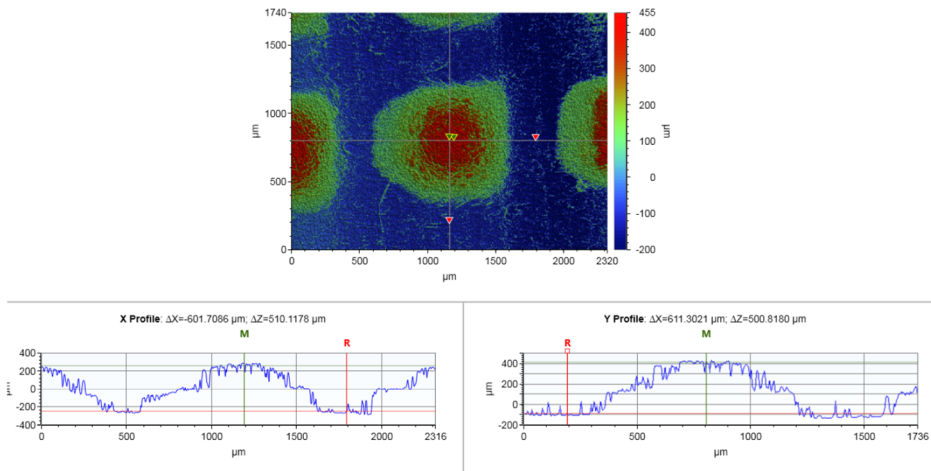


Figure 25 Showing 3D profiling of mesh interface design. The ΔZ (depth) values calculated from two reference points in X and Y profiles. Adapted from original publication III.

6 Discussion

6.1 General Discussion

This series of in vitro studies aimed at investigating various mechanical properties of resin composite materials that are intended to be placed in: (i) bulk increments (ii) bi-layered configuration & (iii) regions of high masticatory load. Through each study, the investigated composite materials were tested using different protocols. The results of evaluating the different protocols allows us to identify the best parameters for the composite materials to optimally perform mechanically.

The focus on clinical significance was a recurring consideration in the methodology design in each of the studies, included in this thesis. As in the first study, the thicknesses of the bulk increments were selected to fit the clinical scenario of utilizing bulk-fill resin composites currently on the market which are intended to be placed in increments of 4mm, in deep posterior cavities. Such increments raise doubts to the actual efficacy of light curing in the deepest layer in addition to undermining the integrity of the bond strength between the material and the tooth structure (Flury et al., 2014). Therefore, we investigated both the materials' bond strength to dentin as well as the light irradiance values at different material thicknesses of 2, 4 and 6 mm to determine the effectiveness of the bulk application procedure for bulk-fill and fiber-reinforced composites that were utilized in this thesis study.

These trending bulk-fill and novel short fiber-reinforced materials are often used in clinical situations as base composite materials that are covered occlusally with a capping layer of conventional particulate filler composite material. The capping layer provides appropriate wear resistance against masticatory function (Engelhardt et al., 2016; Garoushi et al., 2017; Tomaszewska et al., 2015). However, the fact that these restorative materials undergo a combination procedure to form a bi-layered restoration made from two different materials inevitably warranted a systematic approach from different angles to identify possible areas of optimization and improvement. Hence, in study II we aimed to study the currently trending bi-layered composites in relation to the following factors: (i) adhesion between the capping and base composite-layers, (ii) changing the thicknesses of the base and capping composite materials, and (iii) the difference in Young modulus between composites.

In study III, the concept of fabricating an interface design was inspired by the DEJ, which is known to be scalloped in nature (Ten Cate, 1998). Several studies have looked closely at this natural interface and closely investigated its structure and behavior (Brauer et al., 2010; Sui et al., 2016). According to Brauer et al., when investigating the DEJ scallop size, they found that the DEJ is indeed complex. Their results showed that posterior teeth subject to higher masticatory loads tend to have larger and more pronounced scallops than anterior teeth. It has been suggested that this gradient-like interface improves mechanical performance.

However, an actual biomimetic model for bi-layered restorations is lacking. Therefore, study III investigated a novel approach of implementing an interface design that can be fabricated chair-side in the dental office using 3D printing technology. The results suggest that the fracture behavior of bi-layered composite structures could be positively influenced by the interface design between the base and capping composite layers.

Another important aspect of optimizing bi-layered composite structures is fracture propagation and fracture resistance. This aspect was evaluated in study IV by using a simplified model of a clinical situation where the fissure of a tooth would be simulated by fabricating a notch in the test specimens. The loading moment simulated simultaneous loading of buccal and lingual cusps of the restored tooth. Limitation of the test set-up is the supporting points of the specimens versus the clinical condition where the entire restoration is supported by the dentin. Nonetheless, it was assumed in the study design that the elasticity and resiliency of dentin (which mostly occurred at the areas of supporting points) allowed the notch area of the specimens to have tensile stress similar to the modified bending test used in study IV. Additionally, the simplified model relates to the notch of the specimens, which was intentionally extended to the interface area of two resin composite layers. This allowed us to further investigate the behavior of the progressing crack front as it came in contact with the composite-composite interface between two different materials.

6.2 Increment Thickness, Dentin Bond Strength and Light Irradiance (I)

One of the two layers in the bi-layered restorations is a base composite material that is often intended to be placed in bulk to facilitate ease of application and to save time during application. This raises the concern of how effective light curing will be at the most bottom layer when cured in bulk increments of 4 mm (Flury et al., 2014). Hence, study I evaluated the quality of light curing at the deepest layers through the measurement of dentin bond strength between the composites and the human dentin tissue. Also, the amount of light irradiance reaching through the depth of the

composite was measured to further identify parameters that could help in predictive analysis that can help optimize the bi-layered approach when restoring deep cavities such as endodontically treated teeth.

Commonly used flowable and packable bulk-fill composites (SDR and Tetric EvoCeram Bulk Fill) were compared with a novel short fiber-reinforced composite (everX Posterior), and a regular resin composite (G-aenial Anterior). The relatively high dentin bond strength values acquired using short fiber-reinforced material (everX posterior) at 4 mm increments illustrate its ability to be placed in bulk increments similar to other bulk-fill composite materials and therefore, make it eligible to be used in the bi-layered framework of the following studies contained in this thesis book.

Furthermore, the sharp decrease of dentin bond strength between increment thicknesses of 2mm and 6mm in everX Posterior and SDR, emphasizes the importance of strictly adhering to 4mm increment thicknesses to avoid significant drops in bond strength and potentially affecting dentin bond strength. Furthermore, it is noteworthy that everX Posterior yielded the highest mean bond strength values in both 2mm and 4mm increments. This could be due to, the potential presence of a micro-mechanical interlocking between the protruding short fibers of everX Posterior and dentin. This may have influenced the bond strength values, especially where shear stress is concerned. However, such protruding fibers are expected to have less influence on tensile stress components that clearly exist within the present study setup.

Moreover, light variance and irradiance was tested, to investigate the irradiance power through the materials and measure how much of it actually reaches the deepest layer as the bulk thickness increases. The data showed that the total energy for particulate filler composite, G-aenial Anterior (control) was significantly ($p < 0.05$) lower than the other investigated composites. Given the nature of the material, PFCs display a lower translucency than BFCs (Bucuta et al., 2014). As light transmission is strongly linked to material opacity (Shortall, 2005) the comparatively lower total energy values might be a result of the increased opacity of PFCs. These observations are in coherence with a previous study, where it was shown that the inherent translucency of PFCs induced a significant reduction in comparative light transmission values when increment thickness was increased (Garoushi et al., 2015). Furthermore, a decrease in translucency has been reported with increase in filler content (Lee, 2008). This was evident from the packable bulk-fill Tetric EvoCeram Bulk Fill showed the lowest irradiance values due to its higher filler content (Table 1). However, Tetric EvoCeram Bulk Fill was still able to exhibit appropriate bond strength values in bulk due to a supplementary initiator system (ivocerin) (*Vivadent Ivoclar. Research and Development Report no. 19. Schaan Liechtenstein*, 2013) that is integrated with camphorquinone (CQ) to enhance absorption of visible light and

improve polymerization of the uncured resin (Moszner et al., 2008). In contrast, everX Posterior's findings can be explained by the fundamental difference of filler content and type being randomly oriented glass fibers (Garoushi et al., 2013). The fibers may further transmit and scatter the light, which therefore reaches deeper and wider area (Le Bell et al., 2003). On the other hand, SDR's decreased filler content and the presence of a special photo-active group, enhances the light transmittance properties allowing higher total energy to pass through and reach the photochemical initiators during the exposure cycle (Uctasli et al., 2005). All these different approaches to improve light curing capabilities allow the aforementioned materials to be placed in bulk.

In addition, 6 mm increment thickness was also tested to mimic clinical situations where the light cure unit tip cannot be placed at close proximity to the restoration surface. The investigated materials still had the ability to allow greater total energy through the material reaching the bottom layer. Which is in line with the depth of cure measurements reported by Garoushi et al., showing that when following recommended manufacturer curing times, everX Posterior showed greater depth of cure values when compared to Tetric EvoCeram Bulk Fill and other conventional PFCs, in addition to showing comparable depth of cure to SDR and other Bulk Fill composites (Garoushi et al., 2008).

Furthermore, the beam profile and spectral emission of the light-curing unit are crucial for the light curing of composites (3M-ESPE, 2011) For instance, light curing units with a highly inhomogeneous beam profile can adversely affect a restoration in localized segments of sub-optimal curing of the resin (Megremis et al., 2014). Thus, it should not be assumed that the total energy measured from an LCU is in reality the amount of energy being received by the entire restoration (Price et al., 2015). Accordingly, Elipar™ S10 was used in all studies in this thesis due to its relative beam homogeneity and uniform spectral emission across the light beam, as reported in a previous study (Price et al., 2014).

According to the findings of this study, FRC can be applied safely in bulks of 4 mm increments similar to other bulk fill composites, although in 2mm thickness the investigated composites showed better performance.

6.3 Composite-Dentin Bond Strength and Failure Assessment (I)

In study I, the failure mode assessment data shown in the results section revealed that the most common failure type was cohesive in dentin, which could be attributed to a good adhesion interface where the point of fracture was at the weakest point lying beneath the hybrid layer. Pearson's chi-square test revealed a non-significant ($p>0.05$) association between increment thickness and fracture type in all three bulk

fill composites (Tetric Evo Ceram Bulk Fill, SDR and everX Posterior). According to this data, we can infer that bulk fill composites behave in a non-predictable manner. They can thus still show a homogenous adhesion interface and result in relatively high percentages of cohesive failures even with the increase in increment thickness. This can lead to relatively higher average SBS values at bulk increments when compared with conventional PFC.

In contrast, the conventional PFC (G-aenial Anterior) exhibited a significant ($p < 0.05$) association, which indicates a more predictable failure type pattern. The observed behavior can be explained by the fact that PFCs are intended to be placed in 2mm increments for optimal polymerization. Thus, increase in material thickness directly translates into a weaker adhesion interface that renders a decrease in cohesive failure types and an increase in adhesive failures. As a result, they exhibit a lower average SBS value when compared to bulk fill composites.

Moreover, the adhesive system used in study I was a 7th generation system - Scotchbond Universal (3M Deutschland GmbH, Neuss, Germany) because it can be applied using a one-step, self-etch system; and this was suggested in a previous study by Van Ende et al., to have a less masking effect over the failure modes when compared to a two-step or three-step adhesive system (Van Ende et al., 2013).

6.4 Interlayer Bonding Interface Influence on Load-Bearing Capacity and Fracture Behavior of Bi-Layered Composites (II, IV)

In the clinical setting, a constant challenge dentists face when restoring deep cavities is optimum isolation of the cavities. It is important to avoid any compromise by saliva contamination or mechanical disturbances during the restoration process as this leads to a poor prognosis for the resin composite restorations involved (Eiriksson et al., 2004). Therefore, studies II and IV evaluated the influence of the quality of adhesion at the composite-composite interface in bi-layered specimens.

In study II, the composite-composite interface had a significant influence on the load bearing capacity of cylindrical bi-layered specimen (dimensions) that were prepared for static loading. When comparing the loading values from all the groups with OA, it was evident that the deteriorated quality of adhesion caused a significant reduction in the load-bearing capacity Figure 17. As the layers become less bonded to one another, the bi-layered specimen starts to lose structural integrity and the load bearing capacity of the whole system is jeopardized. In principle, the weakest failure point for such bi-layered structure lies at the adhesion interface as described in a previous study (Barreto et al., 2016). In studies II & IV, DA was simulated by wet grinding. This also causes removal of the OIL which potentially affects optimal chemical adhesion (Demarco et al., 2012; Truffier-Boutry et al., 2003). Adhesion

quality was confirmed with the bond strength test Figure 16. In OA groups the surface PFC was placed immediately after light-curing the base composite, thus simulating optimal conditions and avoiding any possible degradation to the OIL.

Within each material thickness, FBF and FRC groups showed lower load-bearing capacity at DA in comparison to OA. Between both base materials investigated, there was statistical difference in the load values at 1 mm in the OA group (Table 3). Hence, the quality of this interface is of high significance and should be kept at optimum quality to ensure the maximum performance of such bi-layered composite structures.

In DA, the fracture type was predominantly delaminating fracture whereby the fracture was solely confined to the surface PFC. As previously stated in several studies regarding bonding interface, the point of initial failure is through separation at the deteriorated composite- composite adhesion interface (weakest point (Hatta et al., 2011; Taha et al., 2013)). Such a phenomenon and the rare involvement of the base material can be explained by analysis of the FE stress distribution (Figure 25 A and D). At 1 mm PFC group, high stress region is concentrated at the interface area. Hence, in DA the deteriorated interface does not allow the stresses to dissipate appropriately to the base layer. As a result, the surface layer acts independently of the base material underneath and delamination fracture occurs at a lower load. This can be seen from the load deflection graphs, whereby the initial failure point starts at a lower load value until it reaches a higher loading value that is fracture point (Figure 18).

In contrast, 2 mm surface PFC (Figure 25 C and G) shows that the high stress region is well away from the interface. Consequently, lower stress is able to diffuse towards and beyond the deteriorated interface. Thus, the bi-layered specimen are able to withstand higher loads before failure. Fracture eventually occurs within the surface PFC and the crack front can further propagate to include the base material. With 2 mm PFC thickness, the effect of interface quality decreases and becomes insignificant in FRC groups, which can be seen from the load bearing values (Table 3). However, FBF groups were more sensitive and adhesion quality had a significant effect on the load bearing values at the 2 mm thickness (Table 3).

In OA, the fracture type varied between either complete bulk fractures (FBF and FRC groups) or partial bulk fractures (FRC only). Both these types demonstrate that the bi-layered specimen behaved as single structure, hence fracture was involving both composite layers. However, it is worth pointing out that partial bulk fracture type, was a distinct behavior of FRC groups. This can be explained by the nature of the glass fiber reinforcement within the resin matrix (Van Dijken & Sunnegårdh-Grönberg, 2006). Such crack hindering and deviation properties could be clinically useful in deep restorations as stated in previous studies (Abouelleil et al., 2015; Dere et al., 2010; Garoushi et al., 2015). Therefore, the data in this study strongly suggests

optimum composite-composite adhesion interface conditions to fully utilize potential strengthening properties of fiber reinforcement in FRC.

In study IV however, bi-layered SENB specimens were utilized which gave the study another dimension, i.e. the presence of a pre-crack and its propagation in relation to the adhesion quality between the two composite materials.

The adhesion interface greatly affected the fracture behavior and the fracture type patterns in both materials tested. In FBF OA groups, there was exclusively type 1, i.e. fracture through the midline of the specimen, which is due to the similar material properties of both FBF and capping PFC. The resin fillers allow the crack to propagate through the bulk of the structure with no resistance as has been shown in a previous study (Isufi et al., 2016). However, in FBF DA there was a contrasting behavior of crack propagation, due to the poor adhesion interface. The DA directed the crack front along the path of least resistance which was the composite-composite interface between the materials. Therefore, most of the bi-layered SENB specimens in this group showed the most amount of type 4 fracture i.e. complete debonding between the layers.

In contrast, FRC OA group demonstrated that type 1 fractures through the midline constituted half the tested specimens while the remaining half were either type 2 or 3. This demonstrates that regardless of good adhesion between the layers, the crack front was deflected along the interface and away from the base composite, suggesting that the crack front was influenced by the fiber-reinforced base composite as it was not the path of least resistance in comparison to FBF OA. Such fracture resistance properties when using FRC as a base composite is in line with previous studies that have also demonstrated crack propagation hindering effects when using the same FRC material used in this study (Bijelic-Donova et al., 2018; Ozsevik et al., 2016). This property to deflect fracture away from the bulk of the resin composite would be useful in a clinical situation as it increases the probability of a less extensive fracture that can be restored conservatively instead of an extensive fracture that would require the replacement of the entire restoration, or even involve the tooth and become non-restorable. Furthermore, in FRC DA the fracture types were similar to those of FBF DA, with the contrasting difference of lesser incidence of type 4 delamination fracture patterns (10% and 50% respectively). A possible explanation of the decreased incidence of type 4 delamination fracture pattern in FRC DA can be due to the presence of the fibers as shown in a previous study (Özyürek et al., 2018).

6.5 Individual Layers' Thickness of Bi-Layered Composites (III)

Bi-layered restorations raise the question of whether changes in the thickness of either the base material or surface PFC would have an effect on the mechanical properties, and if so, how does it affect the bi-layered structure as a whole. To the knowledge of the authors, such data is not sufficiently available in the literature. According to the results, it was found that there are statistically significant correlations when changing the thicknesses of the materials within the specimen. In general, the thickness had an anticipated pattern. That is, with the increase of the surface PFC thickness (1.0, 1.5, 2.0 mm), there was an increase in the load bearing capacity of the bi-layered structures. This positive correlation was observed in three groups: FBF (OA) FBF (DA) and FRC (DA).

Conversely, an unexpected pattern was observed when the FRC (OA) group was loaded. As surface PFC thickness increased, the load bearing capacity of the bi-layered specimen decreased. The trend was the opposite of all the other groups and there was a statistically significant correlation.

This inverse phenomenon has also been reported in a previous study by Garoushi *et al.*, where it has been found that the thinner the layer of surface PFC, the more the toughness and fracture resistance, in bi-layered structures with FRC base material (Garoushi *et al.*, 2006). This highlights the possibility that 1 mm thin occlusal composites can safely be used to cover FRC base material and therefore be of utility when restoring deep cavities in the clinical setting.

In OA, there were complete bulk fractures at every thickness when using FBF as a base material. In addition, fractures were catastrophic (shattering in many pieces), rendering the impossibility of repair, for instance, in a clinical situation. Since, both FBF and conventional composites are only particulate fillers reinforced composites, the fracture behavior was similar. These findings are in line with previous studies investigating bulk filling material and mechanical loading behavior (Assis *et al.*, 2016; Ilie *et al.*, 2013). Conversely, when using FRC as a base material and in OA, the thickness had a noticeable effect on the fracture behavior. An increasing incidence of partial fractures (30%, 50% and 70%) correlated with the increase in surface PFC thickness (1.0, 1.5 and 2.0 mm respectively). This interesting finding could be further explained by closely observing the load bearing capacities acquired at those thicknesses. As the thickness of surface PFC increased (1.0, 1.5 and 2.0 mm) the load at which fracture occurred decreased, meaning the load that the fibers had to resist was also decreased. Therefore, at lower loads the fibers were able to appropriately deflect and resist the propagation of crack front. On the same note, the load deflection graph of FRC can also provide an insight into the crack front. The curves had an initial failure point followed by final fracture which is a possible demonstration of the initial failure of the surface PFC and consequently the enforcing

behavior of the fiber reinforced base material before the final fracture. According to a similar study by Rocca *et al.*, investigating the effect of fiber reinforced restorations covered with an overlaying CAD/CAM composite restoration, it was observed that the crack front was first initiated from the contact loading area and then propagated downwards into the rest of the restoration material (Rocca *et al.*, 2015). Such laboratory static loading tests would generate a crack front with a very high stored energy which runs rapidly through the bulk of the restoration with little resistance (Rocca *et al.*, 2015). Similarly, in this study, the loading values experienced by the composite bi-layered structures were also high and reached about 2500 N, while in reality, the human teeth could experience 500-800 N of maximal unilateral occlusal force (Ledogar *et al.*, 2016; Röhrle *et al.*, 2018). Bear in mind that teeth are subjected to more complicated loading, which includes wedging through the cusps as well as compressive loads. Additionally, the modulus of elasticity could have an effect as shown in the FE strain distribution analysis (Figure 24). When the surface PFC was 1 mm, FBF deforms more than FRC. When the surface PFC was 1.5 mm, FBF still experiences deformation whereas FRC deformation is negligible. FBF (7 GPa) is having a quite low modulus so it is not supporting surface PFC as FRC (12.3 GPa).

Moreover, in DA, specimens with both FBF and FRC base materials had an exclusive incidence of delaminating fracture whereby the surface PFC material shattered individually. However, as the surface PFC material thickness increased, the fracture was more likely to involve the base material (Table 7).

This phenomenon could be explained by the loading value at which the fracture happened and a crack front with a high stored energy. Thus, as the thickness of surface PFC increased, the load bearing capacity increased and hence there was a higher chance for the fracture to propagate beyond the surface PFC and also involve the bulk base composite. This behavior is also found from FE analysis.

6.6 Interface Surface Design and Fracture Behavior of Bi-Layered Composites (III)

In study III, the results related to the maximum load in compression test suggest that preparing a flat interface design yields the relatively highest load in compression for a bi-layered specimen. One possible explanation is that a flat design avoids the introduction of any voids that may act as areas of stress concentration (Petersen, 2017; Scotti *et al.*, 2016). Hence, a more extensive and gradient-like design interface could ultimately present weak points in the bi-layered structures and cause fracture at lower loads. This phenomenon can be observed from the results acquired in study III. We found that extensive designs such as mesh and pyramid demonstrated a more extensive and gradient-like design interface. These designs also showed the lowest relative maximum loads in the compression tests (1800 N \pm SD 270). However, the

maximal unilateral occlusal force posterior composites could experience during function is between 500-800 N (Ledogar et al., 2016). Therefore, the maximum load in compression of all investigated interface designs can be considered clinically ideal because they are able to withstand the occasional high occlusal forces experienced during occlusal function (Röhrle et al., 2018).

The maximum load in compression acquired by FBF and FRC are in line with a similar study by Özyürek et al., that utilized the same materials to restore endodontically treated teeth. It was observed that even though FRC groups did not demonstrate the highest fracture loads; they had more restorable fractures in conservative endodontic cavities (Özyürek et al., 2018).

In terms of the effect of interface design and fracture behavior, the results gained could be explained by the fact that the relatively greater surface area in the pyramid and mesh design provided unique crumple zones. These are similar to those present in cars and human skulls (Malterud, 2014). Consequently, it dissipates any incoming crack through the gradient-like interface design and into the bulk material at a lower energy, and the fracture front would have lower energy as it goes through the base material (Rocca et al., 2015). In FBF, this means less incidence of catastrophic shattering, while in FRC this translates into the fibers being able to deflect crack propagation away from the bulk of the material and to the peripheries, as was shown in this study. The results are also in line with a previous study showing that in pre-planning for failure in restorations it can be useful to have predictable and favorable failures, when using fibers (Malterud, 2014). The presence of such energy-absorbing and stress-distributing fibers results in a higher possibility for repair if failure occurs (Karbhari & Strassler, 2007). Similarly, owing to the presence of collagen fibers, it has been reported that the structure of DEJ is comparable to that of a fiber-reinforced structure (Dong et al., 2003; Lin et al., 1993).

With regard to using different bulk-fill materials and their effect on the fracture behavior of bi-layered restorations, there was a general difference between the FBF and FRC groups as shown in the results. The catastrophic and unfavorable fracture behavior observed in FBF groups is in line with previous studies investigating the mechanical behavior of bulk-fill composites and conventional PFC (Assis et al., 2016; Ilie et al., 2013). Their fundamentally similar material properties thus do not offer significant improvement of fracture propagation such as partial bulk fractures acquired with FRC reinforced groups (Isufi et al., 2016).

Interestingly, FRC groups did not have any incidence of catastrophic failure, which is most likely due to the positive reinforcement that fibers provide against fracture as reported in several studies (Bijelic-Donova et al., 2018; Ozsevik et al., 2016). Such improvement in fracture behavior and toughness was observed in a different study by Abouelleil et al., where despite the fact that fractures had occurred

within test samples, the fibers within the matrix held separate fracture ends together (Abouelleil et al., 2015).

Additionally, 3D profiling was performed on the top surface of bulk-fill composites to confirm whether the designs created on the molds by 3D printing were transferrable to the composite surface to create the interface design intended (Figure 26). There was a small variance between the mold and the impression of 0.15 mm. A possible cause could be the viscosity of the bulk-fill materials used in this study (Pfeifer, 2017). On one hand, FRC is a packable and more viscous material, thus imprinting an interface design could be appropriately fabricated using a firmer pressure and rigid mold that can apply more force on the surface. On the other hand, FBF is flowable and less viscous, thus an interface design can be imprinted easily with the current PSE mold used in this study.

A surface microhardness test was performed in this study to evaluate the effect of the light curing protocol used to create the different interfaces (Miletic et al., 2017). The lack of reading observed from protocol 1 and 2 infers that interface design imprinting and the light curing protocol did not have an effect on the formation of oxygen inhibition layer. This observation can be due to the presence of an oxygen inhibited sticky gel like layer on the surface of the composite materials, as reported in previous studies (Gauthier et al., 2005; Vallittu, 1999). This layer of unreacted monomers has a decreased degree of conversion and would thus be soft, and due to the softness of the area, the material simply recoils back to normal as the indenter is removed. Therefore, an indent of 245.3 mN, was not able to produce any permanent deformations. According to a previous study, it was shown that protecting the composite surface from ambient oxygen molecules in the air for up to 360 min allowed for higher hardness and effective polymerization (Aromaa et al., 2018; Bijelic-Donova et al., 2015). This finding is in line with the results for test protocol 3 that used a glass slab to cover the surface of the composite for 360 min. Overall, surface microhardness testing is usually most effective when used for metals (Gauthier et al., 2005). As a consequence, when applying a load of 245.3 mN, test protocols 1 and 2 showed no measurable indentations in comparison to protocol 3 which was harder and hence indentable.

6.7 Crack Propagation and Fracture Toughness of Bi-Layered Composites (IV)

The following null hypotheses were tested: (i) fracture behavior would not be affected by the simulated adhesion interfaces; (ii) there would be no significant differences in fracture behavior using different base composite materials. The test set-up of the loading condition was a simplified model of a clinical situation where the fissure is the notch in the specimen and the loading moment is simulating

simultaneous loading of buccal and lingual cusps of the restored tooth. Limitation of the test set-up is the supporting points of the specimens versus the clinical condition where the entire restoration is supported by the dentin. It was assumed in the study design that due to elasticity and resiliency of dentin, which mostly occurred at the areas of supporting points, allowed the notch area of the specimens to have tensile stress as in the bending test. Another aspect of the simplified model relates to the notch of the specimens, which was intentionally extended to the interface area of two resin composite layers. Clinically the fissures are most often located in the capping resin composite.

In contrast to the study setup used in study IV, the neutral axis between compression and tension is normally present at the middle height of a homogeneous SENB specimen. However, the difference in the modulus of elasticity of the resin composite materials used in the bi-layered SENB specimens will shift the position of the neutral axis away from the middle height of the specimen. The neutral axis shifts towards the stiffer material with a higher modulus of elasticity. Thus, due to base composite layer in FRC groups having higher modulus than capping PFC, the neutral axis is shifted within the base composite. This further promotes pre-crack propagation into the base composite layer. This can be seen from the rare incidence of debonding fracture of capping PFC even in the DA groups. Conversely, in FBF groups the neutral axis is shifted toward the stiffer material which in this case is the capping PFC (Yamamoto et al., 2018). Therefore, the tension side would be confined within the capping PFC and would demote the propagation of the pre-crack into the base composite. This is evident in FBF groups with DA, whereby the fracture mainly involved the capping PFC and caused delamination fractures.

An unexpected result of this study was that the maximal load and MST required to damage the specimens were higher for the specimens with deteriorated adhesion than that of good adhesion between the layers of resin composites. The result suggests that the energy of the static loading event is in the first instance distributed by the notch to the interface of the resin composite layers, letting the base composite to behave like a separate unnotched specimen. In the case of better adhesion, the specimens behaved as a mono-block with a notch which effectively allowed the fracture to propagate through the specimen.

The higher load and MST values of the FRC base composite specimens versus FBF specimens are likely related to the extrusions of fibers from the base composite surface which changed the stress distribution at the interface during the loading event (Nagata et al., 2016).

6.8 Clinical Considerations and Perspectives of Future Studies

Globally, direct restorations are known to be the most utilized restorative treatment (Schwendicke et al., 2016). The number of resin composite restorations placed in year 2015 alone was 800 million, of which 80% were posterior restorations (Heintze et al., 2012). However, according to a study by Heintze et al., it was estimated that about 32 million of those posterior restorations are prone to failure from fracture and will eventually need replacement or repair by the year 2025 (Heintze et al., 2016). This can be due to the presence of cracks or voids in resin composite restorations and the high loads experienced at the posterior region (Petersen, 2017; Sarrett, 2005).

High masticatory forces have been shown to magnify the presence of cracks and voids in resin composite materials which can propagate and eventually result in fracture of the restorations (Elbishari et al., 2012). Previous studies also suggest that fracture behavior of the currently used resin composite materials is not fully predictable (Karbhari et al., 2007; Rocca et al., 2015). Some fractures might be extensive, catastrophic and non-restorable, while others could be smaller, partial and restorable (Assis et al., 2016; Ilie, Bucuta, et al., 2013). This variation in fracture behavior with consideration to the trending bi-layered posterior restorations, inspired the inception of this thesis. The enclosed series of studies are aimed to help grow the current body of literature in terms of understanding how the resin composite restorations fail, and how to favorably influence this failure in order to avoid the risk of losing more tooth-tissue (Malterud, 2014).

Moreover, the current literature suggests that the recent dental resin composite materials developed are aimed to overcome the short comings of previous materials by improving their properties. Improved properties include better wear resistance, modified viscosities to improve adaptability to cavity margins, and the ability to be placed in bulk increments of 4 mm to reduce the time needed to restore deep cavities (Bucuta et al., 2014; Heintze et al., 2016; Leprince et al., 2014; Omran et al., 2017). Other recent innovations of resin composite materials include introduction of novel fillers such as discontinuous glass fibers that improve the mechanical properties (Nicola et al., 2016). Interestingly, such fiber-reinforced resin composite materials have demonstrated a noteworthy behavior to fracture, where the glass fibers hinder crack propagation and lead to improved fracture toughness of the materials (Fonseca et al., 2016; Garoushi et al., 2018). Such fracture resistance properties are similarly observed in the natural dentition due to the presence of the collagen fibers in dentin as well as the dentin-enamel junction (Chan et al., 2011). This similarity highlights the potential of having a biomimetic approach to direct restorations (Omran et al., 2019).

Furthermore, another naturally occurring phenomenon that allows the teeth to have unprecedented longevity when compared to current resin composite materials,

is the presence of a bi-layered structure consisting of two contrasting tooth tissues; enamel and dentin (White et al., 2005). This contrasting material properties causes the tooth to act as a dynamic heterogeneous structure when compared to the homogeneous structure of a single-material resin composite restoration (Zaytsev et al., 2014). This fundamental bi-layered structure of teeth has not been deliberately addressed when utilizing resin composite materials in direct restorations.

Bi-layered approach however, seems sensitive to several parameters as shown in the results compiled in this thesis. Which warrants extra care and clinical consideration when placed in the clinics. Dental practitioners are encouraged to follow optimal placement protocols to help avoid the technique sensitivity of application as well as to optimize the bi-layered restoration as a whole. Areas such as increment thickness, composite-composite quality of adhesion as well as the combination of base composite and capping composite materials have been demonstrated to have an effect on both the load-bearing capacities as well as the fracture behavior in case of its occurrence. Both the materials themselves as well as their application procedures have been evaluated in the series of studies included in this book.

Which sheds on the novelty of the methods used in this thesis. For instance, in study III, the fabrication and application of composite-composite interface designs, is studied in depth for the first time in the restorative field. While the methodology used can be applicable in a chair side setting, shedding the light on more sophisticated and progressive dental restorative procedures using 3D designing and printing technologies. Additionally, in study IV laboratory tests were modified and adapted to bi-layered composites and further investigation is warranted to identify fracture toughness of bi-layered composites as a single structure. The novel methodology utilized can inspire new focus areas for further research of bi-layered restorations.

This thesis evaluated several parameters to help isolate the confounding factors that affect and possibly predict the fracture behavior of bi-layered composite restorations. For if and when the fracture happens, we as clinicians can architect a more favorable pathway of fracture. Which has been a constant observation in this series of studies. FRC are demonstrating fracture hindering capabilities which warrants further research in the future to look at the fatiguing behavior of these materials when placed in a bi-layered configuration. Such a test can have tremendous clinical value due to the more constant low-load repetitive in the oral cavity.

Another yet important area of further research would be to quantify a larger set of composite materials in terms of moduli of elasticity that is similar to that of the tooth structure (enamel and dentin). This can be of high value to clinicians as all they will need to predict the behavior of materials would be a basic material science knowledge of the favorable combinations based on parameters such as moduli of

elasticity and possible microstructure (FRC vs PFC) and they can start choosing the best criteria to provide the possible best prognosis based on the case at hand. It is noteworthy to mention, that the moduli of elasticity of the composite materials investigated in this thesis as well as those currently used in the dental practice, are still not close to the human dental structures. This limitation highlights the need for improvement of the enamel-replacing and dentin-replacing composite materials to be used in the future.

In depth FEA as well as micro-CT can be valuable assessment and diagnostic tools respectively that enable us to further study how the fracture behavior is influenced with interface designs. Also, further studies involving other interface designs can help us create a blueprint for fabricating designs at the micro-level that are similar to the existing DEJ design. That would however, need more advancement in 3D printing technology to accurately print micro-gradient like DEJ directly using fiber-reinforced composite materials.

The notable behavior of fiber-reinforcement in bi-layered configuration warrants further investigation as well as continuous improvements at the material composition level. For instance, as per the current writing of this thesis, new flowable fiber-reinforced materials are being introduced to the market that could provide mechanical improvements as well as easier handling properties while restoring deep cavities (Lassila et al., 2019).

Nonetheless, it is worth mentioning that the tests were conducted through a standardized laboratory protocol, and the clinical situation is variable in terms of handling and placement of material. The correlation of such laboratory findings to the clinical environment has been investigated in a recent systematic review by Heintze *et al.*, whereby it was found that fracture toughness as a laboratory parameter was mostly correlated with clinical fracture (Heintze et al., 2016).

Furthermore, to further clinically validate the results of the current studies, fatigue testing should be conducted to quantify the long-term effects of repetitive low occlusal forces on bi-layered composite structures.

Future dynamic mechanical tests utilizing longer cyclic loads, e.g. 1.2 million cycles can equate to 5 years clinical performance (Alraheam et al., 2019; Heintze et al., 2010). Such studies can help clinicians identify what parameters (composite-composite adhesion interface, Interface-design, each material thickness, each material moduli of elasticity) are the most influential on the long-term survival of bi-layered composites as well as the fracture behavior.

An important aspect that this thesis repetitively investigated was the ability to make the bi-layered restorations predictable when it came to fracture behavior. In efforts to try to assimilate the tooth, which has a higher degree of predictability when comes to mechanical performance, the aspects investigated in this study shed the light on the importance of each of the parameters investigated. The term technique sensitive

is indeed descriptive of direct restorations using resin composite materials and hence adding two materials adds more room for biases and variations. Hence, this project aimed at standardizing several parameters for which their collected empirical data can help draw new guidelines for future bi-layered restorative procedures.

7 Conclusions

Based on the studies and the results of the experiments reported in this thesis, the following can be concluded:

1. As increment thickness increases, the light transmission and shear bond strength decreases for all resin composite, including bulk-fill materials. Discontinuous fiber-reinforced composite showed the highest value of curing light transmission in comparison to other investigated bulk-fill composite materials, which also leads to improved bonding strength to the underlying dentin surface.
2. Bi-layered composite structures are sensitive to the quality of adhesion between the base and surface composite layers. Load-bearing capacity increases with optimum adhesion as the bi-layered structure acts as a dynamic solitary structure.
3. Fracture behavior of bi-layered composite structures could be affected by the gradient-like interface design between the base and surface composite layers. Fracture behavior is expected to improve when using short fiber reinforced composite as the base material along with an intricate interface design that can improve material failure.
4. The combination of innate fracture toughness properties and modulus of elasticity of resin composite materials used in the bi-layered specimens can further promote or demote crack propagation, which warrants further research to identify what are the most favorable material combinations to mimic the natural bi-layered configuration of dentin and enamel.
5. The presence of the O₂ inhibition layer at the composite-composite interface affected the fracture behavior and fracture toughness of bi-layered structures. Therefore, adhering to optimal adhesion protocols are fundamental for the optimal mechanical performance of bi-layered restorations.

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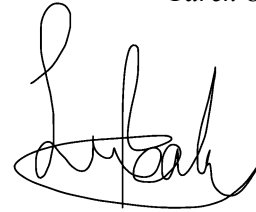
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The last person I need to dedicate an exclusive paragraph for is you Khalil—Professor Dr. Khalil Shahramian. We started our journey from dental school together—the exams, the long drives and the sleepless nights; side by side. When we became dentists, we decided to start a challenging journey. We left our families, friends and loved ones so that we can go to Finland, to pursue knowledge, science and to develop ourselves; side by side. We shared more than just time together; we shared meals, rooms and even kitchen floors. We have both worked on our doctoral degrees; side by side. It was even funny when we introduced ourselves, because only one of us had to say the story while the other said “#metoo”. With strong conviction ($p < 0.00001$) I am certain I wouldn’t be here without your companionship and selfless support. My dadash, you were my backbone since 2008 that only gets stronger as the years go by. Khalil you are a gift from God, that I can never repay back. I thank your family baba-e Ali-Akbar mama-ni Zohreh, and khâhar Zahra for bringing you and shaping you to this world. You are the one person who has truly demonstrated the real meaning of selflessness, enta gada3, enta keda! I am looking forward to the great things we shall do together to positively influence our dental field, our communities and ultimately the world; side-by-side!

San Francisco, October 17th 2019

Tarek Omran

A handwritten signature in black ink, appearing to read 'Tarek Omran', with a large, stylized initial 'T' and 'O'.

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