



**UNIVERSITY  
OF TURKU**

# **BONDING OF ORTHODONTIC BRACKETS TO ENAMEL**

**Studies on the Clinical Outcome of Bracket  
Bonding and Approaches to Increase the  
Bond Strength of the Adhesive Interface**

**Leeni Kilponen**





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*To my family*

*There is nothing like looking, if you want to find something.*

*- J.R.R. Tolkien, the Hobbit, 1937*

UNIVERSITY OF TURKU

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Science

LEENI KILPONEN: Bonding of orthodontic brackets: studies on the clinical outcome of bracket bonding and approaches to increase the bond strength of the adhesive interface

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## ABSTRACT

An extensively used form of orthodontic treatment is the use of fixed appliances, where brackets are attached to the teeth for the duration of the treatment, often with light curing adhesives. The adhesion between the brackets and enamel must withstand the forces generated during the treatment, but on the other hand, an easy removal of the appliances at the end of the treatment without causing damage to dental hard tissues is desirable. Sometimes bracket failure occurs during the treatment, in which case a new bracket must be bonded on the tooth, and here the changes in enamel surface after initial bonding procedures may influence the rebonding.

In the studies presented in this thesis, the failure rate of brackets in clinical practice and certain methods to improve the bonding and rebonding of brackets were investigated.

The failure rate of orthodontic brackets in this study cohort was 7.9%. More brackets were debonded from mandibular teeth than from maxillary teeth, and the selected bonding system and the operator's bonding technique had an effect on the bonding success.

A glass fiber weave placed in the adhesive interface improved the degree of cure of the adhesive under metal brackets, and transillumination through teeth was possible, but the irradiance diminished quickly as the thickness of the tooth slices increased.

Ceramic brackets yielded highest bond strengths (21.9 MPa) compared to metal (8.14 MPa) and polycarbonate brackets (10.47), and a silane based primer improved the bond strength of ceramic brackets even further (26.45 MPa). However, this improvement of bond strength was associated with enamel fractures. To achieve adequate bond strength values after bracket failure, a small amount of enamel needed to be ground off before rebonding.

It can be concluded, that a clinically significant number of brackets fail during the treatment, and therefore these methods that improve bonding are of importance. Yet a higher bond strength must not come at the detriment of intact dental hard tissues.

**KEYWORDS:** orthodontic bracket, bond strength, dental bonding, bracket failure

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## TIIVISTELMÄ

Oikomishoidossa paljon käytetty hoitomuoto on kiinteä kojeistus, jossa kaari-lankojen kiinnikkeet, braketit, kiinnitetään hammaskiilteeseen. Kiinnityksessä käytetään useimmiten valokovetteisia kiinnitysmuoveja. Sidoksen täytyy kestää hoidon aikaiset voimat, joten sidoslujouden parantaminen on tärkeää. Toisaalta liian vahva kiinnitys saattaa johtaa hammaskudoksen vaurioihin hoidon päättyessä, kun braketit irrotetaan hampaista. Jos braketti irtoaa kesken hoidon, täytyy hampaaseen kiinnittää uusi braketti ja tällöin ensimmäisen sidostuksen aikaansaamat muutokset hammaskiilteessä saattavat vaikuttaa uudelleensidostamiseen.

Näissä tutkimuksissa selvitettiin brakettien irtoamisfrekvenssiä, sekä pyrittiin parantamaan brakettien sidoslujutta erilaisin keinoin.

Tutkitussa potilasaineistossa 7,9 % braketeista irtosi ennen aikaisesti kesken hoidon. Irtoaminen oli yleisempää alaleuan hampaissa kuin yläleuassa. Valittu kiinnitysmuovi ja sidosaine, sekä operaattorin sidotustekniikka vaikuttivat sidostuksen onnistumiseen.

Lasikuituverkko braketin alla paransi kiinnitysmuovin kovettumisastetta metallisten brakettien ja suurempien kojeiden alla. Kiinnitysmuovin valokovetus oli mahdollista dentiinin läpi, mutta vain alle 4 mm paksuuteen asti. Vertailtaessa erityyppisten brakettien sidoslujuuksia, keraamisten brakettien sidoslujuus oli korkeampi (21,9 MPa) kuin polykarbonaatti- (10,47 MPa) tai metallibrakettien (8,14 MPa). Silaanipohjainen esikäsitellyaine braketin pohjassa paransi keraamisten brakettien sidoslujutta (26,45 MPa), mutta vahvempi sidos johti kiille vaurioihin kiinnikkeitä irrotettaessa. Braketin ennenaikaisen irtoamisen jälkeen pieni määrä kiillettä täytyi hioa pois, jotta uutta brakettia kiinnitettäessä saavutettiin alkuperäiseen sidostukseen verrattavissa oleva sidoslujuus.

Yhteenvetona voidaan todeta, että kliinisesti merkittävä osa braketeista irtoaa hoidon aikana, joten kiinnitystä on tärkeää parantaa, mutta toisaalta voimakas sidostuminen ei saa johtaa hampaan kovakudosten vaurioihin.

AVAINSANAT: braketti, sidoslujuus, sidostaminen, brakettien irtoaminen

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# Abbreviations

ANOVA	Analysis of variance
ARI	Adhesive remnant index
ATR	Attenuated total reflectance
bis-GMA	Bisphenol-A diglycidyl ether dimethacrylate
°C	Degrees Celsius
CI	Confidence interval
DC%	Degree of cure
DEJ	Dentino-enamel junction
DMAEMA	2-(dimethylamino)ethyl methacrylate
EBPADMA	Bisphenol-A-bis(2-hydroxyethyl ether) dimethacrylate
E-glass	Electrical glass
FTIR	Fourier transform infrared (spectrometer)
GIC	Glass-ionomer cement
LCU	Light curing unit
LED	Light emitting diode
MDP	10-methacryloyloxydecyl dihydrogenphosphate
mm	Millimeter
MPa	Megapascal
MPS	3-methacryloyloxypropyltrimethoxysilane
OH	Hydroxyl group
PMMA	Poly(methylmethacrylate)
RMGIC	Resin-modified glass-ionomer cement
s	Second
SBS	Shear bond strength
SD	Standard deviation
SEM	Scanning electron microscope
SEP	Self-etching primer
Si	Silicon
SiC paper	Silicon Carbide grinding paper
SiOH	Silanol
TEGDMA	Triethylene glycol dimethacrylate

UDMA	Urethane dimethacrylate.
w-%	Weight percentage
μm	Micrometer
Ø	Diameter
4-META	4-methacryloxyethyl trimellitate anhydride

# List of Original Publications

This dissertation is based on the following original publications, which are referred to in the text by their Roman numerals:

- I Kilponen L., Uusitalo E., Tolvanen M., Varrela J., and Vallittu P.K. (2016). Photopolymerization of Light Curing Adhesives Used with Metal Orthodontic Brackets and Matrices. *Journal of Biomaterials and Tissue Engineering*, 6(8): 659-664.
- II Kilponen L., Lassila L., Tolvanen M., Varrela J., and Vallittu P.K. (2016). Effect of Removal of Enamel on Rebonding Strength of Resin Composite to Enamel. *BioMed Research International*, vol. 2016, Article ID 1818939, 7 pages.
- III Kilponen L., Varrela J., and Vallittu P.K. (2019). Priming and bonding metal, ceramic and polycarbonate brackets. *Biomaterial Investigations in Dentistry*, 6(1): 61-72
- IV Kilponen L., Suominen A., Vallittu P.K., and Varrela J. Orthodontic Bracket Failure *in vivo*: Bonding System and Technique Affect Bonding Success. Manuscript.

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# 1 Introduction

Orthodontics is a field of dentistry, which aims to achieve a functional and aesthetic occlusion by influencing the growth of the jaws and positions of the teeth. Malocclusion is a prevalent problem worldwide (Alhammadi et al. 2018), and in a pursuit of a balanced occlusion, an extensively used treatment modality today is the use of fixed appliances, which constitutes of the use of archwires and brackets that are attached to the teeth.

A beautiful smile consists of many factors, one of which is the alignment of teeth (Manjula et al. 2015). People have shown concern for this from early on: the earliest literary description of malocclusion dates back to Hippocrates in 400 BC (Hippocrates, translated Smith 1994). It has been speculated, that gold bands found on the teeth of Etruscan and Egyptian mummies would have been an early form of orthodontic treatment, but this is controversial, and the bands may have played more of cosmetic role than a functional one (Forshaw 2016). A more contemporary form of orthodontics began in the late 1800s and early 1900s with Edward H. Angle, who is often referred to as the “father of modern orthodontics”. He separated orthodontics as its own specialty from general dentistry, and established classification of malocclusion that is still used to this day (Asbell 1990). In those days, the appliances constituted of bands that circled the crowns of the teeth, which caused various problems, ranging from aesthetic concerns to gingival trauma (Gange 2015). In 1955 Michael G. Buonocore introduced the acid etching technique of the dental enamel, which revolutionized dentistry, including the practice of orthodontics (Buonocore 1955). From then onwards, the use of brackets bonded on the enamel replaced the use of bands. The first brackets were made of stainless steel. Stainless steel as an orthodontic appliance material was introduced in the 1920s, and stainless steel brackets and other appliances are still in wide use today (Rossouw 2010). Aesthetics of orthodontic brackets progressed from the 1960s through 1980s, when tooth-colored and translucent plastic and ceramic brackets were introduced (Wahl 2008).

More recent innovations in fixed appliance treatment include e.g. lingually bonded brackets, although brackets bonded on the labial surfaces of the teeth are still predominantly used. The brackets are bonded on enamel, mainly with light curing adhesives, and are to remain attached to the teeth for the entire duration of the

treatment. New material development has been ongoing and fast since the first orthodontic light curing adhesives came into market in the 1990s (Wahl 2008).

The interactions at the interfaces of the bracket, adhesive and enamel are intricate, and an understanding of the mechanisms at play is important. Achieving an adequately strong bond between the tooth and the bracket is essential for successful orthodontic treatment because the bond must withstand forces generated during the treatment. However, easy removal of the brackets at the end of the treatment, without harming the enamel surface, is also desirable. The problems still present with fixed appliance treatment include bracket failure during the treatment. Accidental debonding creates additional work for the orthodontist and extra visits for the patient, thereby creating more costs. Furthermore, repeated bonding can have detrimental effects on the enamel surface and result in inconsistent bond strengths (Bishara et al. 2000). If the variables affecting the success of orthodontic bonding can be identified, and the methods of the bonding procedure improved, the amount of unwanted detachments can perhaps be diminished.

This thesis focuses on the interfaces and adhesion between orthodontic brackets, adhesive, and enamel, with the aim of finding methods to improve bonding.

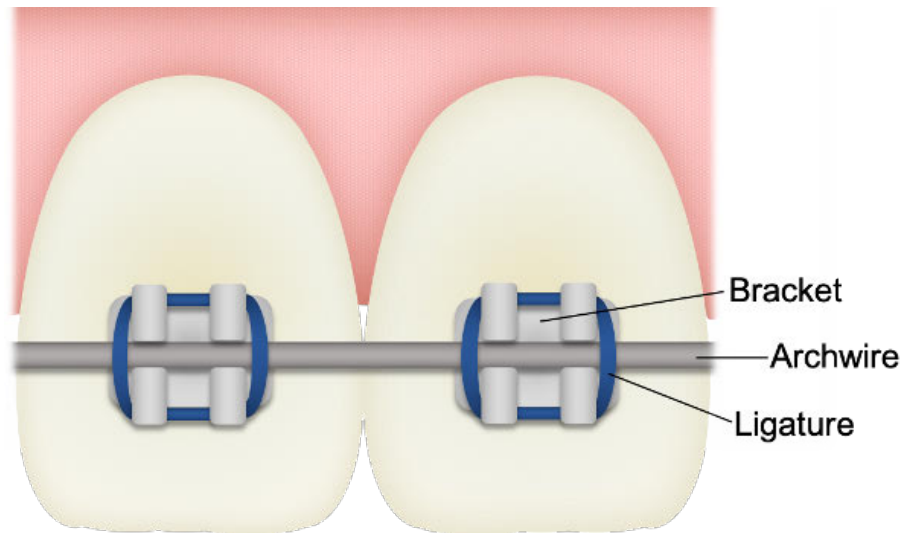
## 2 Review of Literature

### 2.1 Fixed Appliance Treatment

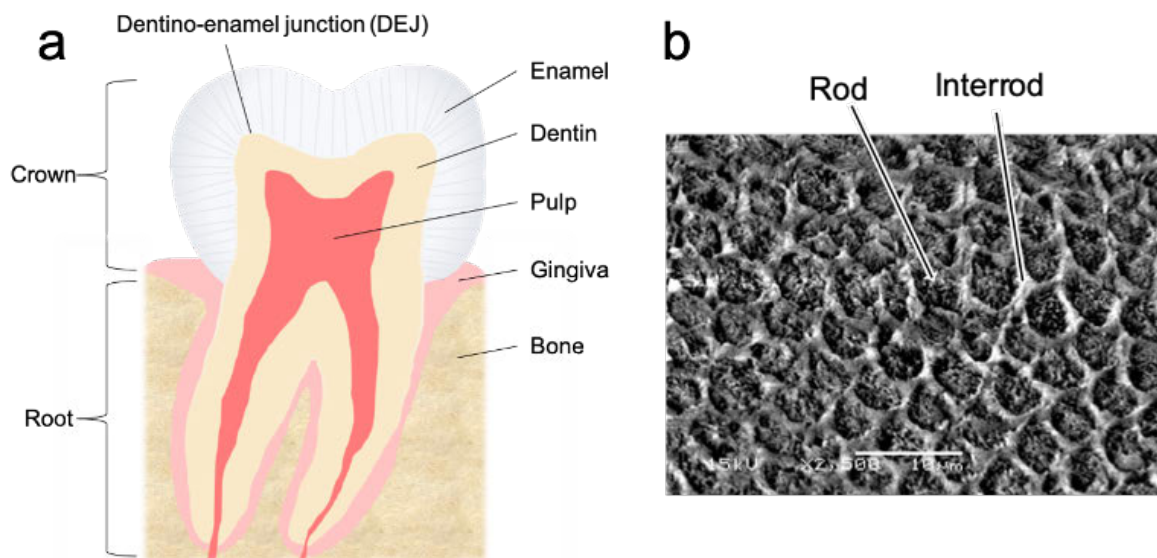
Fixed appliance treatment is a traditional and widely used form of orthodontic treatment to correct malpositions of the teeth and occlusal discrepancies within and between the dental arches. The average duration of fixed appliance treatment is 20-30 months (Skidmore et al. 2006, Tsihlaki et al. 2016, Stasinopoulos et al. 2018). The movement of teeth is achieved by forces generated and directed to the teeth via archwires and brackets, that are attached to the dental enamel (Figure 1).

The treatment consists of an active period during which the tooth movements are carried out, and an equally important retention period, where the stability of the treatment outcome is ensured (Reitan 1967). During the active treatment, the archwires are changed as the treatment progresses, but the brackets remain attached to the enamel for the whole active treatment period. In modern orthodontics, the brackets are usually bonded to enamel with resin-based light curing adhesives. The bonding procedure usually consists of three steps: conditioning the enamel with phosphoric acid, treating the enamel surface with a liquid adhesive primer, and cementing the bracket on the enamel with an adhesive cement.

The bond strength is affected by the properties of the enamel, the adhesive cement layer, and the bracket, and by the interaction between them at their interfaces. It has been suggested, that a bond strength of 6-8 MPa would result in an attachment that is clinically acceptable (Reynolds 1975). Since there is a need to ensure that the bracket remains attached to the tooth during the entire treatment time, the bond must be sufficiently strong. However, too high bond strength may lead to breakage inside the enamel or even at the dentino-enamel junction (DEJ) when the bracket is removed (Joseph and Rossouw 1990).



**Figure 1.** Schematic representation of fixed appliances (brackets, archwire and ligatures) on upper central incisors.



**Figure 2.** **a)** tooth anatomy and **b)** enamel structure after acid etching (scanning electron microscope, magnification X2500, bar in the image is 10 μm).

## 2.2 Enamel

### 2.2.1 Enamel Structure

Dental enamel is the hardest structure in the human body. It is of ectodermal origin, and consists of approximately 96 w-% mineral content, mainly hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ), less than 1 w-% of organic matter, and 4-5 w-% of water. The enamel structure is formed of hydroxyapatite crystals, which are tightly packed into



enamel rods or prisms. The diameter of the enamel prisms is approximately 3  $\mu\text{m}$  (Gentile et al. 2015). The prisms run from the dentino-enamel junction perpendicularly towards the surface of the crown. The thin layer of enamel between the rods, where the crystallites are more loosely packed and oriented in a more random manner, is called interrod or interprismatic enamel (Figure 2).

The thickness of the enamel layer on the surface of the tooth is 1500-2000  $\mu\text{m}$  (Thompson and Way 1981). In the outermost part of a young, intact tooth, there is a layer (15-70  $\mu\text{m}$ ) of condensed, aprismatic enamel, where the usual prism structure is not evident (Ripa et al. 1966, Gwinnett 1967, Whittaker 1982). This is usually worn over time, but also in mature enamel, the outer part is harder than the inner part near DEJ due to a gradient in mineral deposition (Cuy et al. 2002, He et al. 2010, He et al. 2011). There can also be differences in the composition of enamel due to properties of saliva and fluoride concentration of drinking water, because salivary calcium and phosphate ions can mineralize the enamel and fluorine ions can convert the hydroxyapatite to fluoroapatite ( $\text{Ca}_5(\text{PO}_4)_3\text{F}$ ), which is less soluble than hydroxyapatite (Abou Neel et al. 2016).

## 2.2.2 Acid Etching

For successful orthodontic treatment, brackets need to be firmly attached to the teeth. In the earlier days of orthodontics, brackets were welded to bands that were cemented on the teeth covering a large part of the crown (Rossouw 2010). After the emergence of the acid etch technique (Buonocore 1955), enamel etching has been a corner stone of bonding resin composites to enamel. Acid etching results in a porous enamel surface, and the adhesive resin can flow into these pores resulting in a formation of tags, increased micromechanical retention and a stronger bond (Gwinnett and Matsui 1967, Buonocore et al. 1968). The bond strength of resins to etched enamel averages approximately 20 MPa (Barkmeier et al. 1986, Gilpatrick et al. 1991).

Phosphoric acid is the acid of choice when etching enamel, and the highest bond strength values are achieved with a concentration of 10-30%, since it causes maximum enamel dissolution, but the deposits are easily removed by thorough water rinsing (Soetopo et al. 1978). Concentration of 30–40% is commonly used in clinical work. With a conventional 37% phosphoric acid, etching can reach depths of 50  $\mu\text{m}$  (Fjeld and Øgaard 2006). The etching pattern depends on the direction of the enamel prisms, and there are three distinct pattern types: type I, where the prism center is dissolved, type II, where the periphery is dissolved, and type III, where no clear prism structure is evident (Silverstone et al. 1975, Lopes et al. 2007). There can be variation in the etching result in different parts of the etched area due to differences in enamel structure, e.g. in aprismatic areas. When using 32-35% phosphoric acid, a

distinctly porous surface is achieved leading to equal bond strength values for both intact and ground enamel surfaces (Kanemura et. al. 1999, Shinohara et al. 2006). Posterior teeth have been shown to exhibit poorer etching results compared to anterior teeth (Mattick and Hobson 2000, Hobson and McCabe 2002). However, achieving an “ideal” etching pattern is not a prerequisite for adequate bond strength (Hobson and McCabe 2002), and concentrations as low as 2% have also been suggested to yield clinically acceptable bonding (Carstensen 1995).

Prolonging the etching time has been found to increase the depth of the etching in some studies (Hermsen and Vrijhoef 1993, Osorio et. al. 1999, Cehreli and Altay 2000), but not in others (Barkmeier et. al. 1986). Osorio et. al (1999) found stronger shear bond strength with 60 s etching time compared to 15 s, whereas Surmont et. al. (1992) concluded that similar bond strength is achieved with both 15 s and 60 s etching times. Gilpatrick et. al. (1991) found that the strongest bond strength was achieved with 15 s of etching, but other etching times between 5 s and 60 s produced nearly equal bond strengths. On the other hand, Olsen et. al (1996) concluded that not etching at all, or extremely short etching time of 5 s led to a low bond strength, but everything between 10 s and 30 s produced similar bond strengths. With very long etching times, such as 90 s, the etching can dissolve the aprismatic layer and reveal the prismatic layer underneath (Kodaka et. al. 1993). Presently, 15 s of etching is commonly recommend for clinical work.

### 2.2.3 Self-etching Primers (SEPs)

In addition to phosphoric acid etching, products combining the acid and the adhesive primer are available and gaining popularity. The self-etching primers (SEPs) combine the acidic and the methacrylate component by containing a methacrylated phosphoric acid ester, which etches and primes simultaneously. Combining these two steps into a single product significantly shortens the time needed for the bonding procedures (Aljubouri et. al. 2004, Bishara et al. 2005, Banks and Thiruvengkatachari 2007, Elekdag-Turk et al. 2008a, Khalha 2008, Atik et. al. 2019). SEPs cause a more conservative etching pattern and less enamel loss compared to phosphoric acid etching (Hosein et. al. 2004, Cal-Neto et. al. 2006a, Fjeld and Øgaard 2006). In most studies, the use of SEPs results in somewhat lower but still clinically acceptable bond strengths compared to a conventional system of phosphoric acid and a separate adhesive primer (Bishara et al. 2001, Pashley and Tay 2001, Aljubouri et. al. 2003, Romano et al. 2005). The results vary depending e.g. on the specific SEP used in the study, and some researchers have found their bond strengths to be equal to (Arnold et. al. 2002, Korbmacher et al. 2002, Velo and Carano 2002, Cal-Neto et al. 2006b, 2006c, Elekdag-Turk et al. 2009, Hellak et al. 2016), or even higher than those obtained by the conventional system (Buyukyilmaz et. al. 2003). Different

SEPs can have slightly different components, and their acidity varies: their pH is approximately 0.5-3, whereas the pH of phosphoric acid is 0.1-0.4 (Di Hipólito et. al. 2005, Ostby et. al. 2008, Pashley et. al. 2011). More acidic SEPs cause a more aggressive etching pattern, but a deeper etching does not translate into a stronger bond (Pashley and Tay 2001, Ostby 2008).

## 2.3 Orthodontic Brackets

Orthodontic brackets consist of a base, wings and a slot for the archwire. They are commonly bonded on the buccal and labial surfaces of the teeth, although palatinally and lingually bonded brackets are also used. The archwire is secured into place in the bracket slot by placing a rubber or metallic ligature around the bracket wings (Figure 1). The standard type of bracket is metallic, but tooth-colored or translucent ceramic and polycarbonate brackets are also available. The material and design of the brackets influence the bond strength, each having their specific advantages and drawbacks.

### 2.3.1 Metal Brackets

Metal stainless steel brackets were the first to come on the market and are still the most widely used. Their mechanical properties, e.g. strength and wear resistance, are excellent, but they are less pleasing aesthetically. Metal brackets block the curing light from reaching the whole adhesive layer underneath the bracket, resulting in an incomplete degree of cure and variable bond strength values (Eliades et. al. 1995a, Eliades et al. 2000, Mirzakouchaki et al. 2012, Chalipa et al. 2016, Mirzakouchaki et al. 2016, Arash et al. 2017, Delavarian et al. 2019). Hence, there can be a higher risk for bracket failure, but the removal of brackets is relatively easy. When debonding metal brackets, the break-off typically happens at the bracket-adhesive interface, and enamel is left intact (Dickinson and Powers 1980, Ødegaard and Segner 1988, Leão Filho et al. 2015). The bonding is based on mechanical retention, and the design of the bracket base is therefore important (Knox et al. 2000, Sharma-Sayal et al. 2003, Bishara et al. 2004, Wang et al. 2004, Shyagali et al. 2015, Henkin et al. 2016). In mesh-based metal brackets, larger mesh apertures lead to higher bonding values, because resin is able to better penetrate into the base and air can easily be displaced from under the bracket (Hudson 2011).



**Figure 3.** Metal orthodontic brackets (photo: Nielson2000 / CC-BY-SA-3.0).

### 2.3.2 Ceramic Brackets

Ceramic brackets consist of aluminum oxide in mono- or polycrystalline form. Their popularity is based on their better aesthetics compared to the metal brackets, since they are translucent. Monocrystalline brackets have the best optical clarity of the two forms (Eliades et. al. 1995b). Ceramic brackets also have high strength, they resist wear and deformation, and their color stability is very good, which prevents staining. A downside to ceramic brackets, especially to those made of monocrystalline, is their low fracture toughness, which can cause bracket fractures (Scott 1988, Swartz 1988, Bishara and Trulove 1990, Flores et al. 1990, Bishara et. al. 1993, Theodorakopoulou et al. 2004, Bishara et al. 2008, Nishio et al. 2009). Fractured pieces can cause problems in case they are swallowed or aspirated. If a piece of a fractured bracket remains on the enamel, it must be removed with a diamond bur, which can cause enamel damage.

Because of the translucency of ceramic brackets, a higher degree of cure of the adhesive under the bracket and higher bond strengths are obtained (Ødegaard and Segner 1988, Joseph and Rossouw 1990, Viazis et. al. 1990, Forsberg and Hagberg 1992, Eliades et al. 1995b, et al. 2000, Theodorakopoulou et al. 2004, Chalipa et al. 2016). The high bond strengths can increase the risk for enamel damage when ceramic brackets are debonded (Jeiroudi 1991, Forsberg and Hagberg 1992, Wang et. al. 1997, Årtun 1997, Gittner et al. 2012, Leão Filho et al. 2015) although several studies have reported no enamel damage after the removal of ceramic brackets (Ødegaard and Segner 1988, Chen et al. 2007, Habibi et. al. 2007, Suliman et al. 2015). Ceramic brackets derive their retention from mechanical retention caused by

the design of the base, chemical retention achieved by silanation of the base, or with a combination of both. Chemically retentive brackets have been reported to have a stronger or an equally strong bond strength to enamel compared to those with mechanical retention (Joseph and Rossouw 1990, Forsberg and Hagberg 1992, Habibi et. al. 2007).

### 2.3.3 Polycarbonate Brackets

Plastic brackets, initially made of acrylic, are presently made of polycarbonate, a thermoplastic polymer synthesized from bisphenol-A and phosgene gas. Like ceramic brackets, polycarbonate brackets are also translucent. However, polycarbonate is not a strong material, and polycarbonate brackets exhibit problems such as bending and slot deformation (Nishio et al. 2009). To improve their mechanical properties, they are often reinforced with fillers or fibers, and to achieve necessary torque, the slot is reinforced with metal (Feldner et al. 1994, Alkire et al. 1997, Sadat-Khonsari et al. 2004). Polycarbonate brackets are often reported to yield lower bonding values than ceramic or metal brackets (Chaconas et. al. 1991, Guan et al. 2001, Ökangzcan et. al. 2008).

### 2.3.4 Bracket Design

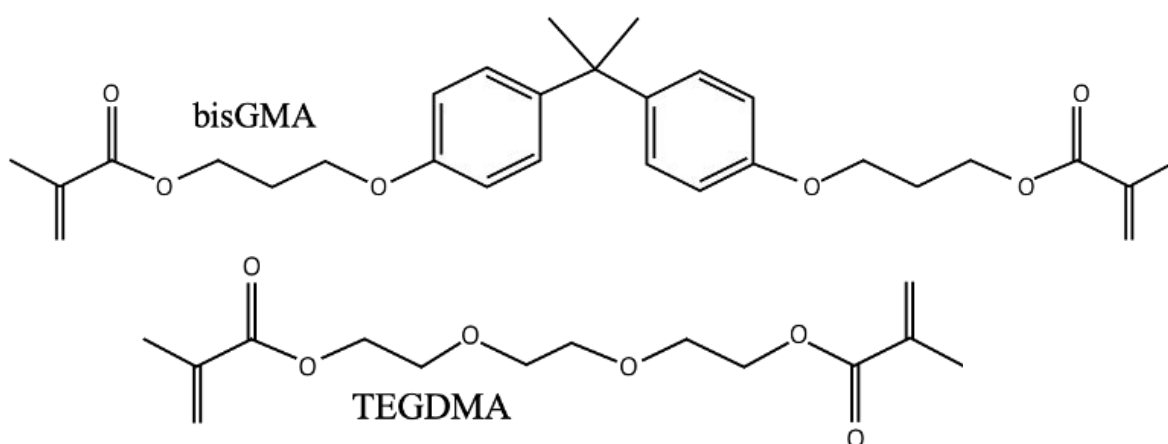
Regardless of the bracket material, the design of the bracket base is of primary importance. The most important type of retention between the bracket and the adhesive in all bracket types is the mechanical retention created by the shape of the bracket base. There are several types of base designs aimed at creating sufficient mechanical retention, e.g. a mesh, grooves or beads on the base can be used to create undercut areas for the adhesive to fill. The surface roughness and thereby mechanical retention is increased with increasing irregularity of the base (Kang et al. 2013). Even with ceramic brackets, which usually yield high bonding values, there can be notable differences in bond strength values because of different base designs (Ansari et al. 2016).

## 2.4 Adhesives and Light Curing

### 2.4.1 Light Curing Adhesives

Light curing adhesives used in orthodontic bonding are dimethacrylate based adhesive resins that contain monomers such as bis-GMA (bisphenol-A diglycidyl ether dimethacrylate) and TEGDMA (triethylene glycol dimethacrylate) (Figure 4). After etching the enamel with phosphoric acid, a liquid adhesive is applied on the

enamel. The liquid adhesive contains methacrylate monomers and a solvent, usually ethanol. The liquid adhesive fills the porosities on the enamel surface created by the acid etching, and excess solvent is evaporated by air drying. Then a more viscous adhesive cement is applied on the base of the bracket, and the bracket is placed on the enamel surface. The structure and length of the resin tag formation varies, but generally it reaches depths of 5-50  $\mu\text{m}$  (Gwinnett 1971, Fitzpatrick and Way 1977, Soetopo et. al. 1978, Shinci et. al. 2000, Pashley and Tay 2001, Fjeld and Øgaard 2006). However, the bond strength does not increase in proportion to the tag length (Shinci et. al. 2000). When SEPs are used, the tag length is shorter than with the use of phosphoric acid (Pashley and Tay 2001, Fjeld and Øgaard 2006).



**Figure 4.** Structure of bis-GMA (bisphenol-A-diglycidyl ether dimethacrylate) and TEGDMA (triethylene glycol dimethacrylate).

### 2.4.2 Light Curing

The adhesives are cured by a free radical polymerization reaction, which is initiated by photons delivered from a light source that emits blue light (wavelength 470 nm) to the photosensitive initiator-activator system in the adhesive. Irradiance expresses the power per unit surface area ( $\text{mW}/\text{cm}^2$ ), and with a constant irradiance, the total energy delivered will be determined by the exposure time. In the polymerization reaction, the double bonds in the methacrylate groups open and react with each other forming a cross-linked, solid polymer. The degree of double bond conversion is a measure of the cure of the resin (DC%, degree of cure), and is usually 55%-65%, indicating that about 60% of the double bonds have reacted. The DC% is a matter of interest, because it affects the mechanical properties (Ferracane and Greener 1986) and other qualities of the material, such as solubility of the acrylates (Eliades et al. 1995c). Monomer leaching is potentially harmful (Tell et al. 1988, Ahrari et al. 2010, Kloukos et al. 2013). It can occur when the appliance blocks the curing light and DC% remains low (Eliades et al. 2011, Sunitha et al. 2011). Highest risk for leaching

occurs when the monomer comes in direct contact with the oral environment, as can happen with lingual retainers and brackets (Eliades et al. 2007, Eliades et al. 2011).

The most commonly used type of light curing unit these days is a light emitting diode (LED), which has many advantages compared to the previously widely used halogen light. Although there are no differences between these light sources in terms of bracket failure (Fleming et al. 2013), there are other factors in favor of LEDs. Whereas the halogen light emits lots of energy as heat, which can damage the pulp, and only a small fraction in a wavelength of blue light, the heat produced by a LED is minimal (Fleming et al. 2013).

It has been concluded, that there is no lower limit to the irradiance ( $\text{mW}/\text{cm}^2$ ) needed to cure adhesives, since even with lower irradiance values, it is possible to attain satisfactory results by increasing the exposure time (Musanje and Darvell 2003). It has been stated that the required radiant exposure that is needed for stainless steel bracket bonding is  $12 \text{ J}/\text{cm}^2$  (Staudt et al. 2006), which can be achieved by multiple combinations of exposure times and irradiance values. However, issues concerning LCU design and their use can cause problems if they are not taken into consideration. A nonuniform beam profile of the LCU can cause very inhomogeneous light output with varying “hot spots” and “cold spots” across the tip area (Price et al. 2015, Price 2017). It should also be taken into consideration that as the distance between the LCU tip and the adhesive increases, the light diverges more and its radiant exposure decreases, which results in a reduced irradiance over the specific target area (Lindberg et al. 2005, Price 2017).

### 2.4.3 Other Adhesives

In addition to light curing dimethacrylate adhesives, chemically cured adhesives and adhesives of different composition, such as methacrylate, glass-ionomer (GIC), or resin-modified glass ionomer (RMGIC) cements are available. Light curing dimethacrylate adhesives are presently the most widely used option, although there is no clear large-scale evidence of their superiority to other adhesive types (Mandall et al. 2018). Glass-ionomer cements bond to enamel via calcium bridges, hydrogen bonds or van der Waals forces, but the bond strength remains lower than with resin adhesives. They were initially promoted because they release fluoride and were therefore thought to be cariostatic (Benson et al. 2005). Because the bond strengths of GICs were weak, resin-modified glass ionomer cements (RMGIC) were introduced. They contain components of both resin composites and conventional glass-ionomers, and bond via both polymerization and an acid-base reaction. It has been suggested, that although resin-modified glass-ionomer cement provides lower bond strengths compared to regular resin-based adhesives, these values would still be clinically sufficient (Summers et al. 2004).

## 2.5 Bracket Failure

Accidental debonding of brackets, referred to as bracket failure, is not uncommon. Reported failure rates vary between 0.6% and 17.6% (Elekdag-Turk et al. 2008a, Reis et al. 2008,) but are usually within the range of 3-8% (Hitmi et al. 2001, Adolfsson et al. 2002, Kula et al. 2002, Linklater and Gordon 2003, Murfitt et al. 2006, Pasquale et al. 2007, Khalha 2008, Romano et al. 2012, Ahangar Atashi and Shahamfar 2013, Hammad et al. 2013, Özer et al. 2014, Mohammed et al. 2016, Krishnan et al. 2017, Mavreas et al. 2018). Detachment of the brackets during the treatment prolongs the treatment time and requires more patient visits (Skidmore et al. 2006, Faruqi et al. 2018). Patient compliance can decrease as treatment progress is delayed, and this can compromise the success of the treatment.

Factors related to the patient that may potentially affect the longevity of the bond are e.g. the age, sex, facial anatomy and diet of the patient. Several studies have examined the effect of age and sex on the failure rate of brackets, but the results have been conflicting. While a few studies have concluded that age and sex play no role in bond failure rate (Khalha 2008, Atik et al. 2019), others have reported that males are more prone to bracket failures (Adolfsson 2002, Murfitt 2006, Hammad et al. 2013). Males have stronger biting forces than females, which could explain the higher failure rate, although the difference is not significant before puberty (Varga et al. 2011). In addition, the facial anatomy of the patient can play a role in bracket debonding, since people with a shorter face tend to have stronger occlusal forces than people with a longer face (Proffit et al. 1983). Furthermore, hard foods and acidic soft drinks have been reported to cause debonding (Önçağ et al. 2005).

The different bonding systems (conventional, SEP, GIC, RMGIC) and different bracket types affect the bonding success, with conventional acid etching technique and ceramic brackets usually exhibiting strongest bonds and least failures. Nevertheless, most available bonding systems are reported to yield clinically acceptable bond strengths of 6-8 MPa (Reynolds 1975). Ceramic brackets are usually found to exhibit stronger bond strengths and less failures (Hitmi et al. 2001), but some studies have reported better bonding success with metal brackets (Mirzakouchaki et al. 2016, Stasinopoulos et al. 2018).

Most studies attest that survival of brackets bonded with self-etching primers is equal to those bonded with conventional acid etching (Arnold et al. 2002, Aljubouri et al. 2004, Banks and Thiruvengkatachari 2007, Elekdag-Turk et al. 2008a, Khalha 2008, Reis et al. 2008, Cal-Neto et al. 2009, Romano et al. 2012, Dominguez et al. 2013, Özer et al. 2014, Atik et al. 2019). However, other studies have reported more failures when SEPs were used (Ireland et al. 2003, Elekdag-Turk, et al. 2008 b).



## 2.6 Methods to Improve Bonding

### 2.6.1 Transillumination

Because metal brackets block the curing light, it has been suggested that the DC% of the adhesive could be improved by curing the adhesive also from the palatal or lingual side through the teeth (Oesterle and Shellhart 2001). However, enamel and dentin significantly attenuate the intensity of light (Oesterle and Shellhart 2001, Turrioni et al. 2013, Uusitalo et al. 2016), with dentin absorbing more light than enamel (Thompson and Way 1981, Fried et al. 1995, Uusitalo et al. 2016). It seems possible that transillumination could successfully be used in the anterior teeth, which are thinner than posterior teeth, and only have a thin layer of dentin between the layers of enamel. On the other hand, transillumination can lead to pulp damage due to heat created by light emitted through the pulp, particularly if the achievement of sufficient radiant exposure would require prolonged curing times (Arikawa et al. 2004).

### 2.6.2 Glass Fibers

Glass fiber reinforced composites are used in many applications in dentistry, e.g. to enhance the strength and toughness of resin based materials and dentures (Vallittu 1999, Kim and Watts 2004, Vallittu 2018). Glass fibers could also be used to conduct and scatter light to improve the curing of resin based materials, especially when the light cannot reach the resin directly, as with metal brackets (Shinya et al. 2009, Durgesh 2015). Two wavelength-dependent coefficients of the glass fibers and the resin matrix that affect the light propagation and curing of the resin matrix, are the refractive index and the extinction coefficient. The refractive index describes how light is refracted by the material, and the extinction coefficient describes the attenuation of light per unit length. It has been shown, that polymerization increases the refractive index and decreases the extinction coefficient of dimethacrylate resin systems (Lehtinen et al. 2008, Vallittu 2015). Therefore, E-glass fibers and the dimethacrylate matrix enhance scattering of light from the glass fibers especially after the curing has begun and progressed (Lehtinen et al. 2008, Vallittu 2015).

### 2.6.3 Bracket Base Conditioning

Primers are compounds that are used in dentistry to achieve adhesion between dissimilar substrates that do not naturally form bonds with each other. Primers are substrate specific, which means that there are primers that are particularly suitable for certain ceramics or resin composites. However, despite of their substrate

specificity, a common and important property of all primers is the improvement of surface wettability, which enhances adhesion between two materials by allowing them to be in closer contact with each other. Using some primers and substrates chemical bonding can be also achieved, e.g. when using silane primers with glass ceramics. Silane based primers have also been utilized in improving the bonding with metal substrates, but there are also specific metal primers. Polymer composite substrates too have specific primers designated for bonding with them. Recently, so-called universal primers have been introduced, and they can be used with various types of substrates (Lung and Matinlinna 2012).

### 2.6.3.1 Silanes

Silanes are alcohols containing silicon (Si) in the chemical structure. They are effective in bonding organic materials, e.g. resin composite, to inorganic substrates, such as porcelain or glass-ceramics. There are several theories or models of the mechanism of action of silanes in improving the adhesion. These include the surface wettability model, the deformable layer model, the restrained layer model and the chemical bonding model (Rosen 1978). The chemical bonding model is most often mentioned to be the primary mechanism of action although the surface wettability model may also have a significant role in the adhesion. Prior to use the silane is hydrolyzed to form silanol ( $-\text{SiOH}$ ), which allows a polycondensation reaction between the hydroxyl groups of the ceramic surface and hydrolyzed silanol. Commercially available silanes can come as a pre-hydrolyzed one-bottle product, or in two bottles, one containing an unhydrolyzed silane monomer and the other aqueous acetic acid. Using the two-bottle system, the compounds are mixed immediately before use, which prevents a problem of excess formation of inactive siloxane polymers that takes place in the one-bottle system over time (Matinlinna et al. 2018).

In dentistry, the most commonly used silane is 3-methacryloyloxypropyltrimethoxysilane (MPS), which contains a methacrylate group and three alkoxy-groups attached to a Si-atom. To form a bond between resin composite and a substrate, the methacrylate group in the silane reacts with the methacrylate groups in the composite resin. Acidic silanol groups are formed by hydrolyzing the alkoxy groups in the silane, and these silanol groups then form bonds with the hydroxyl ( $-\text{OH}$ ) groups that are spontaneously emerged from the ambient moisture on the surface of the substrate, e.g. glass ceramic. (Matinlinna et al. 2004, Aboushelib et al. 2008, Lung and Matinlinna 2012). When bonding to substrates that contain silica, siloxane linkages ( $-\text{Si}-\text{O}-\text{Si}-$ ) are formed, and this is the strongest form of bond that is attainable with silanes. Weaker adhesion is achieved to metals ( $-\text{Si}-\text{O}-\text{M}-$ ). Silanes are not universal bonding agents, and they cannot sufficiently bond to

chemically more inert substrates, e.g. oxide ceramics such as fully crystallized zirconia (Kern and Wegner 1998). The surface of inert ceramics can however be conditioned to be chemically reactive with silanes, e.g. silane-based chemical bonding to zirconia is possible with tribochemical silica-coating, where silica-coated alumina powder is blasted and embedded into the substrate surface (Kern and Strub 1998, Matinlinna et al. 2006). In addition, other adhesion promoters such as organophosphate ester monomers (e.g. 10-methacryloyloxydecyldihydrogen-phosphate, MDP) can be used to improve bonding to oxide ceramics (Kern and Wegner 1998, Tanaka et al. 2008, Kitayama et al. 2010, de Souza et al. 2014). Other methods of further increasing the bond strength include improving the mechanical retention of the substrate, e.g. air abrasion or selective infiltration etching (Aboushelib et al. 2008). Poor hydrolytic stability is a problem with silane promoted bonding, and it can lead to bond deterioration over time (Aboushelib et al. 2009, Heikkinen et al. 2013), but the hydrolytic stability can perhaps be improved by adding a so-called non-functional silane in the mixture, which forms a cross-linking network with the functional silane, and thus stabilizes the system (Matinlinna et al. 2018).

The influence of silane on the base of ceramic brackets on bond strength is a question of interest. In previous studies, it has been reported that MPS does not have an influence on the bond strength of ceramic brackets (Özcan et al. 2008), and that MDP on the base of the bracket enhances bonding of ceramic brackets to ceramic substrates (Falkensammer et al. 2013).

### 2.6.3.2 Other Primers

To improve adhesion to polymer materials, composite primers can be used. They can interact either with the inorganic filler particles in the composite or by dissolution and polymerization of the polymer matrix. Typically, composite primers contain solvents and methacrylate monomers, along with photo initiators for achieving polymerization (Tezvergil et al. 2003, Perea et al. 2015). Achieving dissolution of the polymer substrate surface requires a linear structure of the polymer substrate, and therefore cross-linked polymers cannot be dissolved. The actual bonding is based on formation of an interpenetrating polymer network in the interface of the adhesive and the substrate (Vallittu 2009). Some composite primers are mixtures of monomers and silanes, but the function of the silane component is questionable because the silanes have been shown to be inactivated in the mixtures during the shelf-life time (Pilo et al. 2018).

If polycarbonate brackets contain glass fibers as fillers, their bond strength can be significantly improved by first exposing the fibers with air-particle abrasion (so-called sandblasting), and then adding silane as a coupling agent

(Guan et al. 2001), so that the silane can then bond with the OH-groups of the exposed glass substrate.

To improve the bond strength of metal brackets, the base can be e.g. air-particle abraded, microetched and silanized, and the use of a metal primer containing 4-META (4-methacryloxyethyl trimellitate anhydride) has also been found to improve bond strength (Siomka and Powers 1985, MacColl et al. 1998, Atsü et. al. 2006, Faltermeier and Behr 2009, Cal-Neto et al. 2013). To increase the amount of silica on the substrate's surface, air-particle abrasion can be made with silica coated particles of aluminium oxide. The method is called tribochemical silica coating and it is used with silane coupling agents (Özcan and Vallittu 2003, Bömicke et al. 2019, Emsermann et al. 2019).

## 2.7 Debonding

Once the orthodontic treatment is completed, the brackets need to be removed from the teeth. A strong bond between a tooth and a bracket is relevant for successful treatment, but at debonding an easy removal is desirable without causing damage to the enamel surface. When a bracket is removed, the breakdown of the bond can take place either at the interface between the bracket and the adhesive or between the adhesive and the enamel, or at both. After the bracket is debonded, adhesive remnants are removed from the enamel surface. Sometimes the enamel is damaged by the debonding procedures (Janiszewska-Olszowska 2014).

### 2.7.1 Adhesive Remnant Index (ARI)

The debonding type can be classified with adhesive remnant index (ARI) based on the amount of adhesive left on the enamel surface or on the bracket base after the removal of a bracket (Table 1). If most of the adhesive is left on the bracket, the clean-up is easy, but the risk of enamel damage during the detachment can increase. A break-off with most of the adhesive left on the enamel is therefore considered more favorable (Bishara and Fehr 1997), and this is usually the prevalent type (Theodorakopoulou et al. 2004, Kitahara-Céia et. al. 2008, Suliman et al. 2015). Some studies have reported lower ARI scores with self-etching primers indicating that less residual adhesive remained on the enamel compared to conventional bonding system (Bishara et al. 1998, Velo and Carano 2002, Cal-Neto et al. 2006c).

**Table 1.** Adhesive remnant index (ARI), definition of scores. From original publication III.

<b>Score</b>	<b>Definition</b>
<b>0</b>	No adhesive remained on enamel
<b>1</b>	Less than 50% of adhesive remained on enamel
<b>2</b>	More than 50% of adhesive remained on enamel
<b>3</b>	All adhesive remained on enamel

## 2.7.2 Enamel Damage

It is possible that during the processes of preparing the enamel, bonding and debonding the brackets, and cleaning up the enamel surface, small amounts of enamel are lost.

Before etching, the enamel surface is usually cleaned with a pumice paste to remove the organic pellicle on the tooth surface. The pumicing can be done with a bristle brush or a rubber cup, the effects between these two methods being slightly different. With a rubber cup, the amount of enamel lost during pumicing is 3-7  $\mu\text{m}$  while with a bristle brush it exceeds 10  $\mu\text{m}$  (Pus and Way 1980, Thompson and Way 1981, Hosein et. al. 2004). In addition to creating a porous surface, acid etching can partly remove the top layer of enamel. With the commonly used combination of 32-37% phosphoric acid and 15 s etching time, the amount of enamel lost is 1-5  $\mu\text{m}$ . Prolonging the etching time increases the amount of lost enamel, e.g. with 120 s etching time the enamel loss is approximately 12  $\mu\text{m}$  (Pus and Way 1980, Hermesen and Vrijhoef 1993, Hosein et. al. 2004). Self-etching primers cause less enamel loss than phosphoric acid (Hosein et. al. 2004).

Furthermore, enamel damage can occur when debonding brackets. This has especially been linked to ceramic brackets (Joseph and Rossouw 1990, Årtun 1997, Gittner et al. 2012), although with modern brackets the risk seems to be low (Chen et al. 2007). It has been suggested that enamel damage is less likely if the debonding is carried out with laser (Dostalova et al. 2016). In addition, debonding using a compressive rather than shearing force might diminish the chance of enamel damage

because of a more favorable stress distribution in the enamel (Holberg et al. 2014). However, Özcan et al. (2008) found no difference in bracket failure when debonding using pliers was compared to shearing off the brackets with a testing machine (Özcan et al. 2008). Bishara et al. (1994) found no difference in ARI scores, even if debonding with pliers required 30% less force than debonding with a testing machine (Bishara et al. 1994). Elekdag-Turk et al. (2009) found more ARI scores of 3, e.g. all of the adhesive remaining on enamel after debonding, when the brackets were debonded with pliers instead of with a testing machine.

After removing brackets, the clean-up of remaining adhesive can potentially cause enamel damage. Different techniques and burs can be used in the cleaning, but they all tend to remove small amounts of enamel along with the adhesive remains. Cleaning the adhesive usually removes approximately 5-30 µm of enamel (Thompson and Way 1981, Øgaard and Fjeld 2010, Suliman et al. 2015), but the exact amount depends on the method. Enamel loss up to 40-60 µm has been reported (Fitzpatrick and Way 1977, Brown and Way 1978), but with a carbide finishing bur used at a low speed, the damage can be limited to 3-11 µm (Pus and Way 1980, Hosein et al. 2004). Fractures of ceramic brackets can lead to severe enamel damage if a piece remains on the tooth because due to its hardness, it can only be removed with a diamond bur.

## 2.8 Rebonding

Bracket failure during the treatment creates a need for rebonding of the bracket. A new bracket is bonded with the same technique as the initial bonding but typically a small amount of enamel is lost during the cleaning process and when the enamel is re-etched. The generated etching pattern can be different compared to the initial etching, since the structure of the surface enamel varies depending on whether the enamel is intact or instrumented.

The properties of a previously treated enamel surface can affect the bonding strength when a new bracket is rebonded after a failure. The rebonding strength can be diminished by remaining adhesive because it will decrease the roughness of the enamel surface (Bishara et al. 2000, Bishara et al. 2002). On the other hand, it has been suggested that the adhesive remnants could provide a surface for the new adhesive to bond to, chemically or mechanically (Montasser et al. 2008). In previous studies, the bond strength values achieved in rebonding have been inconsistent: some studies have found the rebonding strength to be lower than the initial bond strength (Bishara et al. 2000, Bishara et al. 2002, Oshagh et al. 2013), whereas others have reported higher rebonding values, and attributed this to increased enamel roughness created by the removal of the residual adhesive (Eminkahyagil et al. 2006).

## 3 Aims

### 3.1 Objectives of the Study

The aim of the studies presented here was to investigate the interfaces between orthodontic brackets, adhesive and enamel. Particular interests were to study the frequency of bracket failure *in vivo* and to examine methods to improve bonding in orthodontic treatment and aspects which relate to the bonding properties. Working hypothesis of the study was that by using certain methods, the curing of the adhesives and the bond strength of brackets to enamel can be improved. Specific objectives were:

- I To study the influence of a bidirectional continuous glass fiber weave in the resin interface under a metallic bracket on the DC% of the adhesive, and to investigate if transillumination through teeth is possible and efficient for initiating and completing the curing of the adhesives.
- II To investigate the effect of enamel removal before rebonding on the rebonding strength and etching patterns of enamel.
- III To examine the fracture pattern and debonding strength, and to compare the magnitude of enamel damage when debonding metal, ceramic and polycarbonate brackets bonded using different primers.
- IV To determine the frequency of bracket failure *in vivo*, and to study factors affecting failure risk during treatment.

## 4 Materials and Methods

The adhesion of brackets to enamel and the interfaces between the two were studied in three laboratory studies, and the failure rate of brackets was investigated in a clinical study.

The degree of cure (DC%) of two light curing orthodontic adhesives (Enlight and Transbond™ XT) was measured under a metal bracket and under pieces of metal dental matrices simulating larger appliances. In half of the specimens, a bidirectional E-glass-fiber weave (thickness of 0.06 mm) with light curing bis-GMA-PMMA resin matrix system (everStick® NET) was used under the bracket/matrix. The other half of the specimens were studied without the glass-fiber weave. In addition, the efficiency of initiation and completion of light curing through teeth was examined (Study I).

The effect of removing a small amount of enamel on the rebonding strength of orthodontic adhesive (Transbond™ XT) was studied. The bonding surfaces were also analyzed with an optical non-contacting profiler and a scanning electron microscope (SEM) (Study II).

The effect of different primers on the base of brackets on the bond strength to enamel was investigated. Brackets of three different materials (stainless steel, zirconia-alumina ceramic and polycarbonate) were bonded to enamel using bracket material specific and universal primers on the bracket base, with orthodontic adhesive (Transbond™ XT). The bond strength values were measured and the bonding surfaces were analyzed with an optical non-contacting profiler, stereomicroscope and a scanning electron microscope (SEM) (Study III).

The failure rate of brackets in clinical practice was studied in a retrospective cohort study. Patient files of 215 patients who had been treated with fixed appliances in the city of Turku, Finland, were examined. Data was gathered on the following variables: bonding time, bonding system (primer + adhesive), the sex of the patient, the position of the bracket in terms of jaw and tooth, and the date of failure (Study IV).

The materials used in these studies are listed in Table 2.



**Table 2.** Materials used in the studies (wt-% weight percentage). Modified from original publication III.

Material	Manufacturer	Lot No.	Contents	Wt-%
<b>Enlight Adhesive</b>	Ormco (Orange, CA, USA)	3733229	Bis-GMA 3-trimethoxysilylpropyl methacrylate	10-30* 1-5*
<b>Transbond XT Light Cure Adhesive</b>	3M Unitek (Monrovia, CA, USA)	N765918, N568393, FT2IU	Silane treated quartz Bis-GMA EBPADMA Silane treated silica Diphenyliodonium hexafluorophosphate	70-80* 10-20* 5-10* <2* <0.2*
<b>Transbond XT primer</b>	3M Unitek (Monrovia, CA, USA)	N762529	Bis-GMA TEGDMA Triphenylantimony 4-(dimethylamino)-benzeneethanol DL-camphorquinone Hydroquinone	45-55* 45-55* <1* <0.5* <0.3* <0.03*
<b>RelyX™ Ceramic Primer</b>	3M™ ESPE™ (St. Paul, MN, USA)	N759704	Ethyl alcohol Water Methacryloxypropyltrimethoxysilane	70-80* 20-30* <2*
<b>Monobond Plus</b>	Ivoclar Vivadent, (Schaan, Liechtenstein)	V12120	Ethanol 3-trimethoxysilylpropyl-methacrylate Methacrylated phosphoric acid ester	50-100 ≤2.5 ≤2.5
<b>Adper™ Scotchbond™ Multi-Purpose Adhesive</b>	3M™ ESPE™ (St. Paul, MN, USA)	N735458	Bis-GMA HEMA Triphenylantimony	60-70* 30-40* <0.5*
<b>GC Composite Primer</b>	GC (Hongo, Bunkyo-ku, Tokyo, Japan)	1604061	HEMA Tetrahydrofurfuryl methacrylate UDMA	30-60* 10-30* 10-30*
<b>everStick® NET</b>	Stick Tech Ltd (Turku, Finland)	120614	Bis-GMA DMAEMA Hydroquinone	25-50* 0.1-0.5* <0.1*
<b>Scotchbond™ Universal Etchant 32%</b>	3M™ ESPE™ (St. Paul, MN, USA)	626002, 576331	Water Phosphoric acid Poly(vinyl alcohol)	50-60* 30-40* 5-15*
<b>Ortomat Minimot bracket</b>	Ormco (Glendora, CA, USA)	15M378M, 13K480K	Stainless steel	
<b>ICE bracket</b>	Ormco (Glendora, CA, USA)	081650467	Single crystal aluminum oxide Zirconium oxide	
<b>Spirit MB bracket</b>	Ormco (Glendora, CA, USA)	081612367	Filler reinforced polycarbonate	

Bis-GMA indicates bisphenol-A glycidyl methacrylate, EBPADMA bisphenol-A-bis(2-hydroxyethyl ether) dimethacrylate, TEGDMA Triethylene glycol dimethacrylate, HEMA 2-hydroxyethyl methacrylate, and UDMA Urethane dimethacrylate. Methacryloxypropyltrimethoxysilane and 3-trimethoxysilylpropylmethacrylate are different names used by the manufacturers for the same compound.

## 4.1 Measuring the Degree of Cure (DC%) (I)

To measure the DC% of two orthodontic adhesives (Enlight and Transbond™ XT), a stainless steel bracket along with pieces of stainless steel dental matrices were used in the testing. The matrices were hand cut in four different sizes: 1) equal to the size of the bracket used in the study, 2) 0.5 mm larger, 3) 1.0 mm larger, and 4) 2.0 mm larger, in all directions, than the bracket. The groups are shown in Table 3. The matrix group m0 was included to ensure that a matrix with the same size shielded curing light in a similar way to the bracket.

**Table 3.** The groups in the study: the bracket (br) and the matrix pieces with different sizes (m0, m0.5 m1 and m2). From original publication I.

Code	br	m0	m0.5	m1	m2
<b>Dimensions (mm)</b>	3.1x4.2	3.1x4.2	4.1x5.2	5.1x6.2	7.1x8.2

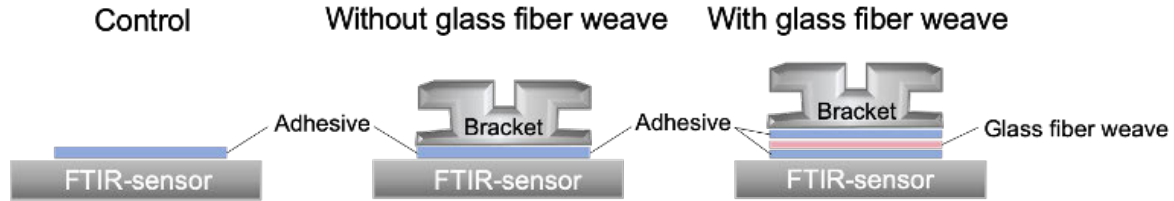
The DC% of the adhesives was measured using a Fourier Transform Infrared Spectrometer (Frontier™ FTIR, PerkinElmer®, Beaconsfield Bucks, UK) with a universal attenuated total reflectance (ATR) sampling accessory. The program with which the samples were analyzed was Spectrum™ (v. 10.4.2, PerkinElmer®). The scans were performed at seven different time points: the first scan before the light curing, the second immediately after the curing and after that every three minutes (0 s, 40 s, 3 min, 6 min, 9 min, 12 min, 15 min).

First, as a control, the DC% of both adhesives was measured without a bracket or a piece of matrix. A small amount of adhesive (thickness 1.0 mm) was applied on the FTIR sensor (ZnSe-crystal, Ø 3.1 mm) and light cured for 40 s with a handheld light curing unit (light-emitting diode, Elipar™ S10, 3M ESPE, St. Paul, MN, USA, intensity 1880 mW/cm<sup>2</sup>) directly above the specimen.

Secondly, the DC% of both adhesives in the five test groups were measured without a glass fiber weave. A small amount of adhesive was applied on the sensor, the bracket/matrix was placed firmly on the adhesive, and the excess adhesive was carefully removed. The adhesive was cured from two sides, 20 s for each.

Thirdly, a glass fiber weave was placed in the adhesive interface before measuring the DC%. A small amount of adhesive was applied on the sensor, a piece of the weave was placed on top of the adhesive, a small amount of adhesive was applied on the bottom of the bracket/matrix, and the bracket/matrix was firmly

pressed on the glass fiber weave (Figure 5). The excess adhesive was removed. The adhesive was cured from two sides, 20 s each. Overall, both adhesives were tested in five different groups with five specimens in each, with and without the glass fiber weave.



**Figure 5.** A schematic representation of the test setting for measuring the DC%: control group, adhesive under the bracket without the glass fiber weave and adhesive under the bracket with the glass fiber weave in the adhesive interface.

The DC% was calculated from the aliphatic C=C peak ( $1638\text{ cm}^{-1}$ ) and the aromatic C=C peak ( $1608\text{ cm}^{-1}$ ) using Equation 1.

$$DC\% = \left[ 1 - \frac{C_{aliphatic} / C_{aromatic}}{U_{aliphatic} / U_{aromatic}} \right] \times 100\%$$

$C_{aliphatic}$  = absorption peak at  $1638\text{ cm}^{-1}$  of the cured sample

$C_{aromatic}$  = absorption peak at  $1608\text{ cm}^{-1}$  of the cured sample

$U_{aliphatic}$  = absorption peak at  $1638\text{ cm}^{-1}$  of the uncured sample

$U_{aromatic}$  = absorption peak at  $1608\text{ cm}^{-1}$  of the uncured sample

**Equation 1.** The equation used for calculating the DC% of the adhesives.

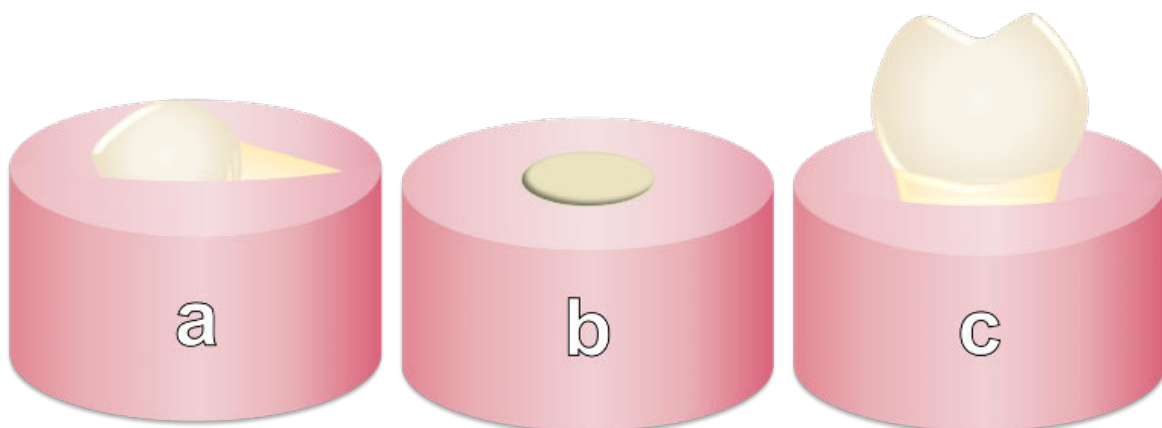
The transmittance of light through dentin was measured with MARC®-spectrometer for determining light irradiation power and analyzed with BlueLight®-program (MARC® Resin Calibrator, BlueLight® analytics inc., Halifax, Nova Scotia, Canada). A slice of dentin was placed on the spectrometer sensor ( $\varnothing 4.0\text{ mm}$ ) and the tip of the light curing unit was placed on the slice. The transmitted light was measured for 20 s. The maximum irradiance of the light curing unit (light-emitting diode, Elipar™ S10, 3M ESPE, St. Paul, MN, USA) was  $1845\text{ mW/cm}^2$  and the wave-length peak according to the manufacturer is  $455\text{ nm} \pm 10\text{ nm}$ .

## 4.2 Preparing the Specimens for Bond Strength Testing (I, II, III)

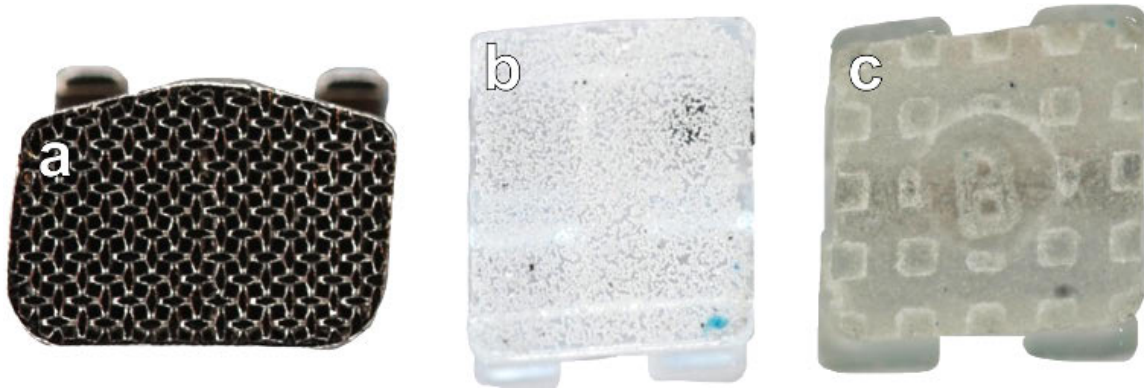
The teeth used in the studies were extracted molars acquired from the Dental Teaching Clinic (Oral and Dental Health Care) at Turku, Finland. According to Finnish legislation (Act on the Medical Use of Human Organs, Tissues and Cells), tissue samples taken for therapeutic or diagnostic purposes can be used for medical research with permission from the health care unit for whose activities the sample was taken, as long as no personal data are used in the surrender or use situation. The teeth were stored in Chloramine-T after extraction and in distilled water after cleaning before testing.

The molars used for testing the transmittance of light were cut with a histological saw (Secotom-50, Struers A/S, Ballerup, Denmark) into vertical slices of different thicknesses (0.5 mm, 1.0 mm, 1.5 mm, 2.0 mm, 3.0 mm and 4.0 mm), five specimens in each group. The slices were stored in distilled water.

The teeth used for bond strength testing were embedded in acrylic blocks, horizontally for Study II and vertically for Study III. In the horizontally embedded specimens, a circular area (minimum diameter 3.6 mm) of enamel was exposed and polished with a polishing machine (LaboPol-21, Struers A/S, DK-2750 Ballerup, Denmark), using two SiC papers: first a 180-grit (FEPA) to remove a bulk of enamel to get a wide enough surface for the bonding, then a 2400-grit to finish and smooth the rough surface. A control group (n=10) of horizontally embedded teeth was also prepared, where the teeth were half-embedded in acrylic resin with intact enamel exposed on the surface (Figure 6). While the horizontally embedded teeth were ground, the vertically embedded teeth were left intact and cleaned with pumice prior to bonding.



**Figure 6.** Prepared specimens: **a)** horizontally embedded group of intact enamel (Study II) **b)** horizontally embedded with a smooth circular area of ground enamel (Study II) and **c)** vertically embedded tooth (Study III).



**Figure 7.** Bonding surfaces of the brackets used in Study III: **a)** metal, **b)** ceramic and **c)** polycarbonate. The surface area of the bracket bases was 13.64 mm<sup>2</sup> for the metal bracket, 13.22 mm<sup>2</sup> for the ceramic bracket and 13.83 mm<sup>2</sup> for the polycarbonate bracket.

### 4.3 Bonding (II, III)

The bonding surfaces of the teeth were etched for 15 s using a 32% phosphoric acid etching gel, thoroughly rinsed with water and air-dried. For the horizontally embedded specimens, a cylindrical (height 5 mm, Ø 3,6 mm) mold was placed on the enamel, the adhesive was dispensed into the mold and light cured for 60 s (20 s from above and 20 s from two sides) with a handheld light curing unit (light-emitting diode, Elipar<sup>TM</sup> S10, 3M ESPE, St. Paul, MN, USA), with the irradiance of 1834.8 mW/cm<sup>2</sup> and the wavelength peak of 455 nm  $\pm$  10 nm according to the manufacturer. After the plastic mold was removed, the specimens were stored in distilled water in 37 °C for four hours. The teeth in the control group of intact enamel were cleaned with pumice, rinsed with water, air-dried and then etched and bonded with the same procedures as the other specimens (Study II).

With the vertically embedded molars, three different types of upper central incisor brackets were used: Inspire ICE by Ormco (a ceramic monocrystalline aluminum oxide bracket with a base covered in small zirconia spheres), Spirit MB by Ormco (a filler reinforced polycarbonate bracket), and Ortomat Minimat by Ormco (a stainless steel bracket) (Figure 7). Molars were used because they are more readily available compared to incisors, but they were chosen so that their bonding surfaces were as flat as possible to simulate the flat surface of incisors. The brackets of each material were divided in three groups (n=10) according to the primer used in the bonding procedure (a control group with no primer and two test groups with different primers). In the context of this study, the word “primer” also refers to the different compounds used here on the base of the bracket, in addition to the standard use of an adhesive primer on the enamel surface. The different primers were selected to match the bracket types based on their universal affinity or material specificity. The primers were a silane based primer (RelyX<sup>TM</sup> Ceramic Primer) and a universal



primer (Monobond Plus) for ceramic and metal brackets, and an adhesive resin (Adper™ Scotchbond™ Multi-Purpose Adhesive) and a composite primer (GC Composite Primer) for polycarbonate brackets. Two different adhesion promoters, methacryloxypropyltrimethoxysilane (MPS) of the silane based primer, and methacrylated phosphoric acid ester (MDP) combined with MPS of the universal primer, were used with ceramic and metal brackets due to their ability to bond with multiple types of substrates. For the polycarbonate brackets, composite primer is specifically aimed at bonding between composite substrates, and the adhesive resin was chosen since a similar solubility parameter between polycarbonate and Bis-GMA could allow the primer to dissolve and penetrate into the polycarbonate. A control group with no primer was used with all bracket types. The brackets and primers used in the study are listed in Table 4.

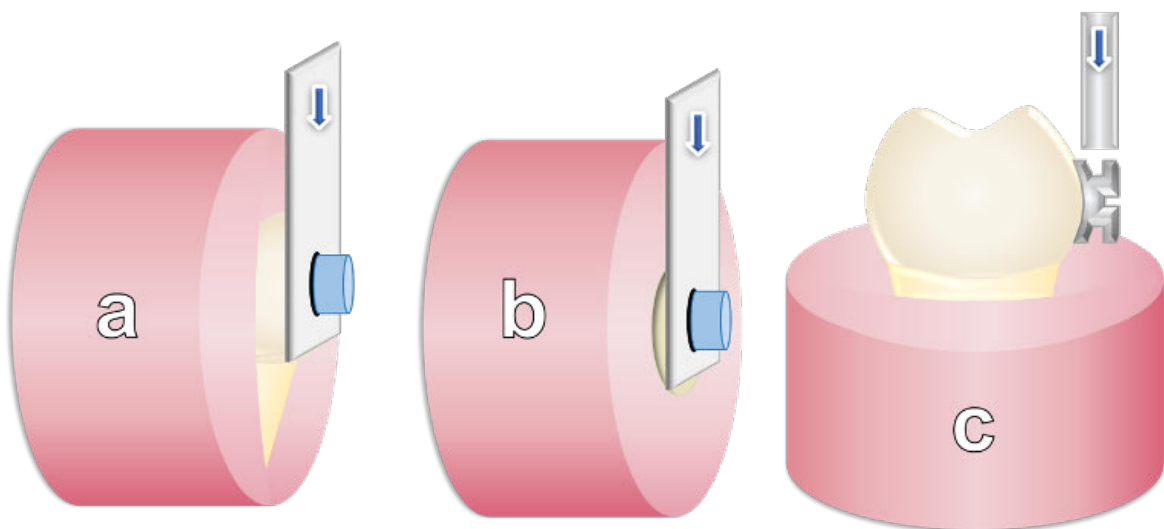
The selected primer was applied on the base of the bracket and air dried or light cured according to the manufacturer's instructions (Table 4). Then Transbond™ XT primer was applied on the enamel, a small amount of Transbond™ XT adhesive was applied on the bracket base and the bracket was placed firmly on the enamel. Excess adhesive was removed with an instrument and the adhesive was light cured for 10 seconds (5 s from both sides) according to the manufacturer's instructions. The specimens were stored in distilled water at 37° C for 7 days (Study III).

**Table 4.** Test groups in Study III, primers and the primer applying procedure. From original publication III.

Bracket type	Primer applied to the base of the bracket	Primer applying procedure
<b>Metal</b>	No Primer (n=10)	-
	Silane (RelyX™ Ceramic Primer) (n=10)	Gentle air drying
	Universal Primer (Monobond Plus) (n=10)	Gentle air drying
<b>Ceramic</b>	No Primer (n=10)	-
	Silane (RelyX™ Ceramic Primer) (n=10)	Gentle air drying
	Universal Primer (Monobond Plus) (n=10)	Gentle air drying
<b>Polycarbonate</b>	No Primer (n=10)	-
	Adhesive Resin (Adper™ Scotchbond™ Multi-Purpose Adhesive) (n=10)	Gentle air drying, 10s light curing
	Composite Primer (GC Composite Primer) (n=10)	Gentle air drying, 20s light curing

#### 4.4 Bond Strength Testing (II, III)

After the bonding procedures, all the specimens for bond strength testing were debonded with static loading using a testing machine (LLOYD Instruments, AMETEK Lloyd Instruments Ltd, West Sussex UK) with so-called shear-bond strength test with cross-head speed of 1 mm/min. Load-displacement curves were recorded. Testing was made in air at room temperature. In the specimens with the cylindrical adhesive, the tip of the testing blade had a round slot that was placed around the adhesive near the enamel, and in the specimens with the brackets the tip of the testing blade was positioned above the bracket wings close to the bracket base (Figure 8).



**Figure 8.** Schematic representation of the positioning of the test blade: **a)** cylindrical adhesive on intact enamel in Study II, **b)** cylindrical adhesive on ground enamel in Study II and **c)** bracket in Study III.

After debonding the specimens with the cylindrical adhesive on ground enamel (Study II), they were stored overnight in distilled water in 37° C. The next day the specimens were divided into four groups (n=10): one group was left untouched, and in the other three groups a small amount of enamel was ground off with an automatic polishing machine (RotoPol-11, Struers A/S, Pederstrupvej 84, DK-2750 Ballerup, Denmark) using a 4000-grit SiC paper (Struers A/SDK-2750 Ballerup, Denmark). All of the groups underwent grinding with the same settings (4000-grit SiC paper, 150 RPM, 5 N), but with different grinding times: 10 s, 20 s and 30 s. The groups and their treatments can be seen in Table 5. After the grinding, all the enamel substrates were etched and bonded again with the previously described procedures, stored for four hours and tested for bond strength again with the testing machine. The amount of enamel that was ground off was determined with additional

measuring-samples: five substrates were each ground for 10, 20 and 30 seconds and measured. The samples were measured with a micrometer (Coolant Proof Micrometer, Mitutoyo Corporation, Japan). Every sample was measured five times to avoid error, and an average thickness was calculated for every sample. The average amount of enamel that was removed was 7  $\mu\text{m}$  ( $\pm 2$ ) for 10 s, 12  $\mu\text{m}$  ( $\pm 1$ ) for 20 s and 16  $\mu\text{m}$  ( $\pm 3$ ) for 30 s. All procedures were performed by the same operator.

**Table 5.** Study II, test groups and their treatments. Modified from original publication II.

<b>Group</b>	<b>Treatment</b>
<b>E</b> (n=10)	Intact enamel, etched and bonded
<b>G0</b> (n=10)	Ground flat enamel surface, etched, bonded and debonded, then re-etched and rebonded
<b>G1</b> (n=10)	Ground flat enamel surface, etched, bonded and debonded, ground for 10 s, then re-etched and rebonded
<b>G2</b> (n=10)	Ground flat enamel surface, etched, bonded and debonded, ground for 20 s, then re-etched and rebonded
<b>G3</b> (n=10)	Ground flat enamel surface, etched, bonded and debonded, ground for 30 s, then re-etched and rebonded

## 4.5 Bonding Surface Analysis (II, III)

After the testing, the specimens with the debonded brackets were analyzed, and adhesive remnant index (ARI) and enamel damage were determined using a stereomicroscope (Wild 3MZ stereomicroscope, Wild Heerbrugg, Geis, Switzerland) (Study III).

The morphology of enamel surfaces and the bracket bases were also imaged for visual analysis using a scanning electron microscope (SEM, JSM-5500, Jeol USA, Inc., Peabody, MA). The substrates were gold-sputtered and imaged. A few enamel substrates of interest of different treatments were selected for examining from the prepared groups. One sample was cut and imaged in transverse section, to observe the depth of the resin tags.

Surface roughness of the enamel specimens and the bracket bases was determined by optical non-contacting profiler (Contour-GT-K1, Bruker, Billerica, MA, USA), and analyzed with a Bruker Vision 64 software (version 5.41, up-date 4, Bruker, Billerica, MA, USA). Microroughness of the enamel surface was reported



as average surface roughness (Ra), and the surface texture of the bracket bases was evaluated.

## 4.6 Patient Data Collection (IV)

To study the clinical failure rate of brackets, files of patients who had been treated with fixed appliances were examined. The study cohort consisted of patients treated in the Health Center of the City of Turku by three experienced orthodontists who had bonded the brackets themselves instead of referring them to dental hygienists. Patients that had brackets bonded on primary teeth, had only partial appliances ( $\leq 6$  brackets/jaw), or had inadequate information of the number of brackets or the bonding procedures were excluded from the study.

Records of the 215 patients included in the study were examined and the following information was collected: the age and sex of the patient, the number of brackets, the teeth the brackets were bonded on, the dates when the treatment started and ended, the name of the orthodontist, the primer and the adhesive used, the number of the brackets that failed during the treatment, and the date of failure (the appointment when the failure was observed).

Altogether, the patients in the study had 3801 brackets. Of the patients, 99 were males and 116 females, aged 9.3-19.1 years (mean 13.2 years, SD 1.65) at the beginning of the treatment. The adhesives and primers that were used in bonding with these patients were GreenGloo™ Light Cure Adhesive with Ortho Solo™ primer (bonding system 1), Transbond™ XT Light Cure Adhesive with Transbond™ XT primer (bonding system 2), GreenGloo™ Orthodontic Adhesive with Adper™ Prompt™ L-Pop™ Self-Etch Adhesive (bonding system 3), and Enlight™ Light Cure Adhesive with Ortho Solo™ primer (bonding system 4). The number of brackets bonded with each bonding system can be seen in Table 10 in Results.

## 4.7 Statistical Analysis (I-IV)

Statistical analyses were performed with SPSS Statistics versions 22.0 and 25.0. and SAS version 9.4.

The data from the FTIR was analyzed with a three-way analysis of variance (ANOVA), with Tukey's HSD test. The transmittance of light was analyzed with a Kruskal-Wallis test and regression curve estimation according to the thickness of the dentine discs (Study I).

To explore differences in rebonding strength after different amounts of ground enamel, a one-way ANOVA was performed with a Tukey's post-hoc test. Regression

analysis was used to demonstrate correlation between grinding time i.e. removal of enamel layer before rebonding and the rebonding strength (Study II).

To compare the bond strength of brackets with different primers, statistical analysis was performed using Kruskal-Wallis test and Regression analysis (Study III).

The number of bracket failures was calculated for sex, jaw, orthodontist and bonding system. Bracket survival distributions with respect to the different variables were compared with Cox regression proportional hazards model with shared frailty, the patient. Due to the fact that the orthodontists largely used different bonding systems, the variables of orthodontist and bonding system was analyzed in separate models. Analyses were conducted using SAS statistical software (SAS Institute Inc., Cary, NC, USA) (Study III).

For all tests, the significance level was  $p < 0.05$ .

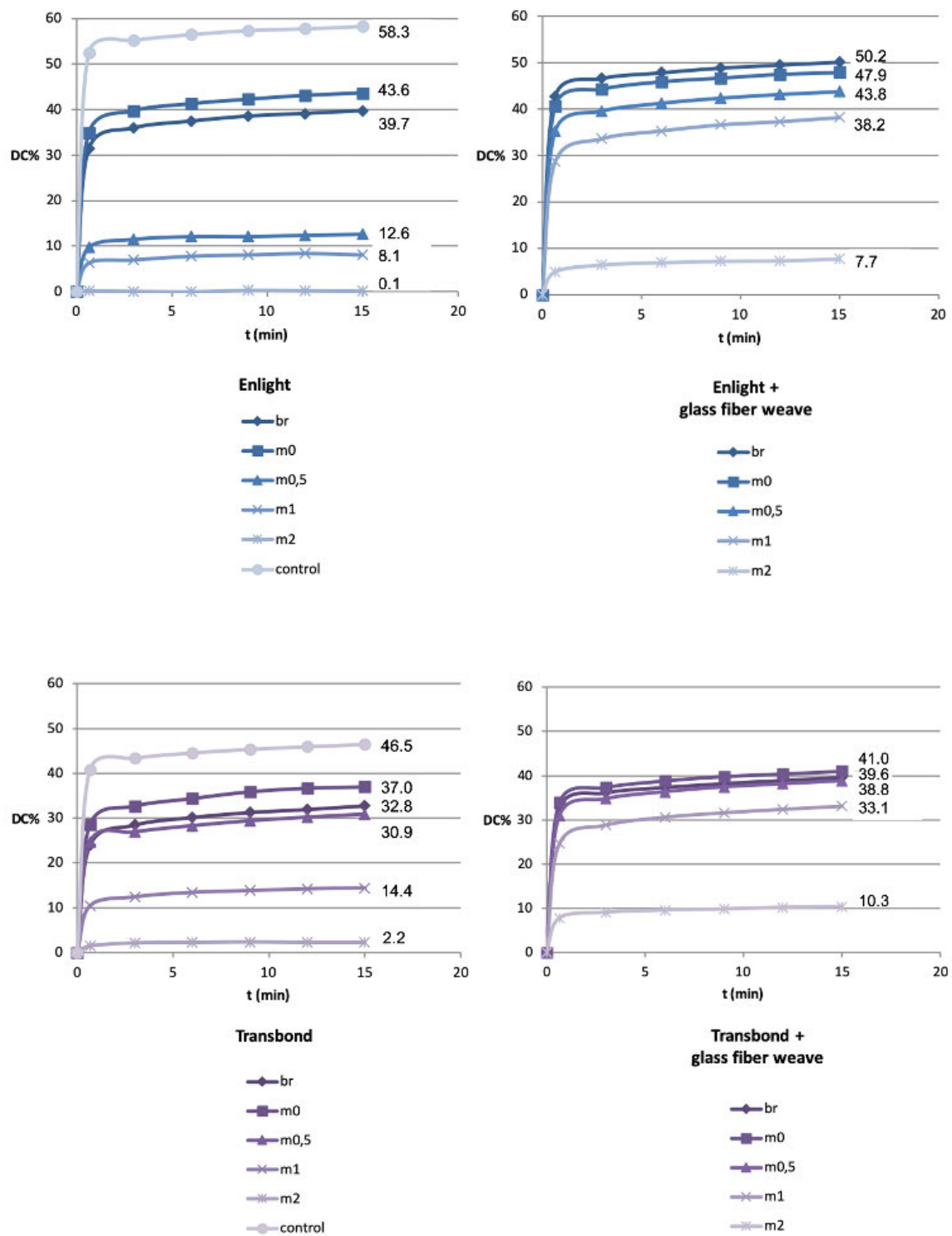
## 5 Results

### 5.1 Degree of Cure (I)

The DC% of adhesive under brackets and metal matrices remained low compared to the control, i.e. direct light curing of the resin. The presence of the glass fiber weave increased the DC% of both adhesives in all groups. The mean DC% values at 15 minutes time point can be seen in Table 6. The three-way ANOVA revealed significant differences in DC% at 15 minutes time point in all the factors and their interactions (Table 7). Tukey's post hoc test showed statistically significant differences between all other groups, but not between br- and the m0-groups, which confirms the success of attempting to simulate orthodontic brackets with the matrix pieces. The changes in the DC% plotted against time from the beginning of curing are shown in Figure 9.

**Table 6.** Mean degree of cure and standard deviation (DC% (SD)) of adhesives Enlight and Transbond™ XT at the 15 min time point with and without the glass fiber weave. The test groups: control) adhesive without a bracket/matrix, br) the bracket, the matrix groups: m0) equal to the size of the bracket used in the study, m0.5) 0.5 mm larger, m1) 1.0 mm larger, and m2) 2.0 mm larger, in all directions, than the bracket. See Table 3. for the exact sizes. From original publication I.

	Enlight			Transbond™		
	DC% without glass fiber weave	DC% with glass fiber weave	Difference (percentage points)	DC% without glass fiber weave	DC% with glass fiber weave	Difference (percentage points)
<b>Control</b>	<b>58.3</b> (±1.0)			<b>46.5</b> (±1.4)		
<b>br</b>	<b>39.7</b> (±2.8)	<b>50.2</b> (±2.3)	<b>10.5</b>	<b>32.7</b> (±2.3)	<b>39.6</b> (±1.4)	<b>6.8</b>
<b>m0</b>	<b>43.6</b> (±2.6)	<b>47.9</b> (±2.9)	<b>4.3</b>	<b>37.0</b> (±1.4)	<b>40.9</b> (±2.1)	<b>3.9</b>
<b>m0.5</b>	<b>12.6</b> (±6.4)	<b>43.8</b> (±3.5)	<b>31.2</b>	<b>30.8</b> (±1.0)	<b>38.8</b> (±1.4)	<b>7.9</b>
<b>m1</b>	<b>8.1</b> (±9.0)	<b>38.2</b> (±6.3)	<b>30.1</b>	<b>14.4</b> (±3.8)	<b>33.1</b> (±1.7)	<b>18.7</b>
<b>m2</b>	<b>0.1</b> (±0.9)	<b>7.7</b> (±10.0)	<b>7.6</b>	<b>2.2</b> (±3.1)	<b>10.4</b> (±11.9)	<b>8.2</b>



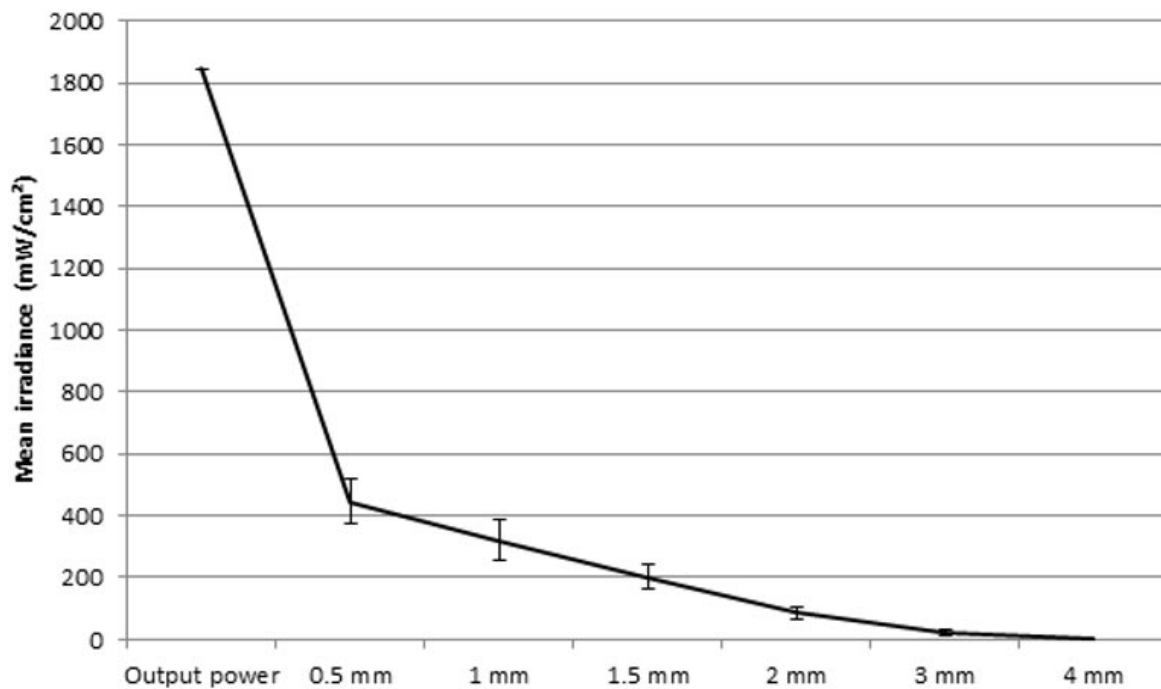
**Figure 9.** Mean degree of conversion (DC%) of different test groups of adhesives Enlight and Transbond™ XT, with and without the glass fiber weave, plotted against the curing time. Modified from original publication I.

**Table 7.** Results of the three-way ANOVA on the effects of the adhesive, glass fiber weave and bracket/matrix group on DC%. Large F and small p indicate statistical significance. From original publication I.

	<b>F</b>	<b>p</b>
Adhesive	1.49	0.226
Glass fiber weave	171.02	<0.001
Bracket/matrix	188.23	<0.001
Adhesive*Glass fiber weave	14.95	<0.001
Adhesive*Bracket/matrix	8.53	<0.001
Glass fiber weave*Bracket/matrix	15.09	<0.001
Adhesive*Glass fiber weave*Bracket/matrix	5.06	0.001

## 5.2 Transmittance of Light (I)

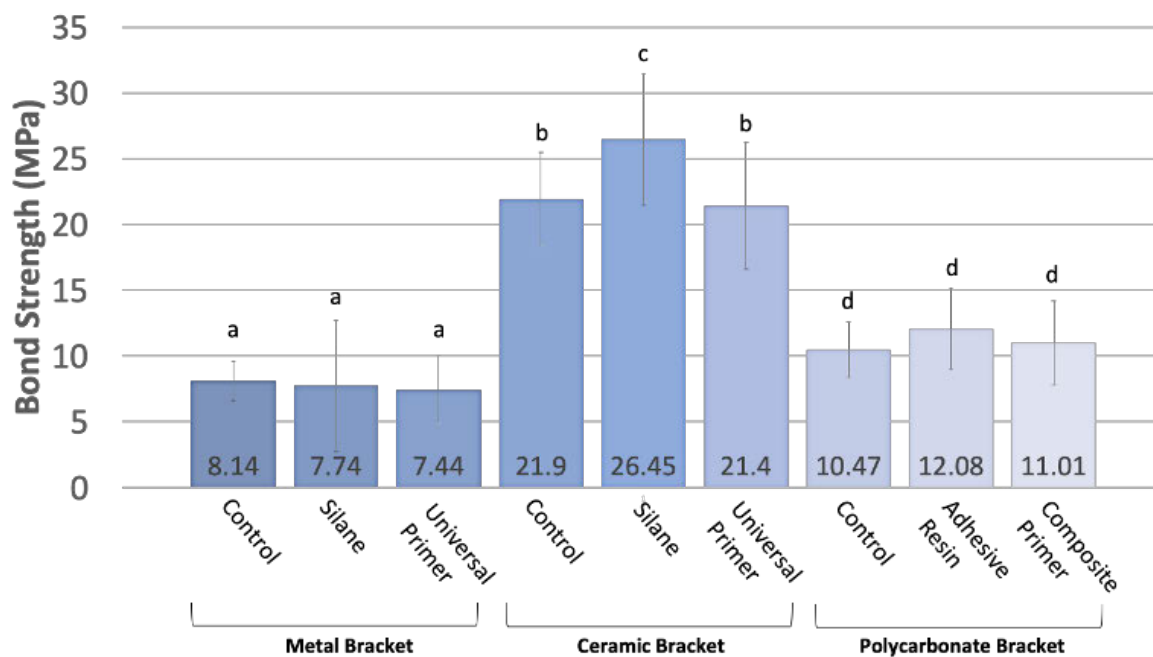
The mean irradiance through the dentin slices can be seen in Figure 10. The light transmission decreased as the thickness of the slices increased. The differences between all the groups were statistically significant ( $P < 0.001$ ). The best fit was the cubic curve (equation  $y=1684-2251x+963x^2-127x^3$ ,  $R^2= 0.92$  and  $r=0.96$ ). Since the necessary radiant exposure for curing the adhesive under the bracket can be reached with multiple combinations of irradiance values and exposure times (Musanje and Darvell 2003, Staudt et al. 2006), the exposure times needed to cure the adhesive through dentin can be calculated. Using the light transmittance values of the present study they approximate 27 seconds for 0.5 mm, 37 seconds for 1.0 mm, 59 seconds for 1.5 mm, 140 seconds for 2.0 mm and 561 seconds for 3.0 mm using an LCU with an irradiance of 1845 mW/cm<sup>2</sup>. However, such long irradiation times are inconvenient and unsuitable for clinical work.



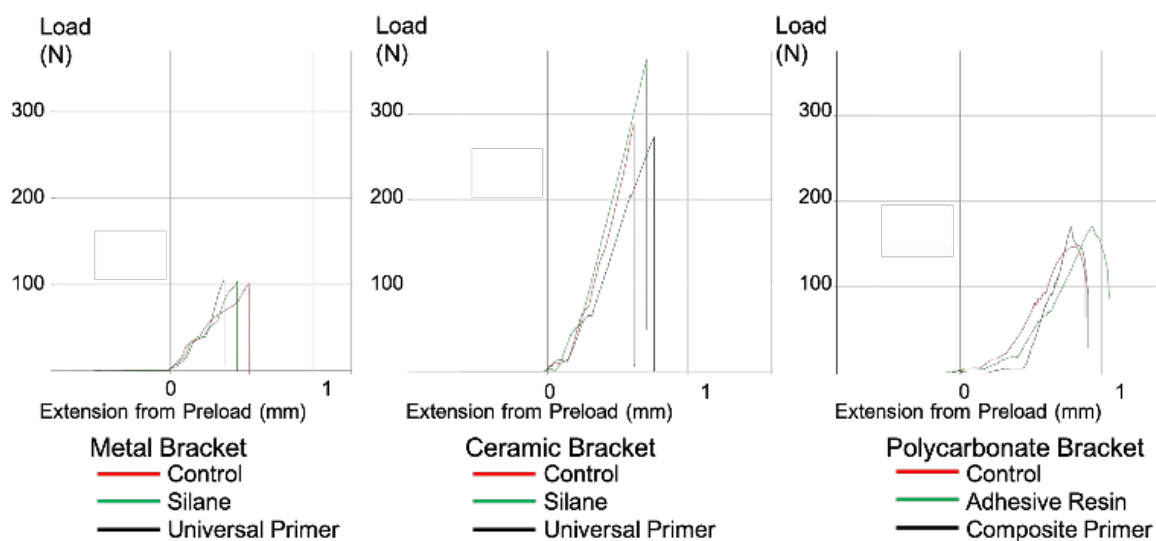
**Figure 10.** Mean irradiance (mW/cm<sup>2</sup>) through dentin slices of different thicknesses with SD bars. The irradiance (output power) of the light curing unit was 1845 mW/cm<sup>2</sup>. From original publication I.

### 5.3 Bond Strength Measurement (II, III)

The bond strength of different brackets without primers was 8.14 MPa ( $\pm 1.49$ ) for metal, 21.9 MPa ( $\pm 3.55$ ) for ceramic and 10.47 MPa ( $\pm 2.11$ ) for polycarbonate brackets, all values differing significantly from each other ( $p < 0.05$ ) (Figure 11.). When using metal or polycarbonate brackets, there were no significant differences between different primers and the control group. The debonding force of ceramic brackets bonded with silane was 26.45 MPa ( $\pm 5.00$ ), which was significantly higher compared to the control group and the universal primer group ( $p < 0.05$ ). The bond strength values are presented in Figure 11. The debonding force - displacement curves can be seen in Figure 12. There was more non-linearity in the loading curves of polycarbonate brackets compared to metal and ceramic brackets, indicating some deformation of the bracket itself. None of the brackets were fractured during testing.



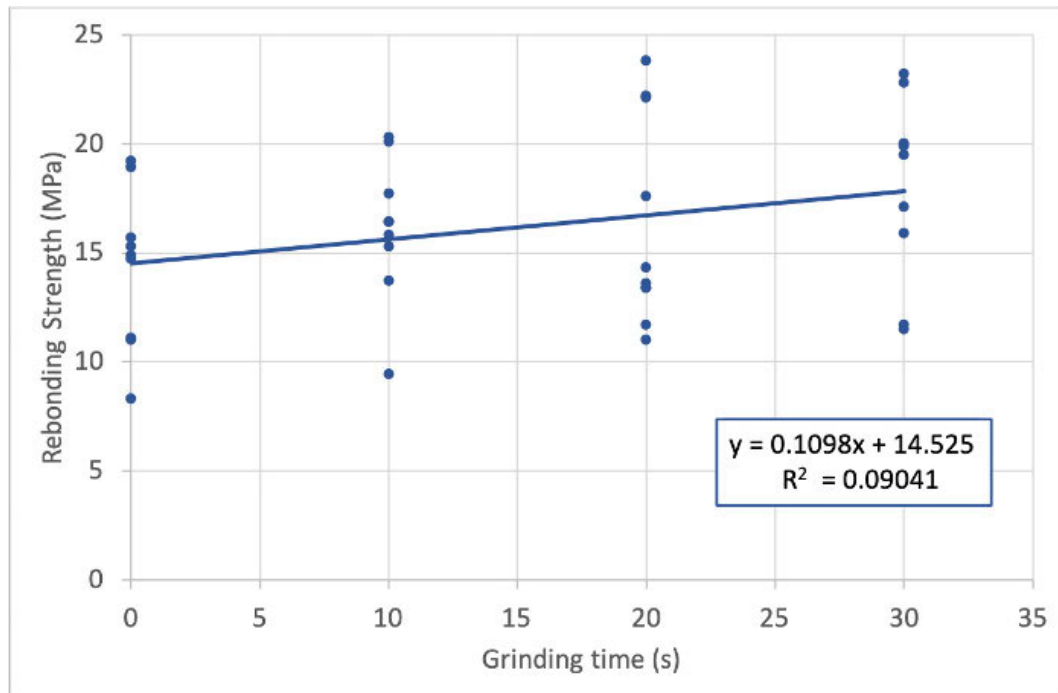
**Figure 11.** Bond strength (MPa) of brackets with different primers, error bars represent standard deviation and the different letters above the columns represent statistically significant difference between groups ( $p < 0.05$ ). Modified from original publication III.



**Figure 12.** Load-extension curves from the bond strength testing of brackets bonded with primers.

### 5.3.1 Rebonding (II)

After debonding, the increase in the grinding time, i.e. increase in the removal of the enamel layer, showed a trend for higher rebonding strength, which was demonstrated by the regression analysis (Figure 13). The changes in bond strength between first bonding and rebonding in different groups can be seen in Table 8. Although there was a positive trend between increasing rebonding strength and grinding time before rebonding (Figure 13), ANOVA did not show statistical differences between the groups ( $p > 0.05$ ). Within the groups, the values between the first bonding and rebonding strengths differed significantly in G0-group ( $p < 0.05$ ), where the rebonding strength was significantly lower compared to the first bonding. Figure 14 shows typical load-displacement curves demonstrating brittle type of debonding failure for the first bonding and minor ductility for the early stage of loading for the rebonded specimens.

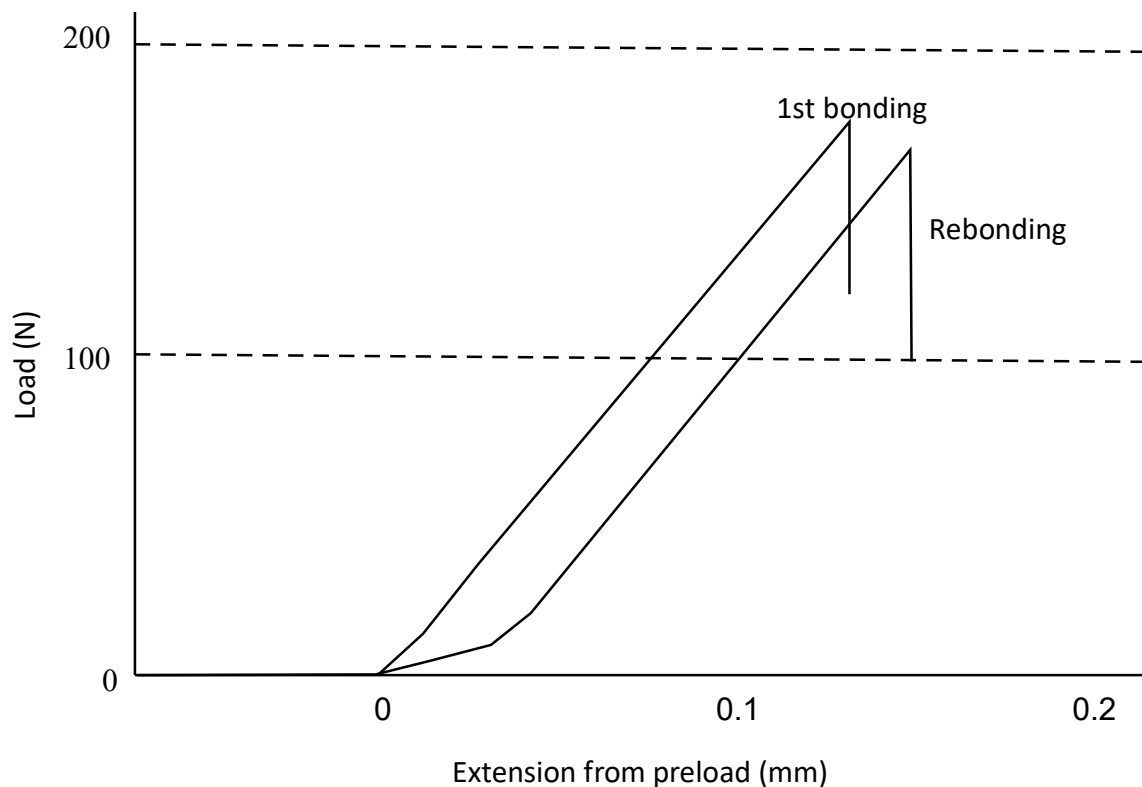


**Figure 13.** Regression line showing a minor trend between the grinding time of enamel before rebonding and rebonding strength. Modified from original publication II.



**Table 8.** Bond strength (MPa) of composite to enamel after the first bonding and rebonding. Enamel substrate has been ground for 0, 10, 20 and 30 seconds (s) and the corresponding removal of enamel is given in micrometers ( $\mu\text{m}$ ). Surface roughness after acid etching of the ground enamel substrate are given as value of average surface roughness ( $R_a$ ). An asterisk \* indicates statistical difference ( $p < 0.05$ ) between values. From original publication II.

	<b>G0</b>	<b>G1</b>	<b>G2</b>	<b>G3</b>
<b>Grinding time (s)</b>	<b>0</b>	<b>10</b>	<b>20</b>	<b>30</b>
<b>Thickness of removed enamel (<math>\mu\text{m}</math>)</b>	-	<b>7(<math>\pm 2</math>)</b>	<b>12 (<math>\pm 1</math>)</b>	<b>16 (<math>\pm 3</math>)</b>
<b>Surface roughness after acid etching</b> (one specimen from each group) ( $R_a$ )	<b>0.301</b>	<b>1.945</b>	<b>0.857</b>	<b>0.343</b>
<b>Bond strength (1st bonding) (MPa)</b>	<b>19.4*</b> ( $\pm 5.2$ )	<b>15.7</b> ( $\pm 5.2$ )	<b>17.5</b> ( $\pm 4.9$ )	<b>20.3</b> ( $\pm 5.9$ )
<b>Bond strength (rebonding) (MPa)</b>	<b>14.3*</b> ( $\pm 3.6$ )	<b>16.1</b> ( $\pm 3.3$ )	<b>16.3</b> ( $\pm 4.8$ )	<b>18.0</b> ( $\pm 4.3$ )



**Figure 14.** Typical load-extension curves from debonding the composite after the first bonding and after rebonding (curves are from groups G0 1st bonding and G0 rebonding). From original publication II.

## 5.4 Bonding Surface Analysis (II, III)

### 5.4.1 SEM Examination (II, III)

The SEM images can be seen in Figure 15. Signs of wear, i.e. pits and grooves, were observed on the intact enamel surface (Figure 15 a).

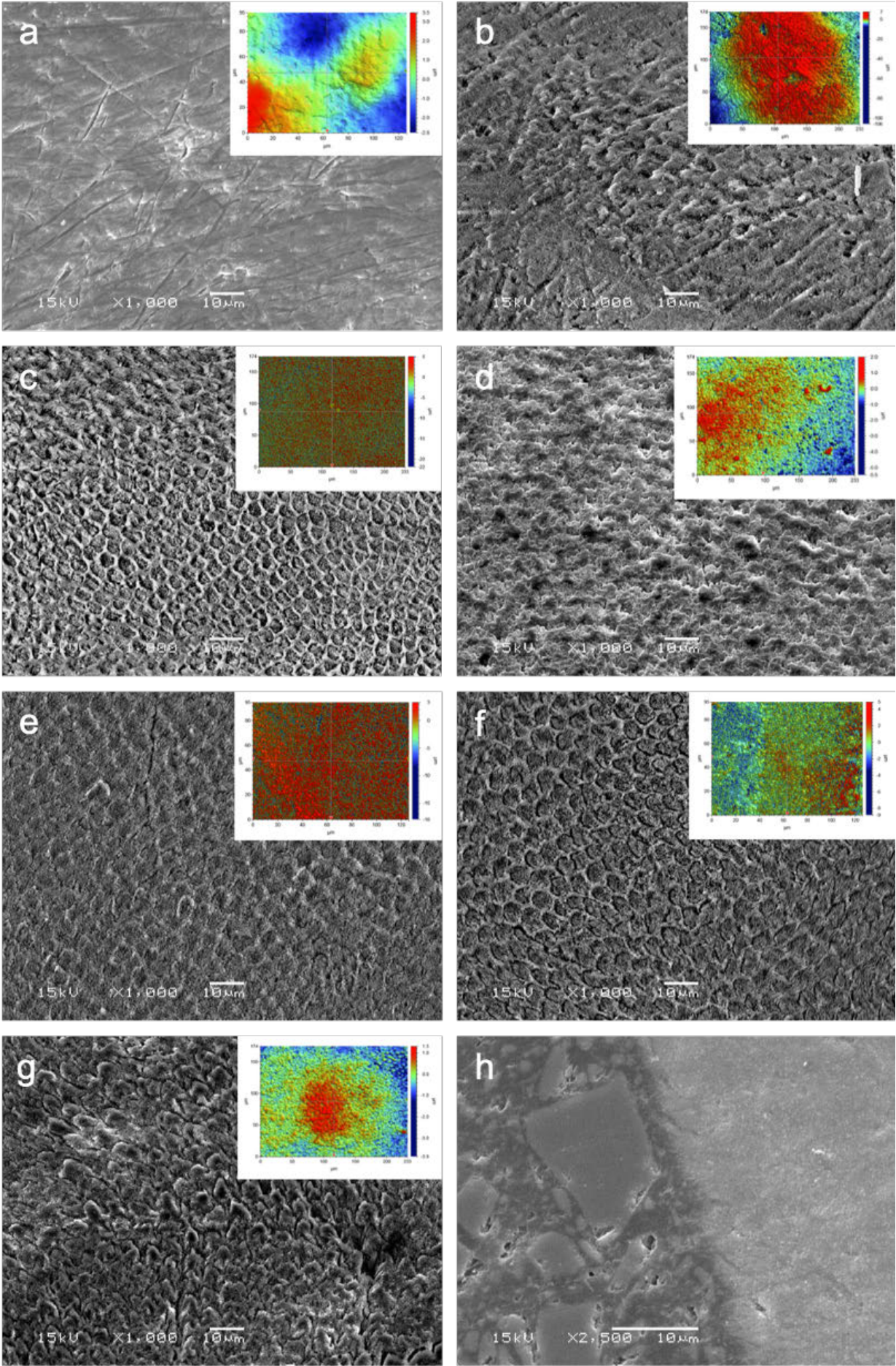
The intact enamel did not exhibit an ideal etching pattern after etching (b), whereas the ground surface etched well (c). The G0 surface that was not ground before re-etching and rebonding, had remnants of adhesive resin on the surface after re-etching (d), whereas the ground surface showed clearly etched enamel, although with different pattern types (e, f, g). It can be seen that the re-etching of the unground surface (G0) did not remove the remaining adhesive resin layer, but rather turned it into “adhesive resin-mash”, whereas grinding the enamel after the first bonding resulted in a similar surface as in the initial surface, indicating that all adhesive was removed by the procedure.

The most common fracture pattern, presented in almost all the specimens, was adhesive failure. The enamel was fractured in three specimens. SEM image of the cross section of the adhesive interface showed the depth of the resin tags to be 5-10  $\mu\text{m}$  into the enamel (Fig. 15, h).

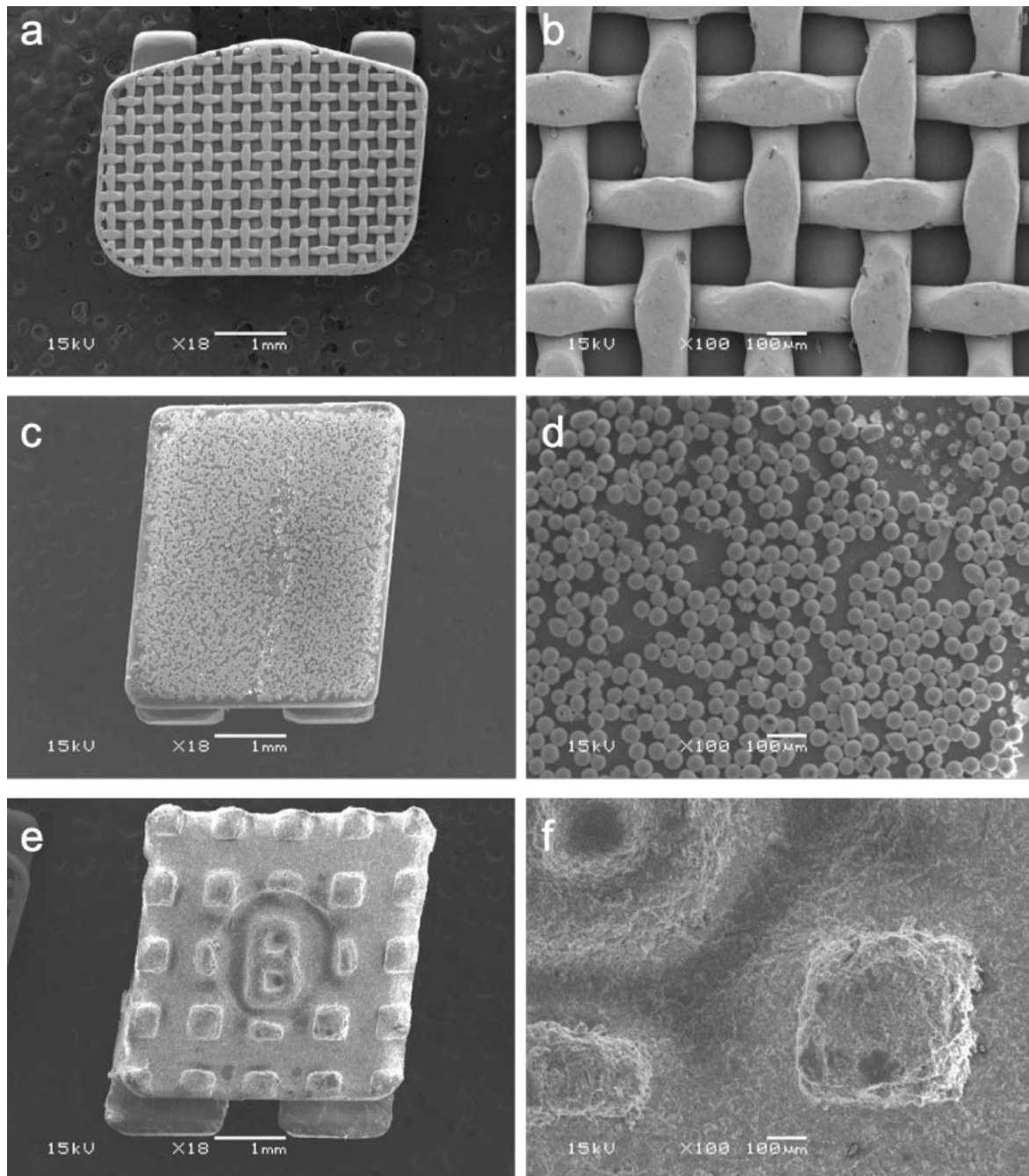
The brackets had different base designs: the metal bracket had a mesh base (Figure 16, a, b), the ceramic bracket base was covered with small spheres ( $\varnothing$  approximately 40  $\mu\text{m}$ ) (c, d) and the polycarbonate base had large square protuberances of varying sizes (approximately 200-500  $\mu\text{m}$ ) (e, f).

**Figure 15.** Next page: SEM images and corresponding optical profilometer image of the enamel surface. All teeth are molars.

- a) Intact, unetched enamel specimen
  - b) Etched unground enamel (Group E) before bonding,
  - c) Ground and etched enamel specimen before 1st bonding,
  - d) Debonded, unground and etched specimen (Group G0) before rebonding,
  - e) Debonded, ground for 10s (Group G1) and etched specimen before rebonding,
  - f) Debonded, ground for 20s (Group G2) and etched specimen before rebonding,
  - g) Debonded, ground for 30s (Group G3) and etched specimen before rebonding.
  - a-g) Original magnification x1000, bar = 10 $\mu\text{m}$ .
  - h) SEM image of the cross section of the interface between etched enamel and resin composite (original magnification X2500, bar = 10  $\mu\text{m}$ ).
- From original publication II.







**Figure 16.** Scanning electron microscope images of the bracket bases.

a, b) metal bracket with a mesh base design, magnification X18 and X100;  
 c, d) ceramic bracket with small spheres on the base, magnification X18 and X100;  
 e, f) polycarbonate bracket with large protuberances on the base, magnification X18 and X100. From original publication III.

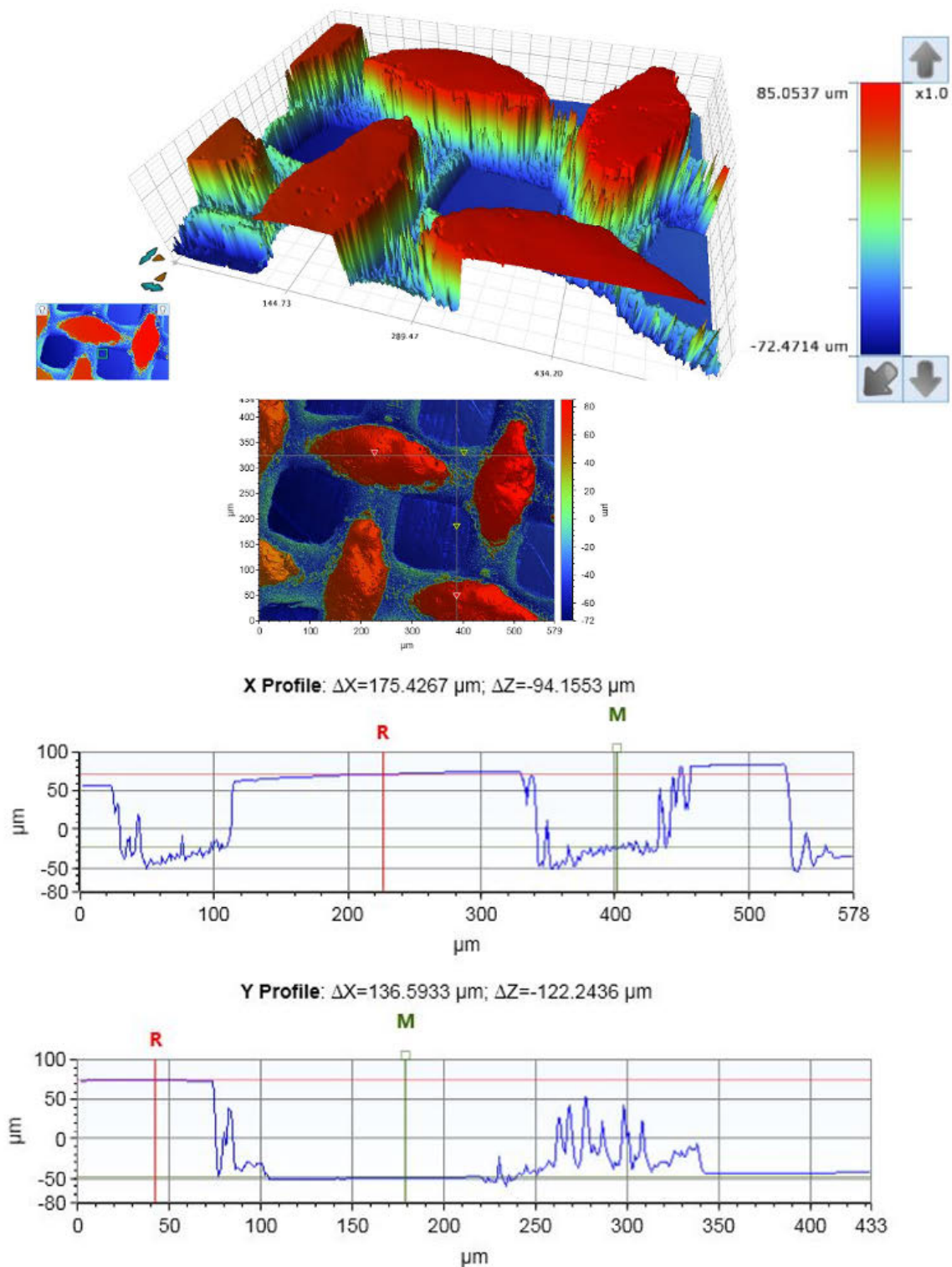
### 5.4.2 Profilometer Analysis (II, III)

The average surface roughness ( $R_a$ ) for the intact enamel was 0.954  $\mu\text{m}$ , for the etched enamel (group E) 2.307  $\mu\text{m}$ , for the group G0 0.301  $\mu\text{m}$ , G1 1.945  $\mu\text{m}$ , G2 0.857  $\mu\text{m}$ , and G3 0.343  $\mu\text{m}$  (Table 9).

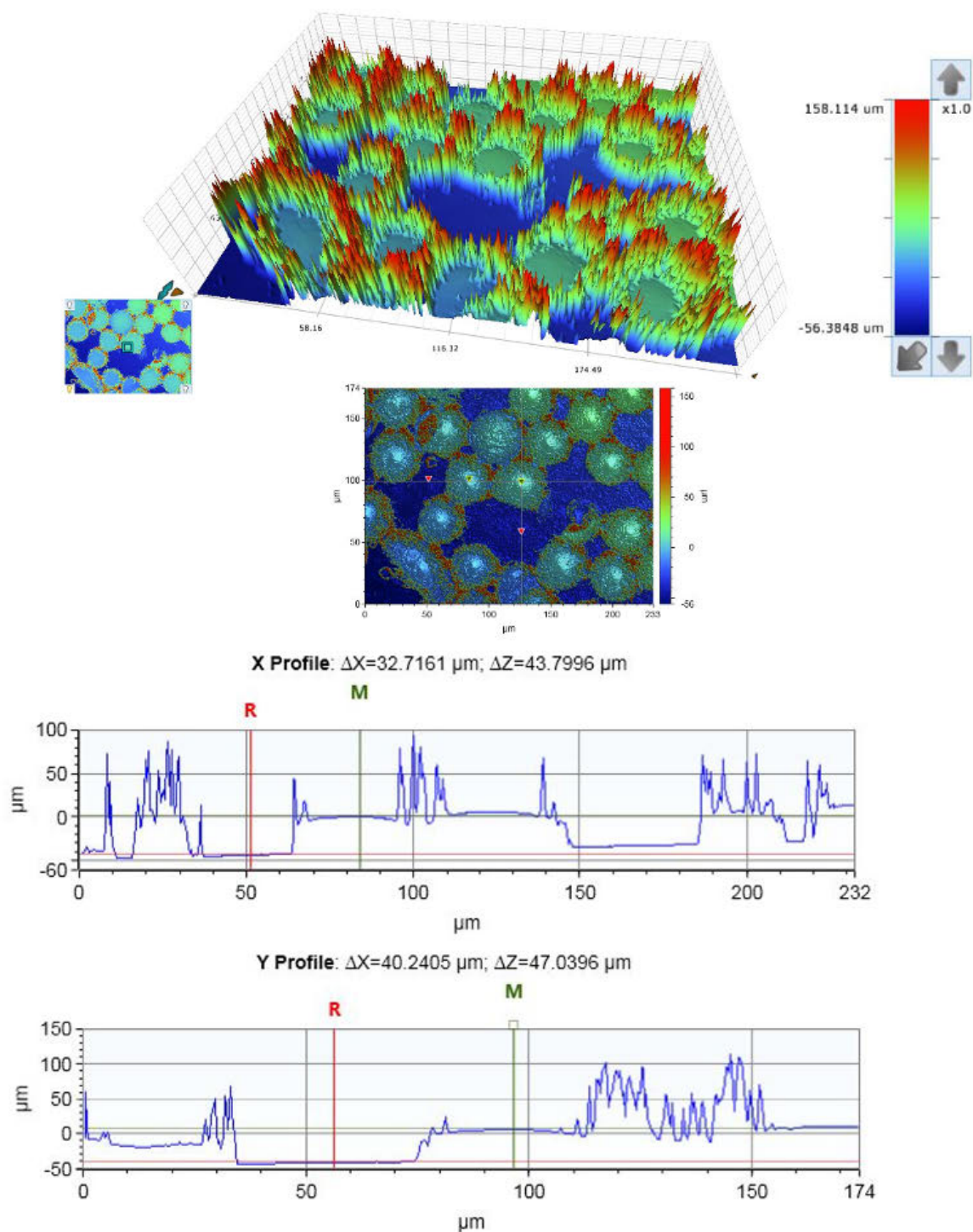
**Table 9.** Surface roughness parameters of  $R_a$  and  $R_t$  of the substrates of test groups in  $\mu\text{m}$ . Roughness average ( $R_a$ ): the arithmetic mean of the height of peaks and depth of the valleys from a mean line. Maximum height of the profile ( $R_t$ ): the vertical distance between the highest and lowest points of the profile. From original publication II.

	Intact enamel	Etched intact enamel (E)	Etched enamel before 1st bonding	Re-etched after debonding (G0)	Ground 10s, etched (G1)	Ground 20s, etched (G2)	Ground 30s, etched (G3)
$R_a$	0.954	2.307	1.928	0.301	1.945	0.857	0.343
$R_t$	6.245	113.359	27.084	7.463	21.344	13.688	5.175

There was a notable difference in the texture of the bracket bases between different bracket types: the difference between the highest and the deepest point in the base was approximately 125  $\mu\text{m}$  for the metal, 50  $\mu\text{m}$  for the ceramic, and 150  $\mu\text{m}$  for the polycarbonate bracket. The profile graphs of the bracket bases are presented in Figure 17.

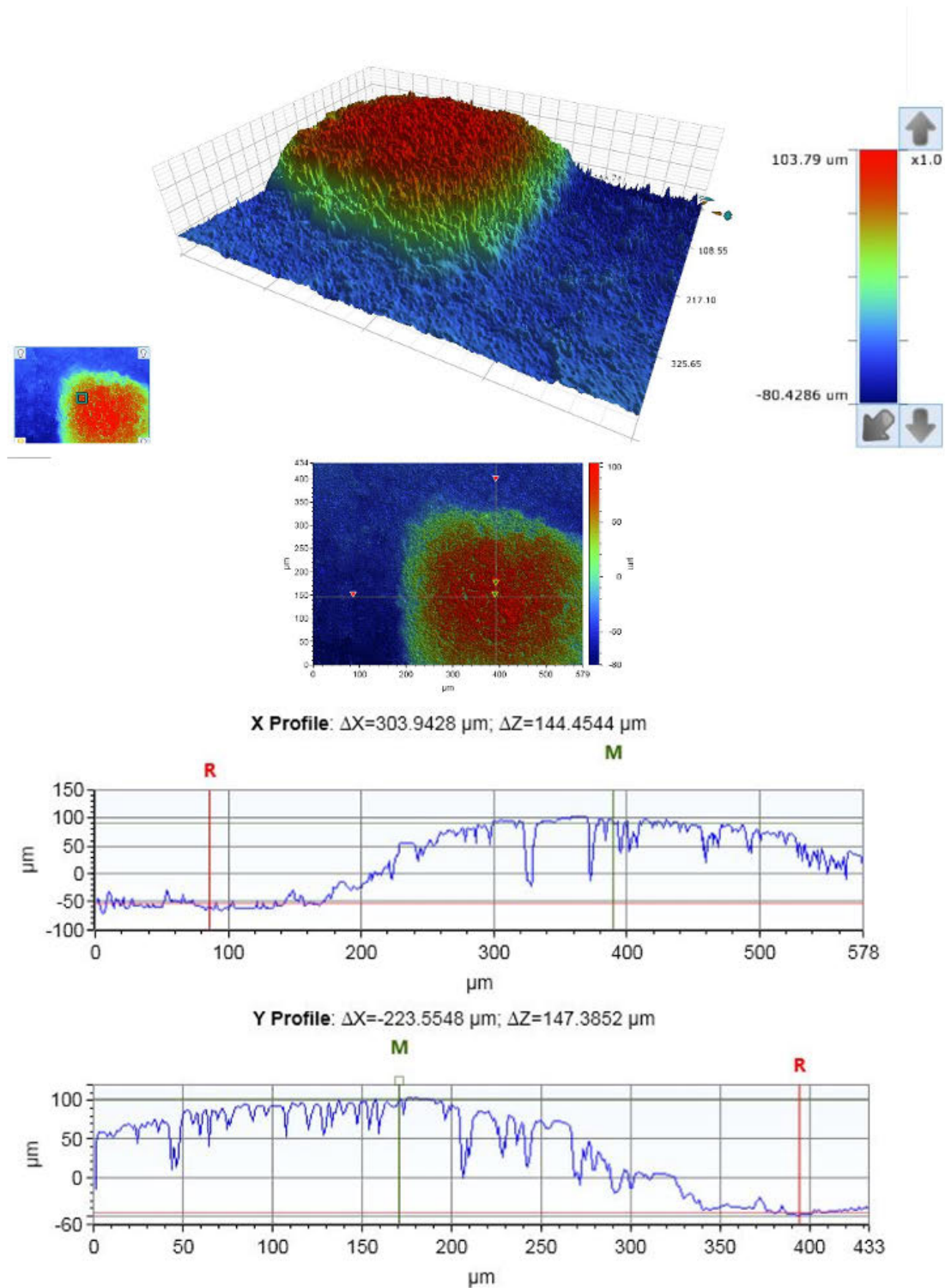


**Figure 17 a.** Surface contour analysis of the metal bracket, a close-up of the mesh on the bracket base and a cross section of the base with the values of the x- and y-axes. The overall difference between the highest and the lowest point of the base is approximately 125  $\mu\text{m}$ . From original publication III.



**Figure 17 b.** Surface contour analysis of the ceramic bracket, a close-up of the small spheres on the bracket base and a cross section of the base with the values of the x- and y-axes. The contour of the spheres accounts for the artefact around the edges of the spheres (the spikes). The overall difference between the highest and the lowest point of the base is approximately 50  $\mu\text{m}$ . From original publication III.



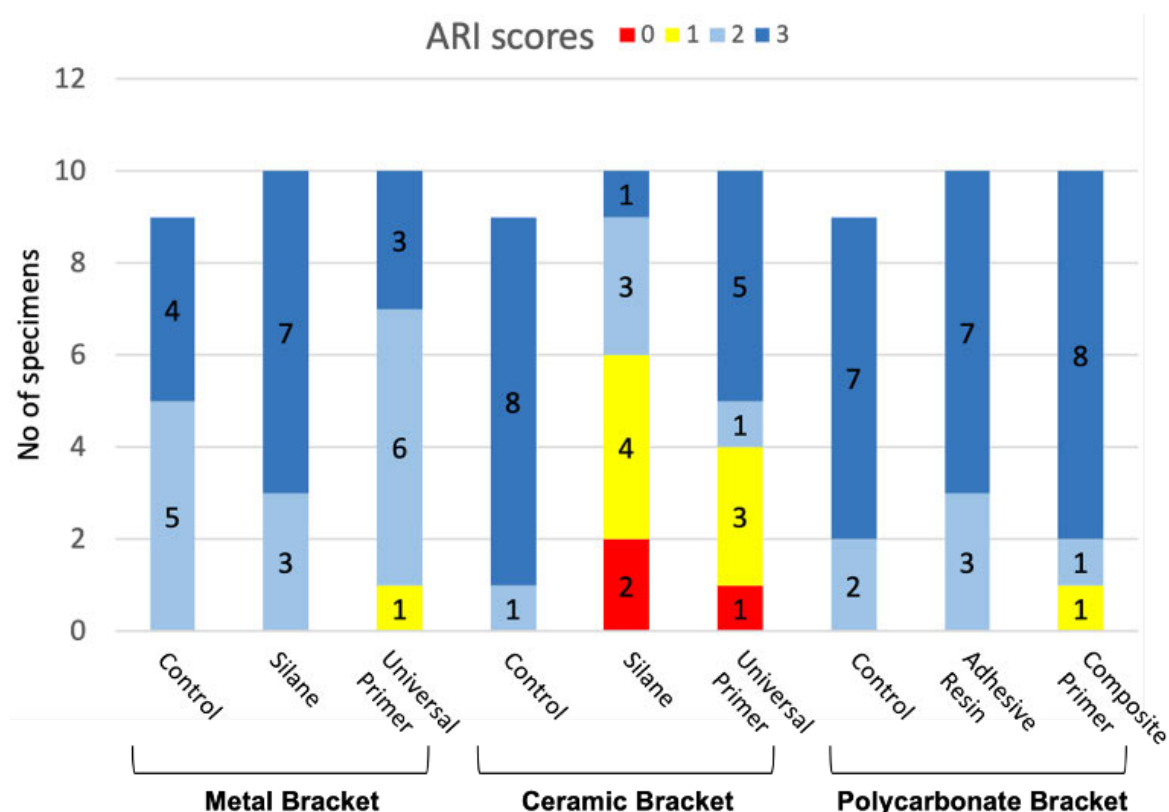


**Figure 17 c.** Surface contour analysis of the polycarbonate bracket, a close-up of one of the square protuberances on the bracket base and a cross section of the base with the values of the x- and y-axes. The overall difference between the highest and the lowest point of the base is approximately 150  $\mu\text{m}$ . From original publication III.

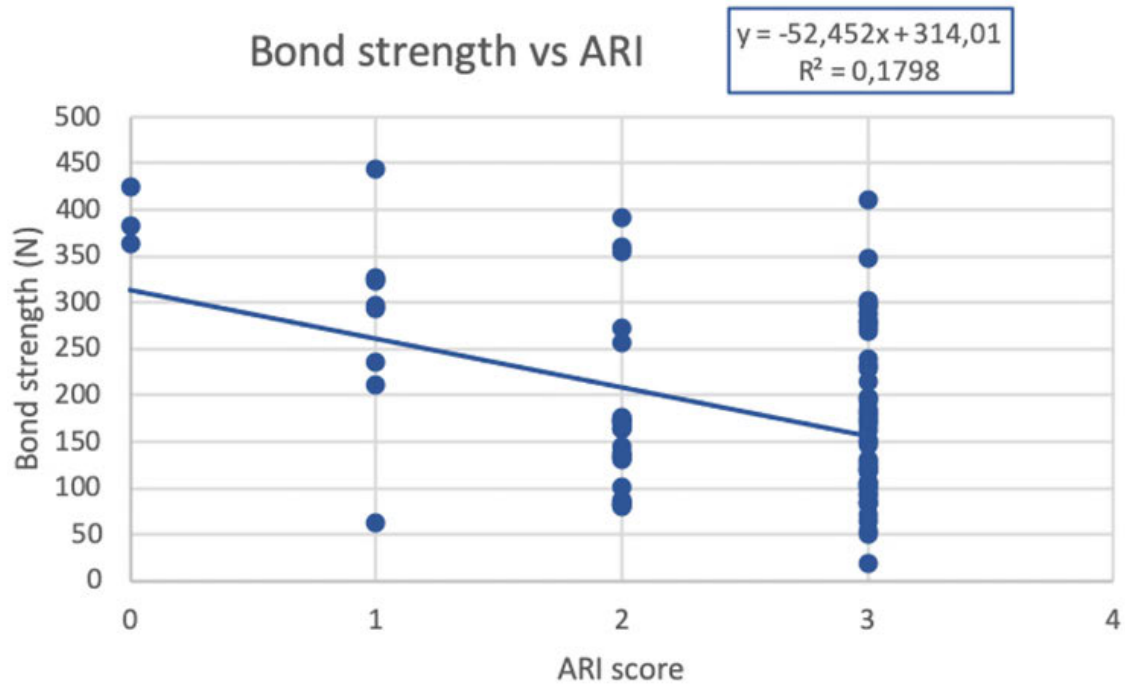


### 5.4.3 Adhesive Remnant Index (III)

After bracket debonding, the ARI scores were mostly 2-3, except when silane was used with ceramic brackets, in which case the ARI score was mostly 0-1 (Figure 18). ARI scores 2-3 indicate that all or above 50% of the adhesive remained on the enamel after debonding, whereas ARI scores 0-1 indicate that all or above 50% of the adhesive remained on the bracket (see Table 1 for ARI score descriptions). Regression analysis showed a correlation between bond strength and ARI score ( $p < 0.05$ ) (Figure 19).

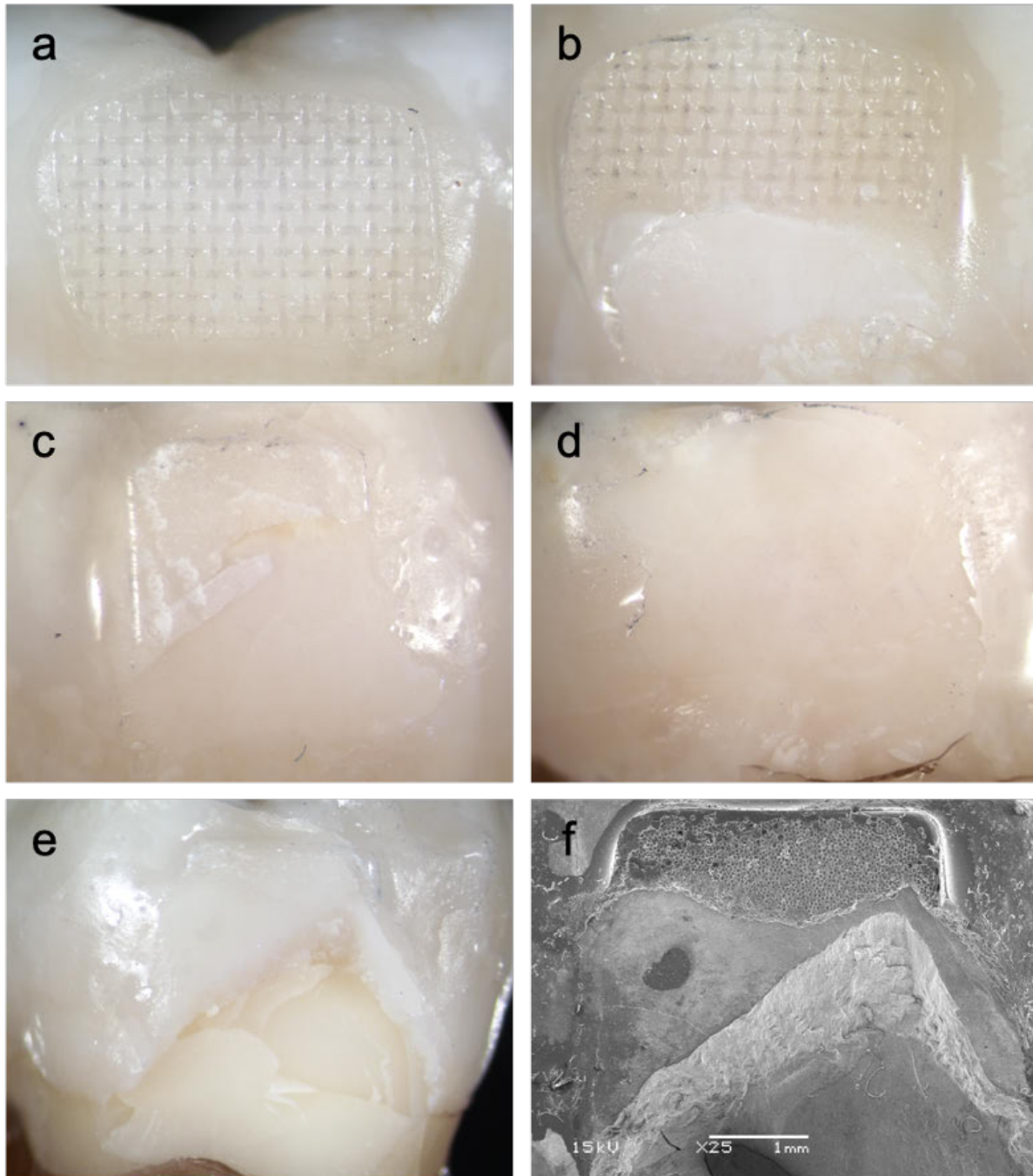


**Figure 18.** ARI scores of the test groups. See Table 1 for ARI score description. Modified from original publication III.



**Figure 19.** Regression line between bond strength and ARI score. See Table 1 for ARI score description. Modified from original publication III.

An enamel fracture was observed in four specimens when ceramic brackets were used: three with the silane based primer and one with the universal primer. No enamel fractures were observed with ceramic brackets without primer, or metal or polycarbonate brackets. Stereomicroscope images of specimens with different ARI scores along with a SEM image of a specimen with fractured enamel can be seen in Figure 20.



**Figure 20.** Examples of ARI scores. a-e) light microscope images,

- a) ARI score 3,
- b) ARI score 2,
- c) ARI score 1,
- d) ARI score 0,
- e) ARI score 1 with enamel fracture,
- f) SEM image of the same sample as image e: adhesive remnants and fractured enamel, magnification X25. From original publication III.

## 5.5 Bracket Failure Rate (IV)

The descriptive statistics and statistical analyses for bracket failure can be seen in Table 10. Altogether 300 brackets failed during treatment, which was 7.9% of the brackets. The failure rate for males was slightly higher (8.7%) than for females (7.2%), but the difference was not statistically significant ( $p>0.05$ ).

The rate of failure for mandibular brackets (9.5%) was significantly higher than that of maxillary brackets (6.4%), and the failure risk for mandibular brackets was 1.78 times the risk for maxillary brackets ( $p<0.05$ ).

The teeth that showed most bracket failures were the left and right mandibular lateral incisors with failure rates of 16.9% and 14.4%, respectively, followed by maxillary right and left first molars, with failure rates of 12.3% and 12.1%, respectively. The teeth that exhibited least failures were left maxillary first premolar (1.9%), followed by left maxillary canine (3.3%), and right mandibular first premolar (3.4%). The number of brackets and their failure rates on all the teeth can be seen in Figure 21. One patient had brackets bonded on lower second molars due to loss of first molars, but because of the low number of cases, the second molars were excluded from further analysis.

The three orthodontists showed different failure rates, 4.4%, 13.5%, and 8.0%, respectively. Bonding system 3 had the most failures at 14.1%, while bonding systems 1 and 4 both had a failure rate of 5.8%. Bonding system 2 was excluded from the statistical analyses due to small number of cases. The differences between the orthodontist 2 compared to 1 and 3 and the bonding systems 1 and 3 were statistically significant ( $p<0.05$ ).

In regards to the bonding system and the orthodontist, the hazard ratio was calculated for a given time (14 days and 90 days).

The failure risk was statistically significantly higher for bonding system 3, compared to bonding system 1 ( $p<0.05$ ). The risk of failure at 14 days for a bracket bonded with bonding system 3 was 3.97 times the risk for a bracket bonded with bonding system 1, and 2.58 times the risk at 90 days.

The failure risk was statistically significantly higher for orthodontist 2, compared to orthodontists 1 and 3 ( $p<0.05$ ). The risk for failure at 14 days for a bracket bonded by orthodontist 2 was 4.82 times the risk for a bracket bonded by orthodontist 1, and 3.01 times the risk at 90 days. Whereas the risk for failure at 14 days for a bracket bonded by orthodontist 3 was 1.93 times the risk for a bracket bonded by orthodontist 1, and 1.72 times the risk at 90 days.

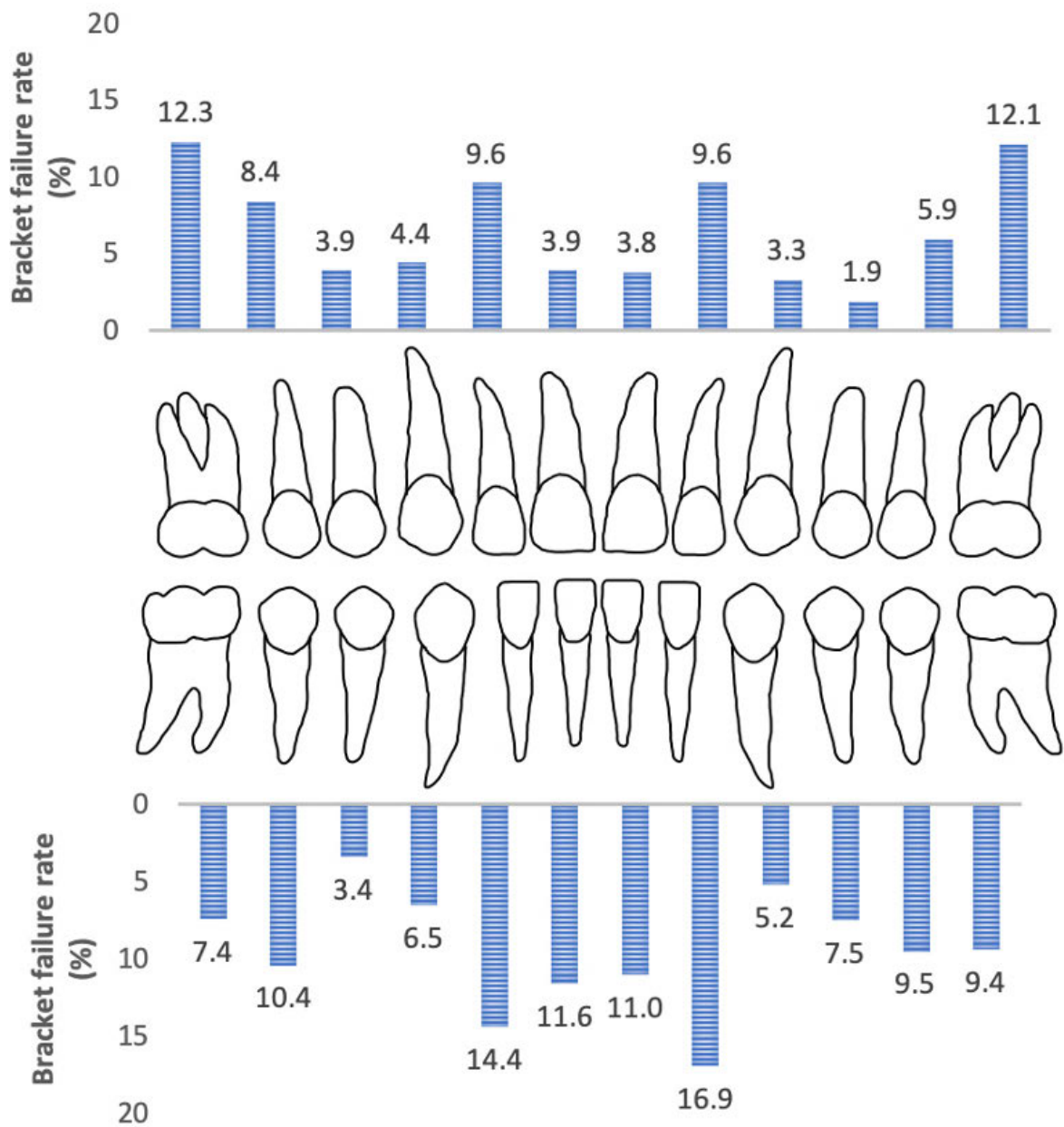
The mean duration of the treatment was 15.9 months (486 days, SD 223 days, range 84-1382 days).

**Table 10.** Descriptive statistics concerning bracket failure and statistical analyses (HR Hazard ratio, SE Standard error).

	NUMERIC VALUES			STATISTICS			
				Bonding System		Orthodontist	
	Number of brackets	Number of failed brackets	Failure-%	HR (95 per cent CI)	SE	HR (95 per cent CI)	SE
<b>Sex</b>							
Male	1761	154	8.7	1.21 <sup>a</sup> (0.88-1.65)	0.16	1.16 <sup>a</sup> (0.85-1.58)	0.16
Female	2040	146	7.2	1.00 <sup>la</sup>	—	1.00 <sup>la</sup>	—
<b>Jaw</b>							
Maxilla	1994	128	6.4	1.00 <sup>la</sup>	—	1.00 <sup>la</sup>	—
Mandibula	1807	172	9.5	1.78 <sup>b</sup> (1.39-2.29)	0.13	1.72 <sup>b</sup> (1.34-2.20)	0.13
<b>Orthodontist</b>							
Orthodontist1	1731	77	4.4	—	—	1.00 <sup>la</sup>	—
Orthodontist2	1047	141	13.5	—	—	9.40 <sup>b</sup> (3.83-23.1)	0.46
Interaction Orthodontist2*time				—	—	0.78 <sup>b</sup> (0.65-0.93)	0.09
Orthodontist3	1023	82	5.8	—	—	2.29 <sup>a</sup> (0.81-6.47)	0.53
Interaction Orthodontist3*time				—	—	0.94 <sup>a</sup> (0.76-1.16)	0.11
<b>Bonding System</b> (Adhesive + Primer)							
1) Greengloo + Orthosolo	2396	138	5.8	1.00 <sup>la</sup>	—	—	—
2) Transbond XT + Transbond XT primer	8	0	0	—	—	—	—
3) Greengloo + Prompt L-Pop	988	139	14.1	7.34 <sup>b</sup> (3.55-15.16)	0.37	—	—
Interaction (Greengloo + Prompt L-Pop)*time				0.79 <sup>b</sup> (0.68-0.92)	0.08	—	—
4) Enlight + Orthosolo	399	23	5.8	1.07 <sup>a</sup> (0.26-4.47)	0.73	—	—
Interaction (Enlight + Orthosolo)*time				0.97 <sup>a</sup> (0.72-1.30)	0.15	—	—

Transbond™ XT not included in statistical analyses due to small number of cases.

<sup>la</sup>Reference category. Superscript letters indicate statistically significant difference,  $p < 0.05$ .



**Figure 21.** Bracket failure rates in different teeth.

## 6 Discussion

### 6.1 Light Curing of the Adhesive

The present study explored questions related to incomplete cure of adhesives under orthodontic brackets. In general, the commonly achieved level of DC% for majority of dental applications is approximately 50-75% (Ferracane and Greener 1986, Eliades et al. 1987, Kauppi and Combe 2003), but because of the light shielding effect of metal, obtaining an acceptable level of cure in clinical situations with metal brackets remains a concern. It should be noted that in the present study, the DC% was measured in the center of the bracket. DC% of adhesive around the perimeter of the bracket would probably be higher. Further research should be aimed at studying the distribution of DC% under the bracket.

The presence of the glass fiber weave underneath the bracket increased the DC% significantly. In addition, the polymerization of the adhesives was more consistent when the glass fiber weave was present. These results are in accordance with previous studies (Shinya et al. 2009, Durgesh et al. 2015). However, very low DC% values were observed under larger metal matrices (DC% 0.1 to 10.3), regardless of the presence of the glass fiber weave. It can be concluded, that even though the glass fiber weave increased the DC%, the light curing may not be adequate when using larger orthodontic appliances.

The mechanisms in which the glass fiber weave enhances the curing of adhesive under a bracket are diverse. It has been suggested that the change in the refractive index and the extinction coefficient of the resin matrix between the glass fibers during the curing process enhance the light curing to proceed to the surrounding adhesive resin (Lehtinen et al. 2008, Vallittu 2015). Another possible explanation for the improved cure of the adhesive in the presence of glass fiber weave could relate to the increased thickness of the adhesive layer under the bracket. Thickness of the glass fiber weave (0.06 mm) will slightly increase the distance between the base of the metal surface and the sensor. That may have allowed more light to enter, and provided better transmittance of the curing light under the bracket.

Additionally, it has been shown that the degree of cure is also influenced by the orientation of the glass fibers. In this study, the glass fiber weave that was used was a bidirectional weave, but even better results can perhaps be achieved with

unidirectional fibers (Durgesh et al. 2015). The effects of glass fibers seem to be promising but further research is needed before its clinical use can be recommended. Especially, studies are needed to investigate the long-term static stress that the brackets and the glass fibers are exposed to during the orthodontic treatment.

It was found that some light is able to penetrate through dentin up to a thickness of 3 mm, but a layer of 4 mm completely attenuated the irradiance of the LCU. Attenuation of irradiance was found to be in accordance with Beer-Lambert law, i.e. in proportion to the thickness of the material sample. Therefore, it is probable that as an aid for low degree of cure of the adhesive under a bracket, transilluminating through teeth could adequately contribute to the curing through incisors only. The dentin slices used in the present study were cut from the middle of the teeth, and therefore did not contain enamel. Because it has been shown that enamel absorbs less light than dentin (Fried et al. 1995, Uusitalo et al. 2016), transmission of light through teeth in a clinical situation could be more significant than through the slices of dentin *in vitro*. This could especially be true in the case of incisors, which are thin and only have a thin layer of dentin inside. The transmission of light through teeth *in vivo* should be further researched. In addition, it should be studied whether there is a threshold intensity, which must be exceeded to initiate the polymerization reaction. Further research is needed to clarify whether the transmission of light is increased with an LCU with a greater irradiance. When the irradiation time is prolonged, the possible rise in temperature needs to be taken into consideration (Arikawa et al. 2004).

## 6.2 Bonding

### 6.2.1 Factors Affecting Bond Strength

Sufficient bond strength of brackets, without causing damage to the enamel, is important for orthodontic treatment with fixed appliances. Therefore, there has been interest in the efficacy of applying primers on the base of brackets of different materials to improve the bonding.

When comparing brackets made of different materials, ceramic brackets yielded significantly higher bond strength than metal or polycarbonate brackets. The bonding of ceramic brackets is based on micro- and macromechanical retention, and chemical reactions between the ceramic surface and adhesive resin. Chemical bonding can be achieved with a silane primer, whereas micro- and macromechanical retention is created by the smaller and larger texture details on the bracket base, such as undercuts, grooves or protruding particles (Eliades et al. 1994, Park et al. 2013). The higher bond strengths, found in previous studies, especially with chemically retentive brackets, were reported to cause enamel fractures when the brackets were



debonded (Jeiroudi 1991, Wang et al. 1997). This was apparent also in the present study, where enamel fractures were observed in three specimens when ceramic brackets were used with silane primer.

Although the universal primer with both MDP and MPS has been suggested to bond to oxide ceramics (Kern and Wegner 1998, Tanaka et al. 2008, Kitayama et al. 2010, de Souza et al. 2014), the present study found no effect on the bond strength. Previous studies have shown that MPS does not have an effect on the bond strength of ceramic brackets (Özcan et al. 2008), and that MDP on the bracket base enhances the bonding of ceramic brackets to ceramic substrates (Falkensammer et al. 2013). The primer that improved bond strength of the ceramic brackets was the silane with MPS only. Besides forming bonds with surface hydroxyl groups, another mechanism of action of silane coupling agents is improving the surface wettability of the substrate for the monomers of the resin. This could explain the increased bond strength of the ceramic brackets used with the silane primer. It seems that the silane was better at improving the wettability of the ceramic bracket than the universal primer.

A significant problem with silane bonding is poor hydrolytic stability, which will over time lead to the deterioration of the bond (Heikkinen et al. 2013, Aboushelib et al. 2009). In this study, the specimens were stored in water for seven days before testing. It is possible that with a longer storage time, the bonds could have started to break down.

In the present study, higher bond strength values were obtained for polycarbonate brackets than for metal ones, even though previously they have been reported to yield lower bond strength values than metal brackets (Guan et al. 2001). In the present study, the composite primer did not have an effect on the bond strength of polycarbonate brackets. A minor increase in the bond strength was observed when adhesive resin was used as a primer, but the differences were statistically insignificant. It is possible that the slight improvement in bond strength could be attributed to a similar solubility parameter between polycarbonate and bis-GMA, since this would allow the primer to dissolve and penetrate into the polycarbonate. Although polycarbonate is a thermoplastic polymer and thus, it is able to be dissolved, the polymer structure may not be easily soluble with the monomers of the adhesive resin. Monomers of the composite primer, namely 2-hydroxyethyl methacrylate, tetrahydrofurfuryl methacrylate and urethane dimethacrylate, may have had some dissolving capacity to the polycarbonate.

Due to the already high bond strengths of ceramic brackets without added chemical retention, the use of a primer to enhance bonding cannot be recommended. To avoid enamel fractures, bonding of ceramic brackets should be based on mechanical retention rather than on chemical bonding, like other studies have also suggested (Viazis et. al. 1990, Forsberg and Hagberg 1992). As the other primers did

not have any significant effects on the bond strengths, it can be concluded that the use of at least these specific primers is not useful.

The design of the bracket base is an important factor in creating mechanical retention, and it largely affects the bonding properties of the brackets. The brackets in this study had very different types of base designs (Figure 7), and they all required a different debonding force. Irregularities on the base of the bracket increase the surface roughness, and therefore also the mechanical retention (Kang et al. 2013). The small spheres of the ceramic brackets provide large surface area and undercut areas. This seems to provide better retention than the mesh on the metal bracket or the square protuberances on the polycarbonate bracket, even though the height difference in the texture (highest and lowest point in the base) of the bracket bases was lowest in the ceramic bracket (Figure 17). Ceramic brackets are somewhat brittle due to their low fracture toughness and bracket fractures are frequently associated with ceramic bracket removal (Scott 1988, Swartz 1988, Bishara and Trulove 1990, Flores et al. 1990, Bishara et al. 1993, Theodorakopoulou et al. 2004, Bishara et al. 2008). However, none of the brackets were fractured in the present study.

Many bracket types are available within the same material category differing considerably in size or in design of the base. Ceramic brackets, for example, include mono- and polycrystalline brackets, and chemically and mechanically bonding brackets. Therefore, findings on a certain type of a bracket cannot be generalized to all brackets of the same material category. Ideally, only findings on the exact same bracket should be compared but this is difficult due to the smaller number of studies and the pace with which new materials are entering the market.

## 6.2.2 Adhesive Remnants and Enamel Damage

A low ARI score indicates that almost all of the adhesive is left on the bracket base when the bracket is debonded. Removal of all or most of the adhesive with the bracket means an easier clean-up process, but at the same time, the risk for enamel fractures is increased. The bond strength of resin adhesive to enamel is quite high, approximately 20 MPa (Barkmeier et al. 1986, Gilpatrick et al. 1991), so when the bond between the bracket and the adhesive exceeds this value, the breakage will happen either at the enamel-adhesive interface, or the enamel itself will fracture. Especially in the presence of predisposing factors, e.g. if the tooth is non-vital (Joseph and Rossouw 1990), fractures are more likely to occur. Therefore, it is considered safer that the breakage happens at the bracket-adhesive interface rather than at the enamel-adhesive interface (Bishara and Fehr 1997). The clean-up process will inevitably cause small damage on the enamel, as well, but it is less destructive than fractures caused by debonding (van Waes et al. 1997). When SEPs are used in the bonding, the etch is less aggressive, and hence the bond between enamel and

adhesive can remain weaker (Bishara et al. 2001, Pashley and Tay 2001, Aljubouri et al. 2003, Romano et al. 2005), and lower ARI scores are perhaps attainable in a safe way.

Results of the present study are in agreement with those of earlier studies indicating that if the bond strength between the bracket and the adhesive is not increased, the breakage usually happens at the bracket–adhesive interface, but with chemical retention, lower ARI scores can be expected (Atsü et al. 2006, Habibi et al. 2007, Kitahara-Céia et al. 2008). ARI score values of 0 and 1 were significantly more common when using primers with ceramic brackets (Figure 18). Without primers, most of the adhesive was left on the enamel, which is in accordance with previous studies (Theodorakopoulou et al. 2004, et al. 2005, Suliman et al. 2015).

Ideally, a strong bond between the bracket and the adhesive could be combined with a slightly weaker bond between the adhesive and enamel to allow the brackets to withstand forces produced by treatment and to enable their removal with the adhesive but without causing damage to enamel. The use of SEPs has been recommended for better bonding and debonding but the *in vivo* failure rates observed in Study IV suggest that use of SEPs can in fact cause accidental failures more often than conventional techniques.

### 6.2.3 Bracket Failure

The rate of bracket failure was 7.9% (Study IV), which is in the higher range of the previous studies (Hitmi et al. 2001, Adolfsson et al. 2002, Kula et al. 2002, Linklater and Gordon 2003, Murfitt et al. 2006, Pasquale et al. 2007, Khalha 2008, Romano et al. 2012, Ahangar Atashi and Shahamfar 2013, Hammad et al. 2013, Özer et al. 2014, Mohammed et al. 2016, Krishnan et al. 2017, Mavreas et al. 2018). One factor that can contribute to the differences in failure rates, is the length of the follow-up period. For example, using the same adhesives in the bonding procedures, Elekdag-Turk et al. (2008a) found very low failure rates of 0.6% for both conventional bonding system and a SEP, but with a follow-up period of six months. On the other hand, Reis et al. (2008) who followed the patients for 18 months, reported failure rates of 17.6% and 15.6% for conventional bonding and SEP, respectively.

The most important factors affecting the longevity of bonding seem to be the position of the bracket in terms of tooth and jaw, along with the bonding system and the technique of the operator. Several studies have reported that mandibular brackets are more prone to failure than maxillary ones (Adolfsson et al. 2002, Linklater and Gordon 2003, Pandis and Eliades 2005, Barbosa et al. 2018), but others have found no difference between the jaws (Elekdag-Turk et al. 2008a, Elekdag-Turk et al. 2008b, Khalha 2008, Petracci et al. 2009, Romano et al. 2012, Hammad et al. 2013). In the present study, most failures were observed in mandibular incisors where in

case of a large overbite the brackets are likely to be exposed to occlusal forces. However, there are conflicting results as to whether a deep bite has a role in accidental debonding (Ahangar Atashi and Shahamfar 2013, Barbosa et al. 2018). The failure rate of different teeth varies considerably from study to study, but premolars are often found to exhibit most failures (Adolfsson et al. 2002, Kula et al. 2002, Linklater and Gordon 2003, Elekdag-Turk et al. 2008a, Hammad et al. 2013, Mohammed et al. 2016, Ahangar Atashi and Shahamfar 2013, Stasinopoulos et al. 2018). Hitmi et al. (2001) found molar tubes to have a failure rate twice as high as the other brackets. Also in the present study, the maxillary molar tubes exhibited high failure rates, approximately 12%. It has been suggested that because of the thick aprismatic layer, enamel on premolars and molars does not etch as well as that on anterior teeth, and this could cause higher failure rates (Whittaker 1982, Mattick and Hobson 2000, Hobson and McCabe 2002, Cal-Neto 2009, Romano 2012).

In the present study, the sex of the patient did not have an effect on the rate of failure. While some previous studies agree with this finding (Linklater and Gordon 2003, Elekdag-Turk et al. 2008a, Elekdag-Turk et al. 2008b, Khalha 2008, Petracci et al. 2009), others have found that boys have higher failure rates than girls (Adolfsson et al. 2002, Hammad et al. 2013). Adolfsson et al. (2002) speculated that girls would be more careful with their appliances. In the present study, boys were slightly more prone to bracket failures but the difference was not statistically significant.

The bonding system and the technique of the operator also play a role in the longevity of bonding. In the present study, there were significant differences between the orthodontists in the bonding success. Of the bonding systems, GreenGloo with Prompt L-Pop was found to give the poorest results. Because GreenGloo with Orthosolo was significantly more successful as a bonding system, it seems that the Prompt L-Pop, a self-etching primer, was the cause of the high failure rate. This is in line with earlier studies that have found that Prompt L-Pop exhibits lower bond strength compared to conventional phosphoric acid etching technique (Bishara et al. 2001, Pashley and Tay 2001, Aljubouri et al. 2003, Sreedhara et al. 2015). However, there are also studies that have found no differences between the two modes of bonding (Velo and Carano 2002, Hellak et al. 2016). Additionally, other SEPs have been found to demonstrate equal or even higher bond strengths than phosphoric acid etching (Buyukyilmaz 2003, Bishara 2005, Cal-Neto 2006b). The present results are at odds with an earlier clinical study that found no differences in failure rate between brackets bonded with Prompt L-Pop and conventional technique (Aljubouri et al. 2003). Prompt L-Pop, with a pH of 0.9-1.0, is in the more acidic end of the range of SEPs (Ostby et al. 2008), a feature that should improve the etching effectiveness of Prompt L-Pop compared to less acidic SEPs (Kanemura et al. 1999, Di Hipólito et al. 2005). It should be noted, however, that Prompt L-Pop is not an orthodontic but

a restorative product, and therefore its use in orthodontic applications is questionable.

#### 6.2.4 Enamel Surface and Rebonding

Bond strength after removing the surface layer of the enamel before rebonding was examined in Study II. A trend indicating higher rebonding strength with increasing enamel removal was observed, although the effect did not reach statistical significance. The testing method measured bond strength predominantly with shear stress but the presence of micro locations of tensile stress cannot be ruled out, which possibly could have increased the standard deviation values affecting the statistical significance of the differences between the groups.

At debonding, the observed fracture type was a brittle fracture, as is evident in the load-extension curve (Figure 14). Brittleness of the fracture relates to the cross-linked polymer matrix of the resin composite. In the group that was rebonded without grinding the enamel (G0), the load-extension curve showed a degree of ductility in the early stages of the loading, which may relate to the presence of partly loose remnants of the adhesive resin on the bonding surface. During loading, these loose particles debond with lower level of stress, seen in the early stage on the load-extension curve.

The present results revealed that the bond strength was lower when the bracket was rebonded directly on the debonded surface rather than on a ground surface. The differences were minor, but if rebonding strength equal to the initial bonding strength is desired, grinding of the enamel surface can be recommended. Since the re-etching step does not remove the residual adhesive from the enamel, fresh enamel surface should be exposed before rebonding. The present results are in line with those of Mui et al. (1999) in showing that the re-etching produced a regular etching pattern on the enamel surface that was ground after debonding. In the present study, a maximum thickness of 17  $\mu\text{m}$  of enamel was removed during the grinding process resulting in a similar bond strength as with the initial bonding of intact enamel. According to literature, approximately 5-30  $\mu\text{m}$  of enamel is normally removed in the clean-up of the residual adhesive, depending on the method used (Thompson and Way 1981, Øgaard and Fjeld 2010, Suliman et al. 2015). In the entire debonding and clean-up procedure, a loss of 40-60  $\mu\text{m}$  of enamel has been reported to occur (Fitzpatrick and Way 1977, Brown and Way 1978). In the present study, grinding of 7  $\mu\text{m}$  created a favorable surface for rebonding. The surface re-etched well as confirmed by the surface roughness measurement for  $R_a$  and  $R_t$ . Interestingly, surface roughness parameters of etched enamel became lower when the enamel was ground further.

In the present study, the bond strength to intact enamel was found to be similar to that to ground enamel, when 15 s of etching with phosphoric acid was used. This was also true for molars where poor etching results have been observed in earlier studies (Mattick and Hobson 2000, Hobson and McCabe 2002). However, the SEM images indicated that the etching pattern on intact molars was not as clear as that on the ground surfaces of the molars, possibly because of an aprismatic layer or hypermineralization of the enamel (Figure 15). In previous studies, Gilpatrick et al. (1991) found that etching time of 5 s was adequate for ground enamel, whereas Olsen et al. found 5 s of etching to be inadequate for intact enamel. The results of the present study suggest that with an etching time of 15 s, phosphoric acid etching will produce similar and adequate bonding strengths on intact and ground enamel surfaces, though the etching pattern on intact enamel surface may not always be ideal.

### 6.2.5 Limitations of the Study

Many factors influencing the bonding of orthodontic brackets to enamel were considered in the studies presented in this thesis, but there are countless aspects that could be elaborated on and require further research. The limitations of the study include at times small sample sizes, which makes it difficult to draw definite conclusions, since the statistical power remains weak.

There are several factors that complicate comparisons of different studies. In laboratory studies molars and premolars are readily available and therefore often used. The age of the patient at extraction can affect the enamel surface of the teeth. In the present study, all teeth were molars, and therefore extrapolating the results to incisors should be done with caution due to differences in the constitution of enamel (Whittaker 1982). When bonding brackets to molars, the bonding surfaces were chosen so that their shape would simulate the flatness of the incisors, but some variation in the results could presumably be perceived if incisors were used in the testing instead.

Additional weaknesses include the fact that in Study I, the transmission of light was studied through dentin only, whereas in vivo enamel probably affects the transmission greatly. In Study II, the standardization of the amount of enamel that was ground off proved to be somewhat difficult.

The storage medium of teeth used in different studies also varies, e.g. thymol, chloramine, saline and distilled water have generally been used, but it seems that the medium does not significantly affect the bond strength to enamel, although it may influence the bonding to dentin (Eliades et al. 2000). In addition, handling of the specimens, e.g. condition of the enamel surface, duration of the acid etching, thermocycling etc., can impact the results.

Also, as already mentioned, the amount of different bracket and adhesive materials makes comparisons between different studies difficult, which was evident especially in Study III, trying to summarize the findings of previous studies and to compare them with the present results. Furthermore, the fact that Study IV was executed as a retrospective study made it difficult to obtain all the information that was wanted, and the distribution of different bonding systems between patients was quite uneven, which complicated the statistical analysis.

Several methods can be applied in laboratory testing of bond strength, mainly using either tension, shear, or torsion forces, the three loading modes producing different non-uniform stress field patterns (Katona 1997). In the most common test method, the shear bond strength (SBS) testing, the applied force acts through a moment arm and creates also tensile stress components. Thus, in addition to the shearing effect, the force tends to peel the bracket away from the tooth. While most in vitro testing is made with the SBS test, pliers used in clinical removal of brackets, exert several types of forces (shear, peel and torque). This should be considered when comparing SBS test results with those of clinical studies.

## 7 Conclusions

The present study showed that bracket failures during orthodontic treatment are of clinical importance. Several methods to improve bonding were tested with mixed results. Adding a glass fiber weave under a metallic bracket seemed to improve bond strength but requires further research. Use of chemical retention was found to lead to an overly strong bond and enamel damage, and it cannot be recommended. In addition, a clean-up of the enamel surface by grinding after bracket failure was found to improve the rebonding strength, but more effective and enamel-saving methods would be needed.

The main conclusions are:

- Bracket failures during treatment occurred frequently (7.9%). The bonding success was affected by the tooth's position on the dental arches, the bonding system, and the technique of the operator. The clinician should pay special attention to the selection of the bonding system and to their bonding technique to avoid failures.
- The low degree of cure of adhesive under a metal bracket is a matter of concern. It was possible to improve curing by adding a glass fiber weave in the adhesive resin interface.
- Bond strength values were highest for the ceramic brackets, followed by polycarbonate brackets, and metal brackets.
- Primers had only a minor effect on the bonding values with the exception of silane primer, which increased the bond strength of ceramic brackets. However, the bond strength was raised to a level that increased the risk for enamel damage at debonding.
- Before rebonding a new bracket, grinding the enamel surface slightly refreshed the surface for bonding and improved the rebonding strength.



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