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(Director: Prof. Dr. M. Kern)

at the University Medical Center Schleswig-Holstein, Campus Kiel

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**Influence of Dentin Bonding Techniques on the  
Fracture Strength and Fracture Mode of Thin  
Occlusal Glass-Ceramic Veneers**

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presented by

**Christine Yazigi**

from **Homs, Syria**

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1<sup>st</sup> Reviewer: Prof. Dr. Matthias Kern

2<sup>nd</sup> Reviewer: Prof. Dr. Helge Fischer-Brandies

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Approved for printing, Kiel,

Signed: .....

(Chairperson of the Examination Committee)

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## **ABBREVIATIONS**

**CAD:** Computer Aided Design

**CAM:** Computer Aided Manufacturing

**CEJ:** Cement-enamel junction

**DBA:** Dentin bonding agent

**DDS:** Delayed dentin sealing

**DEJ:** Dentin-enamel junction

**°:** Degree

**GPa:** Gigapascal

**IDS:** Immediate dentin sealing

**Kg:** Kilogram

**µm:** Micrometer

**min:** Minute

**mm:** Millimeter

**MPa:** Megapascal

**nm:** Nanometer

**N:** Newton

**ROI:** Region of interest

**SCG:** Slow crack growth

**SD-OCT:** Spectral domain optical coherence tomography

**sec:** Second

## 1. INTRODUCTION

### 1.1. Minimally Invasive Restorations

The goal of modern dentistry is to treat functional and esthetic dental problems with minimally invasive, biocompatible, esthetic and durable restorations. The concept of minimally-invasive dentistry has resulted in an increase in the use of all-ceramic partial coverage restorations [77]. Minimally invasive preparation designs in the posterior area could save up to 40% of the prepared tooth structure when compared to their complete coverage preparation counterparts [41].

This trend was facilitated by many factors such as the adhesive bonding technique, computerized and digitalized dentistry, the development of high resistance ceramics and improved bonding materials as well as refined bonding agents and techniques [13, 91, 94, 139].

The success of the adhesive bonding technique eliminated the necessity for concepts of resistance and retention forms of dental restorations, which in turn contributed in favor of minimally invasive preparation designs and maximum conservation of sound tooth structure. The rise of computerized and digitalized technology and its applications in all aspects and fields of dentistry allowed the use of computer-aided design and manufacturing (CAD/CAM) and made the manufacturing of precise complex restorations with ideal fit for non-retentive conservative preparations possible. Moreover, the development of high resistance ceramics facilitated the fabrication of restorations with minimal thickness, which yet still enjoys excellent esthetic and mechanical properties. Additionally, high bond strengths to both the tooth structure and the intaglio surface of the restorative material were achieved by the development of novel bonding materials, and new generations of bonding agents [13, 91, 94, 139].

All of the aforementioned factors and developments contributed in favor of the minimally-invasive trend and the preservation of sound tooth substance, in order to give both the restoration and the tooth structure greater strength and resistance, as well as to achieve durable bonding [9, 13, 85, 139, 146].

## 1.2. Occlusal Defects and Thin All-Ceramic Occlusal Veneers

Clinically, attrition, abrasion, corrosion and abfraction are often associated with erosive wear and extensive destruction of the occlusal enamel and the exposure of dentin causing the loss of occlusal contacts between the maxillary and the mandible in addition to the formation of occlusal defects [58]. These clinical conditions are not only associated with patients with parafunctional activities, but may also be a matter of concern for patients in middle age as a result of tooth wear caused by long clinical service period leading to the loss of a considerable amount of tooth structure [13].

The conventional method for treating such cases and the most commonly described in literature is treatment with full crowns [11, 73, 152]. However, such a treatment involves the sacrifice of substantial sound tooth structure and is associated with many biological complications such as pulpal injuries, postoperative sensitivity and periodontal complications [13]. Moreover, the loss of occlusal contacts in some cases, caused by tooth wear, may be compensated by tooth over-eruption, which in term may restrict the thickness of the required occlusal restorations and makes the preparation for a full crown restoration critical and unrealistic [13, 138].

The treatment with crowns is no longer considered an optimal solution for occlusal defects, because these clinical situations presuppose a minimally-invasive treatment that allows maximum conservation of the remaining intact tooth structure and preservation of tooth vitality [61, 68]. Ultra-thin and thin all-ceramic CAD/CAM occlusal veneers can be a reliable treatment of choice for occlusal defects [139].

Adhesive bonding of all-ceramic restorations is the basis of the minimally-invasive restorative treatment concept, as it allows the restoration of esthetics and functions without jeopardizing sound tooth structure and requires only minimal preparation, which can be especially useful in cases of eroded and greatly destructed teeth [94].

The clinical survival rate of thin and ultra-thin ceramic restorations is influenced by a combination of factors: the preparation design, restoration's geometry and thickness, mechanical properties of the ceramic material, occlusal forces and most importantly the luting material and the bonding technique [1, 59, 86, 123, 127, 143].

Recent laboratory studies concentrating on ultra-thin and thin occlusal veneers showed encouraging results when these restorations were adhesively bonded, even for veneers that had

a thickness as low as 0.3-0.6 mm [2, 61, 137]. Moreover, the similarities between anterior veneers and occlusal veneers allow for the translation of similar concepts to the latter [139].

### 1.3. Adhesive Bonding

Adhesive bonding of ceramic restorations to the tooth substrates increases their fracture resistance, retention and subsequently their survival rate [81, 121]. Other important factors that contribute to the success of these ceramic restorations are the tooth adherent substance and the ability to achieve durable bonding between the ceramic intaglio surface and the luting resin on one hand, and between the tooth substrate and the luting resin on the other hand [94, 99].

#### 1.3.1. Bonding to Dentin

The concept of adhesive bonding has been in use and proved its effectiveness and reliability for more than 50 years especially regarding the resin-enamel-bonds. The resin-dentin bonds have been a subject of interest only during last decades [42, 94].

The work of Nakabayashi et al. is considered to be the milestone in establishing the concept of dentin bonding, in which the monomer's infiltration into the hard tissues of the dentin creates an interphase layer or what is known as the hybrid layer, which, upon polymerization, creates a bond between resin and dentin that is similar to the interphase seen at the dentin-enamel junction level (DEJ) [87, 102, 103]. In reality, the resin infiltration is far from ideal as the adhesive monomers cannot fully penetrate the dentin's collagen network, which leaves the bottom half of the hybrid layer partially exposed without strongly cross-linked polymer network for protection against water and saliva [147, 156]. The exposed unprotected part of the hybrid layer is therefore susceptible to hydrolysis and enzymatic biodegradation, which can subsequently lead to the destruction of the hybrid layer and loss of the resin-dentin bonds [14, 115].

However, the exposure of dentin is still a considerable problem that affects adversely the survival rate and the fracture resistance of all-ceramic restorations [15]. Various studies in the literature showed that anterior veneers adhesively bonded to dentin have an increased risk of failure and significantly inferior fracture strength values compared to those bonded to enamel [23, 40, 52, 119, 122].

The weakest link is usually the dentin-resin interface where the adhesive layer tends to debond from the dentin surface [57, 90]. Many variables might influence the bonding to dentin and could make this process unpredictable such as previous pathologies or injuries, the depth



of the preparation and possible contamination [90, 105, 113]. A known fact is also that at given loads, higher stresses would arise for the adhesively bonded ceramics as the elasticity modulus of the underlying supporting structure decreases [74], which is the case with the dentin (16 GPa) when compared to the enamel (94 GPa) as an underlying structure [5, 83, 154].

### 1.3.2. Immediate Dentin Sealing

Immediate dentin sealing (IDS) is one of the most effective methods to overcome the problems associated with the exposure of dentin in terms of susceptibility to contamination and the risk of the collapse of the hybrid layer which can result in patients' discomfort, teeth sensitivity in addition to decreased bond strength and durability [40, 52, 89, 145]. IDS dictates that the dentin is immediately treated, after the preparation and before taking the impression, by a dentin-bonding agent (DBA) [89, 92]. This concept has been in use since the early 90s and has been refined ever since [8, 90, 116, 117].

IDS has many advantages such as: noticeably lesser formation of gaps, better adaptation of the restoration, a remarkable enhancement in bond strength in addition to maximum conservation of intact tooth structures, as a result of the reported improved retention and cohesive strength which allows overlooking the traditional preparation principles and following a more conservative preparation especially in excessive destruction cases. Additionally, sealing the dentin immediately after the preparation prevents any dentin contamination or bacterial leakage and thus leads to reduced postoperative sensitivity. Moreover, treating the dentin separately, allows the operator to focus on treating and conditioning the enamel and dentin as two different dental substrates, each according to its individual characteristics and qualities, which leads in turn to a maximization in the performance of bonding to both hard tissues [70, 89, 90, 92, 98, 108].

When Paul and Schärer first modified the conventional bonding process and applied instead what they called the “dual bonding technique” and immediately coated the freshly cut dentin after the preparation and before placing the temporary restoration, a remarkable increase in bond strength was reported [117]. This was also the case with Magne and Douglas in 1999, when the application and polymerization of an adhesive layer to the exposed surfaces of the dentin directly after the preparation increased the achieved bond strengths [90]. Another study by Magne et al. [98] investigated the effect of the double application of DBA, i.e. following the IDS protocol, on the bond strength of composite overlays to the prepared dentin surfaces. The

mean microtensile bond strengths obtained in the IDS groups exceeded 45 MPa in comparison to mean values of 1.8 MPa and 11.6 MPa for the delayed dentin sealing groups<sup>[98]</sup>. A recent study by Gresnigt et al.<sup>[57]</sup>, where central incisors were completely prepared in the dentin to receive lithium disilicate laminate veneers, a significantly higher mean fracture strength of 576 N was obtained when immediate dentin sealing was followed as compared to a mean value of 314 N when delayed dentin sealing was applied<sup>[57]</sup>.

In a systematic review in 2016<sup>[125]</sup>, available literature on the subject of immediate dentin sealing for indirect restorations was searched and 40 related studies were found and reviewed. The main focus of these studies was as follows: the influence of IDS on the micro-tensile or shear bond strength<sup>[25, 38, 39, 84, 92, 132, 133]</sup>, interaction of IDS with impression materials<sup>[43, 46, 54, 95, 96, 130, 131]</sup>, post-operative hypersensitivity<sup>[65, 82]</sup>, the influence of bonding system on the bond strength<sup>[39, 92, 132]</sup>, the influence of DBA film thickness on bond strength<sup>[37, 84, 145]</sup>, effects of provisional cements, provisional restorations and provisional period<sup>[16, 22, 89, 98, 129, 133]</sup> and effects of different dentin conditioning agents on the bond strength<sup>[25, 38, 39, 48, 89, 92, 96, 98, 128, 145]</sup>. These studies concluded that immediate dentin sealing of the freshly cut and exposed dentin is advisable and leads to enhanced bond strength, decreased bacterial leakage and post-operative dentin sensitivity as well as fewer gap formation, although a standardized IDS protocol is still needed. The influence of IDS on the fracture strength of all-ceramic restorations was the focus of a study published later in 2016<sup>[57]</sup> and concerning anterior lithium-disilicate laminate veneers. However, no studies were found regarding the possible influence of IDS on the fracture strength of all-ceramic posterior restorations adhesively bonded to dentin<sup>[125]</sup>.

### 1.3.3. Etching of Dentin

Tooth substrate, whether enamel or dentin, can be treated using different etching protocols. Total-etching with phosphoric acid can be used as a separate step to provide a porous tooth surface before applying the bonding agent<sup>[142]</sup>. In an attempt to shorten the sensitive technique of bonding procedure, self-etch adhesive systems have been developed, where it etches and penetrates the tooth structure simultaneously but to a lesser degree especially for the enamel<sup>[27, 67]</sup>.

However, accumulating evidence shows that prior etching of the dentin by phosphoric acid may lead to the creation of a hybrid layer of poor quality, which reflects negatively on the

bonding qualities of the dentin as a substrate and leads to reduced bond strengths and an increase in adhesive failures, in addition to a susceptibility to nano-leakage [18, 135, 136, 148, 151, 153].

Possibly, the bonding performance and survival rates could be improved by selectively etching the enamel with phosphoric acid while omitting the step of dentin etching, a technique in which the enamel bonding strength is optimized while the risk of over-drying or over-etching the dentin is also avoided, which in turn leads to an enhancement in the performance of both dentinal substrates, each according to its unique qualities [28, 49, 51]. However, the possible interaction between the etching protocol applied for dentin and the dentin sealing method has not been investigated [125].

#### 1.4. Cracks in Glass-Ceramics

Glass-ceramics, due to their brittle nature, are predisposed to flaws, such as cracks, that might develop while processing the ceramics, during the luting process or later during the clinical service [12, 50, 93]. Additionally, postoperative crack formation and development might be attributed to various factors such as functional loading, polymerization shrinkage of the luting resin and thermocycling [83, 93]. The load required to initiate a crack formation is influenced mainly by differences in elasticity modulus of the cement, the tooth substrate and the restorative material [34]. It was assumed that the varying results between different studies in terms of ceramic's fracture strength could be explained by ceramic materials' sensitivity to existing cracks that differ in size [55].

Subjecting specimens to cyclic loading under wet conditions leads to a clinically-relevant cracks that develop deep as adhesive interface cracks similar to those observed after clinical failure analysis. In contrast, quasi-static loading tests result in the development of typical cone cracks and Hertzian rings [74].

Fracture of ceramic restorations take place when these cracks, exposed to stresses, develop and is dependent on many factors such as level and mode of the stresses, dimensions and composition of the ceramic, form of cracks, bond strength and durability of these bonds [12, 29, 30, 55]. Slow crack growth (SCG) phenomenon is also critical to the fracture resistance, and is facilitated by the presence of fluids in the oral cavity, and can eventually, with time, lead to catastrophic failures [55, 56, 74].

Crack and microcrack formation during dynamic laboratory loading might be a hint for an increased risk of technical complications under clinical conditions. However, microcracks in

bonded ceramic restorations might behave in a similar way as microcracks in enamel supported by dentin, which exist frequently in natural teeth without resulting in clinical failure<sup>[88]</sup>.

## 1.5. Optical Coherence Tomography (OCT)

The assessment of internal pre-luting and post-luting cracks and microcracks, their extent and progress might be detected by a variety of procedures such as visual evaluation, digital photometer, microscopic inspection, fluorescent penetrant method and acoustic monitoring<sup>[50, 74]</sup>. The technology of optical coherence tomography (OCT) is a new sophisticated and reliable method to scan teeth structures up to a penetration depth of 2.5 mm<sup>[110-112, 140, 141]</sup>.

OCT technology is an imaging and scanning method that allows non-invasive non-radiographic contactless 2D-3D imaging of examined objects in just 10-20 seconds. It gives micron-scale resolution images of the object and uses low coherence interferometry. This technology is well known and established in ophthalmology examinations and gaining recently much interest in the field of dentistry especially in early identification of carious lesions and detecting interfacial adhesive defects in composite restorations<sup>[71, 104, 110-112, 141]</sup>.

The concept of this technology is that the light source is split into a sample arm and a reference arm. The scattered light from the sample arm and the reference arm creates an interference pattern that is compared to a reference and is analyzed by the intensity of light reflected by the examined objects' internal structures and the echo time delay. A point-by-point scanning of the sample creates 2D cross-sectional images, while line-by-line scanning produces a stack of 2D images that could be translated into 3D scans<sup>[110-112, 141]</sup>.

OCT that is currently used for medical and dental purposes has two modalities: Polarization-sensitive OCT (PS-OCT), which has little imaging artifacts, as well as frequently used non-polarization-sensitive Fourier-domain approaches (spectral domain (SD)-OCT, swept-source (SS)-OCT). While the SD-OCT uses a wideband laser source, the latter SS-OCT functions on a frequency-sweeping laser. Both depend on low frequency interferometry<sup>[110-112, 141]</sup>.

OCT has already shown promising results in several aspects of dentistry, but has previously never been used for the purpose of detecting and studying the formation of cracks within ceramic restorations, their progression or their impact on the survival of affected restorations<sup>[110, 111, 140, 141]</sup>.

## 2. AIM OF THE STUDY

As shown in the introduction, scientific data on the possible influence of IDS on the fracture strength of thin all-ceramic posterior restorations adhesively bonded to dentin, the interaction between the dentin etching protocol and the dentin sealing method as well as the effect of the development of cracks on the restorations are missing. Therefore, the purpose of this laboratory study was to:

1. Evaluate and compare the fracture strength of thin occlusal premolar veneers made of lithium disilicate ceramic, with and without the application of immediate dentin sealing.
2. Evaluate the influence of different bonding protocols (total etching/selective etching) on the fracture strength of thin occlusal ceramic veneers.
3. Evaluate the effects caused by artificial aging on the teeth, ceramic material and luting resin after 1,200,000 thermal-dynamic cycles using spectral domain optical coherence tomography (SD-OCT).
4. Evaluate the effect of artificial aging (simulated 5-year of clinical service with thermo-mechanical loading) on their fracture strength.
5. Determine the mode of failure after quasi-static loading and the possible effect of crack progression in the ceramic restorations on the resulting mode of failure.

The null hypothesis to be tested was that there will be no difference in survival rates, load to fracture and failure mode with the application of IDS, different bonding protocols and artificial aging.

### 3. MATERIALS AND METHODS

#### 3.1. Materials

The materials used in the study and their batch numbers are shown in Table 1. All materials used for bonding and its composition are described in Table 2 and shown in Figure 1.

Table 1: List of materials used in the study

Material	Manufacturer	Generic Name	LOT No.
<b>Total Etch</b>	Ivoclar Vivadent, Liechtenstein	Phosphoric acid etching gel	<b>U03546</b>
<b>Adhese Universal</b>	Ivoclar Vivadent, Liechtenstein	Single-component light-curing adhesive for enamel and dentin	<b>U18661</b>
<b>Monobond Plus</b>	Ivoclar Vivadent, Liechtenstein	One component primer for restorative materials	<b>U09067</b>
<b>IPS Ceramic Etching Gel</b>	Ivoclar Vivadent, Liechtenstein	Hydrofluoric acid etching gel	<b>U12483</b>
<b>Variolink Esthetic</b>	Ivoclar Vivadent, Liechtenstein	Dual-curing luting resin (neutral)	<b>T48678</b>
<b>IPS e.max CAD</b>	Ivoclar Vivadent, Liechtenstein	Lithium disilicate ceramic blocks for CAD/CAM applications	<b>U06701</b>
<b>Liquid Strip</b>	Ivoclar Vivadent, Liechtenstein	Air-inhibiting glycerin gel	<b>T29844</b>
<b>e.max CAD Glaze Paste</b>	Ivoclar Vivadent, Liechtenstein	Glaze paste for ceramic	<b>U02995</b>
<b>Virtual</b> -Light body -Putty	Ivoclar Vivadent, Liechtenstein	Putty and light-body polyvinyl- siloxane impression materials	<b>TL4143</b> <b>UL2233</b>
<b>Anti-Rutsch- Lack</b>	WenkoWenselaar GmbH, Hilden, Germany	Artificial gum resin	<b>5504010500C</b>
<b>GC Fujirock EP</b>	GC Corp, Tokyo, Japan	Improved type IV dental stone	<b>1310314</b>

<b>OptraStick</b>	Ivoclar Vivadent, Liechtenstein	Adhesive application instrument	<b>UL3717</b>
<b>Steatite Ceramic Ball</b>	Höchst Ceram Tec, Wunsiedel, Germany	Steatite Ceramic	-
<b>Technovit 4000</b> -Powder -Technovit Syrup I -Technovit Syrup II	Heraeus Kulzer, Wehrheim, Germany	Self-curing polyester resin	<b>010294</b> <b>011065</b> <b>012033</b>
<b>Zwick Z010</b>	Zwick, Ulm, Germany	Universal testing machine	-
<b>Telesto II</b>	Thorlabs GmbH, Dachau, Germany	Spectral-Domain optical coherence tomography apparatus	-
<b>Programat EP 5000</b>	Ivoclar Vivadent, Liechtenstein	Press and ceramic furnace	-
<b>Elipar 2500</b>	3M ESPE, Seefeld, Germany	Halogen curing light	-
<b>UniXS Kulzer</b>	Heraeus Kulzer, Wehrheim, Germany	External curing system	-
<b>INCU-line IL 53</b>	VWR, Radnor, Pennsylvania, USA	Microbiological incubator	-
<b>Microetcher IIA</b>	Danville Materials, Carlsbad, CA, USA	Intraoral air-abrasion device	-
<b>Willytec Kausimulator</b>	SD mechatronik GmbH, Feldkirchen- Westerham, Germany	Dual-axis chewing simulator	-

Table 2: Materials used for bonding and their composition

Material	Composition
<b>Adhese Universal</b>	<p><u>Standard composition: (in wt%)</u></p> <ul style="list-style-type: none"> <li>• Methacrylates 67.0</li> <li>• Water, Ethanol 25.0</li> <li>• Highly dispersed silicon dioxide 4.0</li> <li>• Initiators and Stabilizers 4.0</li> </ul>
<b>Total Etch</b>	<p><u>Standard composition: (in wt%)</u></p> <ul style="list-style-type: none"> <li>• Water dist. 44.3</li> <li>• Phosphoric acid (85%) 43.5</li> <li>• Thickener 12.0</li> <li>• Pigments &lt;0.2</li> </ul>
<b>IPS Ceramic Etching Gel</b>	Hydrofluoric acid: <5%
<b>Monobond Plus</b>	<p>Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate</p> <p><u>Standard composition: (in wt%)</u></p> <ul style="list-style-type: none"> <li>• Adhesive monomers 4</li> <li>• Ethanol 96</li> </ul>
<b>Liquid strip</b>	Glycerin gel
<b>Variolink Esthetic</b>	<p><u>Standard composition: (in wt%)</u></p> <p><u>Monomer mixture 30-38%:</u></p> <ul style="list-style-type: none"> <li>• Urethane dimethacrylate</li> <li>• Methacrylate monomers</li> </ul> <p><u>Fillers 60-68%:</u></p> <ul style="list-style-type: none"> <li>• Ytterbium trifluoride</li> <li>• Spheroid mixed oxides of silica and zirconium oxides</li> </ul> <p><u>Additional contents:</u></p> <ul style="list-style-type: none"> <li>• Initiators and stabilizers 1-2%</li> <li>• Pigments &lt;1%</li> </ul>



### **3.1.1. Total Etch (Ivoclar Vivadent, Liechtenstein)**

Total etch is a 37 wt% phosphoric acid in water. It also contains inorganic fillers in addition to water-soluble polymer to give it an easy applicable consistency. Total Etch can be applied for the total etching technique to etch the enamel (30 sec) and condition the dentin (15 sec) or for the selective etching technique to selectively etch only the enamel (30 sec).

### **3.1.2. Adhese Universal (Ivoclar Vivadent, Liechtenstein)**

This adhesive consists of methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators and stabilizers. It is a one-step single-component, light-cured adhesive for direct and indirect bonding procedures. Adhese universal can be applied in all etching techniques whether total etching, self-etching or selective etching. In order to build up a connection between the hydrophilic tooth substrate and the hydrophobic resin, Adhese Universal contains a combination of hydrophilic, hydrophobic and intermediate monomers (MDP, MCAP, HEMA, Bis-GMA and D3MA).

### **3.1.3. IPS Ceramic Etching Gel (Ivoclar Vivadent, Liechtenstein)**

It consists of <5% hydrofluoric acid. This etching gel is used to etch the intaglio surface of leucite, lithium disilicate and fluorapatite ceramics in order to create microretentions for silicate ceramic restorations and subsequently enhance the bond strength between luting resin and glass-ceramic restorations. The etching time for lithium disilicate ceramics is 20 seconds and it should be followed by 60 seconds of rinsing with water and thoroughly drying.

### **3.1.4. Monobond Plus (Ivoclar Vivadent, Liechtenstein)**

It consists of alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate. This universal restorative primer is compatible with all types of restorative materials as it contains different coupling agents in one bottle. In order to achieve adequate bonding to silica-based ceramics, the silane contains the silanol group which bonds with the hydroxyl group of the silicate-ceramic surface. While for zirconia ceramics, these bonds are achieved through the phosphate ester group found in a phosphate monomer such as methacryloxydecyl-dihydrogenphosphate (MDP) which bonds with the hydroxyl groups on the zirconia surface.

Monobond Plus should be applied to the intaglio surface of the restorative material and left to react for 60 seconds. Subsequently the luting composite is able to wet the restorative material optimally and an enhanced bond can be established.

### 3.1.5. IPS e.max CAD (Ivoclar Vivadent, Liechtenstein)

IPS e.max CAD (Ivoclar Vivadent) is a lithium disilicate glass-ceramic block for the CAD/CAM technology. The material's main component is SiO<sub>2</sub> (57.0-80.0% by weight). Additional contents are Li<sub>2</sub>O (11.0-19.0%), K<sub>2</sub>O (0.0-13.0%), MgO (0.0-5.0%), Al<sub>2</sub>O<sub>3</sub> (0.0-5.0%), P<sub>2</sub>O<sub>5</sub> (0.0-11.0%), ZrO<sub>2</sub> (0.0-8.0%), ZnO (0.0-8.0%) and other oxides (0.0-8.0%). The material is milled in crystalline intermediate stage then the crystallization procedure is applied. The crystallization process at 840-850 °C leads to a transformation of the microstructure as the lithium disilicate crystals grow in a controlled manner. The densification of 0.2% is accounted for the CAD software and taken into account for the milling process. The blocks are available in three levels of translucency (HT, LT, MO) and two sizes (I12, C14).

### 3.1.6. Variolink Esthetic DC (Ivoclar Vivadent, Liechtenstein)

Variolink Esthetic is dual-curing color-stable luting composite resin (self-curing luting composite with light-curing option) that is commercially available in five shades (light, light+, neutral, warm and warm+) and can be used for permanent adhesive luting of metal, glass-ceramic, lithium disilicate glass-ceramic, composite and oxide ceramic restorations. The monomer matrix of Variolink Esthetic is composed of urethane dimethacrylate and further methacrylate monomers. The inorganic fillers are ytterbium trifluoride and spheroid mixed oxides of silica and zirconium oxides. Initiators, stabilizers and pigments are additional ingredients. The filler particle size ranges from 0.04 to 0.2 µm. The total volume of inorganic fillers is 38%. The film thickness is ≤50 µm and the working time (23 °C) is ≥60 seconds.



Figure 1: Materials used for adhesive bonding

## 3.2. Methods

### 3.2.1. Teeth preparation

Ninety-six intact, unrestored maxillary first premolars, recently extracted for orthodontic reasons, as similar as possible in dimension, were collected from patients through informed consent and stored in 0.1% thymol solution (Caelo, Hilden, Germany) at room temperature for two weeks<sup>[63]</sup>. Teeth were thoroughly cleaned using periodontal scalers and then stored in 0.9% NaCl solution at 4 °C until starting the experiments.

First, the roots of the teeth were coated with an artificial periodontal membrane made of a gum resin (Anti-Rutsch-Lack, Wenko-Wenselaar GmbH, Hilden, Germany) 2 mm apical to the cement-enamel junction (CEJ) and of 0.25 mm thickness to simulate the periodontal ligament<sup>[75]</sup>. Teeth were then fixed, along their long axis, in custom made metallic brass cylinders with a diameter of 15 mm by an auto-polymerizing polyester resin (Technovit 4000, Heraus Kulzer, Wehrheim, Germany) 2 mm below the cement-enamel junction. Figure 2 shows the artificial periodontal membrane and Figure 3 shows a tooth fixed in Technovit.



Figure 2: Artificial periodontal membrane.



Figure 3: Embedment in auto-polymerizing polyester resin.

Specimens were randomly divided, according to their initial treatment procedure, into three main groups (n=32). The study groups and group codes are shown in Table 3. In the first main group (**W**) no immediate dentin sealing protocol was applied and the dentin was not treated after the preparation, whereas in the second (**IT**) and third (**IS**) main groups immediate dentin sealing protocol (IDS) was used and the exposed dentin was immediately treated with a dentin bonding agent (DBA) directly after the preparation and before taking the impression. However, groups **IT** and **IS** differed in the etching approach: the second group (**IT**) followed the total etching protocol, whereas the third group (**IS**) followed the selective etching protocol. Table 3 shows an overview of the study outline and the group codes.

The preparation was carried on manually using a series of fissure diamond burs (#837KR.314.012, # 8837KR.314.012, #836.314.014, #8836.314.012, Komet Dental, Brasseler, Lemgo, Germany) under constant water-cooling. The preparation procedure followed the following requirements: all teeth were prepared to receive occlusal ceramic veneers, the preparation depth was created in the dentin with margins in the enamel for all specimens, an angle of  $120\pm 10$  degrees was prepared between the cusps, a wide finishing line was created around the palatal cusp and finally all angles were carefully rounded. All teeth were prepared by the same operator. Figure 4 shows the preparation design and Figure 5 shows the final preparation of a premolar to receive an occlusal veneer.

The preparation was carried on very cautiously and carefully, so that after the enamel was removed and the dentin was fully exposed, with only margins of enamel left, only minimal finishing steps such as final rounding of all angles and smoothening of all sharp edges were conducted.

Table 3: Overview of study outline and group codes

	<u>Group</u>	<u>Subgroup</u>	<u>No. of specimens</u>	<u>1. OCT imaging</u>	<u>Artificial Aging</u>	<u>2. OCT imaging</u>	<u>Fatigue load</u>
96 natural extracted first maxillary premolars	<b>W</b>	WT	16	8	8	8	8
				8	-	-	8
		WS	16	8	8	8	8
				8	-	-	8
	<b>IT</b>	ITT	16	8	8	8	8
				8	-	-	8
		ITS	16	8	8	8	8
				8	-	-	8
	<b>IS</b>	IST	16	8	8	8	8
				8	-	-	8
		ISS	16	8	8	8	8
				8	-	-	8

- (1) W: Without immediate dentin sealing
- (2) I: Immediate dentin sealing
- (3) T: Total etching
- (4) S: Selective etching

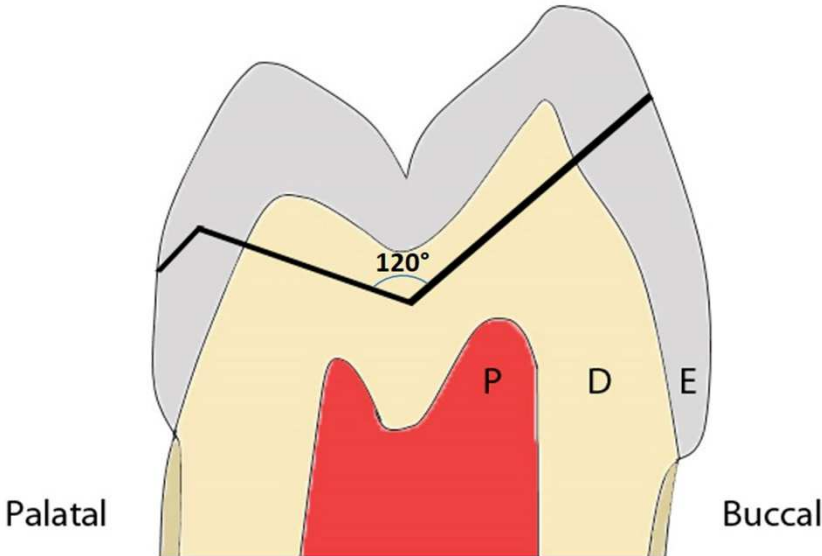


Figure 4: A diagram showing the preparation design



Figure 5: A tooth prepared to receive an occlusal veneer.

Groups **WT** and **WS** followed the conventional adhesive luting protocol rather than the immediate dentin sealing protocol and the exposed dentin during the preparation was not treated with a dentin bonding agent. The preparation was finalized, all angles were rounded and enamel margins were defined. The specimens were then ready for impression taking.

In group **IT** the freshly cut and uncontaminated surfaces were etched with phosphoric acid 37% (Total Etch) as it was applied onto the prepared enamel margins first and then to the exposed dentin. The etchant was left to react on the enamel for 30 seconds and on the dentin for 15 seconds. Whereas for group **IS** the phosphoric acid 37% (Total Etch) was only applied selectively to the enamel margins and left to react for 30 seconds. Then, the etchant was rinsed thoroughly from all surfaces with vigorous water spray for at least 15 seconds and then dried with oil-free air spray until the surfaces appeared chalky white. Figure 6-A shows a specimen following the total etching treatment, while Figure 6-B shows a specimen that was selectively etched.

Subsequently, the bonding agent (Adhese Universal, Ivoclar Vivadent) was applied to the surfaces of both groups with gentle brushing motion for at least 20 seconds and left to diffuse and then cured at first for 20 seconds (Elipar 2500, 3M ESPE, Seefeld, Germany) followed by the application of a thick layer of glycerin gel (Liquid Strip, Ivoclar Vivadent) to the sealed surface, to prevent the formation of an oxygen inhibited layer, and then another 10 seconds of light curing was applied. After that, the glycerin layer was removed by rinsing. Finally, the enamel margins were redefined and rounded. Figure 7-A shows a specimen treated with the conventional delayed dentin sealing protocol, while Figure 7-B shows a specimen treated with the immediate dentin sealing protocol.

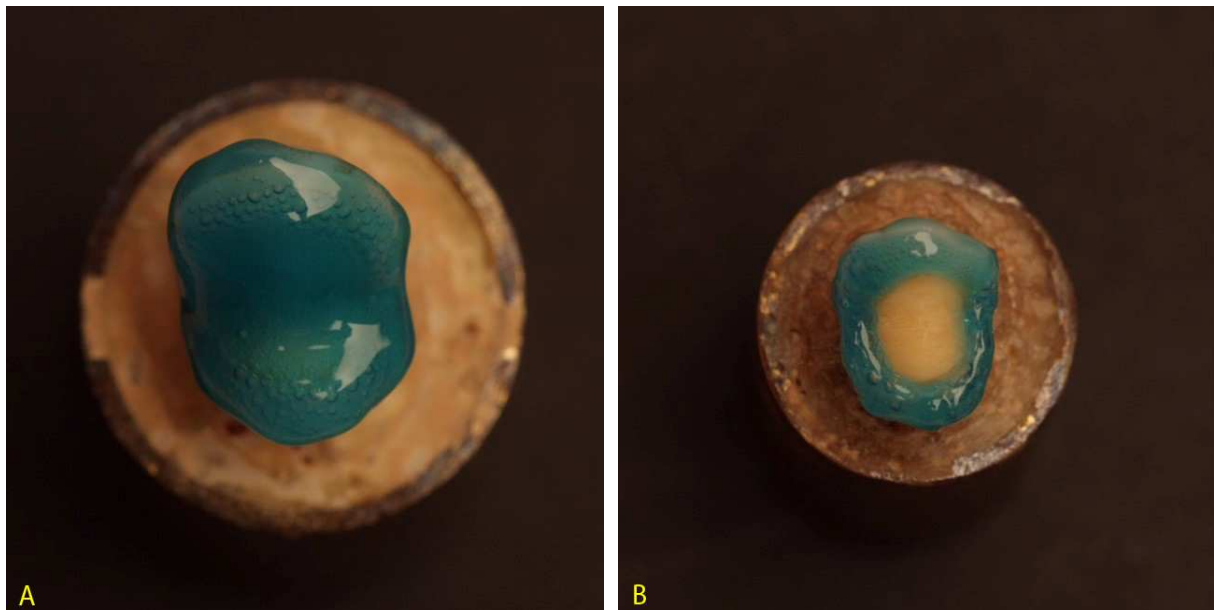


Figure 6: The etching protocol. A: Total etching and B: Selective etching.



Figure 7: The sealing protocol. A: Without immediate dentin sealing and B: Immediate dentin sealing.



### 3.2.2. Occlusal veneers fabrication

Impressions of the individual teeth were taken with a stock tray following the one-step dual-mix impression technique, using putty and light-body polyvinyl-siloxane impression materials (Virtual, Ivoclar Vivadent). Impressions were poured with vacuum mixed improved type IV dental stone (GC Fujirock EP, GC Corp, Tokyo, Japan) to form the master casts. Figure 8 shows the impression technique and the master casts.

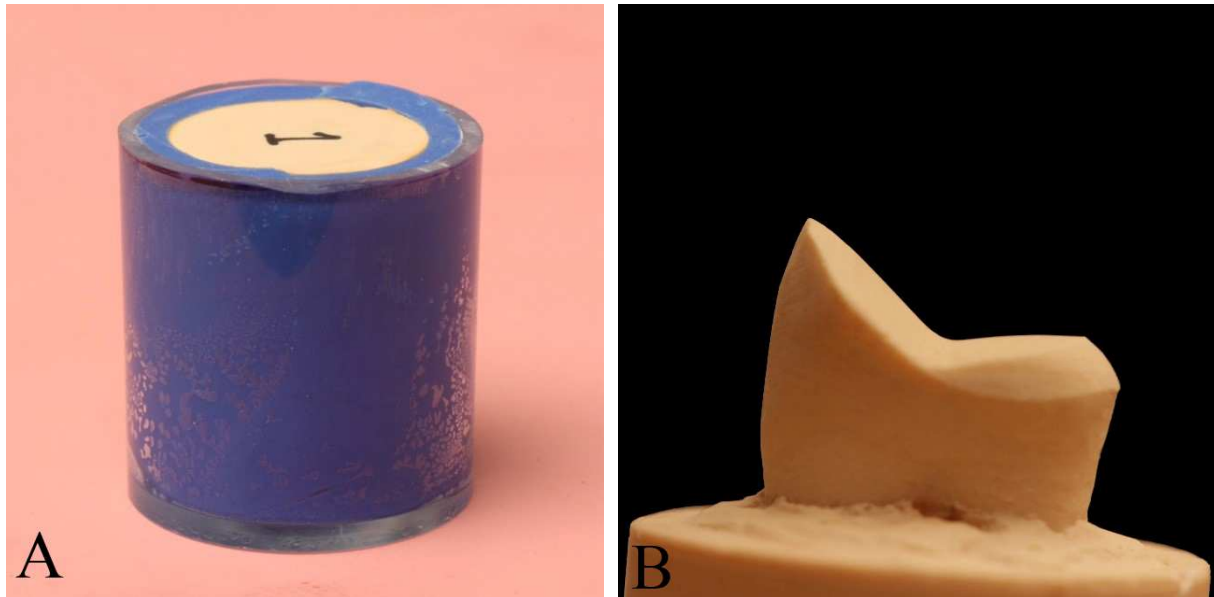


Figure 8: The impression technique. A: An impression taken by the stock tray and cast with stone. B: Master cast.

Master casts were scanned with a 3D scanner (D900 3D scanner, 3Shape, Copenhagen, Denmark). The planned design was created using CAD software (3Shape DentalDesigner Premium 2013, 3Shape). The veneers received a semi-anatomic shaping in the software in order to get a constant ceramic thickness. Then, the area at the fissures was totally reduced and built up manually so that the required thickness could be obtained.

The restoration thickness for all groups was uniform and consisted of 0.8 mm at cusps and of 0.5 mm at fissures. All restorations were digitally designed by the same operator. Figure 9 shows the designing process for an occlusal veneer.

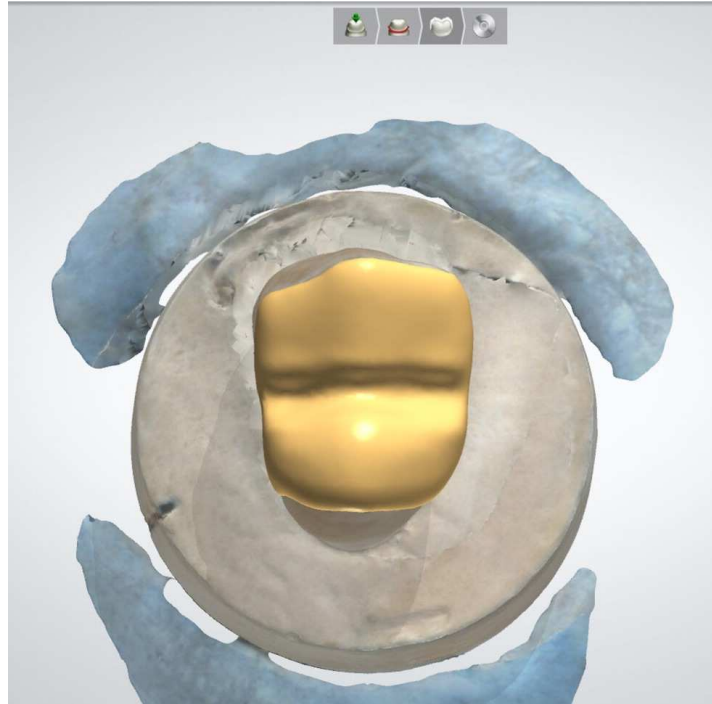


Figure 9: A visual design of an occlusal veneer for one of the specimens.

The veneers were milled (inLab MC XL, Sirona, Bensheim, Germany) out of lithium disilicate ceramic blocks (IPS e.max CAD, Ivoclar Vivadent). After milling, each veneer was separated from the ceramic block and a try-in of the milled restoration was conducted. The veneers were fitted onto the prepared teeth with an adhesive application instrument (OptraStick, Ivoclar Vivadent) and checked for accuracy of fit and marginal integrity. Additionally, the thickness of the restorations was verified and adjusted if needed.

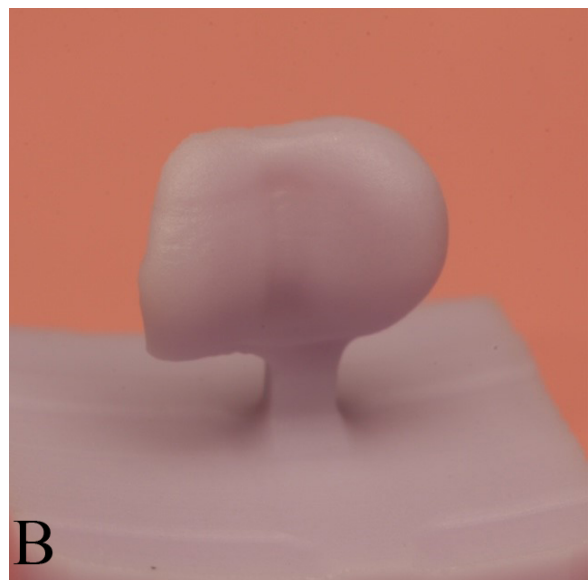


Figure 10: A: An occlusal veneer milled out of lithium disilicate block, B: Closer caption.

After the try-in, the restorations were fixed (IPS Object Fix Putty, Ivoclar Vivadent) onto a firing plate and the glaze paste (e.max CAD glaze paste, Ivoclar Vivadent) was applied to the occlusal surfaces of the restorations. The restorations were then sintered in a furnace (Programat EP 5000, Ivoclar Vivadent) using the combined CAD Crystal/Glaze program at 850 °C according to the manufacturer's instructions. Through this procedure the metasilicate crystal phase was dissolved and the lithium disilicate was crystallized giving the restoration its intended final shade. A final try-in of the restoration was conducted after the crystallization process and before the adhesive luting.

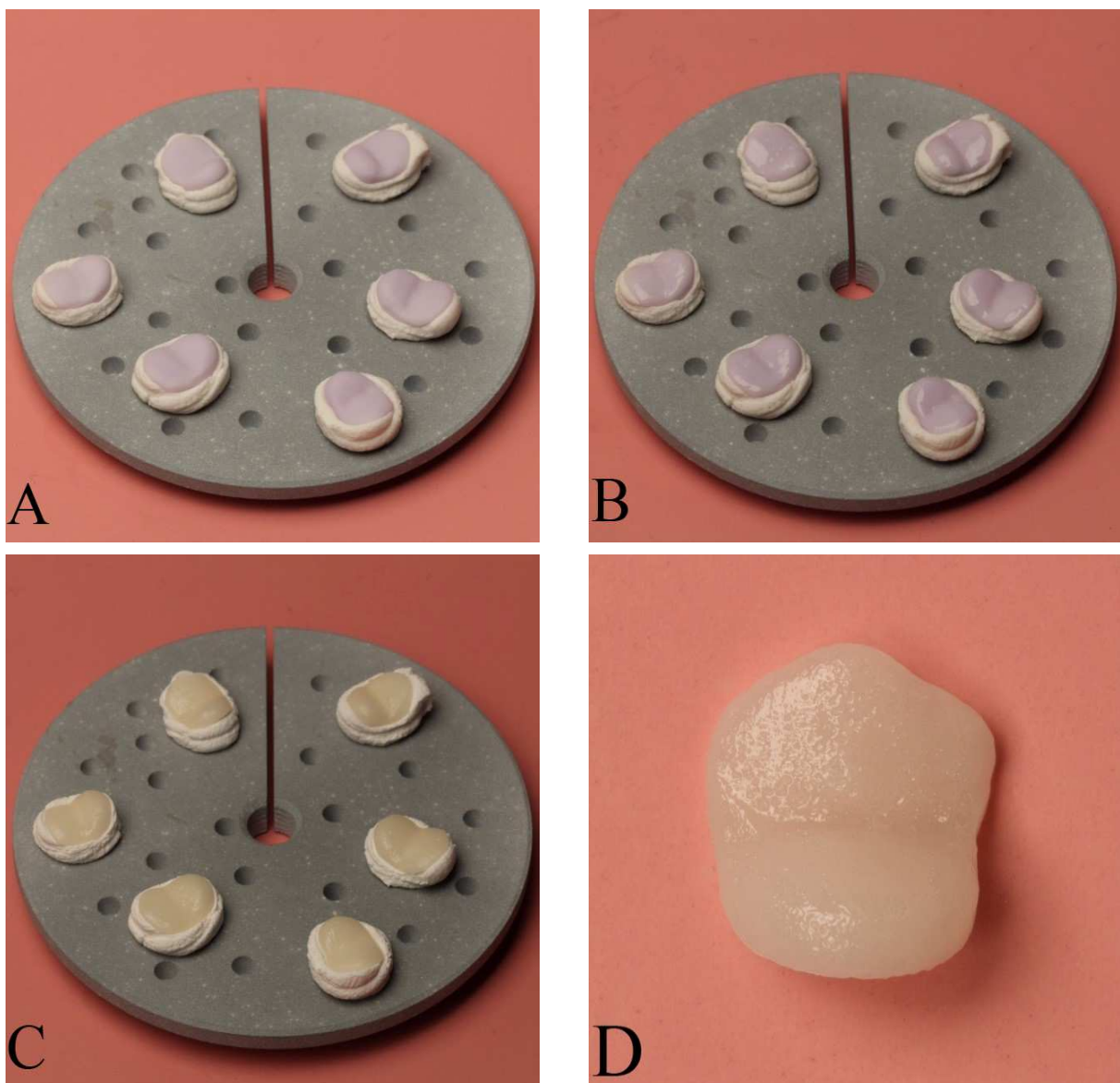


Figure 11: Glass-ceramic occlusal veneers fabrication steps. A: Glass-ceramic veneers after try-in and ready for glazing, B: Glass-ceramic veneers after glazing, C: Glass-ceramic veneers after crystallization, D: Occlusal veneer ready for adhesive bonding.

### 3.2.3. Bonding procedure

The intaglio surface of the IPS e.max CAD restorative veneers was etched with a <5% hydrofluoric acid gel (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 sec, rinsed with water spray for 60 sec, and thoroughly dried with oil-free air. Then a universal restorative primer (Monobond plus, Ivoclar Vivadent) was applied to the pretreated surfaces with a microbrush and was left to react for 60 seconds then the excess was blown with a stream of oil-free air spray.

The sealed dentin of groups **IT** and **IS** was cleaned with airborne-particle abrasion (Microetcher IIA, Danville Materials, Carlsbad, CA, USA) using 50 µm alumina particles at a distance of 10 mm under a pressure of 2 bar until the surface became dull. After that the conditioning of the whole surface of the preparation was carried on conventionally [89, 92].

Each main group (**W**, **IT** and **IS**) was randomly subdivided, according to the pre-bonding surface etching protocol (total/selective), into two subgroups (n=16). The etching procedure for the subgroups was carried on as previously described for total and selective etching protocols. Later on, all prepared surfaces were coated, as previously described, with a layer of the bonding agent (Adhese Universal, Ivoclar Vivadent), which was left unpolymerized until the application of the luting resin.

A dual curing luting resin (Variolink Esthetic DC, Ivoclar Vivadent) was applied to the inner surface of the restorations by an auto-mixing applicator tip. Then, each restoration was seated to its respective prepared tooth using a gentle finger pressure, then with the help of a customized loading apparatus, a constant weight of 750 g was applied onto the occlusal surface of the restoration for 7 minutes. Excess resin was removed gently by foam pellets and margins were covered with glycerin gel and then the polymerization light system (Elipar 2500, 3M ESPE, Seefeld, Germany) was used to polymerize the luting resin for 20 sec at 5 mm distance to each surface of the restoration. Finally, all restorations were additionally cured by an external curing unit (UniXS Kulzer, Heraeus Kulzer) for 90 seconds. Figure 12 shows the adhesive bonding process for the occlusal veneers.

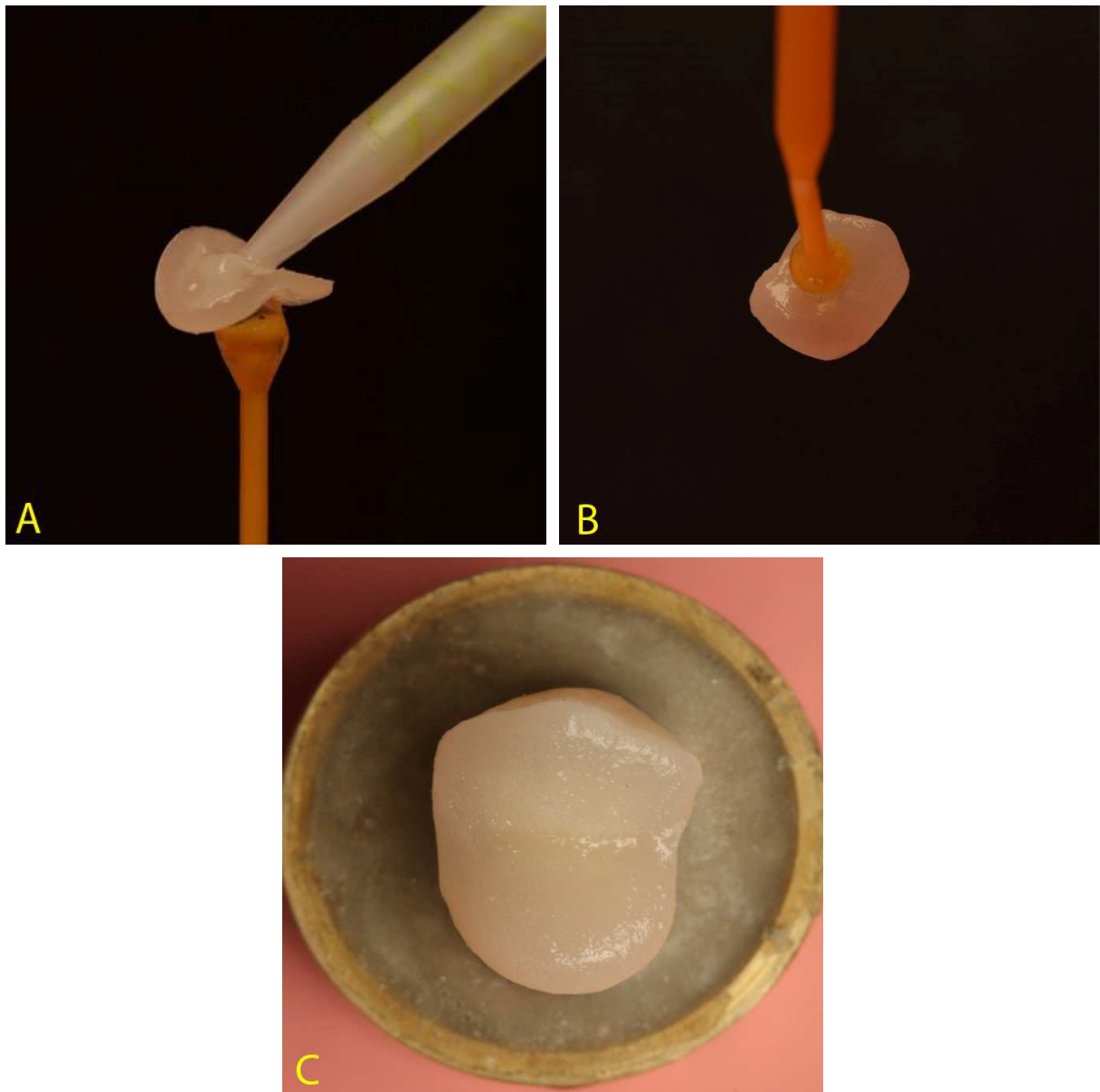


Figure 12: Adhesive bonding of the occlusal veneers. A-B: Luting process, C: Occlusal-view of a veneer adhesively bonded to its respective specimen

### 3.2.4. Storage

The restorations were stored in distilled water at 37°C in microbiological incubator (INCULINE IL 53, VWR, Radnor, Pennsylvania, USA), to ensure that autopolymerization of the luting resin was complete, for one week before the SD-OCT scanning images and two weeks in total before testing.

### 3.2.5. 2D and 3D Imaging by spectral-domain optical coherence tomography

After the adhesive bonding and storage in distilled water for a week, all specimens were imaged by SD-OCT (Telesto II, Thorlabs GmbH, Dachau, Germany) which has a central wavelength of 1310 nm, an axial resolution (air, water) of 5.5  $\mu\text{m}$ , 4.2  $\mu\text{m}$  respectively, a lateral resolution of  $>7 \mu\text{m}$  and a maximum imaging depth of 2.5 mm in teeth (Figure 13).

Specimens were imaged to identify any luting-related defects for all specimens and to obtain before-images for the specimens that will undergo the artificial aging tests, to be able to compare it with the after-images. Five regions of interest (ROI) were 2D imaged for each specimen. Three ROI in the buccal-lingual direction; one ROI to the distal, another to the mesial and one in the center passing through the tips of the cusps, while 2 ROI were 2D imaged in the mesial-distal direction; one at the fissure and the other in the center of the buccal tip where the antagonist is supposed to contact the specimen during the artificial aging process. Additionally, each specimen was 3D scanned as well. The regions of interest were marked on the metal rings with waterproof marker, to ensure that almost the same ROI could be imaged again after the artificial aging process.

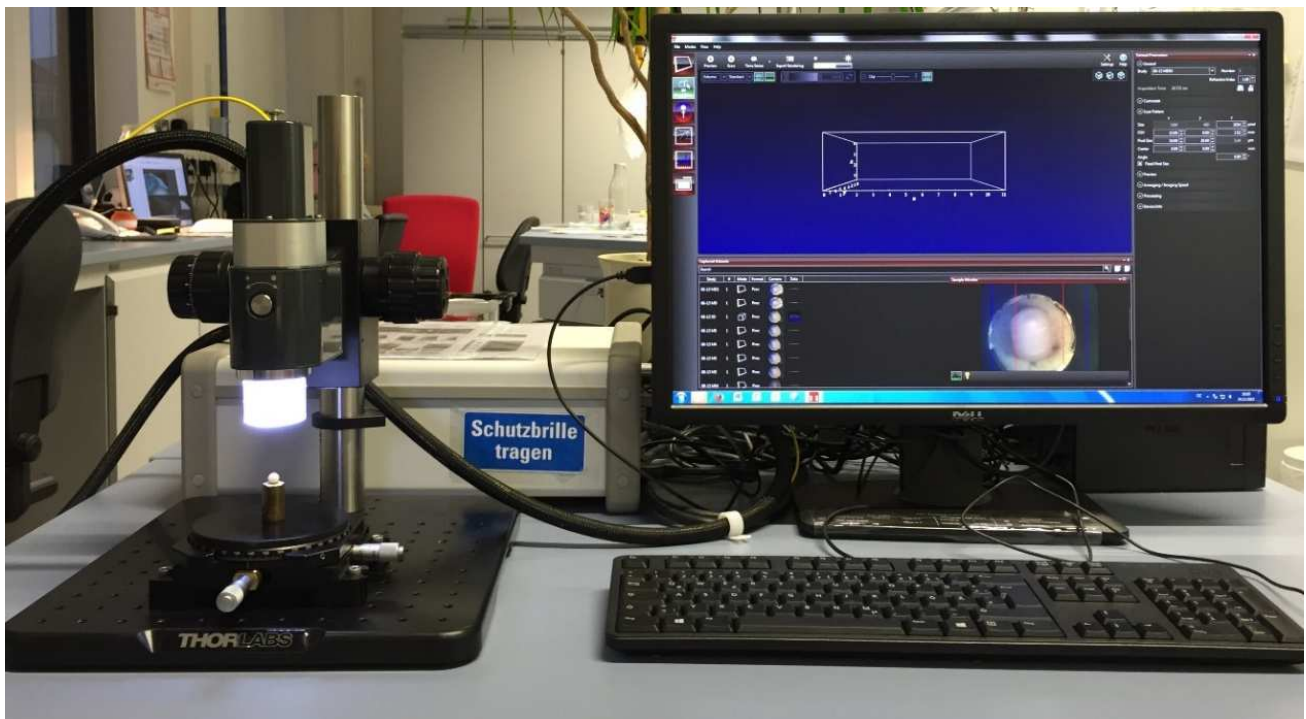


Figure 13: Spectral domain optical coherence tomography (SD-OCT).

### 3.3. Loading tests and statistical analysis

#### 3.3.1. Dynamic loading test

After water storage at 37 °C for two weeks, half of the specimens of each subgroup (n=8) were subjected to 1,200,000 cycles (Figure 14) of thermo-mechanical fatigue in a dual-axis chewing simulator (Willytec Kausimulator, SD mechatronik GmbH, Feldkirchen-Westerham, Germany). A force of 10 Kg was applied using a ceramic steatite ball with a diameter of 6 mm as an antagonist (Steatite Hoechst Ceram Tec, Wunsiedel, Germany). Integrated thermocycling of 5 °C to 55 °C with a 30-seconds dwell time at each temperature and a total of 5,500 thermal cycles at a frequency of 2.4 Hz was used as to simulate the wet conditions and temperature fluctuation in the oral cavity. The load was applied 0.5 mm below the tip of the cusp then moved down with a lateral sliding component of 0.3 mm towards the central fissure. The vertical motion was 6 mm, the descending and ascending velocities were 30 mm/s and 55 mm/s respectively. A video camera was set for each specimen throughout the artificial aging process, in order to retrieve the number of cycles the specimen underwent and survived in case of failure during dynamic loading.

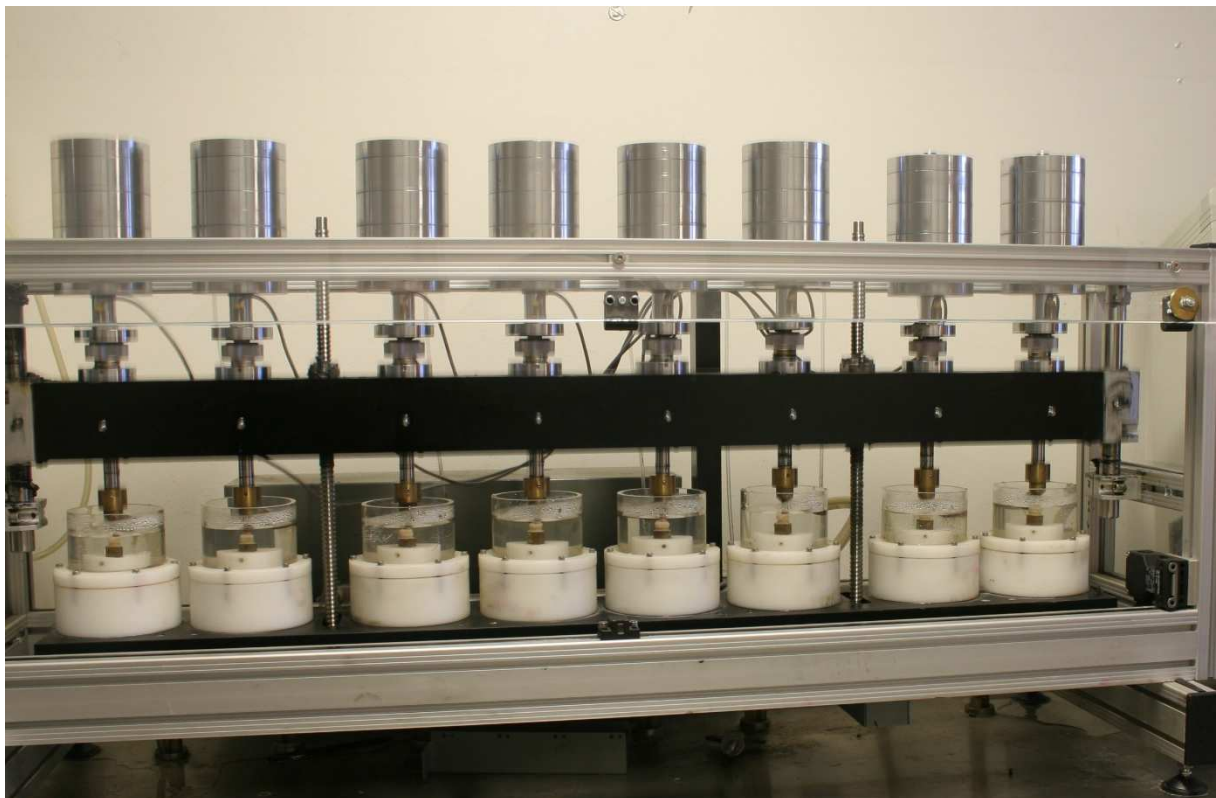


Figure 14: Test specimens during dynamic fatigue loading in the chewing simulator.

### 3.3.2. Microscopic evaluation

After the thermal-dynamic loading tests, all specimens were examined visually and under low power ( $5.8\times$ ) stereo-magnification with the use of an optical microscope (Carl Zeiss, Jena, Germany) and representative photographs of all specimens were taken. The microscopic evaluation was performed to identify the development of cracks, microcracks and any possible fractures that were undetected by visual inspection.

### 3.3.3. SD-OCT evaluation

Specimens that underwent the thermal-dynamic loading were scanned again by means of SD-OCT technology. Possible effects of the artificial aging process were assessed by comparing the before- and after-images of the aged specimens. Images of the same 5 ROI were re-taken. All images were examined to precisely detect any development of cracks and microcracks, especially those located at the luting interface. Additionally, all images were checked to verify any changes, up to a penetration depth of 2.5 mm, that might have occurred to the superficial layer of the dentin, ceramic restorative material or luting resin layer after 1,200,000 of dynamic loading cycles.

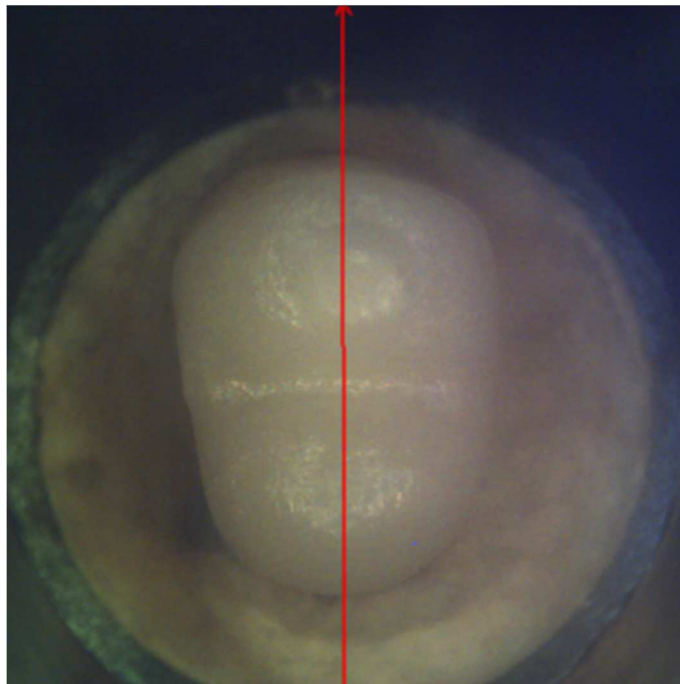
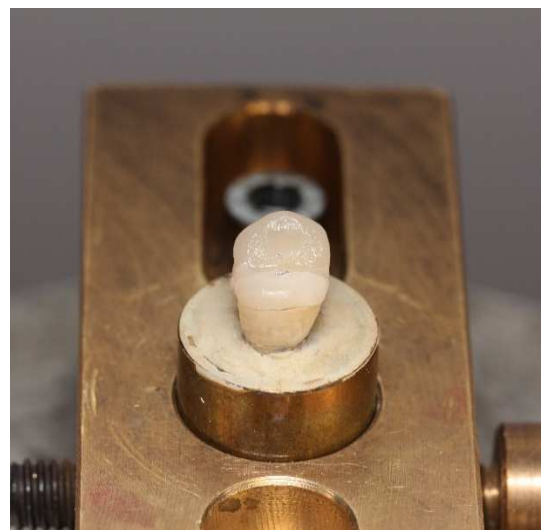
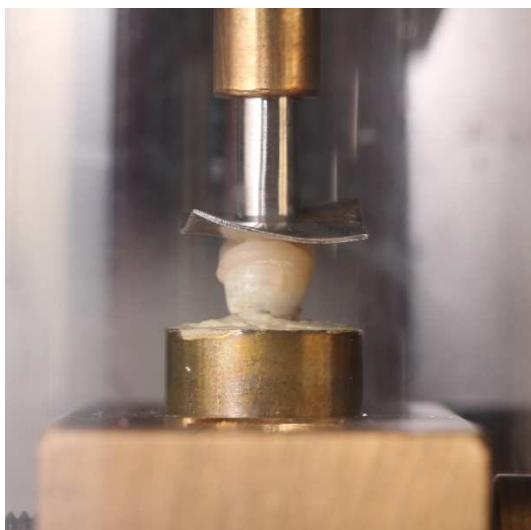
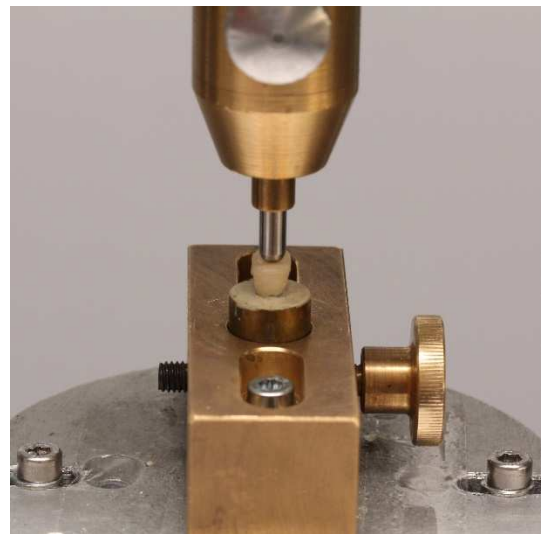
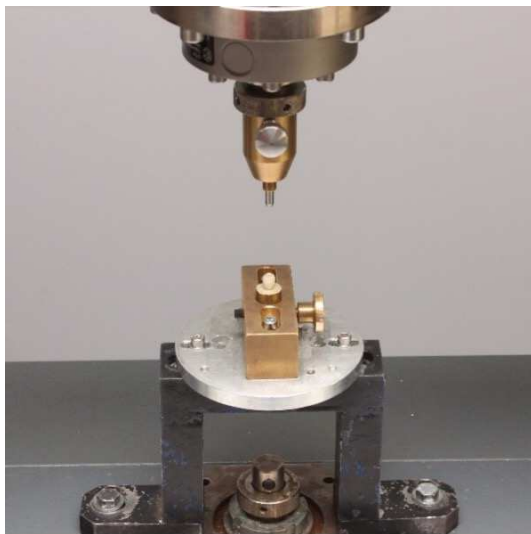


Figure 15: An example of a region of interest (ROI).



### 3.3.4. Quasi-static loading test

All the dynamic loading surviving specimens and the other half of subgroups were subjected to quasi-static loading using a universal testing machine (Zwick Z010/TN2A, Zwick, Ulm, Germany) until failure. A stainless-steel cylinder with a 6 mm ball-like tip was positioned on the center of the main fissure so that the load would be evenly applied to both cusps. A 0.5 mm tin foil (Zinnfolie, Dentaurum, Ispringen, Germany) was placed between the ball-like tip and the specimens to distribute the stresses uniformly. The load was applied at a cross-head speed of 1 mm/min. The loads required to fracture the restorations were recorded using the software of the universal testing machine (testXpert II V 3.3, Zwick/Roell).



Figures 16-19: Quasi-static loading process.

### **3.3.5. Microscopic evaluation**

After the quasi-static loading test, all specimens were examined again visually and under low power (5.8×) stereo-magnification with the use of an optical microscope (Carl Zeiss, Jena, Germany) and representative photographs of all failed specimens were taken. The microscopic evaluation was performed to assess the mode of failure. All tested specimens were examined for development of extensive cracks and any incipient fractures. The mode of failure was classified according to Guess et al. <sup>[61]</sup> into four categories in accordance with the following criteria: (I) Extensive crack formation within the restoration; (II) cohesive fracture within the restoration; (III) adhesive fracture between the restoration and tooth structures; (IV) longitudinal fracture of the restoration and tooth.

### **3.3.6. Statistical analysis**

The collected data was revised, coded, tabulated and introduced to a PC using Statistical Package for Social Science (SPSS 20.0 for Windows, SPSS Inc, Chicago, IL, USA). The Shapiro-Wilk test was performed on all the groups and revealed that all data was normally distributed. Three-way ANOVA was used to detect interaction between different variables and Tukey's post-hoc was performed to list pairwise comparisons between groups. Chi-Square Test and Fischer's Exact Test of Independence were conducted to test the correlation between nominal variables.

## 4. RESULTS

### 4.1. Dynamic loading

Only one specimen in subgroup **IST** did not withstand the artificial aging process and was rated as a failure. All specimens of subgroups **WT, WS, ITT, ITS** and **ISS** survived a total of 1,200,000 cycles of dynamic loading. The failed specimen fractured after 1,100,000 cycles. The fracture occurred at the buccal cusp and the fractured part of the ceramic was lost in the course of the thermo-cycling switching intervals.

### 4.2. Microscopic evaluation

The surviving specimens were rated as a success when no cracks or microscopic cracks were detected, or as partial success when crack formation took place. Seventy-seven percent of the surviving specimens, after artificial aging, did not show any damage and were rated as success, whereas 23% of them developed cracks or microcracks and were therefore rated as partial success. The success and failure rates as well as their distribution after artificial aging are shown in Table 4. Wear of the ceramic at the surface of contact was apparent for all specimens (Figures 20-21).

Table 4: Success and failure rates (in %) for all groups after artificial aging.

Group	Survival		Failure
	Total success	Partial success	
<b>WT</b>	<b>75%</b>	<b>25%</b>	<b>0%</b>
<b>WS</b>	<b>87.5%</b>	<b>12.5%</b>	<b>0%</b>
<b>ITT</b>	<b>75%</b>	<b>25%</b>	<b>0%</b>
<b>ITS</b>	<b>75%</b>	<b>25%</b>	<b>0%</b>
<b>IST</b>	<b>62.5%</b>	<b>25%</b>	<b>12.5%</b>
<b>ISS</b>	<b>75%</b>	<b>25%</b>	<b>0%</b>

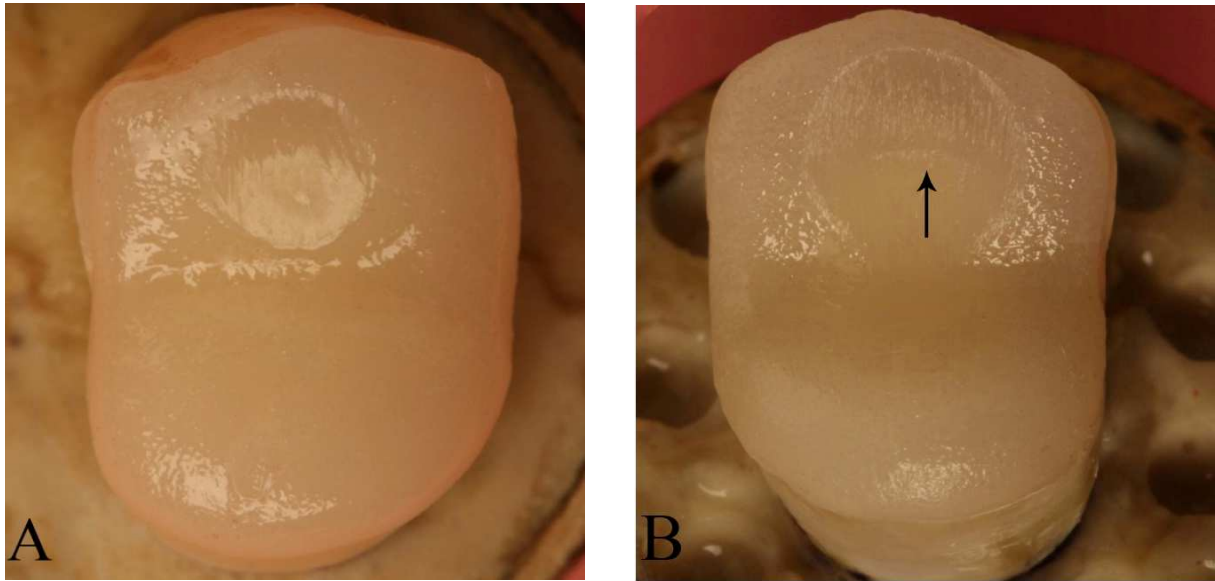


Figure 20: A: Occlusal view of an intact undamaged specimen after the artificial aging, B: Occlusal view of a specimen that developed cracks after the artificial aging

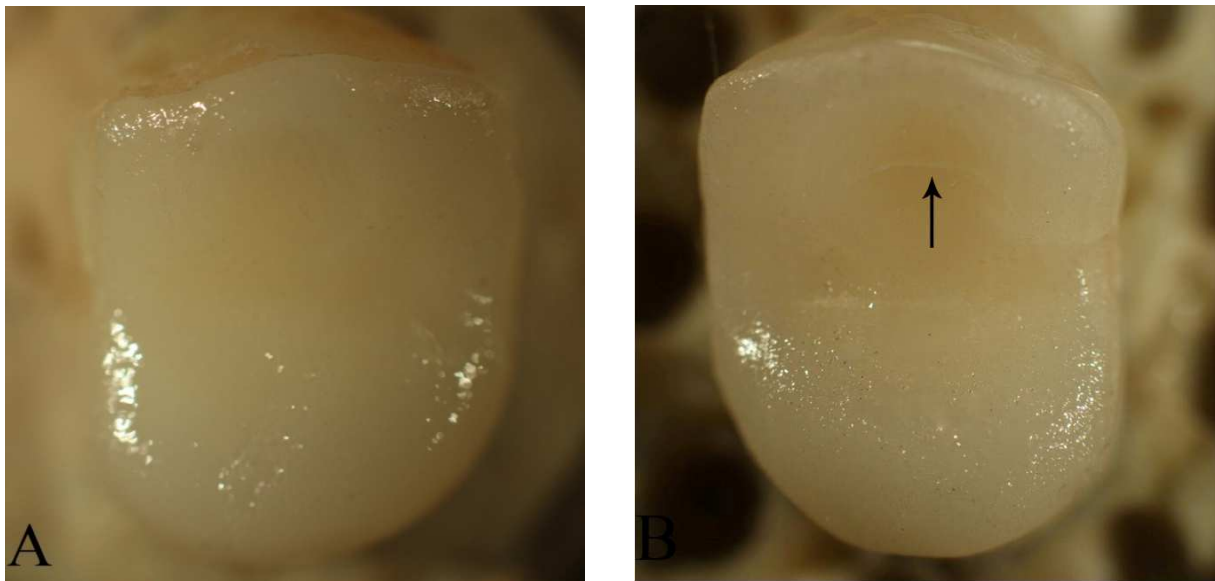


Figure 21: Microscopic photos of the specimens after the artificial aging. A: An intact undamaged specimen, B: A specimen that developed cracks

### 4.3. SD-OCT evaluation

The 2D and 3D SD-OCT imaging of the specimens revealed no changes to neither the tooth structure nor the luting resin after the thermo-dynamic loading in the chewing simulator.

The formation of cracks was detected in 11 restorations as well as the wear of ceramic at the surface of contact with the steatite ball during dynamic loading for all specimens.

The fracture of one specimen during the dynamic loading could not be traced to any defects prior or after the luting process.

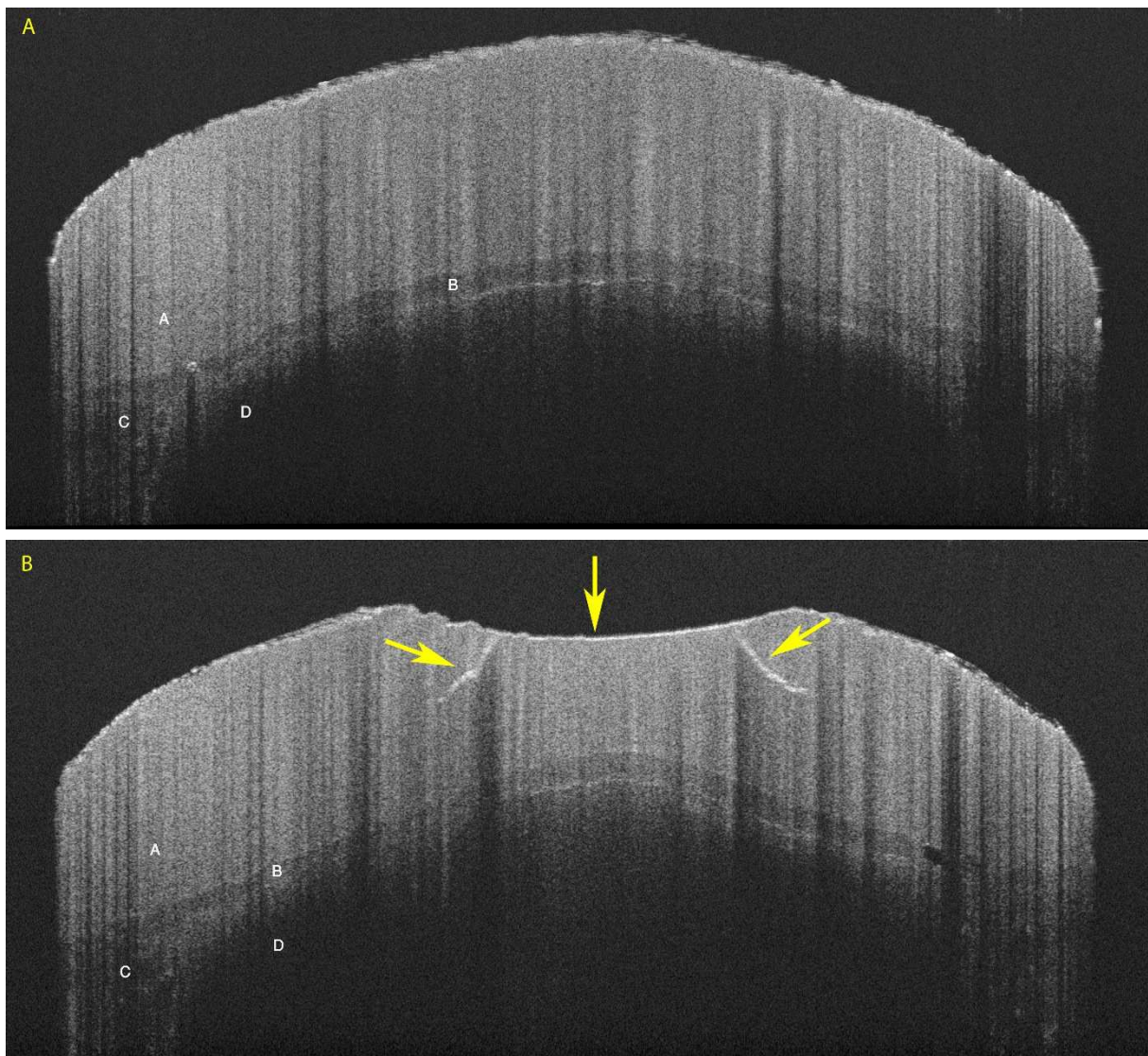


Figure 22: Paired 2D-OCT scans of a mesio-distal section of a specimen. A: Before thermal-dynamic loading, B: After thermal-dynamic loading. (Inside the Pictures: A: Ceramic, B: Luting resin layer, C: Enamel margins, D: Dentin). The arrows indicate the formation of cracks and wear of ceramic at the appointed areas.

The formation of cracks did not follow a unified pattern, and the path of development varied from superficial cone crack formation (Figure 22) closer to the surface to deep progression starting from the adhesive interface (Figure 23).

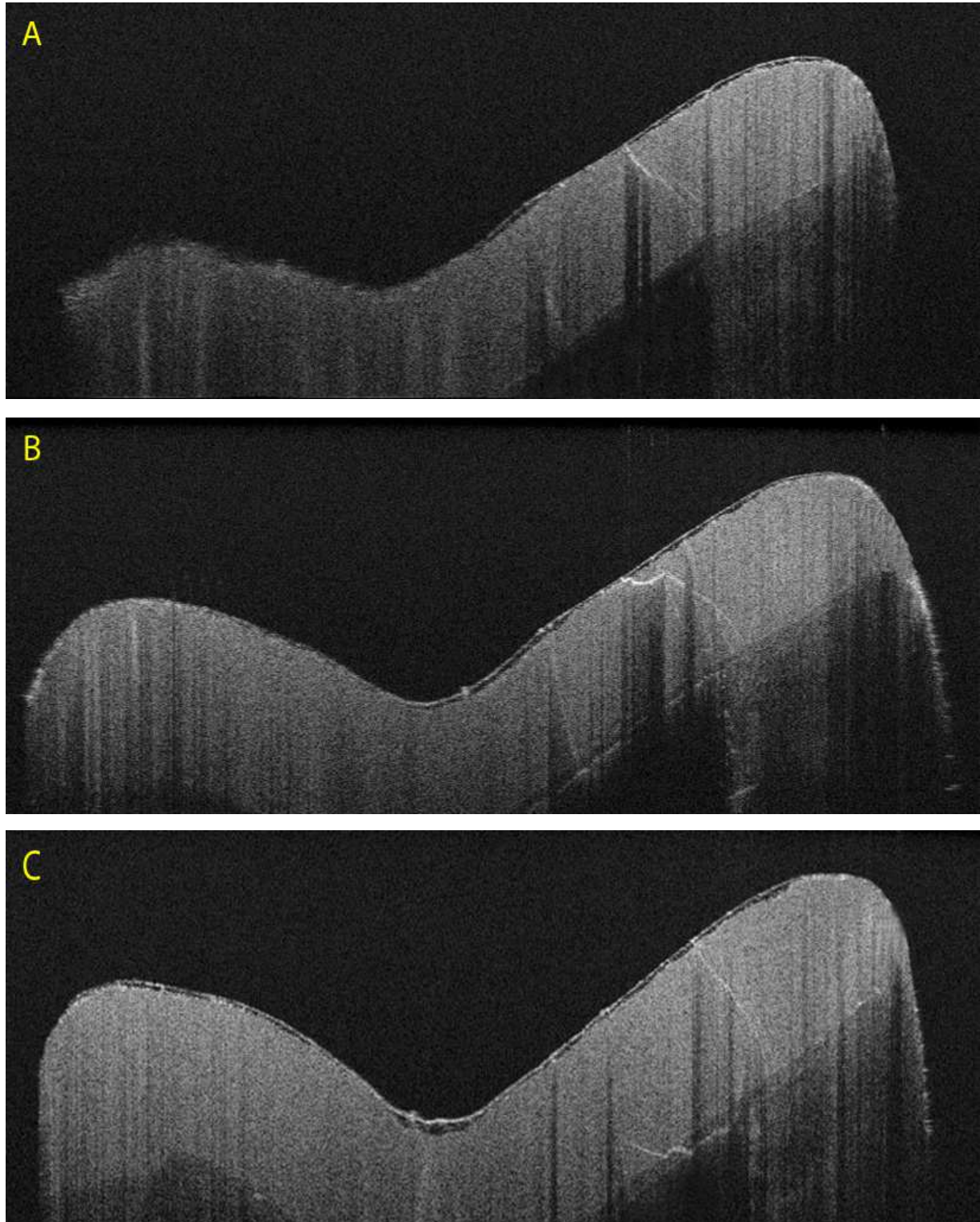


Figure 23: A-C: Three randomly selected images from a 3D-OCT scan showing the development of cracks in a buccal-lingual section of a specimen after dynamic loading.

#### 4.4. Quasi-static loading

All specimens (n=16) of groups **WT**, **WS**, **ITT**, **ITS** and **ISS** and 15 specimens of group **IST** (after failure of 1 specimen during dynamic loading) were subjected to quasi-static loading until fracture using a universal testing instrument (Zwick Z010, Zwick).

The mean fracture load for groups **WT** and **WS** were  $1,198 \pm 379$  N and  $1,344 \pm 175$  N respectively. Each subgroup showed the following mean fracture loads:  $1,275 \pm 427$  N for subgroup **WT** which was subjected to dynamic fatigue loading and  $1,122 \pm 336$  N for subgroup **WT** which was directly subjected to quasi-static loading. The mean fracture loads for **WS** subgroups were  $1,293 \pm 152$  N and  $1,396 \pm 191$  N, respectively.

Artificial aging as a factor did not have a significant effect ( $p>0.05$ ) on the resulting bond strength after quasi-static loading, nor did ( $p>0.05$ ) the followed etching protocol (total/selective). However, the initial treatment protocol (delayed dentin sealing/immediate dentin sealing) influenced the fracture strength significantly ( $p\leq 0.001$ ). When immediate dentin sealing was followed, the specimens showed a significantly higher resistance to fracture loads than those obtained in groups **WT** and **WS** with mean fracture loads of  $1,777 \pm 283$  N and  $1,720 \pm 254$  N for groups **ITT** and **ITS** and of  $1,780 \pm 556$  N and  $1,848 \pm 333$  N for groups **IST** and **ISS**, respectively. Table 5 shows mean fracture loads.

Table 5: Mean fracture loads for all subgroups in Newton.

<b>Subgroup</b>	<b>Artificial</b>	<b>Mean</b>	<b>Total</b>
<b>WT</b>	Yes	1,275	1,198
	No	1,122	
<b>WS</b>	Yes	1,293	1,344
	No	1,396	
<b>ITT</b>	Yes	1,804	1,777
	No	1,750	
<b>ITS</b>	Yes	1,781	1,720
	No	1,659	
<b>IST</b>	Yes	1,814	1,780
	No	1,746	
<b>ISS</b>	Yes	1,853	1,848
	No	1,844	

No significant interaction ( $p>0.05$ ) was detected between the three different variables (immediate treatment/pre-luting treatment/artificial aging) regarding the effect on the resulting bond strength. Table 6 shows the results of Three-Way-ANOVA test.

Table 6: Test of between-subjects effects (Three-Way ANOVA)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	6076535.615 <sup>a</sup>	11	552412.329	4.272	.000
Intercept	249224927.510	1	249224927.510	1927.279	.000
Initial treatment	5616215.146	2	2808107.573	21.715	.000
Pre-luting treatment	66202.510	1	66202.510	.512	.476
Artificial aging	60450.844	1	60450.844	.467	.496
Initial treatment * Pre-luting treatment	167906.396	2	83953.198	.649	.525
Initial treatment * Artificial aging	18006.813	2	9003.406	.070	.933
Pre-luting treatment * Artificial aging	40385.010	1	40385.010	.312	.578
Initial treatment * Pre-luting treatment * Artificial aging	107368.896	2	53684.448	.415	.662
Error	10862407.875	84	129314.379		
Total	266163871.000	96			
Corrected Total	16938943.490	95			

Dependent Variable: Fracture Strength

a. R Squared = .359 (Adjusted R Squared = .275)

Tukey HSD was applied for post-hoc pairwise comparisons between groups. A significant interaction ( $p=0.0001$ ) was detected between delayed dentin sealing group and IDS/Total Etching and IDS/selective etching groups. No interaction was detected ( $p>0.05$ ) between IDS two groups (total/selective). Table 7 shows the results of Tukey's post hoc test.

Table 7: Pairwise comparisons between groups with significant differences (Tukey HSD)

(I) Initial treatment	(J) Initial treatment	Mean Difference (I*J)	Std. Error	Sig.	95% Confidence Interval	
					Lower	Upper
W	IT	-477.1563*	89.90077	.000	-691.6557	-262.6568
	IS	-542.7188*	89.90077	.000	-757.2182	-328.2193
IT	W	477.1563*	89.90077	.000	262.6568	691.6557
	IS	-65.5625	89.90077	.747	-280.0619	148.9369
IS	W	542.7188*	89.90077	.000	328.2193	757.2182
	IT	65.5625	89.90077	.747	-148.9369	280.0619

Based on observed means.

The error term is Mean Square (Error) = 129314.379.

\*. The mean difference is significant at the .05 level.



#### 4.5. Mode of failure

Mode I, which is considered the least catastrophic failure mode, was the least to occur in all groups (Figure 24). Contrariwise, mode III and mode IV which involve fracture in the tooth or both the tooth and the root, and which are considered the most catastrophic types of failure, together amounted up to 75% of the most commonly detected failure mode for groups **WT**, **WS**, **ITT**, and **ISS** and to 50% for groups **ITS** and **IST**. Table 6 shows the distribution of mode of failure amongst the groups.

Table 8: The distribution of failure modes for all groups in %.

<b>Groups</b>	<b>Mode I</b>	<b>Mode II</b>	<b>Mode III</b>	<b>Mode IV</b>
<b>WT</b>	6.25%	18.75%	37.5%	37.5%
<b>WS</b>	6.25%	18.75%	37.5%	37.5%
<b>ITT</b>	6.25%	18.75%	31.25%	43.75%
<b>ITS</b>	12.5%	37.5%	12.5%	37.5%
<b>IST*</b>	6.25%	37.5%	25%	31.25%
<b>ISS</b>	6.25%	18.75%	31.25%	43.75%

\* 1 specimen fractured during dynamic loading, whereas 15 specimens were subjected to quasi-static loading.

Mode I: Extensive crack formation within the restoration;

Mode II: cohesive fracture within the restoration;

Mode III: adhesive fracture between the restoration and tooth structures;

Mode IV: longitudinal fracture of the restoration and tooth

One specimen from group **IST** fractured during the artificial aging process and the failure was of an adhesive nature between luting resin and tooth structure (Mode III). All the other specimens were loaded to failure in the universal testing machine and the mode of failure was examined and determined after the test. Modes of failure are illustrated in Figures: 24-27.

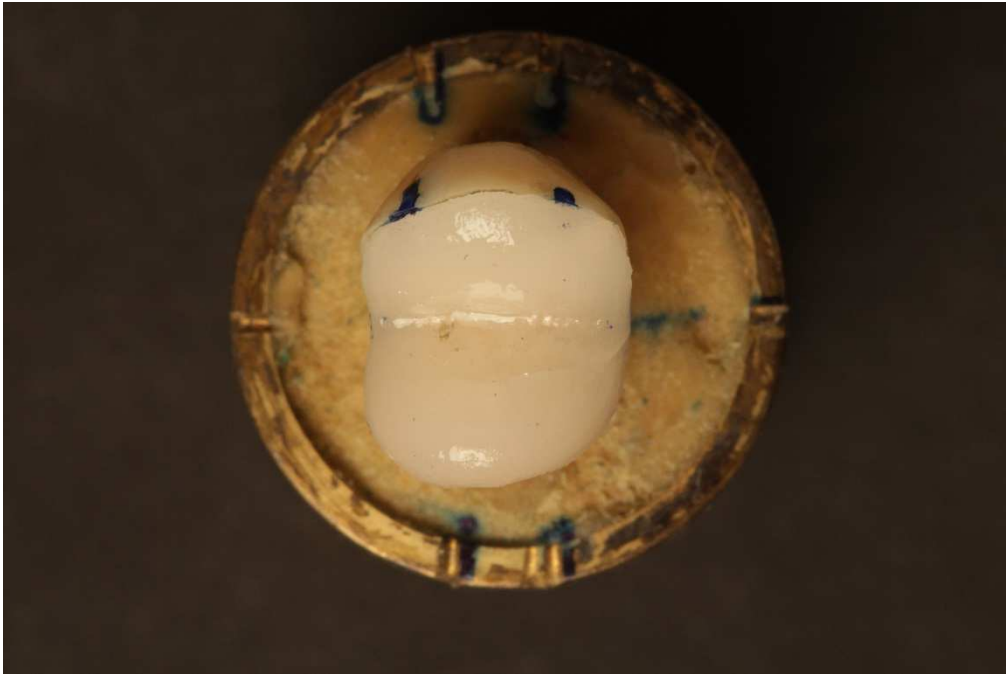


Figure 24: Mode of failure (I).



Figure 25: Mode of failure (II).

The failure mode was neither influenced by the dentin pre-treatment method nor by the etching protocol ( $p>0.05$ ) but it was affected remarkably by the pre-existence of cracks ( $p\leq 0.001$ ). All specimens with pre-existing cracks showed failure mode IV after the quasi-static loading.



Figure 26: Mode of failure (III).

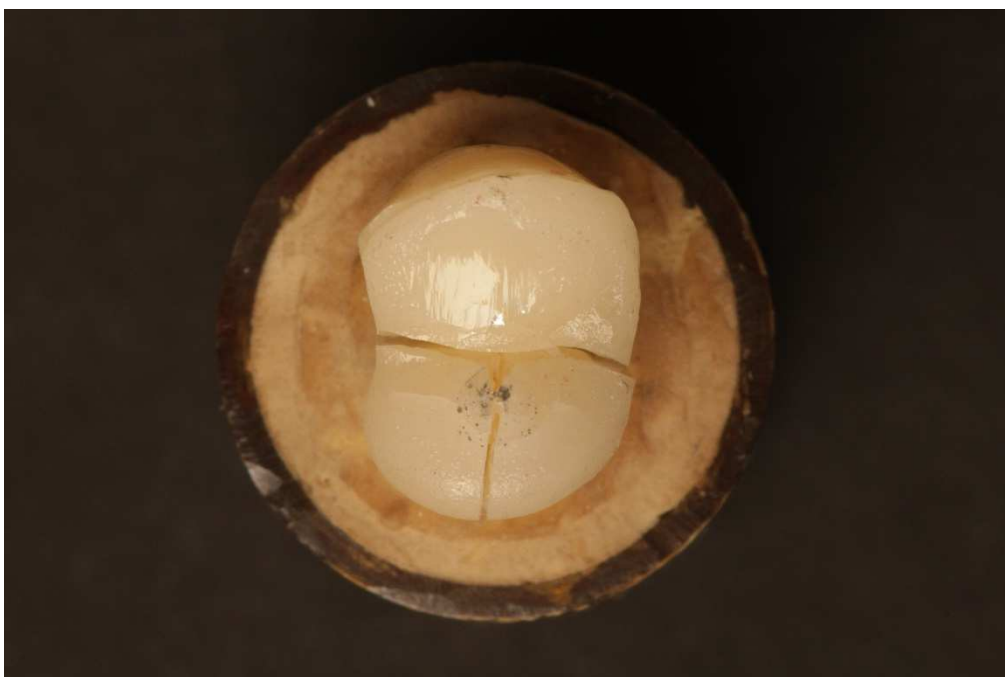


Figure 27: Mode of failure (IV).

## 5. DISCUSSION

### 5.1. Discussion of methodology

In order to minimize the number of variables, the study was designed to limit the variables exclusively to thin occlusal veneers with identical thickness, all made of lithium disilicate ceramic CAD/CAM blocks and adhesively bonded to dentin using the same materials, but different bonding protocols.

#### 5.1.1. Natural teeth

Natural human extracted teeth were used in the present study, as they provide a good clinically-relevant model for research regarding dentin bonding<sup>[150]</sup>. Laboratory evaluations of new bonding materials and methods using extracted teeth are necessary to preliminary determine their effectiveness and limitations before clinical trials should be conducted<sup>[35]</sup>.

#### 5.1.2. Embedding in autopolymerizing acrylic resin

Teeth were coated with a gum resin of 0.25 mm thickness 2.0 mm below the cemento-enamel junction to mimic the periodontal ligaments. This uniform coating was described in previous studies and proved to permit an artificial movement of  $100 \pm 30 \mu\text{m}$  in the horizontal direction and  $65 \pm 21 \mu\text{m}$  in the vertical direction under a force of 5 N, which is similar to the physiological teeth mobility and which in turn allows the absorption and distribution of stresses from the teeth to the alveolar bone<sup>[75, 80]</sup>. Specimens were then fixed in auto-polymerizing polyester resin that mimics the human alveolar bone<sup>[23]</sup> in a technique that proved to be efficient and reliable in previous studies<sup>[6, 23]</sup>, as well as granting a better stress absorption and distribution from the teeth into the alveolar bone.

#### 5.1.3. Lithium disilicate CAD/CAM blocks

All the restorations were milled out of lithium disilicate blocks, which have supreme esthetic and mechanical properties even for restoration with minimal layer thickness as lithium disilicate is the strongest form of glass-ceramics available, with fracture toughness of 2.5-3 MPa.m<sup>0.5</sup>, flexural strength of 500-530 MPa, excellent translucency and esthetics<sup>[3, 60, 64]</sup>. It also allows, according to the recommendations of the manufacturer, a minimum thickness of 0.4 mm for the productions of anterior veneers. Moreover, it can be easily chemically etched

using hydrofluoric acid. High survival rates have been documented when lithium disilicate restorations were used as partial coverage restorations <sup>[2, 62, 137]</sup>, as well as other types of restorations <sup>[44, 53, 124]</sup>.

#### **5.1.4. Thickness of the occlusal veneers**

The thickness of enamel varies widely in human teeth which makes the standardization of preparation's depth not possible, regardless of the control method. Therefore, a standardized location of the preparation; superficial dentin layer just below the EDJ, followed by a standardized thickness of the ceramic restorations (0.5 mm at fissures and 0.8 mm at cusps) were controlled and regulated.

A ceramic thickness of 0.8 mm at cusps and of 0.5 mm at fissures was selected in order to conserve as much as possible the intact tooth structure, especially in cases of severe wear, which are usually associated with a compensated eruption <sup>[139]</sup>.

Clinically, the loss of occlusal contacts caused by attrition, abrasion, corrosion and abfraction may be compensated by tooth over-eruption, which in term restricts the thickness of the required occlusal restorations <sup>[138]</sup>. In such clinical cases, a minimally invasive treatment, that conserves as much as possible of the remaining sound tooth structure, as well as thin restorations, that are better suited for the restricted occlusal space, are the treatment of choice when compared to their full coverage counterparts <sup>[61, 68, 139]</sup>.

A study by Guess et al advocated the use of thin ceramic partial coverage restorations, as it concluded that a lesser number of catastrophic failure modes were observed when the ceramic thickness was reduced for premolar partial coverage restorations <sup>[61]</sup>. Additionally, a study by Saase et al supported the use of occlusal veneers with thicknesses of 0.5 mm at the fissures and 0.8 mm at the cusps with registered fracture resistance of up to 2,355 N.

#### **5.1.5. Adhesive Luting**

The durability and survival of restorations milled out of silica-based ceramics such as lithium disilicate depends basically on the adhesive bonding as it plays a crucial determining factor in this aspect, especially in the absence of a retentive preparation form such as it is the case in occlusal veneers preparation.

The etching and silanization of the intaglio surfaces of lithium-disilicate ceramic restorations substantially improve their bonding strength. When etching lithium-disilicate

ceramics with hydrofluoric acid, the acid reacts with silica-containing glass matrix and forms hexafluorosilicates, resulting in an exposed crystalline structure and a rougher ceramic surface. The resulting roughly etched surface enhances the micromechanical retention pattern of the ceramic surface as well as improving the surface energy before silanization [19, 20, 69, 155]. The silane agent is applied as a chemical coupler to chemically link and bond the inorganics, the glass-ceramic surface, to organics which is the luting resin [4, 10, 72, 78].

The choice of the luting resin was in accordance to the manufacturer's recommendations. Variolink Esthetic is recommended for the adhesive luting of restorations milled out of IPS e.max CAD blocks. It is also recommended by the manufacturer for the adhesive luting of thin and ultra-thin veneers and occlusal veneers owing to its wide-range shade variety and its color stability after illumination test and water storage [126]. Moreover, Variolink Esthetics, in combination with Adhese Universal, which is compatible with all etching protocols, enjoys excellent bond strengths to both enamel and dentin [66, 126]. Additionally, the combination of Variolink Esthetic and the universal primer Monobond Plus was found to result in durable bond strength values even after aging conditions when compared to the following combinations of universal adhesives and luting resins; Scotchbond Universal and Rely X Ultimate, OptiBond XTR Adhesive and NX 3 and All Bond Universal and Duo Link Universal [45]. Thus, Variolink Esthetic, in combination with Adhese Universal and Monobond Plus, is a reliable choice to achieve adequate bonding between thin lithium disilicate occlusal veneers and both enamel and dentin substrates.

#### **5.1.6. Loading tests**

New materials, methodologies, preparation forms and restorations' designs should all be laboratory tested before they could be clinically recommended. Scientific data in regard to the mechanical and biological behavior, as well as efficiency and success evaluation should be collected to support their clinical usage. However, clinical oriented studies are associated with higher costs and are time consuming [76]. Well-constructed and designed laboratory studies are effective alternatives to clinical studies and can give an estimation to their likely clinical success before clinical use is recommended.

In the late 90s Kelly concluded that all-ceramic materials should be pre-clinically tested in similar situations to those of the oral cavity so that the resulting failures and registered results are equivalent and comparable to those resulting in the clinical situation. The fundamental

requirements to obtain relevant significance are: clinically relevant substrates, adequate contact area with the specimen during the tests, cyclic dynamic loading and wet conditions <sup>[74]</sup>.

The clinical survival rate of all-ceramic restorations is influenced by a combination of several factors, such as the preparation design, restoration's geometry and thickness, mechanical properties and microstructure of the ceramic material, processing technique, final surface finishing of the ceramic and most importantly the luting material and bonding technique [31, 59, 60, 86, 107, 127, 143]. Moreover, several factors influence the fracture loads of all-ceramic restorations while tested. The test conditions such as storage conditions, the type of fatigue loading (monotonic or cyclic, dynamic or thermal-dynamic), the direction of the loading forces and the location to which it is applied <sup>[74]</sup>. Therefore, the current study was designed to standardize all these factors as close as possible to the clinical conditions.

Studies have shown that ceramics under the cyclic loading and in presence of wet conditions show a more clinically-relevant modes of failure and damage that cannot be incited or initiated under quasi-static loading <sup>[74]</sup>. This is especially true in cases of bulk fracture, which are caused by subsurface and microstructural weakness in the ceramic resulting from fatigue, and which cannot be induced by only subjecting the ceramics to static loading testing <sup>[74, 118]</sup>. Additionally, cyclic loading is presumed to decrease the fracture strength of all-ceramic restorations <sup>[74]</sup>. Moreover, wet conditions and environment are found to negatively influence the failure loads, facilitate the growth of cracks and causing surface degradation of the material mimicking the conditions that restorations in the oral cavity undergo and thus will lead to a more clinically-relevant results <sup>[21, 101]</sup>.

In order to test the effect of fatigue and dynamic loading on the all-ceramic restorations, and compare it to the effect of single static loading, half of the specimens were subjected to artificial aging, prior to quasi-static loading, by applying thermal-dynamic loading under controlled moisture in a dual-axis chewing simulator with parameters simulating the conditions of the oral cavity and similar to those reported in other parallel studies in the literature <sup>[2, 137]</sup>.

The aim of artificial aging is subjecting the specimens to cyclic fatigue comparable to the physiological fatigue found in the oral cavity. In clinical service, restorations are introduced to an average of 250,000 masticatory cycles per year <sup>[32, 33, 134]</sup>. Therefore, a total of 1,200,000 masticatory cycles are performed through the artificial aging process to correspond to five years of clinical service in the oral cavity <sup>[76]</sup>. In humans, chewing incorporates a high number of low cyclic loads rather than monotonic loads, thus the specimens were introduced to similar

parameters and the chewing simulator was designed to procreate a fatigue of cyclic pattern <sup>[74]</sup>. A ceramic steatite ball was used as the antagonist owing to the fact that steatite ceramic has a hardness index similar to that of the human enamel (Vicker's scale) and therefore makes for a suitable substitute in wear tests <sup>[74]</sup>. The applied force was set to 10 kg, which corresponds to a loading force of 98 N. The occlusal forces vary widely amongst individuals with a range between 10 and 120 N <sup>[61]</sup> but was found to rarely exceed 70 N in an average person with normal chewing <sup>[7, 26]</sup>. The choice of 98 N is therefore representative of the functional forces in the oral cavity and in agreement with similar previous studies on occlusal veneers <sup>[2, 137]</sup>.

In regard to quasi-static loading and although it is considered to induce failures that are not similar to the ones occurring in the clinical situations <sup>[74]</sup>, nonetheless it is helpful in predicting the material's behavior and to compare different variables.

## 5.2. Discussion of results

### 5.2.1. Dynamic loading

In the present study, each test group was divided into two subgroups and one of them was exposed to artificial aging before the fracture strength test was performed. During the fatigue testing of the six subgroups, all specimens of subgroups **WT**, **WS**, **ITT**, **ITS** and **ISS** survived 1,200,000 cycles of exposure to thermo-dynamic loading. In group **IST**, only one specimen did not totally withstand the dynamic fatigue loading and showed fracture of the ceramic at the buccal cusp during the test. The failure occurred at the end of the test after 1,100,000 cycles.

Before surviving specimens were subjected to quasi-static loading, they were checked visually, under a microscope and scanned in 2D and 3D by the SD-OCT technology to detect any incipient cracks, fractures or damages that might be caused by the artificial aging process. The presence of cracks in 23% of the surviving specimens was rated as partial success, whereas crack-absent specimens, which accumulate up to 77% of the surviving specimens, were considered as success. This outstanding performance of thin glass-ceramic occlusal veneers and the ability to withstand and survive thermo-dynamic fatigue equal to five years of clinical service makes it a suitable choice for restoring occlusally damaged premolars.

Other laboratory studies support the use of thin and ultra-thin occlusal veneers as well after exploring and testing their ability to withstand the functional loading equivalent to five years of clinical service <sup>[2, 61, 97, 137-139]</sup>. In one of these studies, occlusal veneers fabricated from



lithium disilicate blocks with a thickness of 0.5-0.8 mm and adhesively bonded to enamel survived 1,200,000 cycles of fatigue loading without showing any signs of fractures, chipping or cracks [2]. In another study, occlusal veneers milled from lithium disilicate ceramic and of three different thicknesses (0.3-0.6 mm/ 0.5-0.7 mm/ 0.7-1.0 mm) were adhesively bonded to dentin and subjected to 600,000 thermo-dynamic cycles, which is equivalent to 2.5 years in the oral cavity, in a chewing simulator. All specimens of groups with thicknesses 0.3-0.6 mm and 0.7-1.0 mm withstood the artificial aging process completely. The survival rate of the second group with the thickness 0.5-0.7 mm was only 50%, as half of the specimens did not survive the artificial aging, which contradicts the results of our study [137]. This contradiction might be explained by the different teeth tested (molars vs. premolars), different convergence angle prepared between the cusps (150° vs. 120°) and the different adhesive bonding protocol and materials used.

### 5.2.2. Quasi-static loading

The average maximum biting forces on posterior teeth are reported to range between a minimum of 150 and a maximum of 665 N [24] and the minimum fracture strength recommended for posterior restorations is 500-700 N [79]. However, bruxism and other parafunctional activities can induce higher bite forces [106]. The specimens of all groups showed a fracture resistance higher than the maximum values of physiological occlusal forces expected in the posterior area. The mean fracture strength ranged from a minimum of  $1,122 \pm 336$  N to a maximum of  $1,853 \pm 333$  N. The lowest recorded value was 690 N for a specimen in group **WT**, a value that is still within the average recommended fracture resistance in the posterior area. Moreover, specimens in groups **ITT**, **ITS**, **ITS** and **ISS** showed a fracture strength that was higher than the maximum physiological occlusal forces in the posterior area and that was significantly higher ( $p \leq 0.001$ ) than the fracture resistance obtained in both groups that followed the DDS protocol (**WT**, **WS**).

The higher fracture strength obtained by the immediate application of the DBA on freshly cut dentin could be attributed to various reasons. For one, the freshly exposed dentin provides the most favorable substrate for bonding and this is only possible at the time of preparation when the dentin is still uncontaminated [92, 116, 117]. Additionally, the pre-curing of the dentin bonding agents during the immediate dentin sealing procedure means that the resin is cured, polymerized and had infiltrated the hybrid layer long before the adhesive luting while through the traditional procedure, where the adhesive layer is left unpolymerized, the pressure

associated with the placement of the luting resin and the seating of the restoration might lead to the collapse of the collagen network or the hybrid layer of the uncured dentin-resin [8, 17, 36, 84, 100]. Furthermore, when the dentin is sealed, at the time of the preparation, and because of the postponed exposure to the customary occlusal forces, the dentin bonds are allowed to develop freely [109].

This conclusion corresponds to that of previous studies concentrating on the application of IDS, in which higher bond strength or fracture strength values were obtained when IDS was followed, although most of the studies in the literature evaluated the positive effects of IDS on the bond strength, whether shear bond strength or tensile bond strength [8, 39, 84, 92, 98, 117, 125], rather than on the fracture strength [57]. When Bertschinger et al. [8] first modified the traditional luting procedure and followed the technique of dual application of DBA, the resulting bond strength values when applying IDS were much higher and ranging from 16.3 to 19 MPa in comparison to values ranging from 0.3 to 14.9 MPa when following DDS [8]. Similar results were concluded by Paul and Schärer [117] when the freshly exposed dentin was immediately coated by a DBA, using shear testing the recorded bond strength values when following IDS where twofold the values recorded when following the conventional DDS protocol [117]. Similar results were achieved by Magne and Douglas [90] when they applied and polymerized an adhesive layer after the preparation and before taking the impression. This improved and optimized method of applying the DBA led to an improved bond and restored the biomimetic behavior of ceramic veneers adhesively bonded to teeth [90].

In another study using microtensile testing for evaluation, following the IDS protocol with a delayed placement of the final restoration up to 12 weeks, resulted in a much higher bond strengths exceeding 45 MPa in comparison to bond strengths ranging from 1.8 to 11.6 MPa with the delayed dentin sealing groups [98]. A recent study evaluating the effects of IDS on the fracture strength of lithium disilicate veneers adhesively bonded to central incisors concluded that the highest mean fracture strength value of  $576 \pm 254$  N was obtained in the group where the whole preparation surface of the central incisors was created in dentin with the application of IDS in comparison to a mean value of  $314 \pm 137$  N for the same design and preparation but without the application of IDS [57]. However, these findings contradict the results of a laboratory study by Falkensammer et al. [48], where premolars were either immediately sealed by a 2 step self-etching adhesive after preparation or sealed just before adhesive luting. All prepared teeth received provisional restorations cemented with eugenol-free temporary interim cement for a week, then were conditioned in 4 different methods, and the final restorations were then

adhesively luted<sup>[48]</sup>. An explanation for the disagreement could be the difference in the applied testing method (shear bond strength vs. fracture strength), different surface treatment methods and the application of eugenol-free provisional cement for a week in the mentioned study, which could have interfered or penetrated the surface of the dentin or the hybrid layer.

The fracture strength achieved in our study by CAD/CAM fabricated lithium disilicate occlusal veneers, with a minimal thickness of 0.5 mm at fissures and 0.8 mm at cusps, was higher than the minimum recommended fracture strength for posterior restorations with values ranging from a minimum of  $1,122 \pm 336$  N to a maximum of  $1,853 \pm 333$  N. These findings are in accordance with the results of a recent study<sup>[2]</sup> comparing the fracture strength of thin premolar occlusal veneers (0.5-0.8 mm) made of four different materials. Although the preparation of the teeth as well as the adhesive bonding were strictly limited to the enamel the recorded median of fracture strength values for the lithium disilicate groups were 1,335 N and 1,560 N, which make it comparable to the values achieved in the current study despite dentin being a supposedly inferior substrate for adhesive bonding of ceramic restorations. Similar results were found in a study by Sasse et al.<sup>[137]</sup> in which thin CAD/CAM lithium disilicate occlusal veneers, with the identical thickness (0.5-0.8 mm), had a median fracture resistance of 1,105 N when adhesively bonded to exposed dentin with margins in the enamel. The study followed the conventional delayed dentin sealing protocol and IDS was not applied and the reported value is in similar range to the ones obtained in the current study for groups WT and WS when immediate dentin sealing was not followed. Interestingly the values are comparable although different teeth (molars vs. premolars) and different convergence for the preparation angle between the cusps ( $150^\circ$  vs.  $120^\circ$ ) have been used.

The effect of artificial aging on the tested specimens was not statistically significant, although one specimen fractured and 23% developed cracks. It also did not affect the ultimate fracture resistance of the specimens during the static loading test, even for specimens that developed cracks. This result contradicts the assumptions that dynamic loading in wet conditions leads to a significant decrease in the fracture resistance of the tested restorations<sup>[74, 144]</sup>. The contradiction in results could be attributed to the different ceramic systems, bonding to different substrates, different restoration types and ceramic thicknesses, as well as different cyclic loading protocols.

With reference to the etching protocol, the tested specimens did not show difference in their fracture resistance regardless whether total etching or selective etching was followed. In general, enamel bonding is less satisfactory when self-etching adhesive systems are used in

comparison to etch and rinse adhesive systems<sup>[114]</sup>. Furthermore, clinical and laboratory studies report a noticeable increase in enamel marginal defects when eliminating the etching step<sup>[120]</sup>. Contrariwise, etching dentin surfaces with phosphoric acid affects negatively the proficiency of self-etching adhesive systems and results in a less desirable hybrid layer morphology<sup>[148]</sup>. Consequently, to skip etching the dentin and only selectively etch the enamel is a procedure that allows maximizing and optimizing the bonding performance of the two different adhesion substrates, each according to its unique properties<sup>[120, 149]</sup>.

The current study shows that the use of a self-etching adhesive with selective pre-etching of the enamel margins is as efficient as the traditional total-etching protocol. These findings are in accordance with the results of a study where the bond strength achieved when only enamel was selectively pre-etched was similar to those obtained with conventional total-etching<sup>[47]</sup>. In another study, testing four different self-etching adhesive systems, the overall performance of the tested systems was distinctly optimized when selective etching was performed on the enamel margins<sup>[51]</sup>.

### 5.2.3. Failure mode

Fractures in all groups were mostly extensive and involved the underlying tooth coronal structure or the coronal tooth structure in addition to the root. The prevalence of mode III and mode IV were 75% for groups **WT**, **WS**, **ITT**, and **ISS**. For groups **ITS** and **IST** the prevalence was 50% and 56.25% respectively.

On the contrary, failure mode I, which consists of an extensive crack progress within the ceramic and which is considered as the least catastrophic mode of failure, was rarely observed in the examined specimens following quasi-static loading.

The prevalence of the catastrophic type of failure could be explained by the dentin being the substrate<sup>[61]</sup>. When minimal preparations are conducted and the underlying supporting tooth structure is preserved and predominantly located in the enamel, the tooth structure is rarely implicated in the fracture. Contrariwise, when the supporting underlying structure is mainly in dentin, catastrophic failures, that include the tooth structure or the tooth structure and the root, are to be expected<sup>[61]</sup>.

These observations were affirmed in a study by Guess et al.<sup>[61]</sup>, when testing different types of restorations. In this study, minimally invasive preparations located predominantly in the enamel mainly led to failure modes I and II following quasi-static loading, whereas

aggressive preparations exposing predominantly the dentin led to failure modes III and IV, of which the tooth structure or the tooth structure and the root were involved in fracture <sup>[61]</sup>.

These findings are consistent with the results of a recent study testing the fracture strength and failure mode of thin (0.5-0.8 mm) occlusal ceramic veneers with the same preparation design but adhesively bonded to the enamel <sup>[2]</sup>. In the aforementioned study, and for the two lithium disilicate restorations' groups; **LD1** which was only quasi-statically loaded and **LD2** which was first artificially aged then quasi-statically loaded, the most commonly detected failure mode was mode I with a frequency of 42% for **LD1** and 35% for **LD2**. Failure mode IV had a percentage of only 10% for the former and 9% for the latter <sup>[2]</sup>. This contrast in the prevalence of failure mode between both studies despite the identical preparation design, restoration's thickness, used restorative material and applied tests shows the major role of the underlying supporting substructure and adherent substrate (enamel vs. dentin), despite the higher fracture resistance reported in our study. In our study, the adhesion surface was mostly the dentin with only enamel margins, whereas, in the mentioned study, the adhesion surface was completely located in the enamel.

However, in another study on fracture strength and failure modes of lithium disilicate laminate veneers adhesively bonded to dentin, with and without the application of IDS, the study used a different failure mode categorization, but still the frequency of failures including a tooth fracture was 0% and 10% for the delayed dentin sealing two groups (partially bonded to dentin vs. completely bonded to dentin), and 40% and 20% for the immediate dentin sealing groups (partially bonded to dentin vs. completely bonded to dentin) <sup>[57]</sup>. This variety in results might be attributed to many reasons such as the different morphology of prepared teeth (centrals vs. premolars), different types of restorations (laminate veneers vs. occlusal veneers) and the different applied testing methods.

Interestingly, there was a correlation between the presence of cracks and the recorded type of failure. All specimens that developed cracks after the artificial aging, and following the quasi-static loading test, showed failure mode IV. This association between the occurrence of cracks in the ceramic and the catastrophic type of failure might be an indication to the role of the development of cracks in the clinical survival of the ceramic restorations and a hint for an increased risk of technical complications under clinical conditions. This conclusion should not be generalized as the number of affected specimens is low and such a correlation needs to be verified.

### 5.3. Study limitations

Laboratory studies try to simulate the clinical conditions but are never able to reflect real clinical conditions. The differences in results, fracture strength values, mode of failures and conclusions between the several available studies can be attributed to numerous factors. The use of different restorative materials with different thicknesses can explain some of the reported varieties. Additionally, the different bonding procedures applied and the use of different products for this purpose make it not possible to compare the resulting data.

The number of tested specimens and the use of water rather than artificial saliva during dynamic loading present limitations of the current study. The absence of the dentinal fluids and their possible influence on both the adhesive bonding and the acceleration of slow crack growth is considered as another shortcoming of the current laboratory study <sup>[74]</sup>.

Another factor that might be considered as a shortcoming of this study is the less than optimal standardization as a result of using natural extracted teeth. While natural human teeth offer the possibility of experimenting on real dentinal substrates, natural teeth as specimens mean variations in dimensions both mesio-distally and bucco-lingually, thus non-standardized specimens. Additionally, due to the requirement of exposing the dentin, the depth of the preparation could not be standardized as well.

Skipping the usage of a provisional restoration or the application of provisional cement, during the provisional stage, and their possible influence on the sealed dentin surface and possibly on the positive effects of immediate dentin sealing present another limitation of the study.

## 6. CONCLUSIONS

Within the limits of this laboratory study, the following conclusions can be drawn:

1. Thin premolar occlusal veneers fabricated from lithium disilicate ceramic and adhesively bonded to dentin demonstrated fracture resistance exceeding the recommended values for restoring posterior teeth, and might therefore be recommended, as an alternative to full coverage restorations, for the restoration of occlusally destructed teeth in the posterior region.
2. Following the immediate dentin sealing protocol can have a beneficial effect on the fracture resistance of the occlusal veneers when dentin is exposed during the preparation.
3. Selective etching of the enamel was as effective as total-etching.
4. The process of artificial aging did not influence the fracture strength of the specimens.
5. The development of cracks in the ceramic restorations during the thermo-dynamic loading tests did not affect the ultimate fracture resistance of the affected specimens.
6. The development of cracks during artificial aging process influenced the failure mode and led to a catastrophic mode of failure during quasi-static loading.
7. Optical coherence tomography is a unique non-invasive technology to examine and scan internal structures of teeth and restorative materials.

## 7. SUMMARY

Minimally-invasive preparations became the trend in dentistry allowing for a maximum preservation of tooth structure, while restoring esthetics and function. In the posterior area, minimally-invasive designs could save up to 40% of the intact tooth structure when compared to their conventional full-preparation counterparts.

This study was designed to measure the fracture strength and determine the failure mode of all-ceramic occlusal veneers fabricated from lithium disilicate and adhesively bonded to exposed dentin and to compare the fracture strength of the groups that followed the immediate dentin sealing to those that were conventionally bonded. Additionally, the influence of artificial aging and etching protocol were evaluated. Moreover, changes in the tooth structure, luting resin layer and ceramic before and after the artificial aging were analyzed.

Ninety-six freshly extracted sound maxillary premolars were identically prepared to receive occlusal veneers. The preparation was designed in the dentin with enamel margins to simulate the clinical condition of severely worn teeth. The teeth were divided into 3 main groups with 32 specimens each: without IDS (**W**), IDS/total etching (**IT**) and IDS/selective etching (**IS**). Restorations were milled from lithium disilicate blocks (IPS e.max CAD) with a thickness 0.5-0.8 mm. According to the surface etching protocol (total/selective) during adhesive luting with dual-curing luting resin (Variolink Esthetic), groups were further subdivided into 2 subgroups with 16 specimens each. One subgroup of each group was subjected to thermo-dynamic fatigue loading for 1,200,000 cycles in a dual-axis chewing simulator with 98 N load. Specimens that were artificially aged were scanned using the technology of spectral domain-optical coherence tomography (SD-OCT). All specimens were then quasi-statically loaded in a universal testing machine until fracture.

One specimen (1%) of group IST fractured during dynamic loading while all other specimens survived. Twenty-three percent of the surviving specimens developed cracks, while the others (77%) survived without showing any signs of cracks. The failure of one specimen and development of cracks in several others could not be traced by SD-OCT to any manufacturing or luting defects.

The mean fracture load for groups ranged from a minimum of  $1,122 \pm 336$  N to a maximum of  $1,853 \pm 333$  N exceeding the physiological bite forces. The fracture resistance of lithium



disilicate premolar occlusal veneers was significantly influenced ( $p \leq 0.001$ ) by the application of IDS. The groups that followed IDS had a significantly higher fracture strength than those that did not follow IDS. Neither artificial aging nor the development of cracks affected the fracture strength of the restorations ( $p > 0.05$ ). Selective pre-etching of enamel margins was as efficient as the traditional etch and rinse protocol.

The most common mode of failure for all groups was mode IV, which consists of a longitudinal fracture in both the ceramic and the tooth and which accumulated up to 39%. Mode I, which is a crack within the ceramic, was the least to take place with a prevalence of 8% for all groups. The mode of failure was not influenced by the dentin sealing method nor by the etching protocol ( $p > 0.05$ ), but the presence of cracks led to a catastrophic mode of failure for the affected specimens ( $p \leq 0.001$ ).

The positive results of the current study support the initiation of controlled clinical trials to evaluate the clinical outcome of thin occlusal ceramic restorations.

## 8. ZUSAMMENFASSUNG

Das minimalinvasive Konzept ist der Trend in der Zahnmedizin. Es ermöglicht eine maximale Erhaltung der Zahnstruktur, bei gleichzeitiger Wiederherstellung der ästhetischen und funktionellen Aspekte. Im Seitenzahnbereich können minimalinvasive Präparationen bis zu 40% der intakten Zahnstruktur im Vergleich zu konventionellen Präparationen schonen.

Ziel dieser Studie war es, die Bruchfestigkeit und den Versagensmodus von vollkeramischen dünnen okklusalen Prämolar-Veneers, die aus Lithiumdisilikat hergestellt und auf freiliegendem Dentin verklebt wurden, und den Einfluss der sofortigen Dentin-Versiegelung (IDS) auf die Bruchfestigkeit zu bestimmen. Dabei sollte der Einfluss einer Kausimulation von 1.200.000 Belastungszyklen sowie der Einfluss des Ätzprotokolls evaluiert werden. Anschließend sollte die Veränderungen, die durch künstliche Alterung auftreten, unter Verwendung von *spectral domain-optical coherence tomography* (SD-OCT) untersucht werden.

Die Okklusalfächen von 96 frisch extrahierten Oberkiefer-Prämolaren wurden schmelzbegrenzt im Dentin präpariert. Die Zähne wurden in 3 Gruppen eingeteilt (n=32): Kein IDS (**W**), IDS/totales Ätzen (**IT**) und IDS/selektives Ätzen (**IS**). Die Restaurationen wurden aus Lithiumdisilikat (IPS e.max CAD) mit einer Schichtstärke von 0.5-0.8 mm hergestellt. Nach unterschiedlichem Ätzprotokoll (total/selektiv) und der Verklebung mittels des dualhärtenden Klebers (Variolink Esthetic) wurden alle Gruppen in zwei Untergruppen von jeweils 16 Proben unterteilt. Eine Untergruppe wurde erst thermisch-dynamisch im Kausimulator (Kausimulator CS-4) belastet. Belastungskräfte von 98 N wurden bis zu 1.200.000 Zyklen aufgebracht. Proben wurden nach der künstlichen Alterung mittels SD-OCT gescannt. Es folgte eine quasi-statische Belastung aller Proben in einer Universalprüfmaschine bis zum Bruchversagen (Zwick Z010).

In der Gruppe IST versagte eine Probe (1%) während der dynamischen Belastung. Alle anderen Proben überstanden die 1.200.000 Zyklen. Von diesen Proben zeigten aber 23% die Entwicklung von Rissen. Die Fraktur in einer Probe und die Entwicklung von Rissen in 23% der Proben konnte nicht, unter Verwendung des SD-OCT, auf einen Defekt zurückgeführt werden.

Die Mittelwerte der Bruchfestigkeit der Gruppen nach der quasi-statischen Belastung reichten von einem Minimum von  $1.122 \pm 336$  N bis zu einem Maximum von  $1.853 \pm 333$  N,

was höher als die im Seitenzahnbereich auftretenden Kaukräfte war. Eine signifikant höhere Bruchfestigkeit ( $p \leq 0.001$ ) wurde durch IDS erreicht. Die künstliche Alterung und die Entwicklung von Rissen hatten keinen Einfluss auf die Bruchfestigkeit ( $p > 0.05$ ). Selektives Ätzen des Schmelzes war genauso effizient wie totales Ätzen.

Die häufigste Versagensart nach der quasi-statischen Belastung war der Modus IV, der aus einer Längsfraktur in der Keramik und Zahn bestand mit einer Prävalenz von 39%. Modus I; ein Riss in der Keramik, trat mit Prävalenz von 8% am seltensten auf. Die Dentin-Vorbehandlung und das Ätzprotokoll hatten keinen Einfluss auf die Versagensart ( $p > 0.05$ ), aber die Entwicklung von Rissen führte beim Bruchversagen zu einer katastrophaleren Versagensart dieser Proben ( $p \leq 0.001$ ).

Die positiven Ergebnisse der vorliegenden Studie unterstützen die Initiierung kontrollierter klinischer Studien, um die klinische Bewährung dünner okklusaler Keramik-Restaurationen zu evaluieren.

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## 11. Appendix

<b>Table 9: Failure resistance (N) and failure mode recorded for specimens in group WT</b>				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	1	Survived	1,570	III
	2	Survived	883	II
	3	Survived	1,380	III
	4	Survived	1,164	III
	6	Survived	1,770	IV
	9	Survived	1,830*	IV
	10	Survived	748*	IV
	15	Survived	851	II
Mean			1,275	
SD			427	
SD (percentage)			33	
<b>2 (Quasi-static load only)</b>	5	N/A	1,550	IV
	7		831	II
	8		1,280	III
	11		804	I
	12		1,350	IV
	13		1,490	IV
	14		980	III
	16		690	III
Mean			1,122	
SD			336	
SD (percentage)			30	

\*: Crack progression after the artificial aging process

<b>Table 10: Failure resistance (N) and failure mode recorded for specimens in group WS</b>				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	1	Survived	1,400	II
	3	Survived	1,050	IV
	6	Survived	1,370	III
	7	Survived	1,230*	IV
	10	Survived	1,120	II
	11	Survived	1,510	III
	12	Survived	1,310	III
	15	Survived	1,350	II
Mean			1,293	
SD			152	
SD (percentage)			12	
<b>2 (Quasi-static load only)</b>	2	N/A	1,440	III
	4		1,530	III
	5		1,310	IV
	8		1,620	IV
	9		1,430	III
	13		1,580	IV
	14		1,150	I
	16		1,110	IV
Mean			1,396	
SD			191	
SD (percentage)			14	

<b>Table 11: Failure resistance (N) and failure mode recorded for specimens in ITT</b>				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	1	Survived	2,230	IV
	2	Survived	1,950	III
	3	Survived	1,660*	IV
	4	Survived	1,670	II
	6	Survived	1,710	II
	7	Survived	1,420	IV
	8	Survived	2,130*	IV
	11	Survived	1,660	II
Mean			1,804	
SD			274	
SD (percentage)			15	
<b>2 (Quasi-static load only)</b>	5	N/A	2,260	IV
	9		1,920	III
	10		1,850	IV
	12		1,350	I
	13		1,460	III
	14		2,000	III
	15		1,560	IV
	16		1,600	III
Mean			1,750	
SD			308	
SD (percentage)			18	



<b>Table 12: Failure resistance (N) and failure mode recorded for specimens in ITS</b>				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	1	Survived	1,510	II
	2	Survived	2,200*	IV
	3	Survived	2,040*	IV
	4	Survived	1,630	III
	5	Survived	1,640	II
	6	Survived	1,910	IV
	7	Survived	1,850	II
	11	Survived	1,470	III
Mean			1,781	
SD			261	
SD (percentage)			15	
<b>2 (Quasi-static load only)</b>	8	N/A	1,910	I
	9		1,630	I
	10		1,590	IV
	12		1,540	II
	13		1,640	II
	14		1,470	IV
	15		1,360	II
	16		2,130	IV
Mean			1,659	
SD			248	
SD (percentage)			15	

<b>Table 13: Failure resistance (N) and failure mode recorded for specimens in IST</b>				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	1	Survived	2,190	III
	2	Survived	2,250	II
	4	Survived	2,370*	IV
	5	Survived	1,500*	IV
	6	Survived	1,630	I
	7	Survived	2,130	II
	8	Fractured	98	-
	10	Survived	2,340	II
Mean			1,814	
SD			765	
SD (percentage)			42	
<b>2 (Quasi-static load only)</b>	3	N/A	2,000	III
	9		1,530	II
	11		1,590	IV
	12		2,240	II
	13		1,490	III
	14		1,870	IV
	15		1,760	IV
	16		1,490	II
Mean			1,746	
SD			274	
SD (percentage)			16	

<b>Table 14:</b> Failure resistance (N) and failure mode recorded for specimens in ISS				
<b>Subgroup</b>	<b>Specimen no.</b>	<b>Fatigue Test (Survival)</b>	<b>Fracture Strength (N)</b>	<b>Failure Mode</b>
<b>1 (Fatigue load)</b>	2	Survived	1,140	III
	3	Survived	1,760	I
	6	Survived	1,890	III
	7	Survived	1,850	III
	8	Survived	2,180	III
	10	Survived	1,760	III
	13	Survived	2,120*	IV
	15	Survived	2,120*	IV
Mean			1,853	
SD			333	
SD (percentage)			18	
<b>2 (Quasi-static load only)</b>	1	N/A	2,100	II
	4		2,080	IV
	5		1,640	II
	9		1,550	IV
	11		1,160	IV
	12		2,130	IV
	14		1,970	II
	16		2,120	IV
Mean			1,844	
SD			357	
SD (percentage)			19	