

T.C.
YEDİTEPE UNIVERSITY
INSTITUTE OF HEALTH SCIENCES
DEPARTMENT OF PROSTHETIC DENTISTRY

**EVALUATION OF WEAR CHARACTERISTICS OF
DIFFERENT CAD/CAM RESTORATIVE
MATERIALS BY INCREASED CHEWING FORCES
VIA CHEWING SIMULATOR**

DOCTOR OF PHILOSOPHY THESIS

İZİM TÜRKER, DDS, PhD

İstanbul- 2021

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İZİM TÜRKER, DDS, PhD

SUPERVISOR
Prof. Pınar KURSOĞLU

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THESIS APPROVAL FORM

Institute : Yeditepe University Institute of Health Sciences

Programme : Doctoral Program of Prosthodontics

Title of the Thesis : EVALUATION OF WEAR CHARACTERISTICS OF DIFFERENT CAD/CAM RESTORATIVE MATERIALS BY INCREASED CHEWING FORCES VIA CHEWING SIMULATOR

Owner of the Thesis : İzim TÜRKER, DDS, PhD

Examination Date : 08.06.2021

This study has approved as a Doctorate Thesis in regard to content and quality by the Jury.

	Title, Name-Surname (Institution)
Chair of the Jury	Prof. Ender KAZAZOĞLU Yeditepe University, Faculty of Dentistry
Supervisor	Prof. Pınar KURSOĞLU Yeditepe University, Faculty of Dentistry
Member	Prof. Nuray ÇAPA YILDIRIM Yeditepe University, Faculty of Dentistry
Member	Prof. Volkan GÜNAY Yeditepe University, Faculty of Engineering
Member	Assoc. Prof. Hüseyin Emir YÜZBAŞIOĞLU Bahçeşehir University, Faculty of Dentistry

This thesis has been deemed by the jury in accordance with the relevant articles of Yeditepe University Graduate Education and Examinations Regulation and has been approved by Administrative Board of Institute with decision dated 14.06.2021 and numbered 2021/06-01

Prof. Bayram YILMAZ

Director of Institute of Health Sciences

DECLARATION

I hereby declare that this thesis is my own work and that, to the best of my knowledge and belief, it contains no material previously published or written by another person nor material which has been accepted for the award of any other degree except where due acknowledgment has been made in the text.

08.06.2021

İzim TÜRKER



DEDICATION

This thesis is dedicated to my lovely family.

ACKNOWLEDGEMENTS

First, my deepest thanks go to my lovely family. They were always there right beside me. Their presence gave me strength in all fields of my life.

I would like to express my deepest appreciation to my dear supervisor **Prof. Pınar KURSOĞLU**. She has been sincerely giving and always there whenever I needed her. For the most part it is a great honour to be her doctorate student.

I would like to say a special thank to **Prof. Ender KAZAZOĞLU**, the Head of Department of Prosthodontics. He was always there with his sincere approach. I am so grateful to have a mentor like him.

My deepest thanks go to **Prof. Nuray ÇAPA YILDIRIM**. She means a lot to me. She always shared me her valuable knowledge and positive energy whenever I needed her which means a lot to me.

I am grateful to the jury member of my thesis, **Prof. Volkan GÜNAY** for all his efforts and supports in this period. He gave me helpful advices and always be interested in my work.

I kindly would like to thank all my mentors in our department for sharing their knowledge and experiences.

My deepest thanks go to my friends and colleagues, especially the one who I walked this way together for 10 years, **DDS. PhD. Elifnaz ÖZEN**. Also, the one who means lots to me **Mert Can BİÇİCİ**. Thank you for being always there and right beside me.

I am also grateful to my dear **Assist. Prof. Elif Çiğdem ALTUNOK** for her friendly guidance and sharing all her knowledge with me about the statistical analyses of my thesis.

I would like to express my deepest appreciation to **Prof. Tosun TOSUN** and **Prof. Hüseyin KURTULMUŞ** as they provided me an opportunity to study with the chewing simulator machine in Istanbul Aydin University Faculty of Dentistry and for their valuable support.

Finally, I would like to give special thanks to **İsmehan DERE**, hard tissue laboratory technician and **Burak ÜNAL**, prosthodontics laboratory technician, for their support and guidance during the experimental stages of my thesis.

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LIST OF SYMBOLS and ABBREVIATIONS

μm	Micrometer
%	Percent
$^{\circ}\text{C}$	Celsius
2D	Two-dimensional
3D	Three-dimensional
ADA	American Dental Association
Al_2O_3	Aluminium oxide
ATZ	Alumina-toughened zirconia
Bis-EMA	Bisphenol A diglycidyl methacrylate ethoxylated
Bis-GMA	Bisphenol A glycidyl methacrylate
CAD/CAM	Computer-aided design/Computer-aided manufacturing
CaO	Calcium oxide
$\text{Ca}_5(\text{PO}_4)_3\text{F}$	Fluorapatite
DD	Distilled and deionized
FSZ	Fully stabilized zirconia
GPa	Gigapascal
GPT	Glossary of Prosthodontic Terms
HV	Vickers hardness
ISO	International Organization for Standardization
K_1C	Fracture toughness
K_2O	Potassium oxide
$\text{K}_2\text{O}-\text{Al}_2\text{O}_3-4\text{SiO}_2$	Leucite
$\text{K}_2\text{Al}_2\text{Si}_6\text{O}_{16}$	Potassium feldspar

LaB ₆	Lanthanum hexaboride
La ₂ O ₃	Lanthanum oxide
Li ₂ SiO ₃	Lithium metasilicate
MgAl ₂ O ₄	Magnesium aluminate
MBF	Maximum bite force
MVC	Maximum voluntary clenching
min.	Minute
ML	Mechanical loading
mm	Millimetre
MPa	Megapascal
MPa.m ^{1/2}	Fracture toughness
MVC	Maximum voluntary clenching
Na ₂ O	Sodium oxide
nm.	Nanometre
PICN	Polymer-infiltrated ceramic network
PSZ	Partially stabilized zirconia
Ra	2D surface roughness parameter, arithmetic mean of the surface
Sa	3D surface roughness parameter, arithmetic mean of the surface
SB	Sleep bruxism
SEM	Scanning electron microscopy
Si ⁴⁺	Silicon atom
SiO ₂	Silica
SiO ₄ ⁴⁻	Silicate ion (Si-tetrahedron)
SiO ₂ -Li ₂ O	Lithium disilicate

Std.	Standard
TEGDMA	Triethylene glycol dimethacrylate
TC	Thermocycling
TCML	Thermocycling and mechanical loading
TWI	Tooth wear indices
TZP	Tetragonal zirconia polycrystals
UDMA	Urethane dimethacrylate
$\bar{x}$	Mean
vol.	Volume
Y-TZP	Yttria-stabilized tetragonal zirconia polycrystals
ZLS	Zirconia-reinforced lithium silicate
ZrO ₂	Zirconium dioxide
ZTA	Zirconia-toughened alumina

ABSTRACT

Turker, I. (2021). Evaluation of Wear Characteristics of Different CAD/CAM Restorative Materials by Increased Chewing Forces via Chewing Simulator. Yeditepe University, Institute of Health Science, Department of Prosthodontics, DDS/ PhD Thesis, Istanbul.

This study was conducted to compare wear characteristics of different CAD/CAM restorative materials and their enamel antagonists under increased chewing forces. The aim was to evaluate the patient-related and material-related factors to decide material selection with high clinical success. Extracted and divided cusps of healthy human third molars-against CAD/CAM materials as Vita Enamic®, a polymer-infiltrated ceramic network (PICN); GC Cerasmart®, a resin nanoceramic; Celtra® Duo, a zirconia-reinforced lithium silicate ceramic; and IPS e.max ZirCAD, a polycrystalline zirconia was exposed to chewing simulation (1.200.000 cycles, load 120 N, 5/55 °C thermocycling). To determine the wear volume loss, each tooth cusp and material sample were scanned with 3D laser scanner and the images were overlapped using the Geomagic software. After measuring the initial surface roughness (Ra) of materials, Vickers hardness (GPa) and fracture toughness (K₁C) were measured (1 kgf ($\approx$ 9.807 N), 15 sec., 5 indentations). Wear characteristics were analysed by scanning electron microscope. IPS e.max ZirCAD group showed significantly lower volume loss ($0.02 \pm 0.01 \text{ mm}^3$) than those of the three other groups (Vita Enamic, GC Cerasmart, Celtra Duo) ($p=0.001$, $p=0.018$ and $p=0.005$, respectively). The wear of cusp/Celtra Duo [0.59 mm^3 ($0.50\text{-}1.63 \text{ mm}^3$)] was significantly higher than cusp/GC Cerasmart [0.17 mm^3 ($0.04\text{-}0.41 \text{ mm}^3$)] ($p=0.007$). The surface hardness and fracture toughness of Vita Enamic and IPS e.max ZirCAD were decreased due to the thermocycling and mechanical loading ($p=0.000$ and $p=0.008$). Zirconia showed totally different wear mechanism than other materials due to its polycrystalline structure preventing microcrack formation. Zirconia may refer as an enamel-friendly material in patients with high chewing forces as it did not result in significant wear of enamel. It is observed that Celtra Duo exhibit an abrasive character against enamel antagonist by ejected zirconia crystals due to wear. GC Cerasmart might prevent the possible wear of enamel antagonist by its force absorbing manner.

Key Words: Wear, Chewing forces, Enamel, CAD/CAM materials, Chewing simulation

ÖZET

Türker, I. (2021). Farklı CAD/CAM Restoratif Materyallerinin Artmış Çiğneme Kuvvetlerini Taklit Eden Çiğneme Simülatörü Aracılığı ile Gösterdiği Aşınma Özelliklerinin Değerlendirilmesi. Yeditepe Üniversitesi Sağlık Bilimleri Enstitüsü, Protetik Diş Tedavisi AD., Doktora Tezi, İstanbul.

Çalışma farklı CAD/CAM restoratif materyallerin ve mine antagonistlerinin yüksek çiğneme kuvvetleri altında aşınma karakterlerinin değerlendirilmesi için yürütülmüştür. Klinikte doğru materyal seçimi ile yüksek başarıya ulaşmak için hasta ve materyalle ilişkili faktörlerin aşınma üzerine etkisinin değerlendirilmesi amaçlanmıştır. Vita Enamic®, GC Cerasmart®, Celtra® Duo, IPS e.max ZirCAD materyalleri ile antagonist mine örnekleri çiğneme simülatörüne yüklenmiştir (siklus 1.200.000, 120 N, 1 Hz, termosiklus 5/55 °C). Doğal diş ve materyal örneklerinin aşınmaya bağlı hacim kayıplarını değerlendirmek için 3D lazer tarayıcı kullanılmıştır. Materyallerin ilk pürüzlülüklerine (Ra) yüzey profilometresi ile bakılmış; Vickers sertlik (GPa) ve kırılma tokluğu (K_{1C}) değerleri (1 kgf ($\approx$ 9.807 N), 15 sn, 5 indentasyon) ölçülmüştür. Tarayıcı elektron mikroskobu ile aşınma mekanizması sonuçları değerlendirilmiştir. IPS e.max ZirCAD, aralarında istatistiksel anlamlı fark bulunmayan diğer üç gruba (Vita Enamic, GC Cerasmart, Celtra Duo) kıyasla en düşük hacimsel kaybı ($0.02 \pm 0.01 \text{ mm}^3$) göstermiştir (sırasıyla $p=0.001$, $p=0.018$ and $p=0.005$). Cusp/Celtra Duo' nun aşınması [0.59 mm^3 ($0.50\text{-}1.63 \text{ mm}^3$)] cusp/GC Cerasmart'a [0.17 mm^3 ($0.04\text{-}0.41 \text{ mm}^3$)] göre anlamlı şekilde daha fazla olmuştur ($p=0.007$). Vita Enamic ve IPS e.max ZirCAD materyallerinin yüzey sertliği ve kırılma tokluğu, termosiklus ve yüksek kuvvet maruziyeti sonucu zaman içinde azalma göstermiştir ($p=0.000$ and $p=0.008$). Zirkonya, mikro çatlak oluşumunu önleyen sağlam polikristalin yapısı sebebiyle diğer materyallerden farklı bir aşınma mekanizması göstermiştir. Karşit dişte anlamlı bir aşınma yaratmaması nedeniyle çiğneme kuvveti yüksek hastalarda karşit mine dostu özellik gösterdiği söylenebilir. Celtra Duo'nun aşınmaya bağlı olarak açığa çıkan zirkonya kristalleri ile karşit minede abraziv bir etki yarattığı izlenmiştir. Kuvveti emme özelliğiyle GC Cerasmart karşit dişi fazla aşındırma ihtimalinin önüne geçebilir.

Anahtar Kelimeler: Aşınma, Çiğneme kuvvetleri, Mine, CAD/CAM materyalleri, Çiğneme simülasyonu

1. INTRODUCTION and PURPOSE

Wear is the loss of material from a surface caused by mechanical contact, movement of a solid or liquid body, chemical action or both chemical and mechanical action simultaneously.¹ According to weartribology, wear is a complex phenomenon and an overall effect of serial related processes.²

Physiological tooth wear that occurs over a lifetime indicates improved chewing efficiency and reduced susceptibility of the dentition to disease and malocclusion.^{3,4} Conversely, excessive tooth wear may result in poor chewing ability, damaged tooth surface, destroyed structural stability and reduced life of prosthetic materials, which are replaced to compensate for partial or total loss of dental structures.⁵⁻⁸

Currently, by the increase in life expectancy, enamel wear is becoming an important issue as teeth are required to last longer.⁹ Abnormal loss of tooth structure can also lead to problems in occlusion, thus impacting chewing efficiency, tooth sensitivity, and aesthetic levels.¹⁰ For this reason, understanding the mechanisms and control factors of wear is critical.¹¹

Patient-related factors as well as microstructural, physical and mechanical material-related factors of the restorative material can influence enamel wear. Patient-related factors include parafunctional habits such as bruxism, masseter muscle thickness, dentition status, age, and gender.¹²⁻¹⁶

In recent years, the prevalence of bruxism has increased in the general adult population and is usually considered as one of the causative factors for tooth wear, possibly due to tooth overload.^{17,18} In patients, especially those with sleep bruxism, daytime chewing forces play a significant role, resulting in non-physiological force and velocity contraction of masseter muscles, which in turn causes morphofunctional changes and muscle strengthening.^{19,20} Patients with morphofunctional changes require a greater amount of muscle fibers during chewing. In addition, a positive correlation has been found between high chewing forces and masseter muscle thickness.²¹

The applied force may also have differed due to the perception of the patient according to the dentition status. Completely edentulous patients with fixed implant-supported prostheses, do not have periodontal mechanoreceptors and lose their perception

toward chewing forces, which may lead to increased loads on teeth and/or restorations.^{22,23}

Regarding age status, younger adults may have higher chewing forces than older adults because muscle forces decrease with age by reviewing normal aging process.²⁴ Meanwhile, the male population may present higher maximum bite force due to higher muscle strength.^{25,26}

Thanks to current technology, researchers are able to simulate human chewing cycles in the laboratory by using specific mechanical loads and frictional forces exerted by chewing simulators.²⁷ Chewing simulators can better reproduce the physiological conditions of chewing, such as applying force, chewing movement characteristics, lubrication, and oral environment to a certain extent. In vitro studies conducted in the laboratory cannot simulate conditions in the oral cavity as accurately as in vivo studies, but they can standardize the various parameters that may influence wear.²⁸

The oral environment, including chewing and variations in temperature and humidity, can affect restorative materials and compromise the long-term success of restorations.²⁹⁻³¹ These effects can be estimated experimentally by using various aging procedures,³¹⁻³³ including thermocycling and mechanical loading (TCML), to mimic intraoral conditions.³⁴⁻³⁷ Thermocycling is only one method to evaluate the aging of materials that may affect wear in a material-dependent manner.³⁸

Physical material-dependent factors that affect wear are related to hardness, fracture toughness, and frictional resistance. These are also related to the abrasiveness of ceramic materials. Ceramic materials should have sufficient fracture toughness, otherwise brittle breakouts may occur during the abrasion process. Brittle chipping can lead to re-sharpening of the fracture edge, with the rate of wear increasing depending on the type of ceramic material.¹²

In prosthetic dentistry, ceramic materials are widely used due to their intrinsic properties such as biocompatibility, chemical integrity, mechanical durability, and optical properties.³⁹ In recent years, digital technologies have rapidly advanced in the field of dentistry, improving the quality of materials through computer-aided manufacturing methods with high precision.^{40,41} New opportunities have opened up with the

development of ceramic base and computer-aided design/computer-aided manufacturing (CAD/CAM) materials.^{42,43}

Most studies have investigated the wear properties of various ceramic materials that antagonize the enamel. With the growing popularity and clinical use of CAD/CAM ceramic restorations and the increasing prevalence of patients with high chewing forces, it is critical to understand the wear potential of both materials and their enamel antagonists.

Therefore, the present study aimed to evaluate the wear behavior of new-generation CAD/CAM restorative materials and enamel antagonists under high chewing forces and to investigate the change in physical properties of the materials due to TCML in order to perform case-specific assessments on material selection and to achieve long-term clinical success. The null hypotheses of this study were as follows:

1) there would be no difference between the materials in terms of their wear volume loss and wear mechanisms due to increased chewing forces, 2) the materials would have no effect on the enamel antagonists due to increased chewing forces, 3) there would be no effect of TCML by increased chewing forces on different materials and no difference between the materials in terms of surface hardness and fracture toughness, and 4) there would be no effect of initial surface roughness of different materials on the wear of enamel antagonist.

2. LITERATURE REVIEW

2.1. Functions and structures of human teeth

Human teeth play an important role in facial aesthetics and the major functions of the mouth, including speaking, breathing, tasting, and chewing.¹⁰ The mouth is arranged in two opposing dental arches to allow for masticatory function. In each arch there are different types of teeth with different functions. The incisors have the function of shearing, the canines have the function for holding food, and the molars have the function for chewing.

The human tooth has a structure of hard enamel shell, tough dentin inside and pulp from outside to inside.^{44,45} The tooth is mainly composed of the enamel layer. Enamel is the most calcified and hardest tissue in the body and consists of 92-96% inorganic substances, 1-2% organic materials and 3-4% water by weight.⁴⁶ Dentin consists of 70% inorganic, 18% organic material and the remaining part is water. It has a higher organic content compared to the enamel layer.¹¹

2.2. Introduction to dental ceramics

In cases of partial or complete loss of teeth, it is necessary to rehabilitate the damaged/missing parts to restore the comfort and functionality of natural human teeth.¹⁰ Dental ceramics are materials developed with the aim of producing dental prostheses to replace damaged/missing tooth structures.⁷ Ceramics are defined as compounds of metallic and non-metallic elements consisting of metal oxides, borides, nitrides, carbides, silicates and complex mixtures of these materials. Si-tetrahedrons, SiO_4^{4-} are typical form of silicates (**Figure 2.1**).⁴⁷ Si-based inorganic materials such as feldspar, quartz and silica are the main components of dental ceramics. The term "porcelain" is often used for traditional feldspar-based ceramics because of the high proportion of silica. The ratio of feldspar, quartz and silica content in the ceramic determines the difference between a normal ceramic and a dental ceramic. A dental ceramic contains a dispersed crystalline phase surrounded by a continuous amorphous glassy phase, which can be referred to as a multiphase system. Ceramics exist in the structural form of crystalline solids or glasses.^{47,48} They are widely used biomaterials in restorative dentistry due to their intrinsic properties of biocompatibility, chemical integrity, mechanical resistance, and optical characters.³⁹ They have attractive clinical properties and aesthetically pleasing due to their colour, shade and luster.⁴⁹

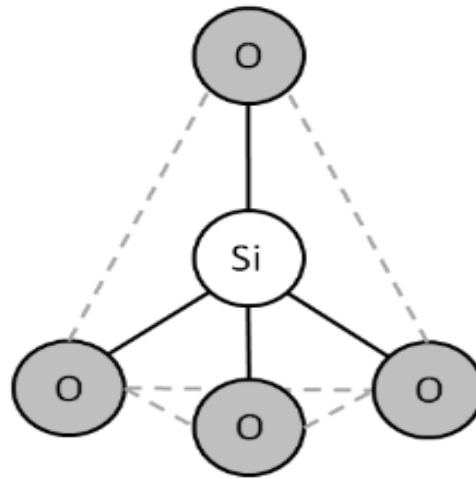


Figure 2.1. Si-tetrahedron (SiO_4^{4-}) configuration of ceramic with a single silicone atom (Si^{4+}) at the center, surrounded by four oxygen atoms (O^-) at the corner.¹

2.3.A brief history of dental ceramics

In 1774, the French researcher Alexis Duchateau had created the first successful dental prosthesis made of porcelain. Dental porcelain was first produced in 1838 with improvements in translucency and shades to match natural teeth.⁴⁹ These improvements were developed from 1838 to vacuum firing in 1949 by Elias Wildman.⁵⁰ In the early 1900s, porcelain jacket crowns containing either feldspar or alumina, also known as aluminous, were first created. However, due to the observation of poor match in thermal expansion coefficients between metal alloy framework bases and veneering ceramics, leucite-containing feldspar ceramics were developed in 1960s. Manufacturers matched the amounts of leucite ($\text{K}_2\text{O}-\text{Al}_2\text{O}_3-4\text{SiO}_2$) with feldspar glass to have more control over the coefficient of thermal expansion of feldspathic ceramics and enabled the fabrication of more reliable metal-ceramic frameworks.⁵¹ In recent decades, researchers have focused on all-ceramic restorations to develop restorations without metal substructures. Today, high-strength core ceramics such as zirconia are more commonly used and have taken the place of metal substructures in prosthetic dentistry.^{52,53}

2.4. Classification of dental ceramics

Dental ceramics can be classified according to different factors:^{48,49}

2.4.1. Classification of dental ceramics according to their clinical applications

There are three main indications for ceramics in dentistry through their clinical use:

¹ https://www.researchgate.net/figure/A-single-SiO4-4-tetrahedron-unit-showing-a-Si-4-cation-surrounded-by-four-O-2-anions-in_fig2_316452121

- metal-ceramic crowns and fixed partial dentures
- all ceramic restorations such as crowns, inlays, onlays, veneers, short-span anterior bridges
- ceramic denture teeth.^{54,55}

Generally, certain ceramics are used to replace the total loss of hard tissue.⁵⁶ However, ceramic laminate veneers are used to cover the facial surface of the tooth by bonding with adhesive systems.⁵⁷

2.4.2. Classification of dental ceramics according to their structure

2.4.2.1. Fusing temperatures

- High-fusing ceramics (1315-1370 °C)
- Medium-fusing ceramics (1090-1260 °C)
- Low-fusing ceramics (870-1065 °C)
- Ultra-low-fusing ceramics (65850 °C)

High-fusing ceramics have higher strength, insolubility, translucency and accuracy in repeated firings than medium- and low-fusing ceramics.^{48,49}

2.4.2.2. Type of restoration

It is useful to classify ceramics according to the type of restoration. There are two main categories such as all-ceramic restorations and metal-ceramic systems (**Figure 2.2**).^{53,58}

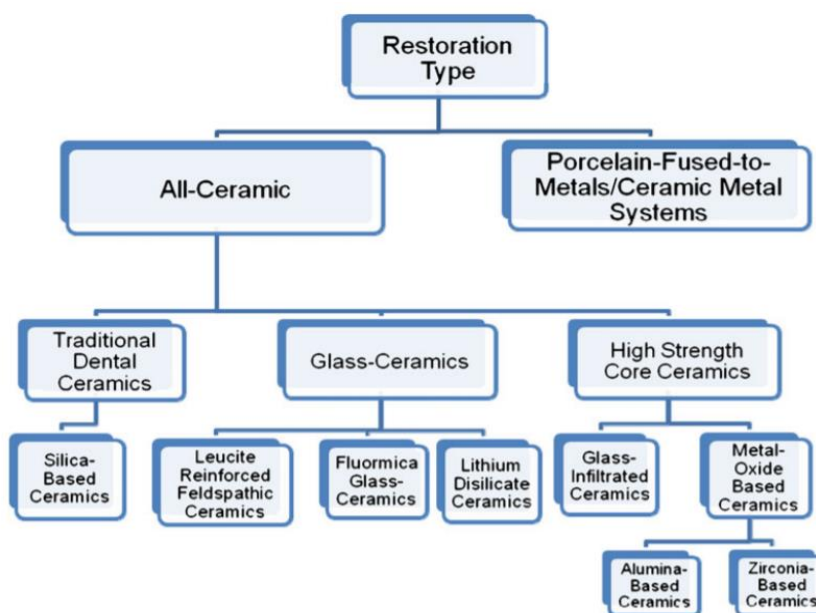


Figure 2.2. Ceramic classification according to the restoration type.⁴⁹

2.4.3. Classification of dental ceramics according to their microstructure:

The glass-to-crystalline ratio in the composition of the ceramic determines its microstructure. Modern dental ceramics containing a higher proportion of the crystalline phase are produced to improve biomechanical properties.⁴⁹ Traditional ceramic materials that have variable microstructures can be classified into four basic compositional categories with several subgroups:

2.4.3.1. Predominantly glass-based materials with high glass content

Glass-based systems mainly consist of silicon dioxide (SiO_2), also known as silica or quartz, and various amounts of alumina/aluminium oxide (Al_2O_3). Naturally occurring aluminosilicates containing varying amounts of potassium and sodium are known as feldspars. Feldspars are modified in various ways for the fabrication of dental ceramics. Glass-based materials were first used to make porcelain denture teeth. Nowadays, the material has indications such as veneering materials for metal or ceramic substructures due to its low flexural strength, which is usually in the range of 60-70 MPa.⁴⁸

2.4.3.2. Particle filled glass-based materials (usually leucite or a lithium disilicate)

The ceramic materials in this category are characterized by a wide range of glass-to-crystal ratios and crystal types. The materials have essentially the same pure glass composition, the difference being due to varying amounts of different crystal types added or grown in the glassy matrix. Currently, the primary crystal types are leucite, lithium disilicate, or fluorapatite.

2.4.3.3. Glass filled crystalline-based materials (mainly alumina)

The material in this category is glass-infiltrated, partially sintered alumina. The system is an alternative to conventional metal-ceramics.

2.4.3.4. Polycrystalline ceramics without glass content (alumina and zirconia)

Materials of this category are solid-sintered, and monophasic ceramics formed by direct sintering of crystals together without any intervening matrix. Alumina and zirconia are polycrystalline solids. Solid-sintered zirconia or alumina frameworks can be produced by various processing techniques.

2.4.4. Classification of dental ceramics according to their processing techniques

Processing techniques represent a greater correlation with the clinical success or failure of dental ceramics. The processing methodology enhances or degrades the final properties. There are a variety of techniques to process different ceramic materials. For dentistry, they can be broadly classified as:

- Powder/liquid technique
- Pressable blocks
- Machinable systems - Computer-aided design/computer-aided manufacturing (CAD/CAM)

2.4.4.1. CAD/CAM technology

Dentistry is an ever-growing field that has been at the forefront of medical disciplines in adopting new technologies such as CAD/CAM. The first application of CAD/CAM technology in dentistry was by Duret and Preston in the 1970s.⁵⁹ In the 1980s, Moermann followed them with his work to continue the development of this system. Since that time, CAD/CAM has been accepted in modern dental laboratories and by most dentists at the dental chairside.⁶⁰

There are three elements based on the development of CAD/CAM as follows: (1) data acquisition, (2) data processing, and (3) fabrication.

Data acquisition requires intraoral scanners. With intraoral scanners, it is possible to create a three-dimensional (3D) model of the oral cavity without taking an impression, casting a model, and then digitizing it. The digital model can be used to design dentures such as crowns, bridges, and frameworks for partial dentures. There are many software options available today for design.

In the past, the digitization, design, and fabrication steps were referred to as a closed system. Currently, it is possible to use open access systems. The system is opened, and it is possible to acquire the components of the CAD/CAM system. It creates an opportunity for data to be acquired from a range of sources and manufacturing techniques. The data can be matched with appropriate design software for fabrication. Crown and bridge frameworks, partial denture frameworks, even custom implants and implant abutments

are the possibilities for fabrication. The most suitable fabrication process and associated materials can be selected.⁴⁰

2.5. Classification of all-ceramic and ceramic-like restorative materials

There are several classification systems for ceramics used in dentistry, as mentioned above, to select the correct material by focusing on clinical applications, composition, microstructure, processing methods, etc.^{61–65} Continuing advances in the properties of ceramics such as strength, aesthetics, and manufacturing methods result in many products for the clinician to select. Classification of dental ceramics according to their microstructure, which is related to the glass content, does not reflect the fundamental development in ceramic technology. The manufacturing process of ceramic materials has evolved, and new restorative materials have emerged. The existing classifications do not include resin matrix CAD/CAM materials that are highly filled with ceramic networks. These materials are recommended as aesthetic alternatives for various clinical indications and have recently been coded as "ceramics" by the American Dental Association (ADA) with their ceramic-like properties, so they should not be ignored in ceramic classification systems.⁶⁶ Thus, a new approach to the classification of dental ceramics has been proposed. Ceramic restorative materials were categorized by Gracis S, et al.⁶⁷ (**Figure 2.3**) in three groups of all-ceramic and ceramic-like materials, based on their compositions as follows:

1. Glass-matrix ceramics

- Feldspathic ceramics
- Synthetic ceramics
 - A. Leucite-reinforced ceramics
 - B. Lithium disilicate and derivatives
 - C. Fluoroapatite-based ceramics
- Glass-infiltrated ceramics
 - A. Alumina
 - B. Alumina and magnesium
 - C. Alumina and zirconia

2. Polycrystalline ceramics

- Alumina
- Stabilized zirconia
- Zirconia-toughened alumina
- Alumina-toughened zirconia

3. Resin-matrix ceramics

- Resin nanoceramic
- Glass-ceramic in a resin interpenetrating matrix
- Zirconia-silica ceramic in a resin interpenetrating matrix

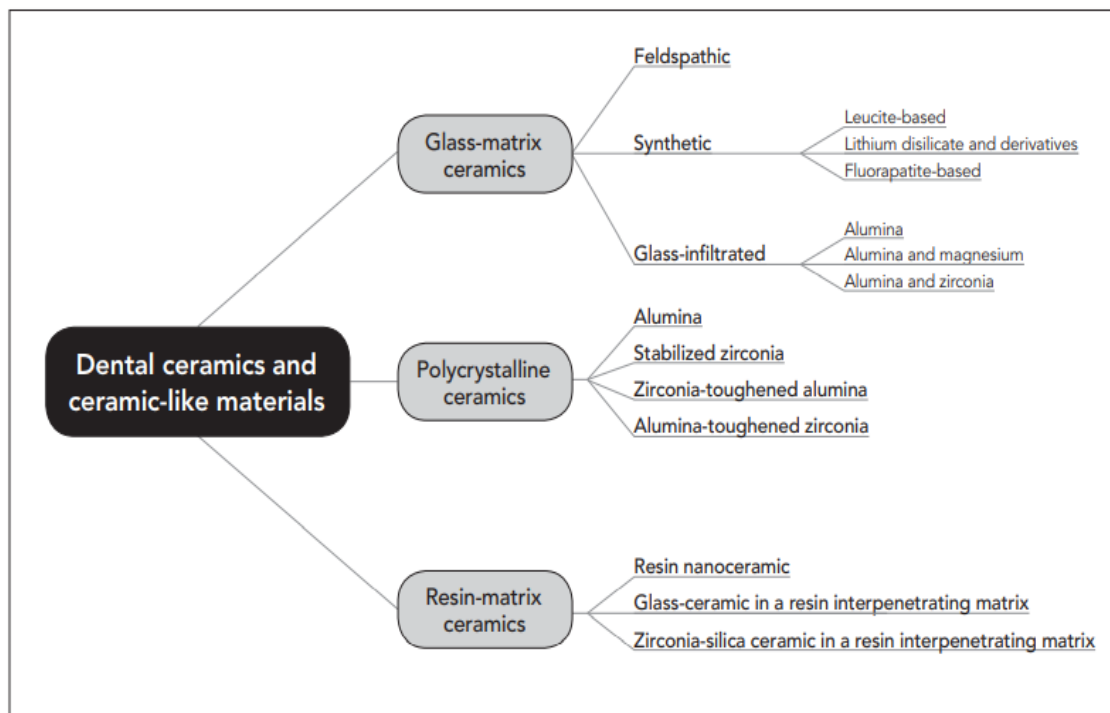


Figure 2.3. Overview of the proposed classification system of all-ceramic and ceramic-like materials by Gracis et al.⁶⁷

2.5.1. Glass-matrix ceramics

Glass-matrix ceramics are referred as traditional ceramics and consist of feldspar, quartz (silica), and kaolin. The non-metallic inorganic ceramic materials contain a glass phase.

2.5.1.1. Feldspathic ceramics

Feldspathic ceramics could be defined as the traditional type of dental ceramics and composed of naturally occurring feldspar (a mixture of potassium and sodium aluminosilicates), quartz (silica), and kaolin (hydrated aluminosilicate). Most ceramics have two distinct phases, a vitreous phase, and a crystalline phase. While the crystalline phase is related to mechanical strength, the glassy phase is often responsible for aesthetic behaviour. Leucite crystals in the crystalline phase formed by potassium feldspar ($K_2Al_2Si_6O_{16}$), increase the inherent strength of the material and make the porcelain veneerable, depending on the amount of potassium feldspar. The veneering ceramic has a coefficient of thermal expansion that is approximately 10% or less lower than that of the framework.^{62,64,65} The crystalline phase has a similar diffraction index to the glassy matrix in feldspathic ceramics. At the same time, it also contributes to the translucency of the overall structure. These materials are still used as an aesthetic option for veneering on metal and ceramic substrates. IPS Empress Esthetic, IPS Empress CAD, IPS Classic by Ivoclar Vivadent; Vitadur, Vita VMK 68, Vitablocs by Vident are the examples of feldspathic ceramic materials.

2.5.1.2. Synthetic ceramics

The ceramic industry has begun to use synthetic materials to avoid dependence on natural sources of raw materials. The composition usually includes silicon dioxide (SiO_2), potassium oxide (K_2O), sodium oxide (Na_2O), and aluminium oxide (Al_2O_3), although the amounts vary depending on the manufacturer. The glass phases can be combined with apatite crystals such as leucite, lithium disilicate, or fluorapatite to improve strength and achieve thermal expansion compatibility with metal frameworks. Crystalline phase reinforced feldspathic porcelains are available with enhanced mechanical properties for use of inlays, onlays, crowns, and three-unit fixed prostheses in the anterior region and use as a substructure material.

A. Leucite-reinforced ceramics

Conventional feldspathic porcelains were reinforced with approximately 45 vol% tetragonal leucite crystals which are responsible for high compressive strength and high modulus of rupture.^{39,68} It was reported that higher leucite content of porcelain

leads to higher fracture modulus and compressive strength compared to conventional feldspathic porcelain for metal ceramics.⁶⁹ Numerous studies have reported that the high leucite content in the material also contributes to a higher thermal contraction coefficient than that of the glassy matrix, which leads to the development of tangential compressive stresses in the glass around the leucite crystals during cooling. These stresses can act as crack arrestors and help to increase the resistance of the weaker glassy phase to crack propagation.^{62,65} Leucite reinforced ceramics are suitable for the fabrication of veneers due to their good optical properties.⁷⁰ The low refractive index of these crystals makes ceramic materials translucent even with high crystalline content⁶³, resulting in improved flexural strength (160-300 MPa), which also depends on the shape and volume of the crystals. The glass matrix is infiltrated by microscopic leucite crystals, resulting in a highly filled glass matrix.⁷¹ Leucite-containing feldspathic glass is referred to as "feldspathic porcelain", although other ceramic categories may also have a feldspathic-like glass. The leucite crystal content changes slightly to moderately. IPS Empress, IPS Empress CAD, and IPS Classic (Ivoclar Vivadent) are examples for leucite-reinforced ceramics.

B. Lithium disilicate and derivatives

- **Lithium disilicate**

Glass ceramics enriched with lithium disilicate crystals ($\text{SiO}_2\text{-Li}_2\text{O}$) were developed by Ivoclar Vivadent for the lost wax/hot press process (IPS Empress 2 and IPS e.max Press) and later for the CAD /CAM technology (IPS e.max CAD). The high crystal content (70% by volume) is significantly higher than that of leucite materials. The ceramic structure consists of strongly interlocked lithium disilicate crystals with a length of 5 mm and a diameter of 0.8 mm.^{48,72} IPS Empress 2 has an improved flexural strength (360 MPa), which is more than twice that of leucite-based IPS Empress and is indicated for anterior and premolar crowns as well as three-unit FDPs in the anterior region. In 2005, IPS e.max Press was launched with improved physical properties (flexural strength 400 MPa) compared to the previous IPS Empress.^{63,71,73} It also consists of a lithium disilicate glass-ceramic, but with a refined crystal size, which exhibits improved physical properties and translucency due to a different firing process.⁷⁴ Due to the relatively low refractive index of the lithium disilicate crystals,

this material exhibits high translucency despite the high crystalline content.⁷³ The IPS e.max Press pressable lithium disilicate ceramic can be used in monolithic application for inlays, onlays and posterior crowns or as a core material for crowns and three-unit FDPs in the anterior region.⁶³ Machinable lithium disilicate blocks (IPS e.max CAD) have recently been developed for the CAD/CAM machining technology. These blocks undergo a two-stage crystallization process. In the first stage, 40% by volume lithium metasilicate crystals- Li_2SiO_3 (with an average size of 0.2-1.0 μm) are formed, resulting in a flexural strength of 130 MPa to 150 MPa, which allows easier machining and intraoral occlusal adaptation. During the final crystallization process, 70% of the crystal volume (with an average size of 1.5 μm) is incorporated into a glass matrix, which increases the final strength (**Figure 2.4**). Additionally, at this stage, the blue colour of the pre-crystallized block is changed to the selected tooth colour. The final restoration has a flexural strength of 360 to 420 MPa; these are indicated for anterior or posterior crowns, implant crowns, inlays, onlays and veneers.^{63,72}

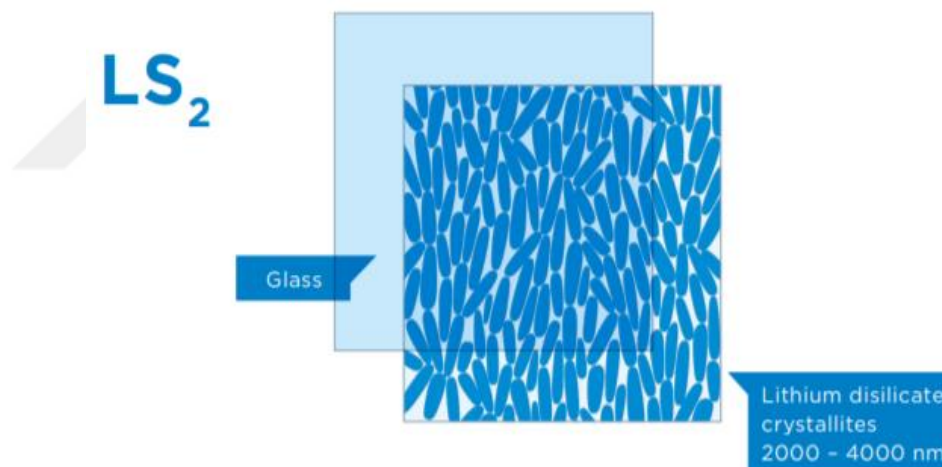


Figure 2.4. Structure of lithium disilicate ceramic²

- **Zirconia-reinforced lithium silicate**

The research and development of new restorative materials with the aim of achieving high mechanical and aesthetic performance has led to the introduction of zirconia-reinforced lithium silicate (ZLS) ceramics, which can be used with CAD/CAM technologies. In 2013, ZLS was developed by two companies, Vita Zahnfabrik and **Dentsply Sirona** and marketed separately as different products: Vita Suprinity and

Celtra Duo.⁷⁵ These materials have similar microstructures with a homogeneous glassy matrix containing a crystalline component of round and submicrometric elongated grains of lithium metasilicates and lithium orthophosphates, tetragonal zirconia fillers to increase the strength values up to 360 MPa on average. This sub-micron sized dispersed zirconia particles embedded in a fine-grained glass matrix of 500 to 700 nm (**Figure 2.5**), and 10% by weight, and enhance mechanical properties by increasing the flexural strength and fracture toughness compared with conventional lithium disilicate ceramics.⁷⁶ After a crystallization process, lithium disilicate grains are formed. Lithium metasilicate is reported to have grown larger in Celtra Duo than in Vita Suprinity (up to ~1 or ~0.5 μm in length).^{77,78} This structural typology was developed to combine favourable optical properties with improved mechanical properties compared to other glass-ceramics, although this assumption remains controversial to this day.^{79–82} Moreover, the fracture strength has been reported to withstand physiological occlusion forces and to increase after a firing protocol.⁸³ The monolithic Vita Suprinity blocks are offered in a meta-sintered stage to facilitate grinding, and the later piece undergoes a final crystallization firing. Celtra blocks are available in a pre-crystallized (Celtra CAD) or fully sintered (Celtra Duo) stage. Inlays, onlays, veneers and crowns are the indications of usage.^{77,84}

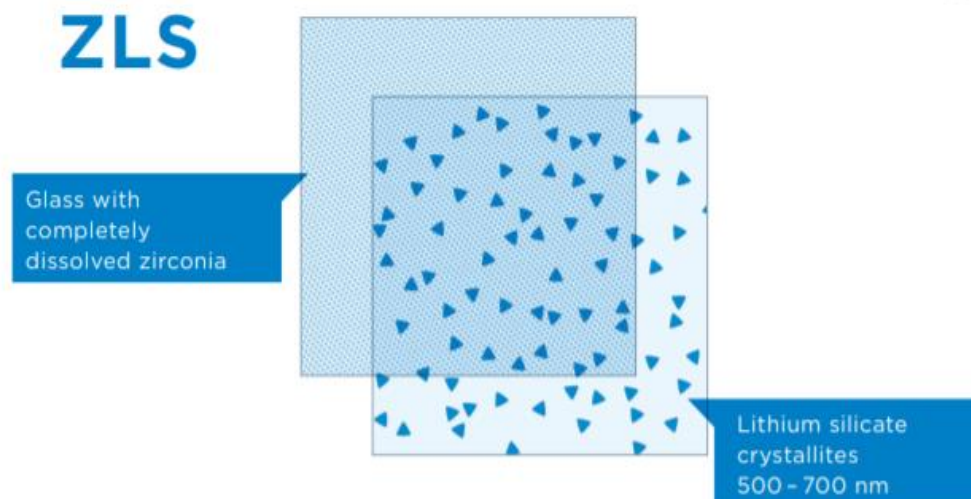


Figure 2.5. Structure of zirconia-reinforced lithium silicate ceramic²

² Dentsply Sirona; 2017- Celtra® Duo technical product information data sheet. n.d.

C. Fluoroapatite-based ceramics

Fluorapatite glass-ceramics contain fluorapatite crystals $\text{Ca}_5(\text{PO}_4)_3\text{F}$ embedded in the glassy matrix in various sizes; the crystals responsible for the opalescence of the material are smaller than 300 nm in length and 100 nm in diameter, while the crystals along the longitudinal axis are larger, 2-5 μm in length and less than 300 nm in diameter. IPS e.max Ceram and IPS e.max ZirPress (Ivoclar Vivadent) are the examples of fluoroapatite-based ceramics. IPS e.max Ceram is a nano-fluorapatite layering ceramic in the form of a powder used for the fabrication of veneers or as a veneering material for glass or oxide ceramics, while IPS e.max ZirPress are pressable ingots suitable for the fabrication of veneers and veneering of zirconia substructures using the press-on-zirconia technique. The flexural strength of IPS e.max Ceram is significantly lower than that of IPS e.max ZirPress, which is due to the pores present in the material as a result of the incorporation of air bubbles when mixing the ceramic powder with the mixing liquid.⁸⁵ Due to the high silica content of approximately 60 % of weight and the very low alumina content of ≈ 12 % of weight, the flexural strength is lower compared to other all-ceramic materials (only 90-110 MPa), which means that they cannot be used to fabricate the substructure of the restorations. Fluorapatite glass-ceramics appear to have similar properties to other glass-ceramics in terms of mechanical properties, milling efficiency and etchability, but in addition this material has biocompatible properties such as fluoride release. The flexural strength is about 120 MPa, Weibull modulus is higher, while Vickers hardness is lower compared to feldspathic, lithium disilicate and resin matrix ceramics.⁸⁶

2.5.1.3. Glass-infiltrated ceramics

In 1989, the first glass-infiltrated material In-Ceram Alumina was introduced and produced by slip casting. Due to the opacity of the material, a porcelain veneer was required. According to the manufacturer, the composition is Al_2O_3 (82%), La_2O_3 (12%), SiO_2 (4.5%), CaO (0.8%) and other oxides (0.7%).

In 1994, In-Ceram Spinell was introduced and manufactured in a similar manner, with the difference that the glass is infiltrated into a synthetically manufactured porous magnesium aluminate (MgAl_2O_4) core. As a modification of In-Ceram Alumina, In-Ceram Zirconia is introduced by adding partially stabilized zirconia (PSZ) to the slip

composition of the Al_2O_3 to strengthen the ceramic. The selection of this class of materials decreased due to the increasing popularity of lithium disilicate and zirconia, especially for CAD/CAM fabrication. In-Ceram Alumina, In-Ceram Spinell, and In-Ceram Zirconia were introduced by Vita Zahnfabrik.

2.5.2. Polycrystalline ceramics

The fine-grained crystalline structure, as the main characteristic of the ceramics classified in this group, provides strength and fracture toughness, but tends to exhibit limited translucency. In addition, there is no glass phase in polycrystalline ceramics. This makes it difficult to etch these ceramics with hydrofluoric acid, which requires higher temperatures or long etching times.⁸⁷

2.5.2.1. Alumina

This material, consisting of high-purity Al_2O_3 (to 99.5%), was first introduced in the mid-1990s by Nobel Biocare with a brand name of Procera AllCeram as a core material for fabrication by CAD/CAM. It has extremely high hardness values from 17 to 20 GPa and relatively high strength. It has highest elastic modulus among all of dental ceramics as 300 GPa⁶³, which has resulted in susceptibility to bulk fracture.^{88,89} The tendency toward core fracture and the improvement in mechanical properties of materials, such as the transformation toughness capability found in stabilized zirconia, have led to decreased use of alumina.

2.5.2.2. Stabilized zirconia

There are three allotropic forms of pure zirconia: first, the monoclinic form, which is stable up to 1170°C. Then it converts to the second tetragonal form, and when the temperature exceeds 2370°C, to the third cubic form (**Figure 2.6**).^{90,91} Shear strain and 4% large volume increase accompany the conversion of the tetragonal to the monoclinic phases. This increase in volume allows cracks in the material to be closed and large increases in fracture toughness to be observed. In order to use this property of transformation toughness in practice, the tetragonal or cubic phases must be stabilized at room temperature by alloying pure zirconia with oxides such as yttrium, magnesium, calcium and cerium. These elements must be either fully or partially stabilized.⁹² A

proposed microstructural classification of zirconia ceramics can be as fully stabilized zirconia (FSZ), partially stabilized zirconia (PSZ), and tetragonal zirconia polycrystals (TZP).⁹³ Dental zirconia all belong to the TZP type, which is most commonly stabilized with yttria as Y-TZP. This form achieves the highest strength and fracture toughness after machining and sintering procedures.

Zirconia ceramic is a material that has historically been used to fabricate framework material for prosthetic veneering. Today, it can also be used to fabricate monolithic restorations. It can be used as monochromatic uniform material and, if needed it can be stained by infiltration. There has been a significant increase in the use of polychromatic (or mixed) CAD /CAM fabrication materials as blocks and disks to mimic dentin to enamel colour variations. In addition, there are now material options that are fabricated with increasing translucency. **IPS e.max ZirCAD (Ivoclar Vivadent)**, Katana Zirconia ML (Kuraray), Lava Plus (3M Espe) are some examples of stabilized zirconia.

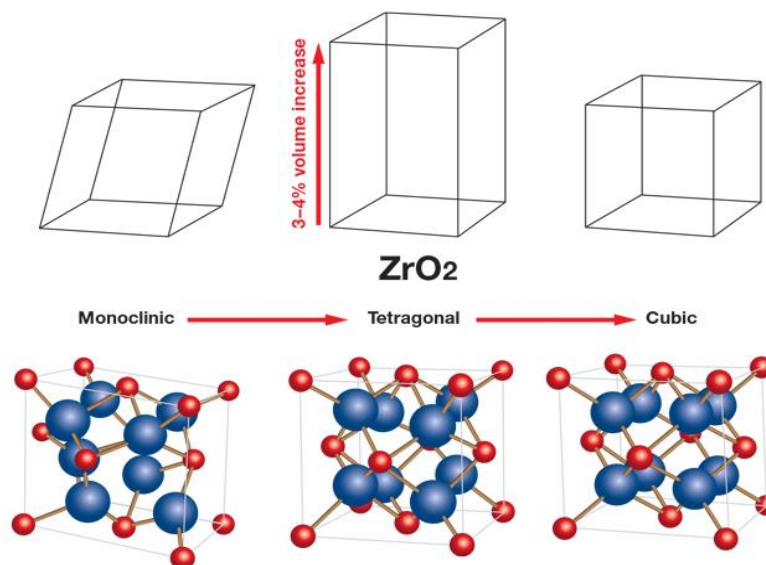


Figure 2.6. Three allotropic forms of zirconia with a uniform, polycrystalline structure³

³ <https://glidewell dental.com/education/chairside-dental-magazine/volume-11-issue-3/bruxzir-full-strength-vs-anterior>

2.5.2.3. Zirconia-toughened alumina and alumina-toughened zirconia

In general, zirconia remains partially stabilized in the tetragonal phase, and alumina presents moderate toughness, so alumina-zirconia (zirconia-toughened alumina [ZTA]) and zirconia-alumina (alumina-toughened zirconia [ATZ]) materials have been developed.^{94,95} In 1976, Claussen was the first researcher to describe that the addition of unstabilized zirconia to alumina increases the fracture toughness of alumina due to the interaction between the crack and pre-existing microcracks that occur during the conversion from tetragonal to monoclinic zirconia.^{96,97} The percentage of zirconia or alumina in the composition can be manipulated accordingly and changed as needed. There are some methods in which nanoparticles of zirconia are deposited on alumina microparticles prior to the sintering process.^{98,99} Compared to Y-TZP, these materials have advantages such as lower temperature degradation, higher strength and fracture toughness^{100,101}, and more than twice cyclic fatigue strength of Y-TZP.¹⁰² In these variants of polycrystalline materials, the glass is infiltrated into the surface of alumina or zirconia substrates. This infiltration creates a more damage-tolerant and aesthetic system for improved clinical performance.¹⁰³ Limiting the crack initiation greatly improves the fatigue life of both alumina and zirconia.¹⁰⁴

2.5.3. Resin-matrix ceramics

Resin-matrix ceramics have an organic matrix content that is highly filled with ceramic particles binding together through coupling agent (**Figure 2.7**).^{54,105} The presence of an organic matrix does not meet the traditional definition of ceramics as: non-metallic inorganic materials commonly processed by firing at high temperature to achieve desirable properties".^{51,106} However, according to the 2013 version of the ADA Code on Dental Procedures and Nomenclature⁶⁶ the term porcelain/ceramic is defined as "pressed, fired, polished, or milled materials containing predominantly inorganic refractory compounds, including porcelains, glasses, ceramics, and glass-ceramics." This definition adds the resin-matrix ceramic category to the ceramic classification. Manufacturers developed resin matrix ceramic materials to (1) provide a material that simulates a closer modulus of elasticity to dentin than conventional ceramics, (2) provide a material that is easier to mill and customise compared to glass matrix ceramics, such as lithium disilicate family synthetic ceramics or polycrystalline ceramics, and (3) allow repair or modification of the material with composite resin. They referred to this category as

"hybrid" ceramics because of their microstructure. It is likely that several new materials being introduced for aesthetic dental restorations will fit into the category of resin-matrix ceramics.

According to the manufacturers, the indications for these ceramic-like materials in restorative dentistry are very diverse. In addition to their compositions vary considerably, they are specially formulated for the CAD /CAM system. Nowadays, the resin-matrix ceramic materials can be divided into several subcategories according to their inorganic composition.

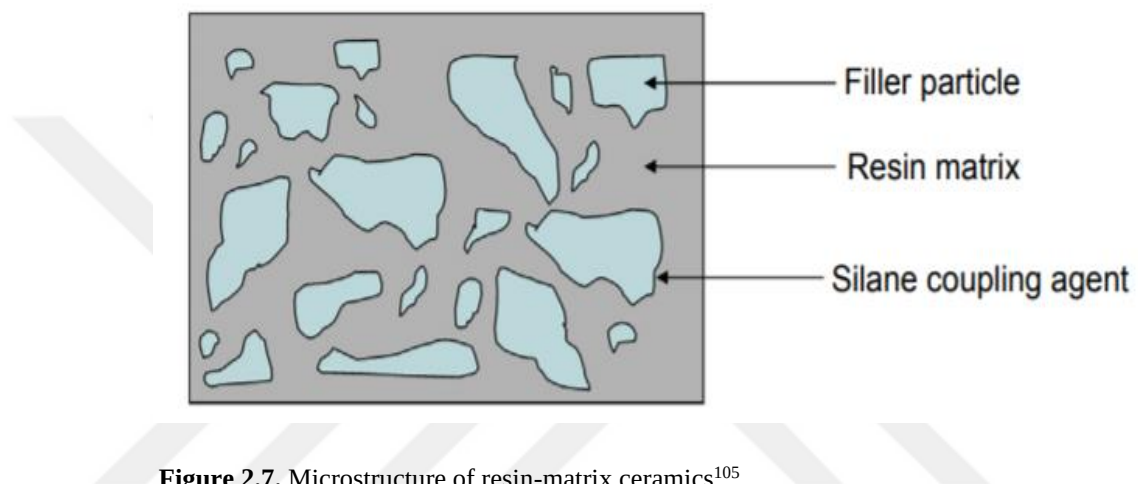


Figure 2.7. Microstructure of resin-matrix ceramics¹⁰⁵

2.5.3.1. Resin nanoceramic

It has a content of a highly cured resin matrix reinforced with nanoceramic particles. A resin nanoceramic material **GC Cerasmart (GC Corporation)** referred as "flexible nanoceramic", consists of relatively small and uniformly distributed particles of alumina-barium silicate embedded in a polymer matrix. It has approximately 71% by weight of silica and barium glass nanoparticles and 29% composite resin. Lava Ultimate (3M Espe), another material in this category, has approximately 80% by weight of nanoceramic particles and 20% composite resin. The material composed of discrete silica nanoparticles (20 nm diameter), zirconia nanoparticles (4 to 11 nm diameter), and zirconia-silica nanoclusters (bound aggregates of nanoparticles) to allow for this high nanoceramic content. The composite resin composition contains urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), bisphenol A glycidyl methacrylate (Bis-GMA), bisphenol A diglycidyl methacrylate ethoxylated (Bis-EMA). The flexural strength (about 242 MPa) is significantly higher, while the flexural modulus

(10.0 GPa) and Vickers hardness (about 64.1 HV) are much lower than Lava Ultimate (170.5 MPa/14.5 GPa/97.9 HV).¹⁰⁷ The modulus of elasticity of 3.07 ± 0.45 MPa is the highest compared to other ceramics or hybrid materials.¹⁰⁸ Due to the polymer content, the low flexural strength and the high elasticity, hybrid ceramics can only be used for single-tooth restorations such as veneers, inlays and onlays (Lava Ultimate), as well as for anterior and posterior crowns and implant-supported crowns (GC Cerasmart).⁸⁴

2.5.3.2. Glass ceramic in a resin interpenetrating matrix

To optimize the performance of these restorative materials, ceramics and resin composites have been combined into a single material by one manufacturer. This fact seems to be a good alternative to combine the dentin-like elastic modulus of resin composite with the long-term aesthetic stability of ceramic.¹⁰⁹ This material, named "polymer-infiltrated ceramic network (PICN)" has recently been introduced and referred to as a hybrid material. The material, **Vita Enamic (Vita Zahnfabrik)** is an example of this category. Typically, it is composed of a dual network, such as 86% by weight of feldspathic ceramic network and 14% by weight of polymer network. The composition and microstructure of dental materials determine their mechanical and physical properties. In particular for ceramics, the microstructure is dependent on sintering and significantly affects the coefficient of thermal expansion, resistivity values, chemical solubility, translucency, and optical appearance.¹¹⁰ Compositional analysis of PICN reveals a dominant ceramic network with leucite as the main phase and zirconia as a minor phase associated with a polymer-based network. A large amount of carbon exists in the polymer phase.¹⁰⁹ The composition of the polymer network is UDMA and TEGDMA. The flexural strength of this hybrid material can reach a value of about 150-160 MPa, much higher than that of a porous ceramic (below 30 MPa) and a polymer (135 MPa) alone. The superior flexural strength of the resin infiltrated ceramic material compared to the individual components indicates a strengthening mechanism of the polymer network to the dominant ceramic network.¹¹¹ The values of elastic modulus, hardness and fracture toughness are as follows: 30.14 GPa, 2.59 GPa, and $1.72 \text{ MPa}\cdot\text{m}^{1/2}$, respectively,¹¹² which are all between those for human dentin and enamel. With the highest filler content (73.1 mass-%) compared to other hybrid ceramics and composites, Vita Enamic has the highest Vickers hardness of 189.8.¹⁰⁷ The indications are minimally invasive restorations and

posterior crowns, veneers, inlays and onlays for posterior teeth and implant-supported crowns.¹¹³

2.5.3.3. Zirconia-silica ceramic in a resin interpenetrating matrix

These materials have an inorganic content comprises more than 60% by weight and are prepared by various organic matrices with the variation of ceramic weight percentage, such as silica powder, zirconium silicate, UDMA, TEGDMA, micro-fumed silica, pigments. Shofu Block HC (Shofu) and Paradigm MZ100 (3M ESPE) are the examples of this category.⁶⁷ A composite consisting of 85% ultrafine zirconia ceramic particles (spherical 0.6 μm) embedded in a polymer matrix of Bis-GMA, TEGDMA and a patented ternary initiator system.

2.6. Weartribology

Wear is the loss of material from a surface caused by mechanical contact, movement of a solid or liquid body, chemical action, or simultaneous chemical and mechanical action. This terminology may differ somewhat from the terminology used in industry, as some mechanisms that may be important in industrial processes do not occur in the mouth. Wear in the mouth is usually caused by different mechanisms acting simultaneously. Wear is a major consequence that can be caused by occlusal interactions. If left uncontrolled, it could lead to poor masticatory function with a reduction in quality of life and probable deterioration in systemic health.^{114,115} According to weartribology, wear is a complex phenomenon and an overall effect of serially interrelated processes described by tribologists in six terms.^{1,2}

2.6.1. Abrasive wear

This is the type of wear caused by hard particles or protuberances that are forced against and move along a solid surface. Two-body wear and three-body wear are the two categories of abrasive wear. Two-body wear is one type in which the cutting asperities are fixed on one or both surfaces, and three-body wear is another type in which the abrasive particles are loose in the slurry (**Figure 2.8**).

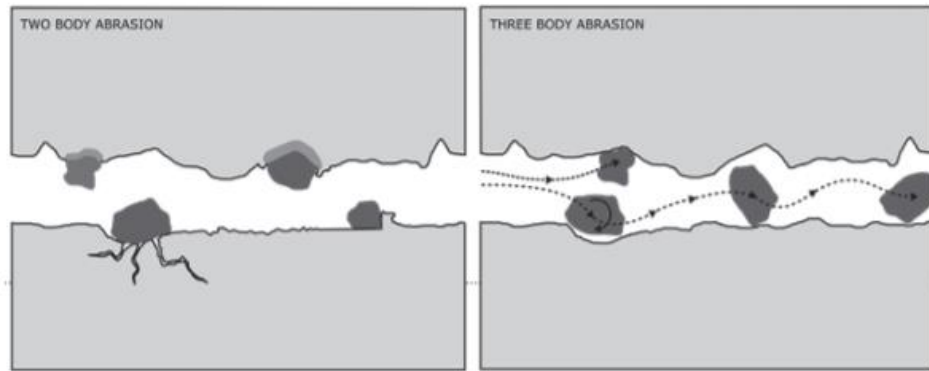


Figure 2.8. Schematic of two-body and three-body wear⁴

2.6.2. Attrition

It is a type of two-body abrasion where teeth or restorations are in occlusal contact. Attrition is usually the result of more than one abrasion mechanism.

2.6.3. Adhesive wear

This is a type of wear that is due to a local bond between contacting solid surfaces, causing a transfer of material between the two surfaces or a loss of one of the two surfaces.

2.6.4. Corrosive wear

This type of wear is caused by chemical or electrochemical reaction with the environment. Corrosive wear can result from interaction with chemicals that have a softening effect on the surface, causing the surface to be abraded from an opposing surface. Corrosive wear can result from erosion or abrasion.

2.6.5. Erosion

This is a tribological progressive loss of original material from a solid surface due to the mechanical interaction between that surface and a fluid, multicomponent fluid, or impinging liquid or solid particles.

2.6.6. Fatigue wear

This is the wear of a solid surface due to fracture caused by fatigue of the material. This situation is often observed due to rolling instead of sliding of surfaces.

⁴ https://www.burckhardtcompression.com/wp-content/uploads/2016/09/efrc16_session_41-2.pdf

2.7. Wear of enamel

Enamel wear is becoming a significant issue as teeth are required to last longer due to increasing life expectancy.⁹ Tooth wear is being identified with the loss of hard tissue caused by factors other than caries or dental trauma.¹¹⁶ It is a normal physiological process throughout life.⁴ Enamel is exposed to the loads on tooth and the chemical environment in the mouth. Wear on occlusal surface of tooth generally begins from the enamel.⁵ When this layer is lost, the underlying dentin is exposed. The dentin layer of the tooth is much softer and more malleable than the enamel. Therefore, it acts as a cushioning layer and reduces the effect of chewing loads on the enamel.¹¹ The wear depth of enamel under normal circumstances is 15-40 μm per year. This physiological wear maintains the health of the stomatognathic system.¹¹⁷ In the early stages, it can eliminate premature contacts, establish stable and wider occlusal contacts. In the middle stage, it can reduce the height of cusps, thus reducing lateral forces and their negative effects on periodontal tissues.¹¹⁸ Finally, in the final stages, it can coordinate the crown-root proportion and reduce the stress of periodontal tissues.¹¹⁹ Moderate tooth wear is useful for reducing the susceptibility of the dentition to malocclusion or disease, and improving masticatory efficiency.³ However, when wear accelerates and becomes excessive, it can lead to uncontrolled situations such as poor masticatory function, some dental diseases, dentine hypersensitivity, pulp exposure and even periapical diseases.^{8,120,121}

Tooth wear involves chemical, physical, and mechanical processes. First, during chewing, the maxillary and mandibular teeth impact to cut and torn foods. Then the teeth slide relatively on each other to grind food particles. The combination of impact and sliding wear results in occlusal surface wear of the tooth structure.^{122,123} The wear mechanism of human enamel is related to its microstructure. The water content within the microstructure has an undeniable influence on the wear process of enamel. Fresh enamel exhibits surface damage by plastic deformation in the early stages of impact wear.⁵ The accumulation and spread of plastic deformation results in iridescent rings appearing around the impact mark. The surface of the enamel begins to delaminate as the impact wear progresses, so that the iridescent rings gradually disappear in the early stages of wear.⁵ The loss of structure increased rapidly with the increase in impact cycles. It was observed that the impact cycles increased the surface hardness of the enamel and no cracks appeared in the cross section of the wear scar. The long hydroxyapatite rods near the enamel surface with a high degree of orientation make it difficult for protein shells to

slip between these rods as a dislodgment effect.¹²⁴ Enamel wear exhibits severe delamination, which is considered to be the dominant mechanism in two-body wear. In addition, the wear behaviour of the enamel does not exhibit subsurface cracks and shows metal-like properties to some extent. In three-body wear with increasing load, significant scratches accompanied by small delamination pits were the dominant wear mechanism.¹²⁵ The surface properties of the enamel against wear depend on various factors related to the behaviour of the antagonistic restorative material.¹²⁶

2.8.Wear of dental ceramics

Restorative materials are used to ensure the function of teeth by restoring their form.¹²⁷ An ideal restorative material should be as close as possible to the natural tooth structure in terms of appearance, biocompatibility, strength, and resistance to wear by opposing teeth or restorations.^{110,128} The wear rate of human enamel should be accurately measured to define the wear of restorative materials.¹¹⁷ While the enamel can lead to wear of material, the material can cause aggressive wear of the enamel.¹²⁹ The severe wear process can lead to premature failure of the ceramic restoration and/or irreversible damage to the enamel by exposing the dentin.¹³⁰ The wear of the antagonist depends on the ceramic material used.¹³¹ While the metal alloys and ceramic materials are generally very wear resistant, composites and especially unfilled polymers can suffer loss of material within a very short period of time depending on the material.^{132,133}

Regarding the wear mechanisms of dental ceramics, the stages of wear should be evaluated. There are three prominent wear stages during the overall wear process such as running-in wear stage, steady wear stage and severe wear stage. In the running-in phase, a higher wear rate is observed, and the ceramic surfaces show severe wear. In the steady wear phase, the ceramic restorations exhibit lower wear activity, and the wear rate remains at a lower level. However, in the steady-state phase, subsurface crack propagation may begin. The dramatic increase of structural loss due to crack penetration and porcelain separation is inevitable in the severe wear phase. The mechanism of wear behaviour shows the transition from surface abrasion to subsurface cracks of brittle fatigue by catastrophic propagation with the development of wear stages. Consequently, it is important to keep ceramic materials in a steady wear phase as long as possible.

In previous studies, attention has been paid to the final value of wear loss of materials after predefined wear cycles by simulating chewing cycles of a human to estimate and evaluate wear resistance.^{134–136} However, there might be a relationship

between some features of wear behaviour hidden beneath the surface of the material. This may lead to inconstant wear behaviour of ceramic material under different experimental settings. Therefore, it is possible to say that the wear behaviour is influenced by various parameters and material-related factors.¹³⁷

2.9. Material-related factors influencing wear

2.9.1. Physical factors

Physical factors affecting wear are related to surface hardness, fracture toughness, and frictional resistance. These are particularly related to the abrasiveness of ceramic materials. The factors evaluated in this thesis are as follows:

2.9.1.1. Surface hardness

Hardness is the resistance of the material to plastic deformation. It is measured using a pointed, pyramid-shaped diamond indenter with a square base of the Vickers hardness tester with a predetermined force to leave a permanent deformation or indentation on the surface of the material (**Figure 2.9**).^{138,139} The force applied by the indenter and the average diagonal size are automatically used to calculate Vickers hardness (HV).

$$HV = 1.8554 \times P_1 / (d_1)^2$$

(HV: Vickers hardness, P: load, c: diagonal of crack length)

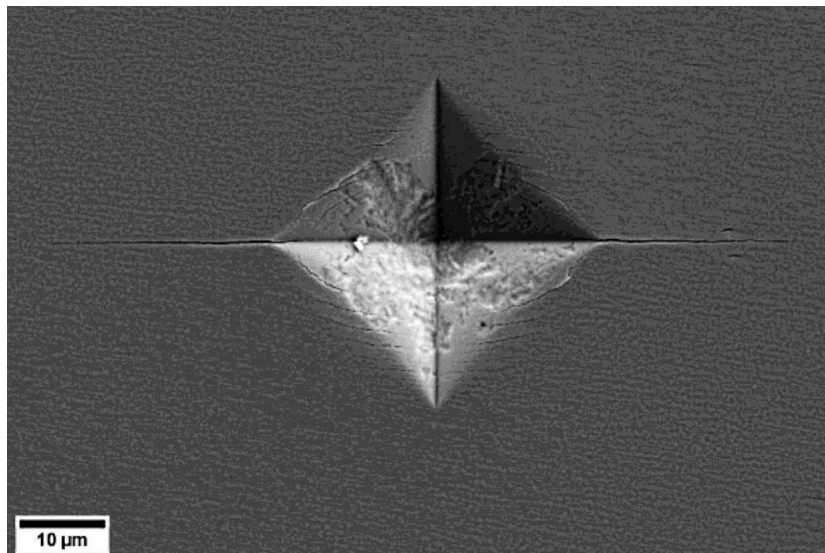


Figure 2.9. Indentation area remaining from Vickers hardness indenter⁵

⁵ <https://metalblog.ctif.com/2019/02/18/methodes-de-caracterisation-locale-des-proprietes-mecaniques>

Hardness is one of the significant properties used to characterize ceramics. Several attempts have been made to relate the hardness of dental materials to their greater abrasiveness and wear resistance.^{140–144} However, other material-related factors have also been shown to influence the wear properties of a ceramic, such as microstructure, porosity, crystal content, surface roughness, and environment.¹⁴⁵

It is not useful to define a strict correlation between hardness and wear of brittle materials, as they wear by subsurface fractures and not by plastic deformation, unlike metals and enamels. As for crystal content, crystalline materials can fail under indentation loads due to dislocation mechanisms of crystals in a glassy matrix. The inhomogeneous distribution of crystals in a glass matrix ceramic is a crucial factor in determining the failure mode of the material based on microhardness indentations. In non-crystalline materials, indentation damage occurs primarily by plastic deformation and subsequent fracture of the weaker glassy matrix. The orientation of the indenter to the crystals also determines the microhardness of crystalline materials. This variability may explain the poor relationship between material hardness and enamel wear.^{12,134}

2.9.1.2. Fracture toughness (K_{IC})

Fracture toughness (K_{IC}) is determined as the relative resistance of the material to rapid crack propagation under tensile stress.⁵¹ This intrinsic material property depends on the stress intensity at a crack tip in the material and the size of the crack relative to the microstructural characteristics of the material such as crystal size, aspect ratio, and orientation/distribution of the glass phase, and porosity.

Fracture toughness can be calculated as follows by the formula according to crack lengths:¹⁴⁶

$$K_{IC} = 0,0824 \cdot P/c^{3/2}$$

(K_{IC} : fracture toughness, P: load, c: half diagonal of the crack length)

K_{IC} of a ceramic material may be relevant to the wear process because failure of the ceramic occurs without detectable plastic deformation. This type of wear process begins with the formation of a crack, with subsequent growth of the crack over time, and finally the catastrophic failure/fracture of the material. Ceramic materials should have sufficient

toughness to resist fracture, otherwise brittle chipping can occur during the abrasion process. Brittle chips can cause re-sharpening of the fracture edges with an increase in wear rate. The sharp edges of broken glass do not lose as quickly because they have a higher hardness depending on the type of ceramic material.

2.9.2. Microstructural factors

The microstructural factors are related to porosity, crystal content, ceramic layer and shading. The crystal size is the microstructural factor that may play a role in this thesis are as follows:

2.9.2.1. Crystals

Crystals incorporated into the glassy matrix may have potential abrasiveness on opposing teeth depending on the type, content, morphology, and distribution of crystalline particles in the material.⁵⁰ The lower abrasiveness of a ceramic may be related to the crystal type, crystal distribution pattern, and crystal sizes. However, it is not correct to predict the wear properties of a ceramic material based only on the abrasiveness of the crystals. There are several material-related factors that affect the wear behaviour of the material, such as the grain size and shape, the presence of glassy and/or crystalline phases, the nature of the reinforcing crystalline phase, and the ceramic composition, especially the presence of some elements or oxides.^{147–149} To improve the wear behaviour and mechanical properties of ceramic, several factors, such as the proportion, type, morphology, and distribution of crystalline particles in a glassy matrix, must be optimized. In general, except for materials with polycrystalline structure, materials contain grains bound by a relatively weak glassy or polymeric matrix. Deformation of these bonding phases is associated with interfacial microcracks that facilitate crystal detachment and dislocation. In lithium disilicate materials, the propagation of microcracks begins along weak crystal-glass interfaces, a weakness responsible for the machinability of this material and the resulting propagation of ejected debris due to wear.¹⁵⁰ However, it has been reported that zirconia-reinforced lithium silicate (ZLS) glass-ceramic has a relatively homogeneous fine crystalline structure, unlike other lithium disilicate glass-ceramics with the characteristic needle-shaped and fine-grained crystals. The homogeneous fine structure has a positive effect on the flexural strength, which reduces the possibility of wear.⁷⁹

2.9.3. Effect of surface finish

The surface finish of the restorative material determines the wear rate of enamel.¹⁵¹ Ceramic materials have non-homogeneous distribution of crystals in a glassy matrix, which may introduce intrinsic multiple defects. During the processing of ceramics, additional defects may develop that reduce the strength of the material and increase enamel wear.¹⁵² Sealing the defects with surface treatment can improve strength and reduce the abrasiveness of the dental ceramic to the opposing tooth structure. Surface glazing of ceramics at a relatively low temperature produces a smooth, glassy, aesthetic, and hygienic surface and increases the strength of the ceramic system.^{153,154} Glazing is also considered a step that reduces wear of the restoration and opposing teeth. However, the glaze layer is only about 30-50 µm thick and can be easily removed immediately after function or by chairside occlusal adjustment.¹⁵⁵ According to many studies, ceramics with polished surfaces cause less enamel wear than glazed ones.^{143,151,156-158} Lower enamel wear is expected on polished material surfaces because of the lower coefficient of friction. However, the presence of an inadequately polished exposed surface may result in accelerated abrasive wear of the opposing dentition, increased plaque accumulation, and reduced strength of the ceramic materials.^{138,140} Several authors have studied the effects of surface polishing and finishing on the wear resistance of dental ceramics,¹⁵⁹⁻¹⁶¹ once surface roughness is one of the factors that increase the coefficient of friction and wear of the materials.

2.9.4. Surface roughness

As a measure of surface texture, roughness can affect the way a surface wears.¹⁶² When two materials touch and slide against each other, the resistance to lateral sliding (frictional resistance) varies depending on the relative motion, the properties and structure of the contacting materials, and the environment. The coefficient of friction is affected by the load and geometric parameters, such as the texture of the grinding surface, the shape of the contacting structure, and the area of the contacting surfaces. Rough surfaces, high loads and high sliding speeds have been shown to increase the coefficient of friction, resulting in increased wear.¹² It is also reported in a study that although the roughness of the ceramic is important in the initial stages of wear, once the effect of roughness disappears as wear progresses, the internal properties of the ceramic begin to affect the rate of wear.¹⁶³

Roughness is often quantified by the deviations of the surface from its ideal shape. If the deviations are small, the surface is considered smooth, but if they are large, the surface is considered rough. Over 100 variants of parameters for measuring surface roughness have evolved since the original international standards were published in 1984.¹⁶⁴ While the surface roughness parameters labelled with the letter "R" refer to two-dimensional (2D) evaluations, the 3D surface texture parameters all start with the letter "S". For example, as an extension of the 3D expression of the arithmetic mean of the surface, Sa; Ra is the 2D expression for this parameter. The selection of 3D surface characterization methods is more reasonable. This is because they could give more specific and solid descriptions about the evaluation of microgeometry and function-related properties for the material surfaces that have the same values in their main indices such as Sa or Ra.¹⁶⁵ However, the most commonly used reporting parameter of roughness is still the surface roughness mean Ra, arithmetic mean (**Figure 2.10**) or the root mean Rq, geometric mean in dentistry.¹⁶⁶ This creates a relative limitation in the qualitative evaluation of the surface by giving only information about fixed heights at regular 2D intervals.

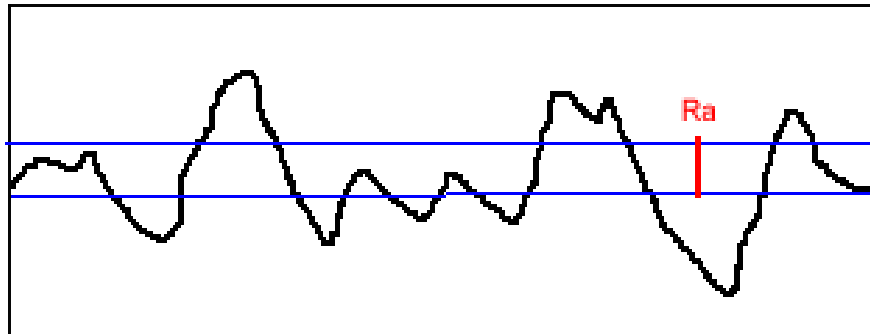


Figure 2.10. 2D surface roughness parameter, Ra (arithmetic mean of the surface)⁶

Surface profilometry, is a method of measuring surface roughness using a contacting stylus directly or a laser profilometer indirectly. Contact stylus profilometry is an older and more established technique. In fact, the current national standards for measuring surface finish are defined by stylus profilometry.¹⁶⁷ In stylus profilometry, a diamond-tipped stylus moves across the surface. The vertical movement is converted to an analogue/digital signal as the stylus is dragged across the surface. Due to the nature of

⁶ <https://www.wisetool.com/surfacefinish.htm>

the technology, the stylus is in almost constant contact with the measurement surface. However, there is a risk of the diamond tip damaging the sample. The standard profilometry method, which refers to measuring the profile of a surface, is still most commonly used to assess surface changes in tooth structure studies.^{168–171} The parameters reported in these studies are often limited and allow little qualification of the tooth surface. Laser profilometers can overcome many of the disadvantages of contact profilometry by providing indirect access to the surface. A light spot, typically less than 100 µm in diameter, is directed onto the surface. In laser profilometers, the surface topography can be profiled either by measuring the deflection of the laser beam or by using the confocal principle with white light. By arranging the map of the surface profile, roughness parameters are often used to report the results. These parameters have no limitations.

2.10. Patient-related factors influencing wear

The wear behaviour of restorative materials and their enamel antagonists is directly related to patient-related factors such as number of chewing cycles, chewing force, bite force, parafunctions such as bruxism, muscle thickness, dentition status, age, gender, salivary characteristics, dietary habits etc.^{12–16,20} The patient-related factors considered in this thesis are as follows:

2.10.1. Parafunction

A parafunctional activity, bruxism is defined as “an oral habit consisting of involuntary rhythmic or spasmodic non-functional grinding, clenching, or clenching of the teeth, in others than chewing movements of the mandible, which may result in occlusal trauma.”¹⁷² According to recent findings, bruxism is known to be a sleep disorder related to waking emotional states such as anxiety or stress, which can trigger muscle tension.¹⁷³ However, the parafunction can occur during a period of sleep or daytime consciousness, referred as sleep bruxism and waking bruxism.

In recent years, the prevalence of bruxism has increased in the general adult population and is usually considered to be one of the causative factors of tooth wear, possibly due to overloading of the teeth.^{17,18} Bruxism can lead to occlusal wear of teeth by causing changes in the functions of masticatory performance by impairing masticatory efficiency and affecting occlusal contacts. This can alter tooth-jaw relationships, leading to muscular fatigue and ultimately compromising both function and aesthetics.^{174–176}

Patients with sleep bruxism (SB) exhibit unconscious, rhythmic masticatory muscle activity and an oral motor dysfunction associated with microarousals, clenching, or grinding of the teeth.¹⁷⁷ These characteristic muscle activities produce non-physiological force and velocity contraction of the masticatory muscles, which can lead to hyperactivity, fatigue, muscle spasm, myofascial pain, and morphofunctional changes in the masticatory system. Patients with morphofunctional changes require a greater number of muscle fibers during chewing. Daytime chewing forces play a significant role in patients, especially those with SB, due to muscle strengthening.^{19,20} A positive correlation has been found between high chewing forces and masseter muscle thickness.²¹

2.10.2. Muscular status

Dynamic and static evaluations of the stomatognathic system are extremely important in understanding the mechanisms and behaviour of muscle arrangements under frequent physical stimuli that cause quantitative and qualitative changes in the structure of the masseter muscles.¹⁷⁸ According to the chewing dynamics during mastication, the jaw muscles elevate and retract the mandible..¹⁷⁹ In addition, the jaw muscles release energy during mastication and swallowing.¹⁸⁰ The process of energy release occurs only during isotonic contractions or repeated dynamics through periods of isometric contraction.¹⁸¹

Muscular status is an important factor during habitual and nonhabitual mastication. Individuals with morphofunctional changes require a greater amount of muscle fibers than healthy individuals to perform the same masticatory function, generating stress, fatigue, and decreased electromyographic activity.²¹ Patients with parafunction, such as bruxism, have more muscle contractions and greater muscle thickness, which can lead to complications later in life.¹⁸²

2.10.3. Forces

The Glossary of Prosthodontic Terms (GPT) defines the “bite force” as the act of incising or crushing between the teeth. Bite refers to incising, so it is not appropriate to use this term in the context of occlusion.¹⁸³ Occlusal force occurs as a result of muscular force exerted on opposing teeth; the force created by the dynamic action of muscles during the physiologic act of mastication. Bite force is considered essential in the diagnosis of stomatognathic system conflicts because it provides valuable information for evaluating jaw muscle function and activity.¹⁸⁴ High bite forces can accelerate tooth

wear.¹⁹ There are several studies, all of which reported that maximum bite force can increase in bruxists.^{19,185} The bite force of some bruxist patients was up to six times that of non-bruxists.¹⁸⁶ However, chewing force and muscle tone play a significant role in individuals with SB due to muscle strengthening.²⁰

Chewing force is synonymous with masticatory force and defined as the force applied by the masticatory muscles during chewing in the GPT.¹⁸³ There are several factors that influence chewing force depending on the individual.¹⁸⁷ Age and gender are the factors that influence chewing force. Males tend to have higher chewing forces than females, and older adults have lower chewing forces than younger adults.¹⁸⁸ While physiologic chewing forces are in the range of 10- 120 N, parafunctional forces are about ten times greater in the range of 200-800 N and can reach up to 1000 N.^{13,189-192} In-vitro chewing-mimicking devices have long been used to determine occlusal wear of restorative materials.¹⁹³ Chewing force is a parameter that can also be mimicked by some in-vitro wear tests.

2.11. Wear testing devices and wear simulation techniques

The wear mechanisms in the mouth are overly complex that may differ from individual to individual. Therefore, it seems impossible to reproduce the different conditions of the process by a single wear test, which are not comparable and not generally accepted. However, the wear test should consider different wear mechanisms. Most wear tests have been proposed to consider mainly a specific aspect of the different mechanisms, some of them even claim to be sufficient to fully characterize the wear resistance of dental materials. Thus, it is reasonable to investigate the wear aspects that occur under clinical conditions using laboratory tests. In vitro wear results from a laboratory test should allow comparison with in vivo findings. It may be possible to predict the complete clinical wear using various test methods.¹ Three main mechanical approaches can be considered to identify different wear simulation techniques.

2.11.1. Tooth-brushing machines

Tooth-brushing machines are used to simulate toothbrush/dentifrice abrasion concept. To design a reliable study, attention should be paid to several set-up variables that play an important role clinically. The concept consists of following elements: Toothbrush, programmable brushing techniques and paths, medium, cycles (20.000-35.600 strokes), time (60–100 min.), and vertical load (50–300 g).¹⁹⁴⁻¹⁹⁶ Brush design,

brushing behaviour and dentifrice type are critical factors in the wear studies conducted with tooth-brushing machines.

2.11.2. Two-body wear machines

The two-body wear simulators were developed to mimic clinical wear. These machines have been used with varying success due to the lack of some wear parameters. It is difficult to interpret the wear results and make comparative conclusions. Two-body wear single-pass sliding, two-body wear rotational counter sample, two-body machine sliding wear, pin-on-disk tribometer (**Figure 2.11**) and grinding wheel designs are some of the examples of these types of machines.

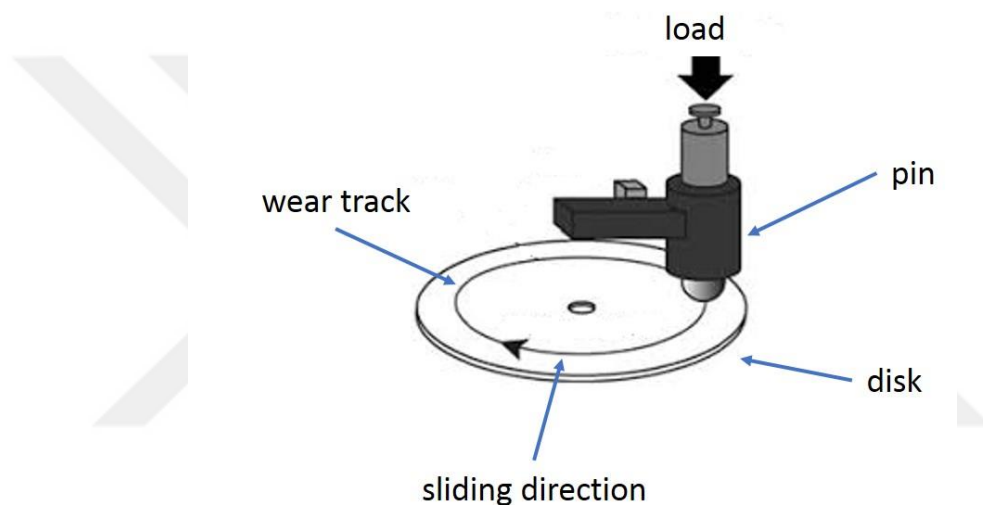


Figure 2.11. Schematic of pin-on-disk tribometer⁷

2.11.3. Three-body wear machines

The three-body wear simulators are designed to mimic the oral environment and biological variables with the aim of determining restorative materials according to their wear resistance compared to reference materials. Chewing simulators are mostly used in in vitro three-body wear machines, which can simulate chewing movements to some extent. These machines could use wear parameters with which they can achieve higher clinical success.^{52,197} ACTA, Alabama, and Willytec (**Figure 2.12**) are some of the examples of these types of machines.

⁷ <https://www.tribonet.org/tribometer/pin-on-disk/>



Figure 2.12. Willytec chewing simulator⁸

After the study design by choosing the wear simulation technique, the following processes should be validated according to the complicating parameters that affect the wear simulation:

- Preparation and storage of specimens
- Material, shape, and size of stylus
- Specification of abrasive medium and its change by time
- Reproducibility of results
- Definition of a standard or reference material

2.12. Wear in-vitro

There are six laboratory methods with different wear generating parameters according to their significance and frequency of citation in the literature such as ACTA, Alabama, Ivoclar, Munich, OHSU, Zurich methods.²⁷

2.13. Parameters influencing in-vitro wear simulation

2.13.1. Standardization of the antagonist materials

In an in-vitro wear testing system, a variety of factors regarding the in-vivo wear, must be considered in order to select the correct antagonist material and achieve an efficient simulation. Evaluation of potential antagonist materials should be supported by investigations of essential mechanical, chemical, and structural properties based on wear

⁸ https://www.researchgate.net/figure/The-Willytec-chewing-simulator_fig1_51713460

simulation.^{198,199} Antagonists should be standardized in shape and have mean values similar to non-standardized cusps found in nature.

2.13.1.1. Composition of the antagonist

Enamel antagonists are preferable to simulate wear in occlusal contact areas. In addition, there are so many options for different wear test methods such as gold, ceramic, composite, stainless steel, annealed chromium-steel antagonists, alumina ball (10 mm diameter), dental porcelain, and a semi-porous ceramic, steatite.²

2.13.1.2. Shape of the antagonist

There are different shapes for different wear test methods, such as flat, spherical or rounded types, flattened enamel surfaces, spherical or hemispherical enamel specimens from extracted human third molars, standardized human enamel cusps.²

2.13.1.3. Force/ load

The selection of the loading force is an essential part of the wear test method. A load of 50 N (~ 5 kg) is generally considered to be the mean value of physiological occlusal forces and has been used in various chewing simulation studies.^{10,41,135,186} It is well known that higher forces result in higher wear. According to another in-vitro wear study, higher forces/loads of 75 N and 100 N result in higher wear rates.²⁰⁰ However, the increase in wear may not be linearly related to the increase in load. The maximum load capacity of a chewing simulator with eight test chambers is 120 N, the upper limit of physiological forces.

2.13.1.4. Size of contact area

During chewing cycles, contact surfaces may change and it is very important to identify them. They indicate whether the material is subject to two-body wear, and they provide an estimate of the distribution of the chewing force. A limitation arises from the impossibility of controlling the forces that occur at the first contact between the loading pin (metal, ceramic ball or enamel antagonist) and the specimen surface. The initial force may be higher than programmed since it cannot be measured. This could result in non-physiological overloading of the specimens.²⁰¹ To avoid the initial overload peak at the contact surface, the simulator should be weight-controlled and operate at a relatively slow rate of descending that results in long-term chewing simulation within a reasonable period of time.²⁰²

2.13.1.5. Descending/ Lifting speed:

The velocity generated when a counter specimen strikes the surface of the specimen can produce a force that may vary at different velocities.²

2.13.1.6. Number of chewing cycles

The number of chewing cycles is an important factor to consider when comparing results from different studies. The ability to correctly simulate human chewing parameters in a uniaxial or multiaxial movement is the main requirement for a realistic chewing simulation device to perform a cyclic loading. Specific studies recommend that 1.200.000 chewing cycles should be performed to simulate 5 years of clinical use.^{203,204}

2.13.1.7. The frequency of chewing cycles

The frequencies of chewing cycles are defined with repetitions of 0.2-1.5 Hz.¹⁹¹

2.13.1.8. Contact time

During the simulation, the contact time between the loading antagonist and the material with clinically relevant lengths should be constant. In certain in-vivo studies, the contact time is reported to be 400- 600 ms/cycle of chewing.^{190,191}

2.13.1.9. Sliding movement

The sliding movement component is very important in a wear test procedure. This is because micro-fatigue of a material occurs as a result of a stress only when an antagonist slides over it. A sliding motion should be incorporated into a wear test to evaluate the fatigue strength of the material.²

2.13.1.10. Lubricant and friction

Various fluids such as water, artificial saliva, alcohol, etc. are used in three-body wear machines. The kinetic energy absorbed between the intermolecular bonds of the lubricant affects the amount transferred to the teeth.² The worn particles located in the interaction zone between the material and its antagonist could be removed by constant water change due to the thermocycling (TC) process, thus reducing the possible abrasive effect of the worn material. The constant water change could help lubricate the contact surface, reduce heat generation due to abrasion, and flush out ejected debris. However, some abrasive particles may still remain in the wear zone and affect contact stresses and wear.²⁰⁵

2.13.1.11. Temperature of ambient medium

Temperature changes of the mouth should be considered. Generally, a constant temperature in the range of 20 °C-37 °C is taken in the in vitro wear simulation studies or the specimens subjected to thermocycling in the range of 5 °C-55 °C.²

2.13.1.12. Thermocycling (TC)

In vitro studies conducted in the laboratory cannot properly simulate oral cavity conditions related to the clinical environment, moisture, and loading of teeth and restorations. Simulation of the oral cavity environment can be accomplished to some degree by aging procedures of teeth and/or restorations.²⁸

Thermocycling (TC) is an aging procedure that affects wear by influencing material-related factors. The temperature ranges, dwell times, and exposure cycles are critical parameters of this procedure.³⁸

According to the recommendations of International Standards Organization (ISO), the suggested temperature regime is 5 °C to 55 °C.²⁰⁶ Various studies have used different dwell time parameters ranging from 5 s to 2 min. ISO considered a protocol of 500 cycles to be sufficient to simulate biomaterial aging. However, for some studies, 500 cycles is too low a number to represent adequate aging time.^{207,208} In 1999, Gale and Darvell assumed that 1 year of clinical use is equivalent to approximately 10.000 thermal cycles.²⁰⁷

Only particular studies had faithfully applied the ISO standard. Most studies used their own procedures and showed only some consistency within the temperature parameter (5-55 °C) and a great deal of variability in the number of cycles and dwell time chosen.³⁸ The biggest problem has always been how to estimate the number of cycles that correspond to one year of physiological aging in the oral cavity. There are still no reports found on the number of thermal cycles per unit time in vivo.

2.13.1.13. Thermocycling and mechanical loading (TCML)

The thermal aging method along with cyclic loading, is the most widely used and necessary method for evaluating the properties of restorative materials. In 2000, a different system was proposed using thermocycling and mechanical loading to simulate the clinical conditions in the oral cavity. It was hypothesized that 50.000 cycles of occlusal loading and 8000 cycles of thermal cycling would simulate 6-8 weeks of function in the oral cavity.²⁰⁹ However, it is known that restorative dental materials are affected by intraoral conditions when used over a long period of time, usually more than 5 years.²⁹⁻³³ 1.200.000 chewing cycles and 50 N of mechanical loading along with 6000 thermal cycles are reported to be equivalent to approximately 5 years of clinical use and are an acceptable estimate of survival.^{30,34,210}

2.14. Wear evaluation methods

Many ways of measuring wear have been developed, including quantitative and qualitative methods.²¹¹ In-vivo and in-vitro wear parameters, which are clinically relevant, can be measured by comparable methods.¹²⁹

2.14.1. Wear quantification methods

Many direct or indirect methods of quantifying wear have now been developed for tooth and/or restorative materials.

2.14.1.1. Direct methods

Clinical tooth wear indices (TWI) are the most commonly used method for direct measurement of tooth wear.^{212–214} It is one of the most popular methods that does not require special equipment, but the evaluation is subjective and it takes a long time to obtain meaningful results.²¹⁵ In addition, the indices do not give a clear classification and quantification of tooth wear, so they are limited in their ability to determine the threshold of acceptable wear and to evaluate the surfaces that are decayed, filled, or covered with calculus.

2.14.1.2. Indirect methods

Measuring tooth surface loss using cast replicas is an indirect method of evaluation of tooth wear.^{117,216,217} Other methods have been developed and used for more accurate quantification of wear. These include indirect techniques such as direct cusp height measurement, image analysis, 3D scanning, computer graphics, and profilometry. The more accurate methods mentioned above used more advanced measurement tools, including stereomicroscope, computerized 3D measuring microscope, customized/commercial profilometer, laser profilometer etc.²¹⁸

3D scanning techniques have been used in various clinical studies and have allowed discrimination of early wear behaviour of restorative materials and identification of wear within 10 μm .^{217–219} These techniques, which allow 3D scanning of the entire tooth and/or material surface without compromising the structure, are highly accurate, quantitative, applicable in both clinical and laboratory settings, and provide storable 3D databases that allow comparison with other 3D databases.^{219,220} However, 3D scanning has the disadvantage of high cost and limited availability to clinical investigators, as it requires specialized hardware and software.^{129,217} From a practical point of view, patterns

must also be accurately matched before and after testing, and reference points must be established for this purpose in some cases, especially when measuring the antagonist **(Figure 2.13)**.¹

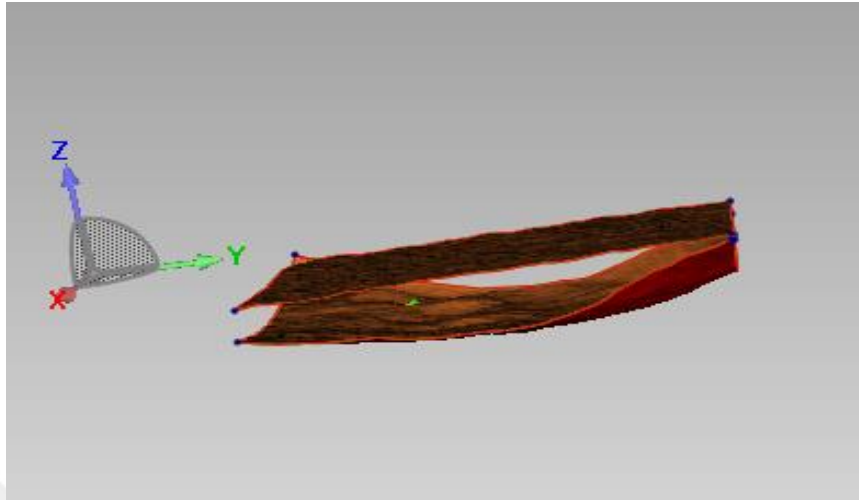


Figure 2.13. Overlap image of 3D profiles of restorative material samples before and after wear test using 3D laser scanner.⁹

The sequential 3D images of surface topologies is also an accurate method for wear measurement.¹²⁹ It is possible to obtain 3D images by using contact profilometers, non-contact white light optical profilometers, microcone CT scanners, laser scanners and CAD/CAM systems.²¹²

There are several studies that quantify only the amount of loss. In addition, there have been relatively few attempts to qualify and describe the remaining surface area beyond descriptors to provide a visual representation of the sample surface. While surface quality is less frequently reported, quantified results are often considered fundamental to comparing laboratory measurements. However, surface quality has the potential to account for certain surface characteristics that determine how future loss may progress.²²¹ Accurate substance loss is very difficult to determine with direct visualisation of surfaces. The textural characteristics of the remaining surface cannot be accurately described even if tissue loss is identified.

⁹ Figure 2.13 belongs to the current study.

2.14.2. Wear quantification parameters

Microtopographic analysis of worn surfaces can reveal different parameters when evaluating wear quantification.⁴¹

2.14.2.1. Wear volume

Wear volume which defined as the removed volume of material, is an obvious choice of wear quantification parameter to measure. The most valid method in quantifying tooth wear is the determination of the change in volume, through which the advantages of the three-dimensional model can be utilized.^{129,211}

Volume can be demonstrated through a wear equation.

$$\text{Volume loss} = k. (F_s \times d) / b$$

It should be assumed that the chewing parameters such as occlusal force, number of chewing cycles, gliding path distance, etc. remain relatively constant with time. Also, opposing materials do not change over the measuring time interval, then the shear strength b , is a constant.

Force times distance ($F_s \times d$) is work. So that, volume loss demonstrates the work done. This refers the volume loss as a material property and is independent of occlusal factors. “ d ” demonstrates the total gliding distance, and calculated as:

$$d = (\text{chewing cycles} / \text{unit time}) \times \text{gliding path} \times \text{time}$$

When d is substituted in the wear equation, and it is assumed that the chewing cycles per unit time and the length of the gliding path are relatively constant over time, it can be asserted that the volume loss is approximately proportional to time. There are particular clinical and in-vitro studies about the relationship of volume with time in the artificial oral environment where the chewing parameters are controlled and are constant with time.^{217,220} Since the modern 3D scanning equipment allows direct imaging of the sample surface, wear can now be identified three-dimensionally as volume loss rather than estimated one-dimensionally as depth or two-dimensionally as area.⁴¹

2.14.2.2. Wear area and depth

Wear area and depth are potential parameters for measuring clinical wear vertically in two and one dimensions. They represent indirect wear measurements that depend on occlusal factors and vary over time, which has some disadvantages. Assuming that the opposing teeth remain in contact during wear, there are three dynamic changes in occlusal parameters: The centric contacts shift, the orientation of the teeth to each other

changes, and the contact surfaces increase. Considering the fact of dynamic quality of wear, the wear area does not always represent active wear. The area of a wear region is an indirect measure of wear depending on occlusal factors. The size of the depth depends on where it is measured and from which direction it is measured, so it is not a good choice.

2.14.3. Wear qualification methods

Wear is assessed not only by measuring the loss of substance, but also by analysing the morphology of the wear surfaces. Microtopographic analysis of worn surfaces can reveal pattern differences between tested materials when evaluating wear qualification. Polarized light microscopy, microradiography, optical coherence tomography, cumulative calcium release, nanoindentation, iodide permeability, surface profilometry, confocal scanning laser microscopy, atomic force microscopy, and scanning electron microscopy are examples of micro- and nanoscopic wear qualification methods.²²¹ Scanning electron microscopy is the one used in this study.

2.14.3.1. Scanning electron microscopy (SEM)

SEM has been one of the most important tools in wear mechanism investigation, including comprehensive analyses of wear particles, worn specimens, and subsurface microstructure. At SEM, an electron beam generated in the filament, which can be tungsten, LaB₆ ceramic, or a field emission gun, is focused to an extremely small size to scan a defined area of a sample surface. During scanning, multiple signals are emitted as a result of physical interactions between the electron beam and the sample volume in the small volume below the beam focused area. These signals are simultaneously collected by different detectors to either generate images or perform spectroscopic analysis. SEM allows for high-resolution 3D images that have a large depth of field (all parts of the image are in focus, although they are at different depths). Due to the high resolution, wear features can be observed down to submicron and nanoscale.^{222–224} Traditional SEM imaging requires the surface to be coated with an electrically conductive material (usually gold). The purpose of this process is to prevent the accumulation of electrostatic charge. The samples are irreversibly altered during the drying and sputtering process.²²⁵

SEM can be used to characterise the microstructural changes in the depth of a worn surface.^{226,227} It provides information on surface topography and composition. This analysis is achieved by making a vertical cross section of the worn surface. Consequently,

SEM can be used to investigate wear-related changes in microstructure, such as deformation, cracking, delamination, transferred layer and intermixing, and tribofilm formation. Most wear features observed on a worn surface, such as scratches and grooves, cracks, and spalling, and transferred layers, are related to the thermal, mechanical, and/or chemical changes at specific depths of the worn sample.



3. MATERIALS AND METHODS

3.1. Materials

All teeth that are planned to use in this study were collected after taking an approval from the Yeditepe University Ethic Committee of Clinical Research (CREC Decision Number:1257). According to the power analysis and sample size software (PASS 15, NCSS LLC, Utah, USA), %95 power was achieved for 40 tooth cusp samples to detect differences with a significance level of 0.05. Tooth cusps were collected from human permanent healthy third molars and were visually inspected to ensure that no caries, damage or fillings/sealants were present. No significant evidence of the attrition was identified.

The CAD/CAM restorative materials used in this study and their main characteristics as well as the manufacturers are listed in **Table 3.1**.

Table 3.1. Materials used in study.

*According to technical product information data sheet, respectively.¹⁰

Material	Classification*	Composition*	Lot and shade/ size	Manufacturer
Vita Enamic	Polymer-infiltrated ceramic network (PICN)	86% feldspathic ceramic 14% polymer	73330 2M2-T/ EM-14	Vita Zahnfabrik, Bad Säckingen, Germany
GC Cerasmart	Resin nanoceramic	71% silica and barium nanoparticles 29% composite resin	1907241 A2 LT/14	GC Corporation, Bunkyo-ku, Tokyo
Celtra Duo	Zirconia-reinforced lithium silicate ceramic	58 % silica, lithium-metasilicate, lithium-disilicate, phosphate crystals, and 10 % zirconia crystals in addition to other minor ingredients	16004062 A2 LT/C14	Dentsply Sirona, Pennsylvania, USA
IPS e.max ZirCAD	Tetragonal polycrystalline zirconia partially stabilized with 3 mol- % yttria (3Y-TZP)	88-95.5 % ZrO ₂	Z000W8 A2 LT/ 98.5-18 mm	Ivoclar Vivadent AG, Schaan, Liechtenstein

¹⁰ Vita Zahnfabrik; Vita Enamic® technical product information data sheet. n.d.
GC Corporation; GC Cerasmart® technical product information data sheet. n.d.
Dentsply Sirona; Celtra® Duo technical product information data sheet. n.d.
Ivoclar Vivadent AG; IPS e.max ZirCAD technical product information data sheet. n.d.

3.2. Methods

3.2.1. Sample Preparation

3.2.1.1. Tooth cusp samples

The third molars were washed with distilled and deionized (DD) water after extraction to remove all blood and adherent tissue mechanically, and then stored in distilled water at 4 °C to avoid deterioration. The teeth were disinfected with a bacteriostatic/bactericidal solution of 1 % chloramine T-trihydrate (Merck KGaA, Darmstadt, Germany) and stored at room temperature for one week. This disinfection and storage procedure was performed according to ISO/ TS:11405.¹ After disinfection, each tooth was divided into 4 parts from crown to root to have 4 independent cusps (**Figure 3.1**). Ten cusp samples (n=10) were prepared for each group. The tooth cusp samples were fixed in supports using acrylic resin (**Figure 3.2**). Each cusp was identified as “cusp/material name”.



Figure 3.1. Tooth sections of third molars which were divided into four parts, from crown to root.



Figure 3.2. A tooth cusp sample which was fixed in support using acrylic resin.

3.2.1.2. Restorative material samples

Each CAD/CAM restorative material (n=10) was sectioned into approximately 6x6x6 mm³ flat-surface samples using a precision saw (Micromet 5114, Buehler, IL, USA) (**Figure 3.3 and Figure 3.4**), and then polished with silicon carbide abrasive papers (600-, 800-, 1200- grit papers, 3M, St. Paul, USA) in a polisher (Phoenix Beta Grinder, Buehler, IL, USA) under water irrigation (**Figure 3.5**). Zirconia reinforced lithium silicate (ZLS)-based ceramic blocks (Celtra Duo, Dentsply Sirona, PA, USA) were subjected to a subsequent firing process at 810 °C for crystallization (Programat P310, Ivoclar Vivadent AG, Schaan, Liechtenstein). Zirconia samples from a disk (IPS e.max ZirCAD, Ivoclar Vivadent AG, Schaan, Liechtenstein) were cut 20% thicker than specified size and then sintered in fire (inFire HTC speed; Dentsply Sirona, PA, USA) at 1500 °C (**Figure 3.6**). Crystallization and sintering were performed according to the manufacturer's recommendations. All samples were then washed with DD water for 10 min for ultrasonic cleaning.

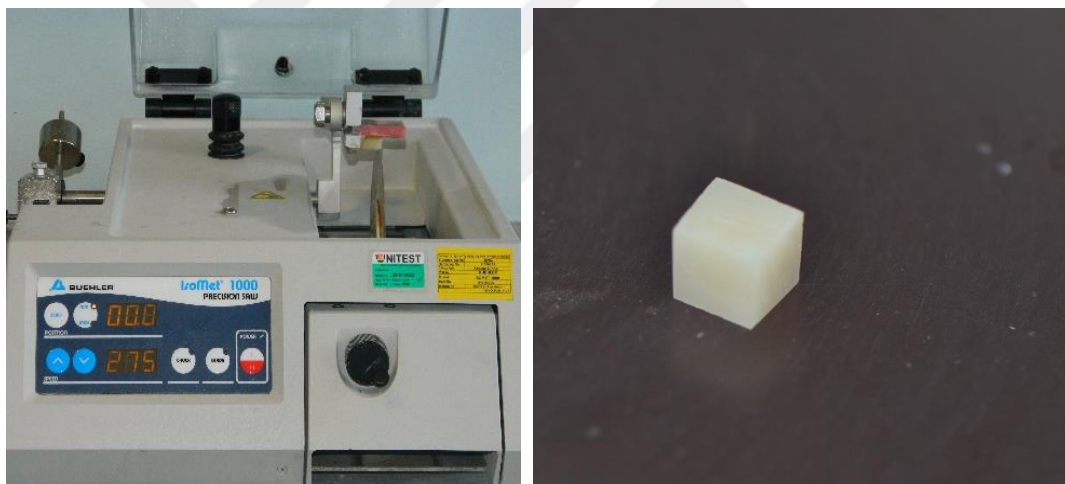


Figure 3.3. The diamond coated precision saw (Micromet 5114; Buehler, IL, USA) and cuboid flat-surface material sample



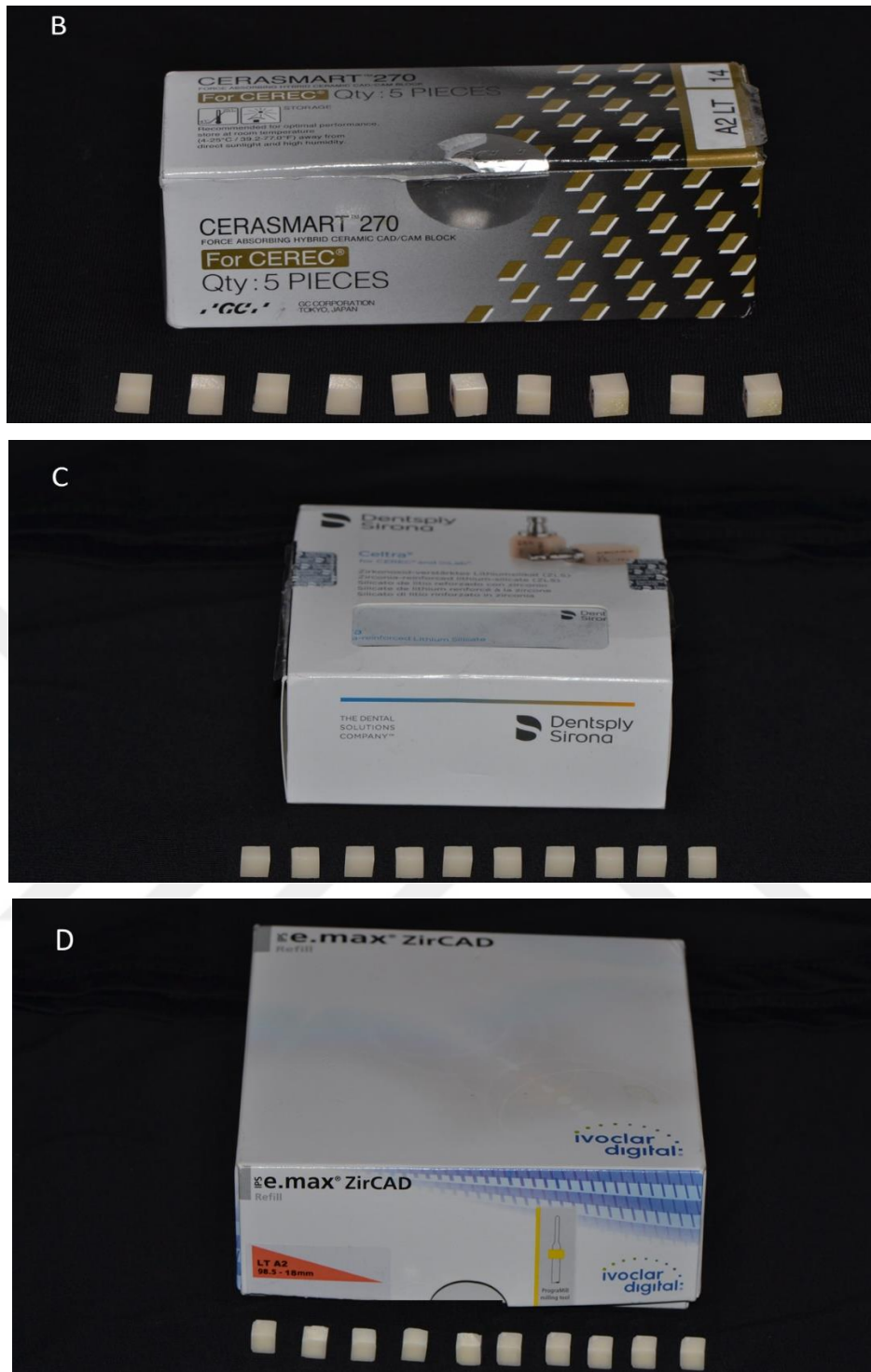


Figure 3.4. Four different groups of CAD/CAM restorative materials and ten samples taken from each group. A. VITA Enamic (Vita Zahnfabrik, Bad Säckingen, Germany), B. GC Cerasmart (GC Corporation, Bunkyo-ku, Tokyo), C. Celtra Duo (Dentsply Sirona, Pennsylvania, USA), D. IPS e.max ZirCAD (Ivoclar Vivadent AG, Schaan, Liechtenstein).

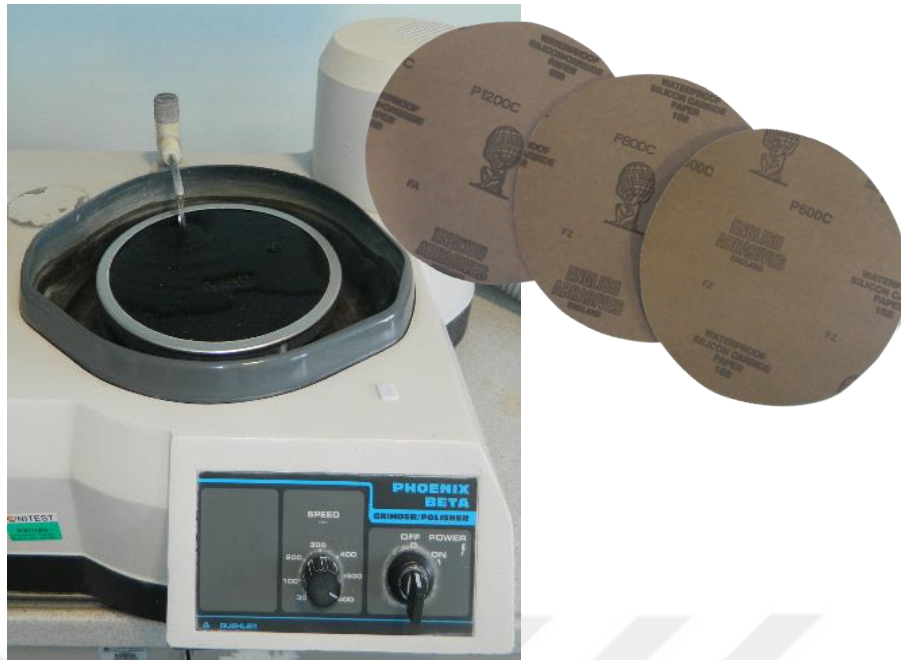


Figure 3.5. Polishing machine (Phoenix Beta Grinder/ Polisher, Buehler, Germany) and silicon carbide sandpapers with granulometers up to 600, 800 and 1200 meshes, respectively



Figure 3.6. Cutting and sintering process of zirconia samples at 1500 °C.

3.3. Wear evaluation studies

The study design is planned to evaluate wear of CAD/CAM restorative materials and tooth cusp antagonists (**Figure 3.7**).

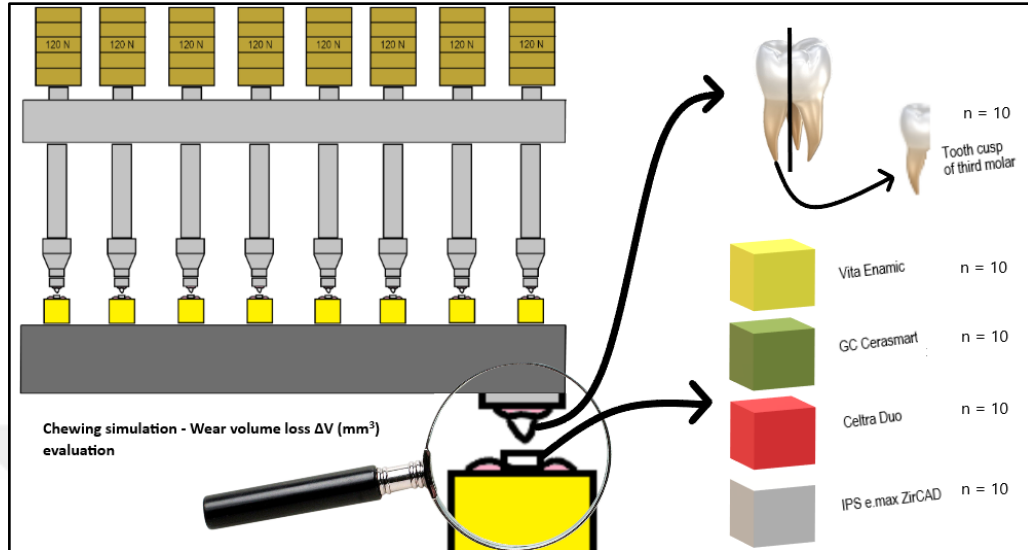


Figure 3.7. The study design (1)

3.3.1. Wear testing

In-vitro wear tests were performed in a chewing simulator machine, Dent-Arge/ACS 8.1 (Benart, Ankara, Turkey) (**Figure 3.8**), to mimic the human mastication as closely as possible.



Figure 3.8. An in-vitro wear testing machine, chewing simulator Dent-Arge/ACS 8.1 (Benart, Ankara, Turkey)

The dental cusps were fixed on a top support, while the material samples were placed at the bottom with a self-curing acrylic resin (Imicryl, Konya, Turkey). The purpose of this placement is to mimic the cusp-fossa relationship (**Figure 3.9**).

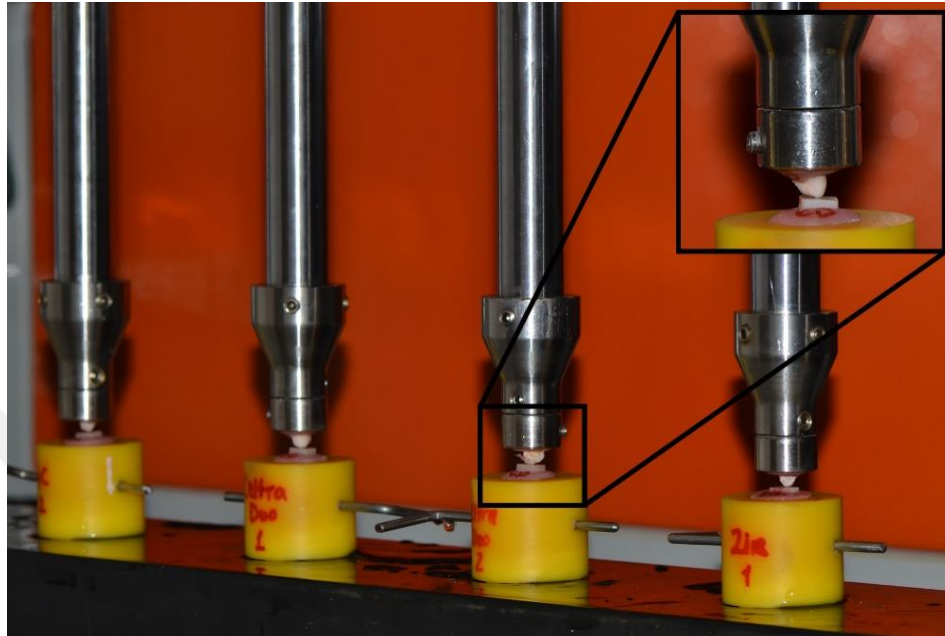


Figure 3.9. Replacing of dental samples on a top support, and material samples at the bottom support of chewing simulator to mimic the cusp-fossa relationship.

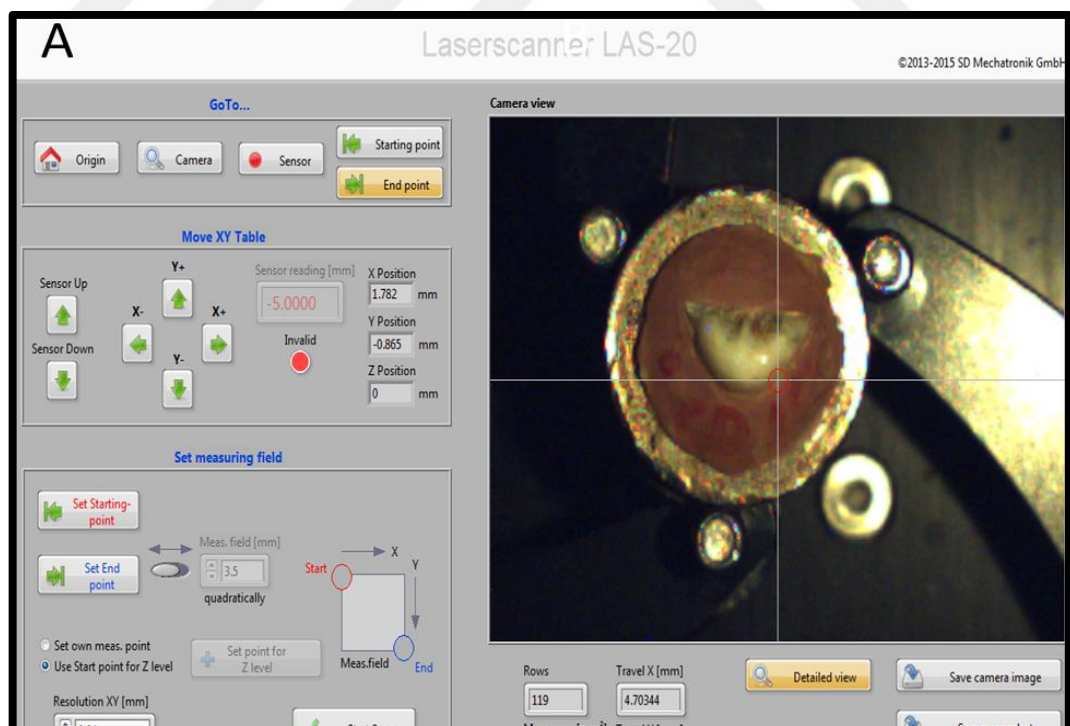
The tests were performed with the following parameters: 120 N of mechanical loading (maximum capacity of the chewing simulator machine to simulate increased chewing forces), repetitions with 1 Hz, lateral motion of 0.7 mm, vertical motion of 2 mm, lateral speed of 20 mm/s and vertical speed of 40 mm/s to simulate 5 years of chewing through 1.200.000 chewing cycles with an additional thermal cycling between 5 and 55 °C at 2 min. intervals.

3.3.2. Wear volume estimation

The 3D profiles of cusps and flat-surface materials were previously obtained using a 3D laser scanner (LAS-20, SD Mechatronic, Feldkirchen-Westerham, Germany) (**Figure 3.10 and Figure 3.11**).



Figure 3.10. The replacement of a material sample into 3D laser scanner, LAS-20 (SD Mechatronic, Feldkirchen-Westerham, Germany).



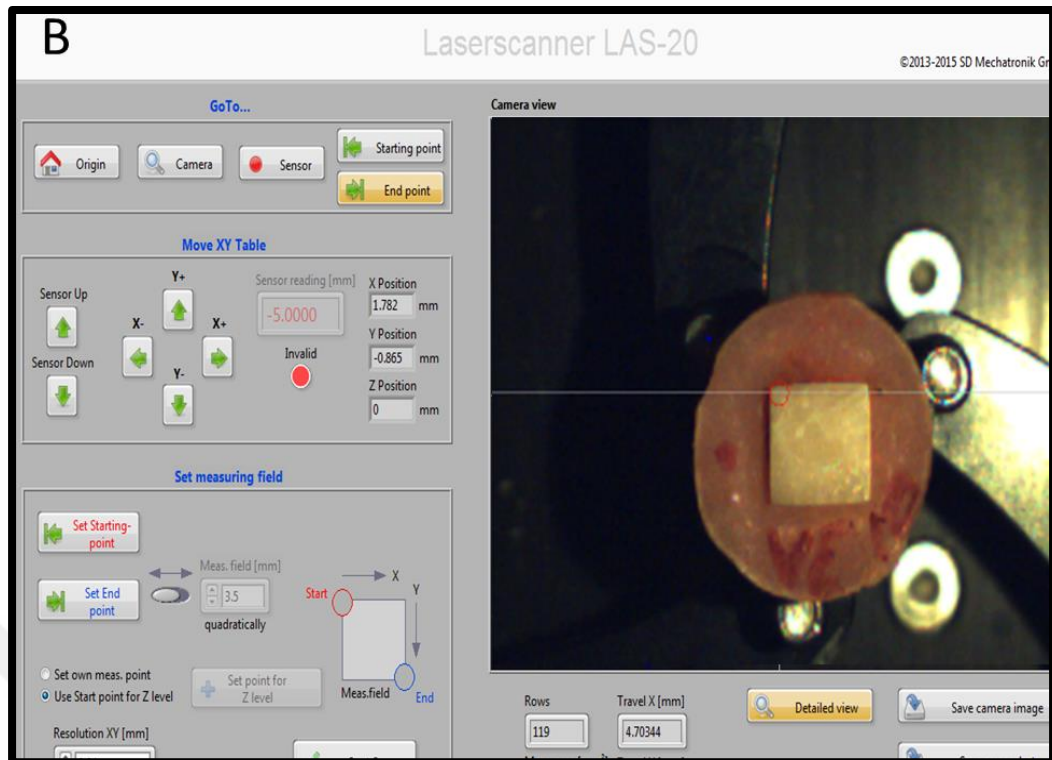


Figure 3.11. A. The screen shot of the interface of LAS-20 before scanning the tooth cusp sample
B. The screen shot of the interface of LAS-20 before scanning the material sample.

3.3.2.1. Wear volume estimation of tooth cusp samples

Before and after the wear test, each cusp was scanned using a dedicated 3D laser scanner (LAS-20, SD Mechatronic, Feldkirchen-Westerham, Germany) to determine the wear of tooth samples (**Figure 3.12**). The 3D images were overlapped using the Geomagic software program (Geomagic Control, 3D Systems Inc., SC, USA). The volumetric loss was automatically estimated.

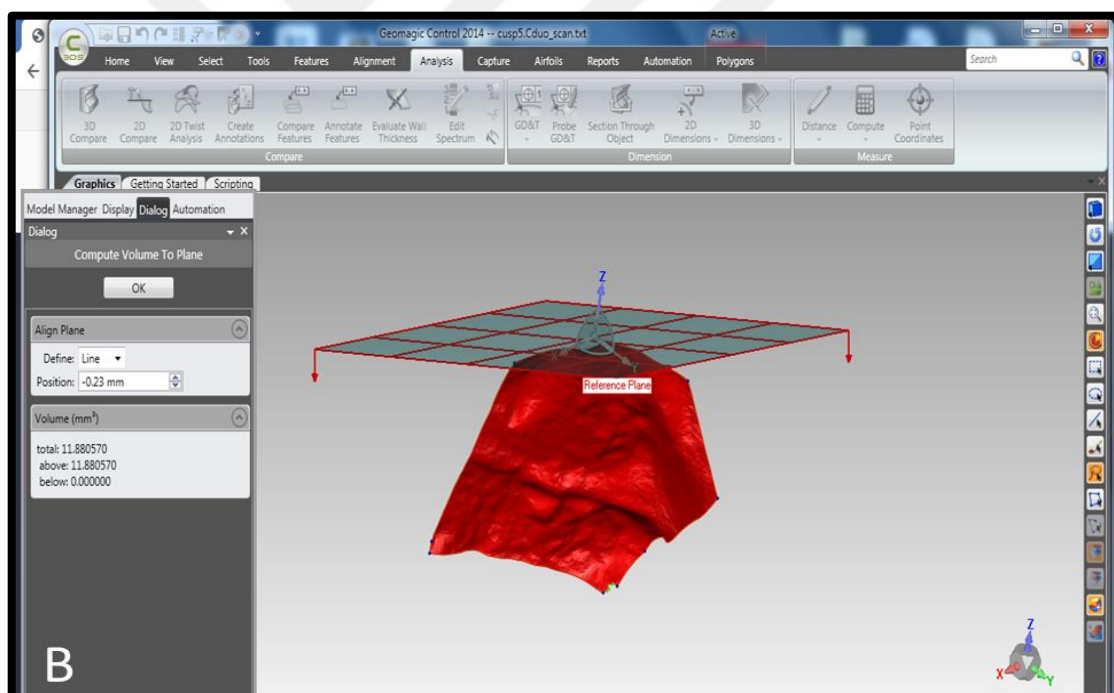
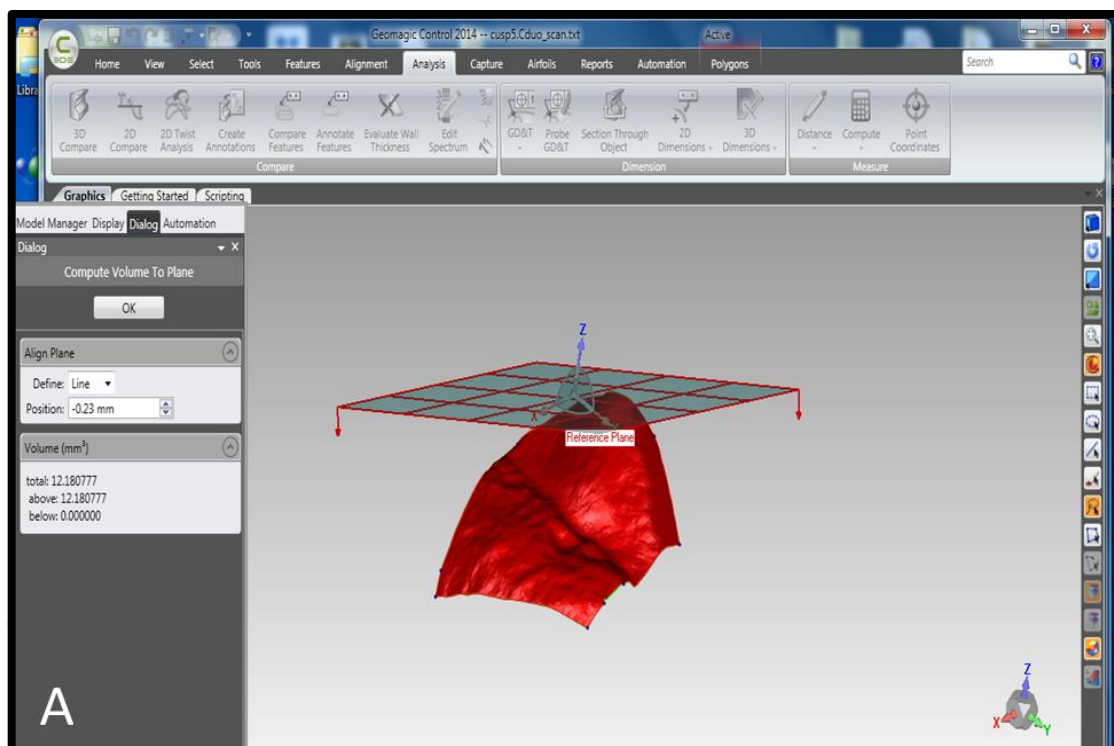


Figure 3.12. The 3D images of cusp/Celtra Duo sample, before (A) and after (B) chewing simulation in Geomagic software (Geomagic Control, 3D Systems Inc., SC, USA).

3.3.2.2. Wear volume estimation of restorative material samples

Before and after the wear test, each material sample was placed and scanned using a dedicated 3D laser scanner (LAS-20, SD Mechatronic, Feldkirchen-Westerham, Germany) (**Figure 3.13**). The 3D images were overlapped using the Geomagic software program (Geomagic Control, 3D Systems Inc., SC, USA).

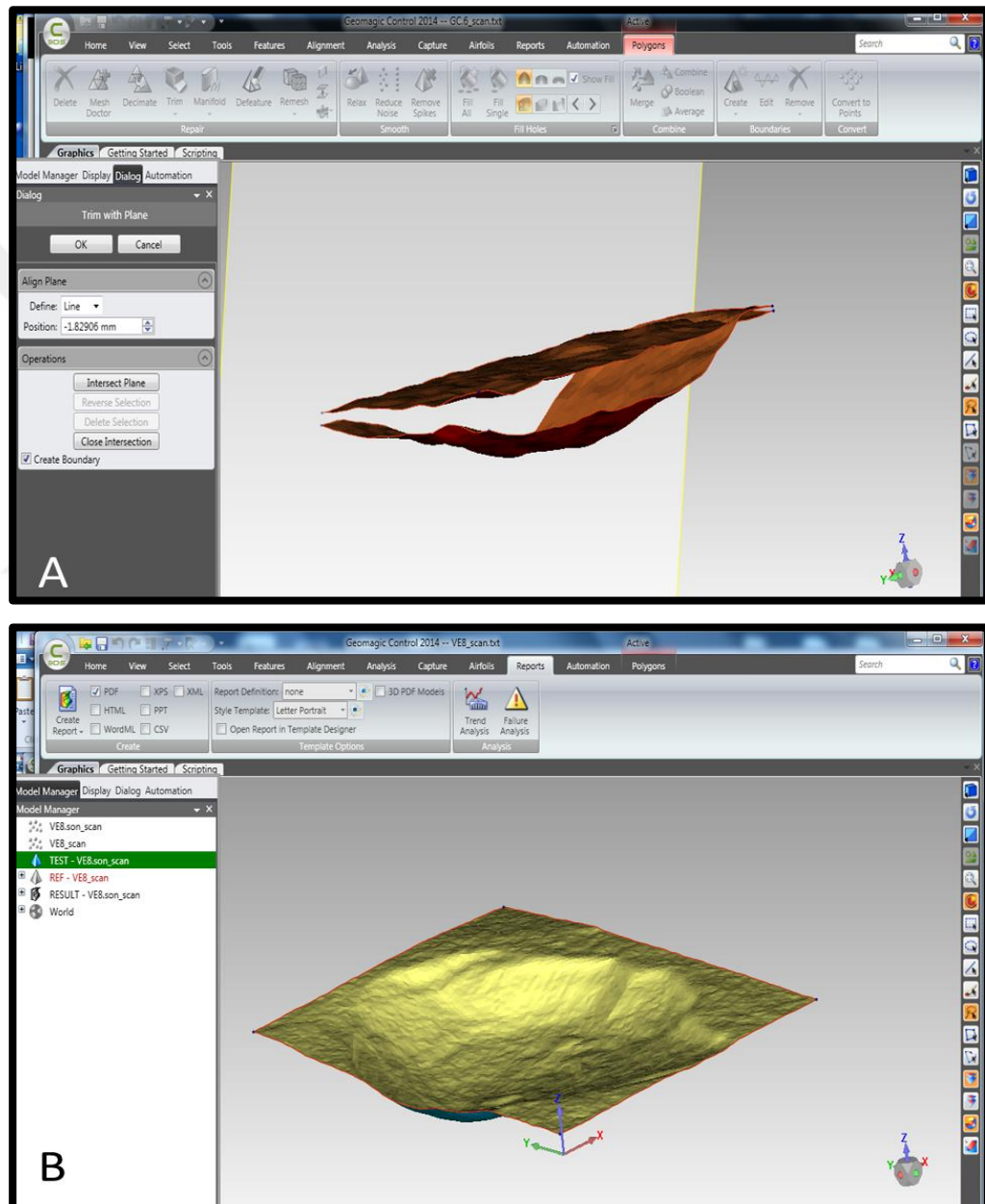


Figure 3.13. A. The 3D images of GC Cerasmart sample, before and after chewing simulation in Geomagic software (Geomagic Control, 3D Systems Inc., SC, USA) B. The 3D image of Vita Enamic sample after chewing simulation in Geomagic software (Geomagic Control, 3D Systems Inc., SC, USA)

3.4. Evaluation of Surface Characteristics of CAD/CAM Restorative Materials

The study design is planned to evaluate surface characteristics of CAD/CAM restorative materials (**Figure 3.14**).

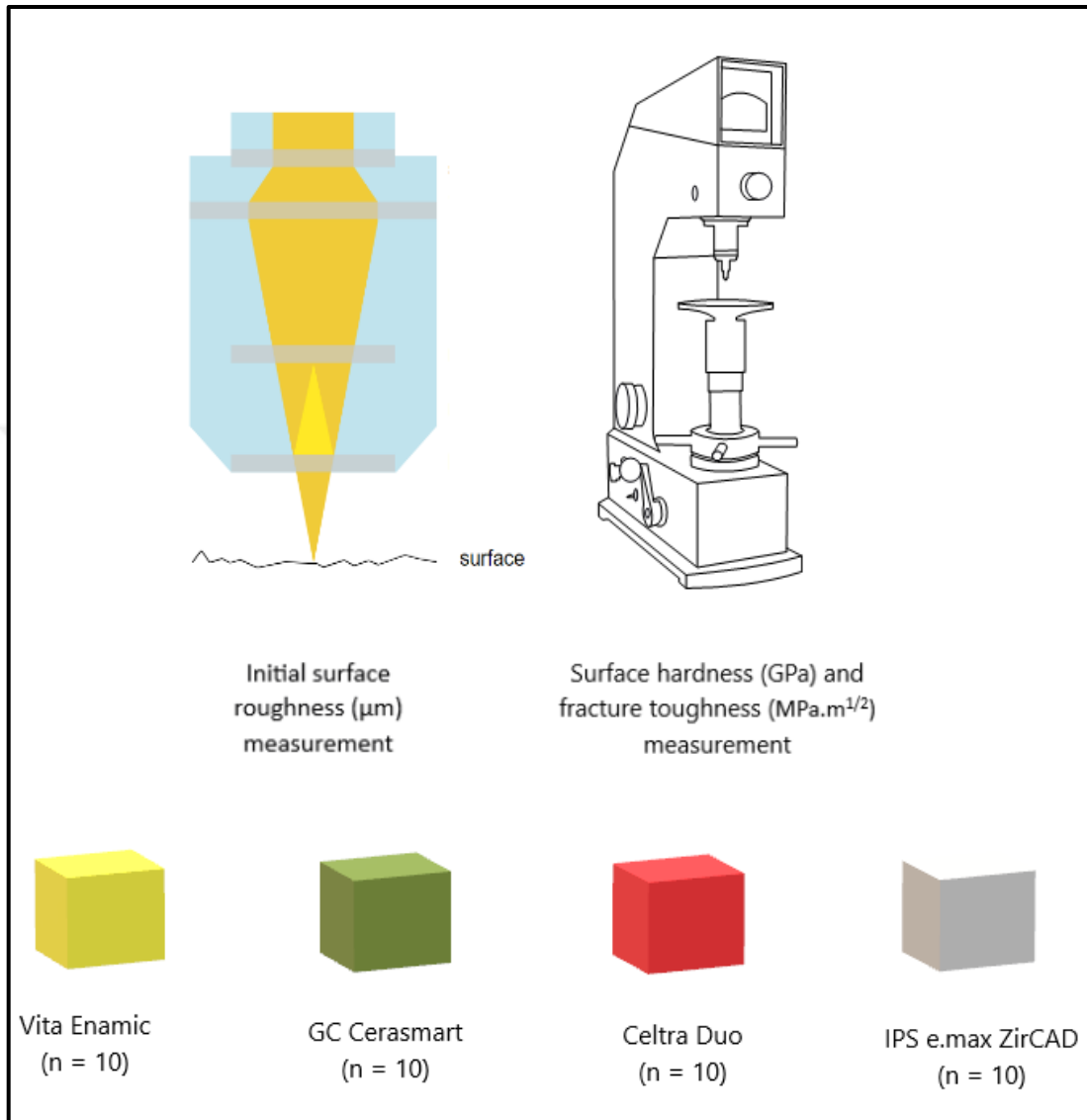


Figure 3.14. The study design (2).

3.4.1. Evaluation of surface roughness of restorative material samples

Before the wear test, each sample was evaluated using a surface roughness tester (Perthometer M1, Mahr, Czech Republic), to determine the wear of the material samples (**Figure 3.15**). Each measurement was performed triplicate. The Perthometer M1 contact profilometer could perform 2D measurements on parallel surfaces. After the wear tests, due to the indentations created by the chewing simulation, there were no flat surfaces on materials over which measurements could not have been made. Thus, only the initial

surface roughness values of the materials could be measured. The Ra values of the materials were used.



Figure 3.15. A surface roughness tester (Perthometer M1, Mahr, Czech Republic) and the measurement screen

3.4.2. Evaluation of surface hardness and fracture toughness of restorative material samples

The Vickers hardness and fracture toughness values of each material sample were determined using the Vickers hardness tester (BuehlerUK Ltd., England) (**Figure 3.16**). before and after TCML process (thermocycling (5/55 °C, 3179 cycles, 2 min. dwell time) and mechanical loading (120 N)) which is conducted by weight controlled loads and thermocycling test chambers of chewing simulator. In accordance with recommendations of ASTM/C1327-15¹³⁹, a controlled load of 1 kgf (≈ 9.807 N) with a dwell time of 15 sec.

was applied to the surface of the sample (**Figure 3.17**). Five measurements were made for each material. Acquired Vickers hardness values in units of HV were converted to units of GPa. In addition, the fracture toughness values (K_{IC}) of the materials were recorded in units of $\text{MPa}\cdot\text{m}^{1/2}$ using the equation of:

$K_{IC} = 0.0824 \cdot P/c^{3/2}$ (K_{IC} : fracture toughness, P: load, c: half diagonal of the crack length)

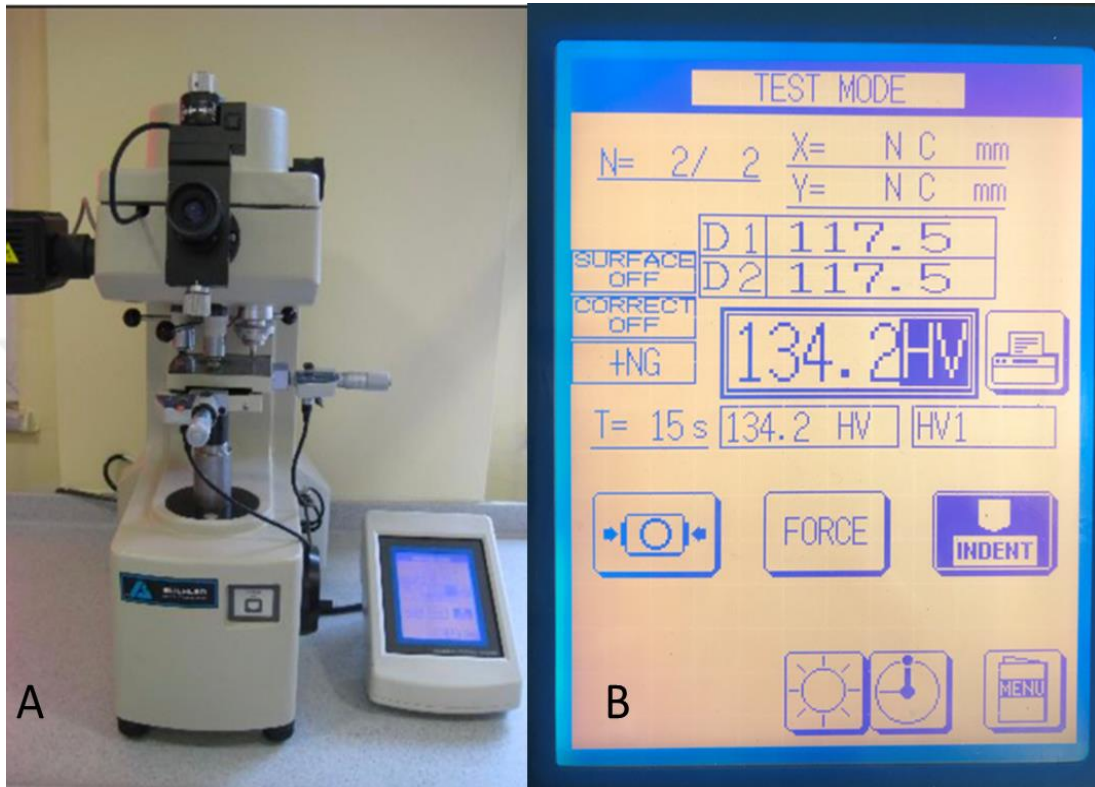


Figure 3.16. A. Vickers hardness test device (Buehler UK Ltd., England), B. Screenshot of the measurement interface

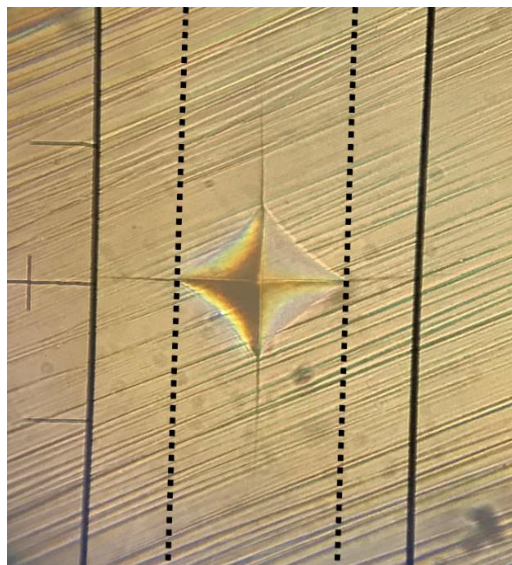


Figure 3.17. Observed indentation mark (straight lines: reference of fracture toughness measurement, dotted lines: reference of Vickers hardness measurement).

3.4.3. Evaluation of wear mechanisms

The samples were coated with a thin film of Au/Pd deposited by sputtering. According to the methodology of this study, wear mechanisms of both tooth cusps and CAD/CAM restorative materials were evaluated using scanning electron microscope (EVO10, ZEISS, Oberkochen, Germany) (**Figure 3.18**).

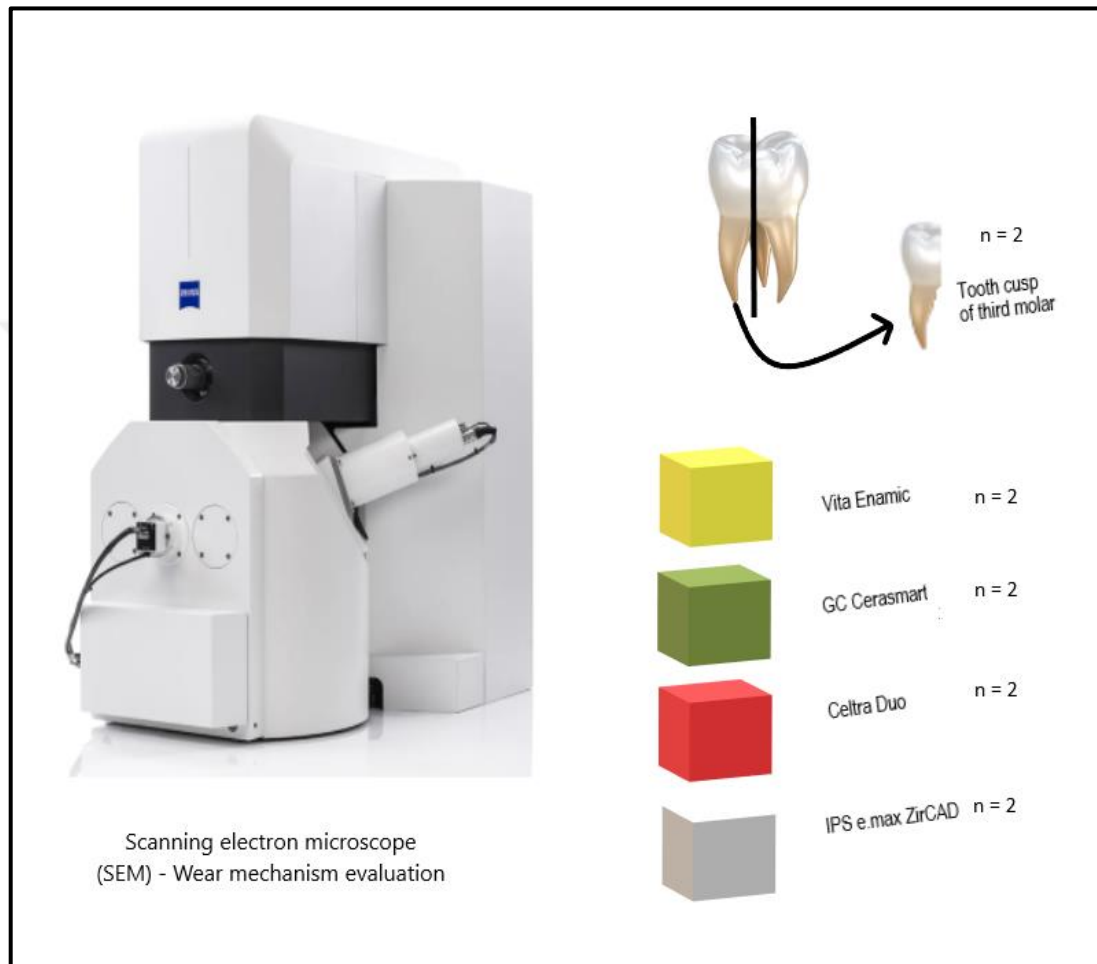


Figure 3.18. The study design (3).

3.5. Statistical Analysis

Results were analysed with SPSS 25.0 program (Statistical Packages of Social Sciences) program. Descriptive statistics of continuous data were presented as mean $\pm$ standard deviation, median, minimum, and maximum values. Normal distributions were evaluated using Shapiro Wilks test. Homogeneity of variances between groups was assessed using Levene's test. For the normally distributed data between groups with homogeneous variances, the ANOVA test was used to compare differences and post-hoc Tukey test was used for differences that were found to be statistically significant. For normally distributed data between groups with inhomogeneous variances, the Welch ANOVA test was used to compare differences. For differences that were found to be statistically significant, the post-hoc Dunnett's T3-test was used. For data that were not normally distributed between groups, the Kruskal Wallis test was used to compare differences, and for differences that were found to be statistically significant, the post-hoc Mann-Whitney u test was also used, with the Bonferroni correction applied to the p-value. For differences with time of normally distributed variables in a group, the dependent samples t-test was used; and for differences with time of non-normally distributed variables in a group, the post-hoc Wilcoxon test was used for differences found to be statistically significant, with Bonferroni correction applied to the p-value. The relationship between pairwise variables was assessed using Pearson correlation coefficient. $p < 0.05$ was accepted as significance level.

4. RESULTS

4.1. Wear volume change (ΔV) values of CAD/CAM restorative material groups

The mean values ($\bar{x}$) and standard (std.) deviations for the wear volume changes (ΔV) of the CAD/CAM restorative material groups were calculated. After determining that all data were normally distributed and variances were not homogeneous, the means were compared using the Welch ANOVA test. Post-hoc Dunnett's T3 test was used for differences found to be statistically significant. ($\alpha=0.05$)

Table 4.1. Wear volume change (ΔV) results of CAD/CAM restorative material groups

Volume Change (ΔV) (mm ³)									
95% Confidence Interval for Mean									
	n	$\bar{x}$	Std. Deviation	Std. Error	Lower Bound	Upper Bound	Minimum	Maximum	p value
Vita Enamic	8	.74	.28	.09	.50	.97	.41	1.17	.000*
GC Cerasmart	8	.82	.52	.18	.38	1.27	.20	1.83	
Celtra Duo	7	.69	.29	.11	.41	.96	.24	1.13	
IPS e.max ZirCAD	10	.02	.01	.004	.01	.03	.003	.04	
Total	33	.53	.46	.08	.37	.69	.003	1.83	

Welch ANOVA * $p<0.05$ statistically significant

The reason for the difference in the “n” numbers in the table is that there were samples that were broken due to the simulation of increased chewing forces during the experiment.

The mean values of wear volume change results (ΔV) between all CAD/CAM material groups were statistically significant. ($p<0.05$)

Table 4.2. Multiple wear volume change (ΔV) comparisons of CAD/CAM restorative material groups

Multiple Comparisons						
(1) Group	(2) Group	$\bar{x}$		p value	95% Confidence Interval	
		Difference (1-2)	Std. Error		Lower Bound	Upper Bound
Vita Enamic	GC Cerasmart	-.08	.21	.998	-.75	.58
	Celtra Duo	.05	.14	.999	-.40	.51
	IPS e.max ZirCAD	.71	.09	.001*	.37	1.06
GC Cerasmart	Vita Enamic	.08	.21	.998	-.58	.75
	Celtra Duo	.13	.21	.985	-.54	.82
	IPS e.max ZirCAD	.80	.18	.018*	.15	1.45
Celtra Duo	Vita Enamic	-.05	.14	.999	-.51	.40
	GC Cerasmart	-.13	.21	.985	-.82	.54
	IPS e.max ZirCAD	.66	.11	.005*	.25	1.07
IPS e.max ZirCAD	Vita Enamic	-.71	.09	.001*	-1.06	-.37
	GC Cerasmart	-.80	.18	.018*	-1.45	-.15
	Celtra Duo	-.66	.11	.005*	-1.07	-.25

Dunnett T3 test * $p < 0.05$ statistically significant

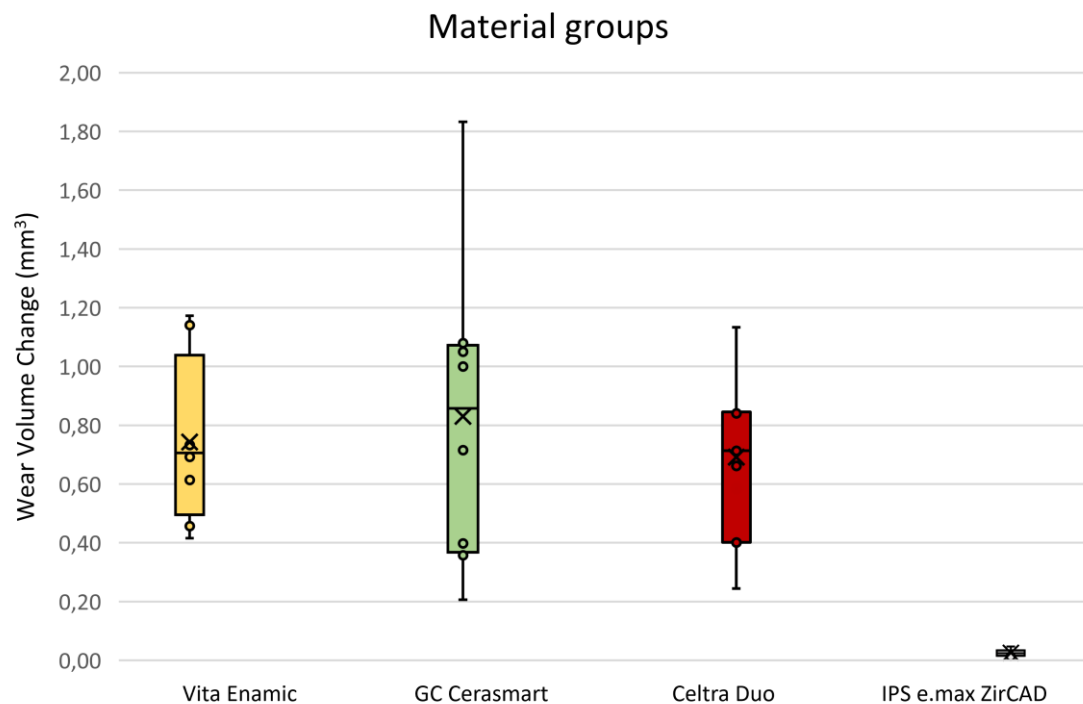


Figure 4.1. Box chart of wear volume change (ΔV) results of material groups

While the materials were arranged as IPS e.max ZirCAD ($0.02 \pm 0.01 \text{ mm}^3$) < Celtra Duo ($0.69 \pm 0.29 \text{ mm}^3$) < Vita Enamic ($0.74 \pm 0.28 \text{ mm}^3$) < GC Cerasmart ($0.82 \pm 0.52 \text{ mm}^3$) according to their wear volume change (ΔV), the only statistically significant result was found in IPS e.max ZirCAD.

IPS e.max ZirCAD material group showed lowest mean value of wear volume change (ΔV) than Vita Enamic, GC Cerasmart, Celtra Duo ($p=0.001$, $p=0.018$ and $p=0.005$, respectively).

4.2. Wear volume change (ΔV) values of cusp/material groups

The means ($\bar{x}$), medians, and standard (std.) deviations for the wear volume changes (ΔV) of the cusp/material groups were calculated. After determining that the data were not normally distributed, the median values were compared using the Kruskal Wallis test. Post-hoc Mann-Whitney u test with Bonferroni correction for p-values was used for differences found to be statistically significant. ($\alpha=0.05$)

Table 4.3. Wear volume change (ΔV) results of cusp/material groups

Volume Change (ΔV) (mm^3)							
Group	n	$\bar{x}$	Std. Deviation	Median	Minimum	Maximum	p value
Cusp/VE	7	.60	.47	.41	.21	1.52	.004*
Cusp/GC	8	.19	.12	.17	.04	.41	
Cusp/Cduo	4	.82	.53	.59	.50	1.63	
Cusp/Zir	8	.29	.13	.22	.15	.49	
Total	27	.42	.38	.26	.04	1.63	

Kruskal Wallis test * $p<0.05$ statistically significant

-VE, Vita Enamic; GC, GC Cerasmart; Cduo, Celtra Duo; Zir, IPS e.max ZirCAD.

The reason for the difference in the “n” numbers in the table is that there were samples that were broken due to the simulation of increased chewing forces during the experiment.

The median values of wear volume change results (ΔV) between cusp/material groups were statistically significant ($p<0.05$).

Table 4.4. Pairwise wear volume change (ΔV) comparisons of cusp/material groups

Pairwise Comparisons of Cusp Groups	
Group 1-Group 2	Adj. p value
Cusp/GC-Cups/Zir	1.000
Cusp/GC-Cusp/VE	.062
Cusp/GC-Cusp/Cduo	.007*
Cups/Zir-Cusp/VE	.670
Cups/Zir-Cusp/Cduo	.094
Cusp/VE-Cusp/Cduo	1.000

Mann-Whitney u test results with Bonferroni adjustment * $p < 0.05$ statistically significant
-GC, GC Cerasmart; Zir, IPS e.max ZirCAD; Cduo, Celtra Duo; VE, Vita Enamic.

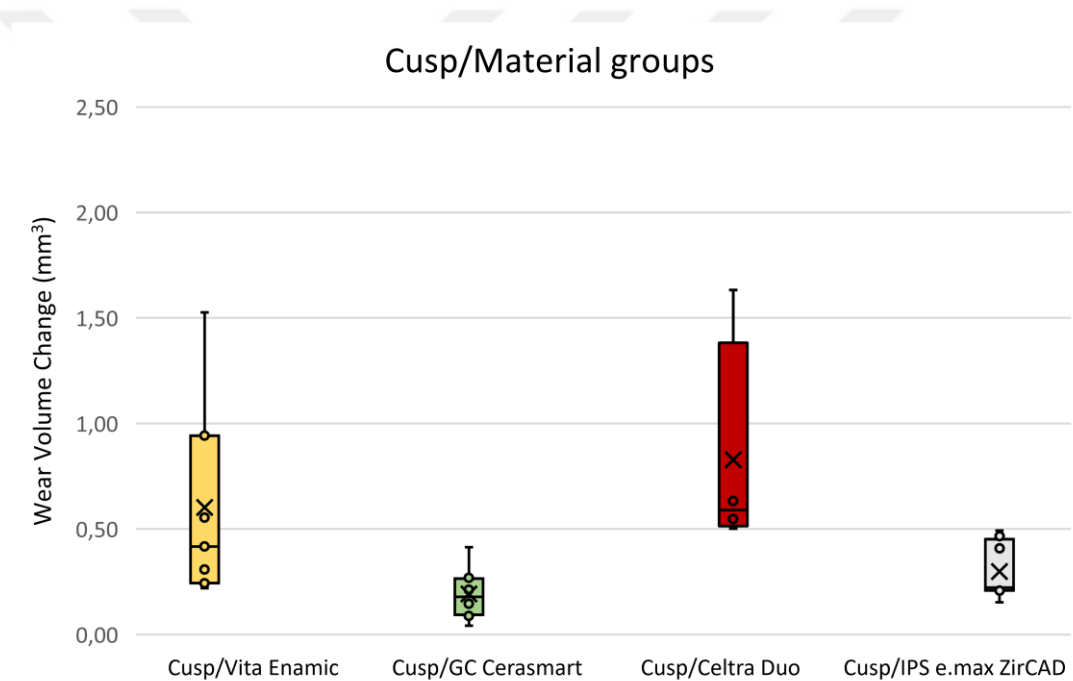


Figure 4.2. Box chart of wear volume change (ΔV) results of cusp/material groups

While the cusp groups were arranged as cusp/GC Cerasmart [0.17 mm^3 ($0.04\text{-}0.41 \text{ mm}^3$)] $<$ cusp/IPS e.max ZirCAD [0.22 mm^3 ($0.15\text{-}0.49 \text{ mm}^3$)] $<$ cusp/Vita Enamic [0.41 mm^3 ($0.21\text{-}1.52 \text{ mm}^3$)] $<$ cusp/Celtra Duo [0.59 mm^3 ($0.50\text{-}1.63 \text{ mm}^3$)] according to their wear volume change (ΔV), the one and only statistically significant difference was found between the cusp/GC Cerasmart and cusp/Celtra Duo groups. ($p < 0.05$)
The wear volume change (ΔV) of cusp/Celtra Duo group was higher than cusp/GC Cerasmart group ($p = 0.007$).

4.3. Initial surface roughness values of CAD/CAM restorative material groups

The means ($\bar{x}$), medians and standard (std.) deviations for initial surface roughness (μm) values of the CAD/CAM restorative material groups were evaluated. After determining that the data were not normally distributed, the median values were compared using the Kruskal Wallis test. For differences found to be statistically significant, a post-hoc Mann-Whitney u test with Bonferroni correction applied to the p-values was used. ($\alpha=0.05$)

Table 4.5. Initial surface roughness (μm) results of CAD/CAM restorative material groups

Ra	Group	n	$\bar{x}$	Std.			Maximum	p value
				Deviation	Median	Minimum		
	Vita Enamic	10	.05	.005	.05	.04	.06	.000 ^{a*}
	GC Cerasmart	10	.03	.003	.03	.02	.03	
	Celtra Duo	10	.04	.070	.02	.01	.23	
	IPS e.max ZirCAD	10	.03	.006	.03	.02	.04	
	Total	40	.04	.03	.03	.01	.23	

^aKruskal Wallis test * $p<0.05$ statistically significant.

The median values of initial surface roughness results (μm) between all CAD/CAM material groups were statistically significant. ($p<0.05$)

Table 4.6. Pairwise initial surface roughness comparisons of CAD/CAM restorative material groups

Group 1- Group 2	Adj. p value
Celtra Duo-GC Cerasmart	.104
Celtra Duo-IPS e.max ZirCAD	.014*
Celtra Duo-Vita Enamic	.000*
GC Cerasmart-IPS e.max ZirCAD	1.000
GC Cerasmart-Vita Enamic	.007*
IPS e.max ZirCAD-Vita Enamic	.061

Mann-Whitney u test result with Bonferroni adjustment * $p<0.05$ statistically significant.

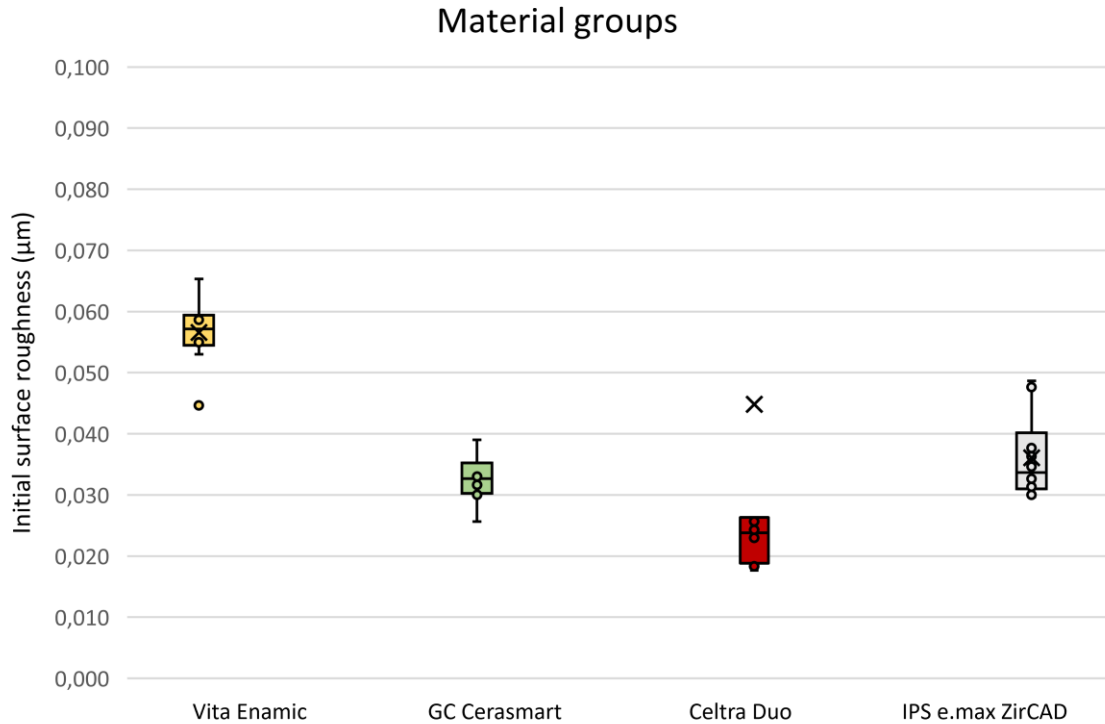


Figure 4.3. Box chart of initial surface roughness (Ra) results of material groups

While the material groups were arranged as Celtra Duo [0.024 μm (0.017-0.023 μm)] < GC Cerasmart [0.031 μm (0.025-0.039 μm)] < IPS e.max ZirCAD [0.033 μm (0.029-0.048 μm)] < Vita Enamic [0.057 μm (0.044-0.065 μm)] according to their initial surface roughness (μm) Ra values, the statistically significant differences were found as follows: The median Ra value of Celtra Duo group was lower than IPS e.max ZirCAD and Vita Enamic group (p=0.014 and p=0.000). Celtra Duo was initially smoother than IPS e.max ZirCAD and Vita Enamic.

The median Ra value of GC Cerasmart group was lower than the Vita Enamic group (p=0.007). GC Cerasmart was initially smoother than Vita Enamic.

4.4. Surface hardness values of CAD/CAM restorative material groups

The means ($\bar{x}$), medians and standard (std.) deviations for surface hardness values (GPa) of CAD/CAM material groups with differences in time were evaluated. The median values of “before thermocycling and mechanical loading (TCML)” data which were not normally distributed, compared with Kruskal Wallis test. For differences found to be statistically significant, a post-hoc Mann-Whitney u test with Bonferroni correction applied to the p-values was used. ($\alpha=0.05$) The mean ($\bar{x}$) values of “after TCML” data were compared using the Welch ANOVA test, after determining that all data were normally distributed, and variances were not homogeneous. For differences found to be statistically significant, the post-hoc Dunnett's T3 test was used. ($\alpha=0.05$)

Table 4.7. Surface hardness (GPa) results of CAD/CAM restorative material groups

	Group	n	$\bar{x}$	Std. Deviation	Median	Minimum	Maximum	p value
Before TCML	Vita Enamic	10	3.63	.34	3.58	3.25	4.38	.000 ^{a*}
	GC Cerasmart	10	1.57	.23	1.55	1.28	2.14	
	Celtra Duo	10	6.25	.49	6.34	4.95	6.73	
	IPS e.max ZirCAD	10	14.19	1.38	13.47	12.69	16.56	
	Total	40	6.41	4.905556	4.67	1.28	16.56	
After TCML	Vita Enamic	10	2.21	.37	2.25	1.4	2.75	.000 ^{b*}
	GC Cerasmart	10	1.46	.28	1.46	1.08	1.89	
	Celtra Duo	10	6.26	.34	6.34	5.62	6.74	
	IPS e.max ZirCAD	10	12.55	.71	12.67	11.26	13.69	
	Total	40	5.62	4.47	4.18	1.08	13.69	
Difference	Vita Enamic	10	1.41	.59	1.23	.56	2.34	.000 ^{b*}
	GC Cerasmart	10	.10	.26	.13	-.41	.58	
	Celtra Duo	10	-.01	.45	-.11	-.92	.70	
	IPS e.max ZirCAD	10	1.64	1.51	1.52	-1.00	4.41	
	Total	40	.78	1.11	.50	-1.00	4.41	

^aKruskal Wallis test ^b Welch ANOVA *p<0.05 statistically significant

4.4.1. Surface hardness values before thermocycling and mechanical loading (TCML)

The median values of surface hardness results between all CAD/CAM material groups “before TCML” were statistically significant. ($p < 0.05$)

Table 4.8. Pairwise comparisons of surface hardness values of CAD/CAM restorative material groups before TCML

Group 1-Group 2	Adj. p value
GC Cerasmart-Vita Enamic	.335
GC Cerasmart-Celtra Duo	.001*
GC Cerasmart-IPS e.max ZirCAD	.000*
Vita Enamic-Celtra Duo	.335
Vita Enamic-IPS e.max ZirCAD	.001*
Celtra Duo-IPS e.max ZirCAD	.335

Mann-Whitney u test result with Bonferroni adjustment * $p < 0.05$ statistically significant.

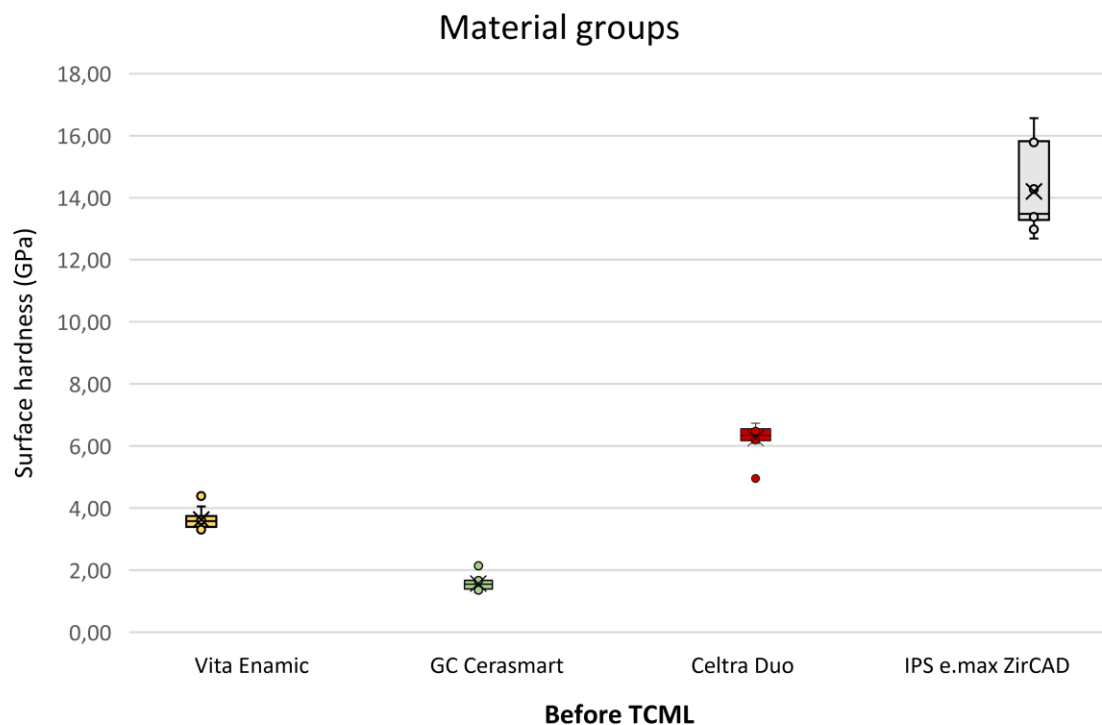


Figure 4.4. Box chart of surface hardness results of material groups before TCML

While the material groups were arranged as GC Cerasmart [1.55 GPa (1.28-2.14 GPa)] < Vita Enamic [3.58 GPa (3.25-4.38 GPa)] < Celtra Duo [6.34 GPa (4.95-6.73 GPa)] < IPS e.max ZirCAD [13.47 GPa (12.69-16.56 GPa)] according to their initial surface hardness (GPa) values, the statistically significant differences were found as follows:

The median surface hardness value of GC Cerasmart group was lower than Celtra Duo and IPS e.max ZirCAD material groups before TCML ($p=0.001$ and $p=0.000$).

The median surface hardness value of Vita Enamic group was lower than IPS e.max ZirCAD material group ($p=0.001$).

4.4.2. Surface hardness values after TCML

Table 4.9. Multiple comparisons of surface hardness values of CAD/CAM restorative material groups after TCML

(1) Group	(2) Group	$\bar{x}$ Difference (1-2)	Std. Error	P value	95% Confidence Interval	
					Lower Bound	Upper Bound
Vita Enamic	GC Cerasmart	.74*	.14	.001*	.30	1.18
	Celtra Duo	-4.05*	.16	.000*	-4.52	-3.57
	IPS e.max ZirCAD	-10.33*	.25	.000*	-11.10	-9.56
GC Cerasmart	Vita Enamic	-.74*	.14	.001*	-1.18	-.30
	Celtra Duo	-4.79*	.14	.000*	-5.21	-4.38
	IPS e.max ZirCAD	-11.08*	.24	.000*	-11.83	-10.33
Celtra Duo	Vita Enamic	4.05*	.16	.000*	3.57	4.52
	GC Cerasmart	4.79*	.14	.000*	4.38	5.21
	IPS e.max ZirCAD	-6.28*	.25	.000*	-7.04	-5.52
IPS e.max ZirCAD	Vita Enamic	10.33*	.25	.000*	9.56	11.10
	GC Cerasmart	11.08*	.24	.000*	10.33	11.83
	Celtra Duo	6.28*	.25	.000*	5.52	7.04

Dunnett T3 test * $p<0.05$ statistically significant

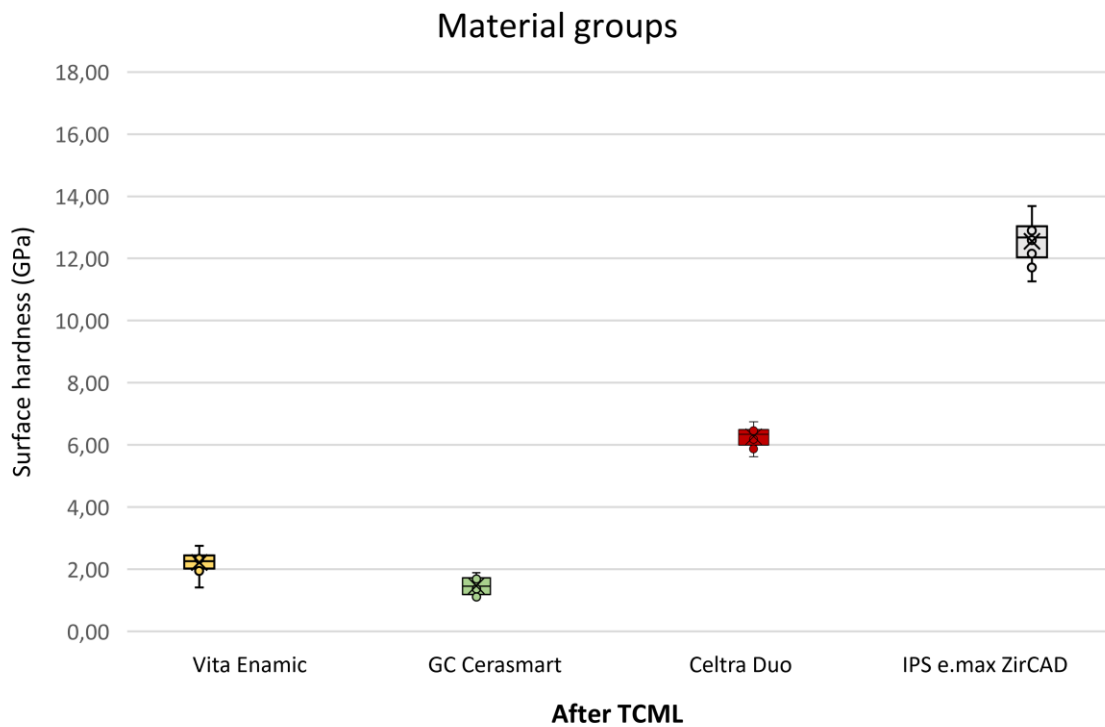


Figure 4.5. Box chart of surface hardness results of material groups after TCML

The mean hardness values of material groups were arranged from lower to higher as follows:

GC Cerasmart (1.46 ± 0.28 GPa) < Vita Enamic (2.21 ± 0.37 GPa) < Celtra Duo (6.26 ± 0.34 GPa) < IPS e.max ZirCAD (12.55 ± 0.71 GPa)

The pairwise comparison of mean values of surface hardness results after TCML were found statistically significant for all material groups. ($p < 0.05$)

4.4.3. Surface hardness difference between CAD/CAM restorative material groups before & after TCML

Post-hoc Dunnett's T3 pairwise comparison test was used to determine the material groups which had statistically significant differences of surface hardness change. ($\alpha=0.05$)

Table 4.10. Multiple comparisons of surface hardness values of CAD/CAM restorative material groups by differences between before & after TCML

(1) Group	(2) Group	$\bar{x}$ Difference		p value	95% Confidence Interval	
		(1-2)	Std. Error		Lower Bound	Upper Bound
Vita Enamic	GC Cerasmart	1.31*	.20	.000*	.67	1.94
	Celtra Duo	1.43*	.23	.000*	.73	2.13
	IPS e.max ZirCAD	-.22	.51	.998	-1.82	1.37
GC Cerasmart	Vita Enamic	-1.31*	.20	.000*	-1.94	-.67
	Celtra Duo	.11	.16	.975	-.38	.61
	IPS e.max ZirCAD	-1.53	.48	.056	-3.11	.03
Celtra Duo	Vita Enamic	-1.43*	.23	.000*	-2.13	-.73
	GC Cerasmart	-.11	.16	.975	-.61	.38
	IPS e.max ZirCAD	-1.65*	.50	.039*	-3.23	-.07
IPS e.max ZirCAD	Vita Enamic	.22	.51	.998	-1.37	1.82
	GC Cerasmart	1.53	.48	.056	-.03	3.11
	Celtra Duo	1.65*	.50	.039*	.07	3.23

Dunnett T3 test * $p < 0.05$ statistically significant

While the surface hardness difference of materials was arranged as Celtra Duo (-0.01 ± 0.45 GPa) < GC Cerasmart (0.10 ± 0.26 GPa) < Vita Enamic (1.41 ± 0.59 GPa) < IPS e.max ZirCAD (1.64 ± 1.51 GPa) the statistically significant differences were found as follows:

The mean surface hardness change value of Vita Enamic was higher than GC Cerasmart and Celtra Duo material groups ($p=0.000$ and $p=0.000$).

The mean surface hardness change value of IPS e.max ZirCAD was higher than Celtra Duo material group ($p=0.039$).

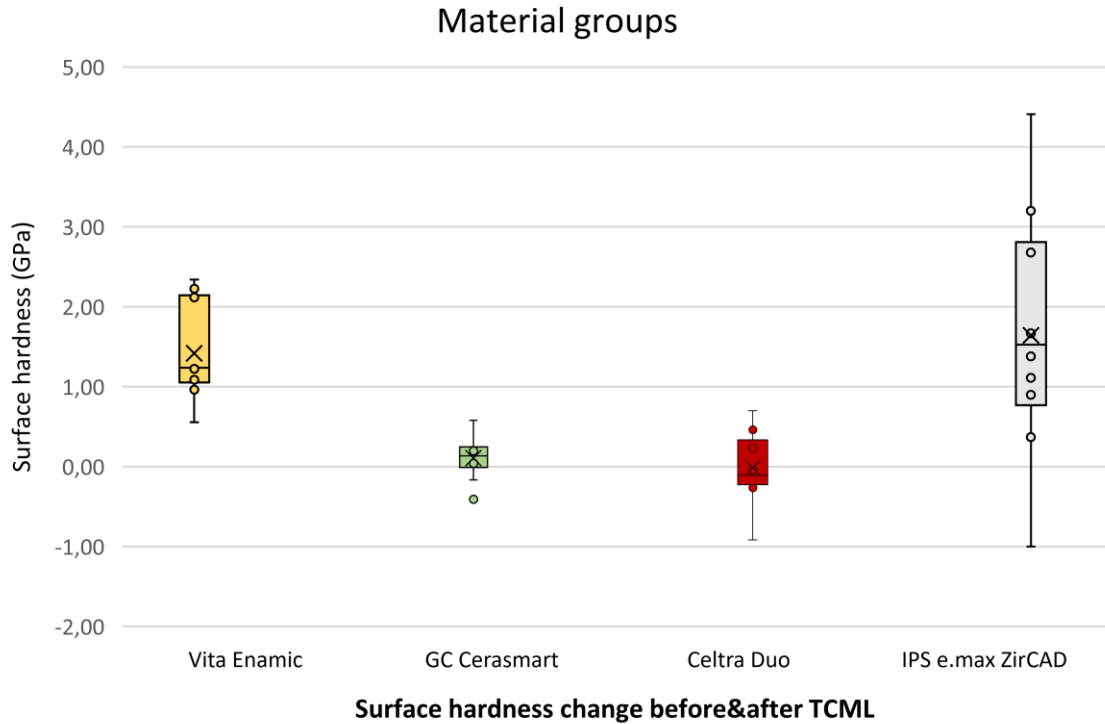


Figure 4.6. Box chart of surface hardness change due to TCML in material groups

4.4.4. Surface hardness change of each restorative material group by differences in time due to TCML

For normally distributed data, paired sample t test; for data which were not normally distributed, Wilcoxon t test was used to evaluate differences in time by each group. ($\alpha=0.05$)

Table 4.11. Comparisons of surface hardness values of every single CAD/CAM restorative material group by differences in time

Group		n	Std.		Median	Minimum	Maximum	p value
			Mean	Deviation				
Vita Enamic	Before TCML	10	3.63	.34	3.58	3.25	4.38	.000 ^{a*}
	After TCML	10	2.21	.37	2.25	1.41	2.75	
GC Cerasmart	Before TCML	10	1.57	.23	1.55	1.28	2.14	.238 ^a
	After TCML	10	1.46	.28	1.46	1.08	1.89	
Celtra Duo	Before TCML	10	6.25	.49	6.34	4.95	6.73	.878 ^b
	After TCML	10	6.26	.34	6.34	5.62	6.74	
IPS e.max ZirCAD	Before TCML	10	14.19	1.38	13.47	12.69	16.56	.008 ^{a*}
	After TCML	10	12.55	.71	12.67	11.26	13.69	

^a Paired sample t test ^b Wilcoxon t test * $p<0.05$ statistically significant

Vita Enamic and IPS e.max ZirCAD were the only groups showing change with differences in time, the mean change values were statistically significant. ($p < 0.05$)

The mean surface hardness values before TCML were higher than after TCML in Vita Enamic ($3.63 \pm 0.34 / 2.21 \pm 0.37$ GPa) and IPS e.max ZirCAD ($14.19 \pm 1.38 / 12.55 \pm 0.71$ GPa) material groups. The surface hardness of Vita Enamic and IPS e.max ZirCAD material groups had decreased due to TCML process.

4.5. Fracture toughness values of CAD/CAM restorative material groups

The means ($\bar{x}$), medians and standard (std.) deviations for fracture toughness values ($\text{MPa.m}^{1/2}$) of CAD/CAM material groups with differences in time were evaluated. The median values of “before and mechanical loading (TCML)” data which were not normally distributed, compared with Kruskal Wallis test. For differences found to be statistically significant, a post-hoc Mann-Whitney u test with Bonferroni correction applied to the p-values was used. ($\alpha = 0.05$) The mean ($\bar{x}$) values of “after TCML” data were compared using the Welch ANOVA test, after determining that all data were normally distributed, and variances were not homogeneous. For differences found to be statistically significant, the post-hoc Dunnett's T3 test was used. ($\alpha = 0.05$)

Table 4.12. Fracture toughness ($\text{MPa.m}^{1/2}$) values of CAD/CAM restorative material groups

	Group	n	$\bar{x}$	Std. Deviation	Median	Minimum	Maximum	p value
Before TCML	Vita Enamic	10	2.75	.67	2.86	.99	3.49	.000 ^{a*}
	GC Cerasmart	10	1.17	.14	1.14	.99	1.39	
	Celtra Duo	10	2.44	.40	2.37	1.82	3.09	
	IPS e.max ZirCAD	10	13.67	1.25	13.52	11.26	15.25	
	Total	40	5.00	5.14	2.72	.99	15.25	
After TCML	Vita Enamic	10	2.14	.48	2.14	1.39	3.19	.000 ^{b*}
	GC Cerasmart	10	1.24	.16	1.27	1.01	1.43	
	Celtra Duo	10	2.79	.75	2.67	1.84	4.11	
	IPS e.max ZirCAD	10	11.66	1.19	11.37	10.51	13.81	
	Total	40	4.46	4.30	2.28	1.01	13.81	
Difference	Vita Enamic	10	.61	.64	.63	-.84	1.58	.005 ^{b*}
	GC Cerasmart	10	-.07	.20	-.10	-.28	.37	
	Celtra Duo	10	-.35	1.02	-.20	-2.29	.77	
	IPS e.max ZirCAD	10	2.00	2.11	2.35	-2.28	4.61	
	Total	40	.54	1.49	.33	-2.29	4.61	

^aKruskal Wallis test ^b Welch ANOVA * $p < 0.05$ statistically significant.

4.5.1. Fracture toughness values before TCML

By evaluating differences in fracture toughness values of material groups “before TCML”, the results were found statistically significant. ($p < 0.05$)

Table 4.13. Pairwise comparisons of fracture toughness values of CAD/CAM restorative material groups before TCML

Group 1 - Group 2	Adj. p value
GC Cerasmart-Celtra Duo	.159
GC Cerasmart-Vita Enamic	.018*
GC Cerasmart-IPS e.max ZirCAD	.000*
Celtra Duo-Vita Enamic	1.000
Celtra Duo-IPS e.max ZirCAD	.005*
Vita Enamic-IPS e.max ZirCAD	.059

Mann-Whitney u test result with Bonferroni adjustment * $p < 0.05$ statistically significant.

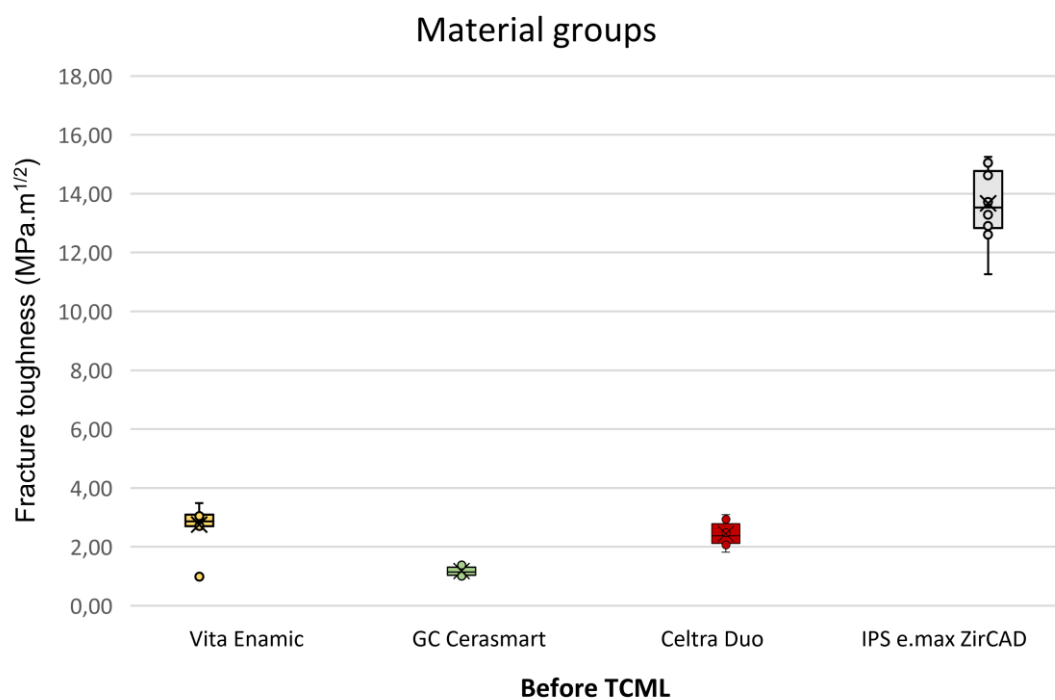


Figure 4.7. Box chart of fracture toughness results of material groups before TCML

While the material groups were arranged as GC Cerasmart [$1.14 \text{ MPa.m}^{1/2}$ ($0.99\text{-}1.39 \text{ MPa.m}^{1/2}$)] < Celtra Duo [$2.37 \text{ MPa.m}^{1/2}$ ($1.82\text{-}3.09 \text{ MPa.m}^{1/2}$)] < Vita Enamic [$2.86 \text{ MPa.m}^{1/2}$ ($0.99\text{-}3.49 \text{ MPa.m}^{1/2}$)] < IPS e.max ZirCAD [$13.52 \text{ MPa.m}^{1/2}$ ($11.26\text{-}15.25 \text{ MPa.m}^{1/2}$)] according to their fracture toughness values, the statistically significant differences were found as follows:

The median fracture toughness value of GC Cerasmart before TCML was lower than Vita Enamic and IPS e.max ZirCAD material groups ($p=0.018$ and $p=0.000$).

The median fracture toughness value of Celtra Duo before TCML was lower than IPS e.max ZirCAD material group ($p=0.005$).

4.5.2. Fracture toughness values after TCML

Table 4.14. Multiple comparisons of fracture toughness values of CAD/CAM restorative material groups after TCML

$\bar{x}$					95% Confidence Interval	
Difference						
(1) Group	(2) Group	(1-2)	Std. Error	p value	Lower Bound	Upper Bound
Vita Enamic	GC Cerasmart	.90*	.16	.001*	.39	1.41
	Celtra Duo	-.64	.28	.186	-1.49	.19
	IPS e.max ZirCAD	-9.51*	.40	.000*	-10.78	-8.25
GC Cerasmart	Vita Enamic	-.90*	.16	.001*	-1.41	-.39
	Celtra Duo	-1.55*	.24	.000*	-2.33	-.76
	IPS e.max ZirCAD	-10.42*	.38	.000*	-11.66	-9.18
Celtra Duo	Vita Enamic	.64	.28	.186	-.19	1.49
	GC Cerasmart	1.55*	.24	.000*	.76	2.33
	IPS e.max ZirCAD	-8.87*	.44	.000*	-10.20	-7.53
IPS e.max ZirCAD	Vita Enamic	9.51*	.40	.000*	8.25	10.78
	GC Cerasmart	10.42*	.38	.000*	9.18	11.66
	Celtra Duo	8.87*	.44	.000*	7.53	10.20

Dunnett T3 test * $p<0.05$ statistically significant

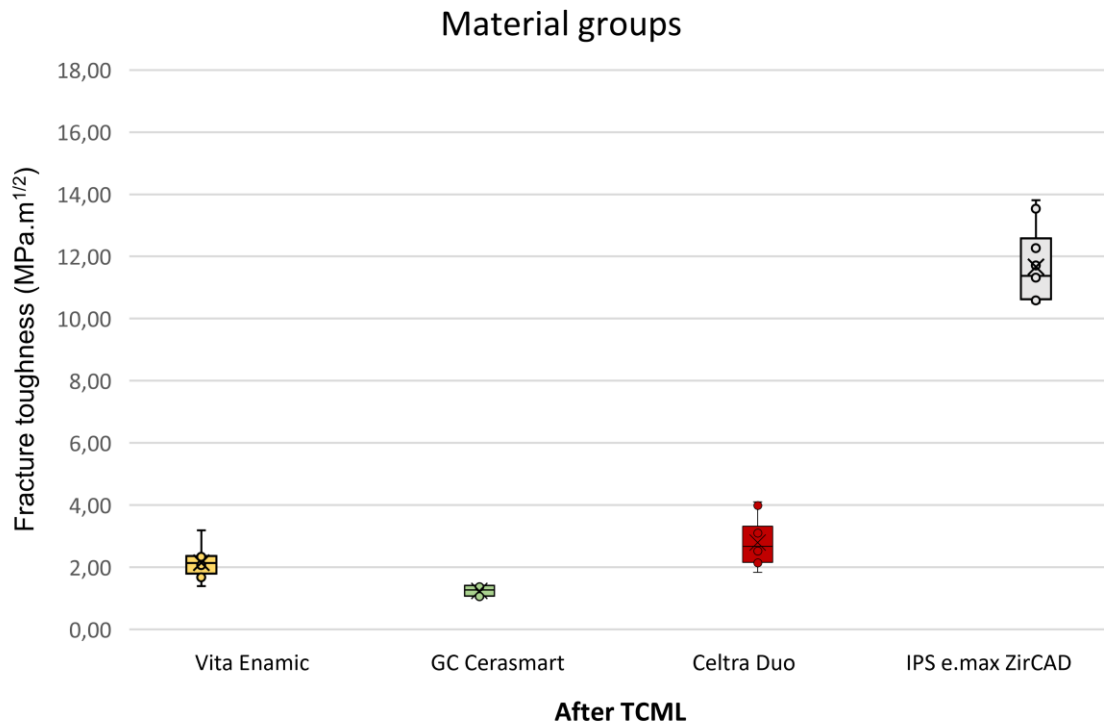


Figure 4.8. Box chart of fracture toughness results of material groups after TCML

While the mean fracture toughness values of the material groups “after TCML” were as following GC Cerasmart ($1.24 \pm 0.16 \text{ MPa.m}^{1/2}$) < Vita Enamic ($2.14 \pm 0.48 \text{ MPa.m}^{1/2}$) < Celtra Duo ($2.79 \pm 0.75 \text{ MPa.m}^{1/2}$) < IPS e.max ZirCAD ($11.66 \pm 1.19 \text{ MPa.m}^{1/2}$) the statistically significant differences were as follows:

The mean fracture toughness value of Vita Enamic after TCML was higher than GC Cerasmart, but lower than IPS e.max ZirCAD ($p=0.001$ and $p=0.000$).

The mean fracture toughness value of GC Cerasmart was lower than mean values of all other material groups (Vita Enamic, Celtra Duo, IPS e.max ZirCAD) ($p=0.001$, $p=0.000$, and $p=0.000$, respectively).

The mean fracture toughness value of IPS e.max ZirCAD was higher than mean values of all other material groups (Vita Enamic, GC Cerasmart, Celtra Duo) ($p=0.000$ for all).

4.5.3. Fracture toughness difference between CAD/CAM restorative material groups before & after TCML

Post-hoc Dunnett's T3 pairwise comparison test was used to determine the material groups which had statistically significant differences of fracture toughness change. ($\alpha=0.05$)

Table 4.15. Multiple comparisons of fracture toughness of CAD/CAM restorative material groups by differences between before & after TCML

(1) Group	(2) Group	$\bar{x}$	Std. Error	p value	95% Confidence Interval	
		Difference (1-2)			Lower Bound	Upper Bound
Vita Enamic	GC Cerasmart	.68*	.21	.049*	.002	1.35
	Celtra Duo	.96	.38	.127	-.18	2.11
	IPS e.max ZirCAD	-1.39	.69	.321	-3.60	.81
GC Cerasmart	Vita Enamic	-.68*	.21	.049*	-1.35	-.002
	Celtra Duo	.28	.33	.941	-.78	1.34
	IPS e.max ZirCAD	-2.07	.67	.064	-4.26	.10
Celtra Duo	Vita Enamic	-.96	.38	.127	-2.11	.18
	GC Cerasmart	-.28	.33	.941	-1.34	.78
	IPS e.max ZirCAD	-2.35*	.74	.040*	-4.62	-.08
IPS e.max ZirCAD	Vita Enamic	1.39	.69	.321	-.81	3.60
	GC Cerasmart	2.07	.67	.064	-.10	4.26
	Celtra Duo	2.35*	.74	.040*	.08	4.62

Dunnett T3 test * $p<0.05$ statistically significant.

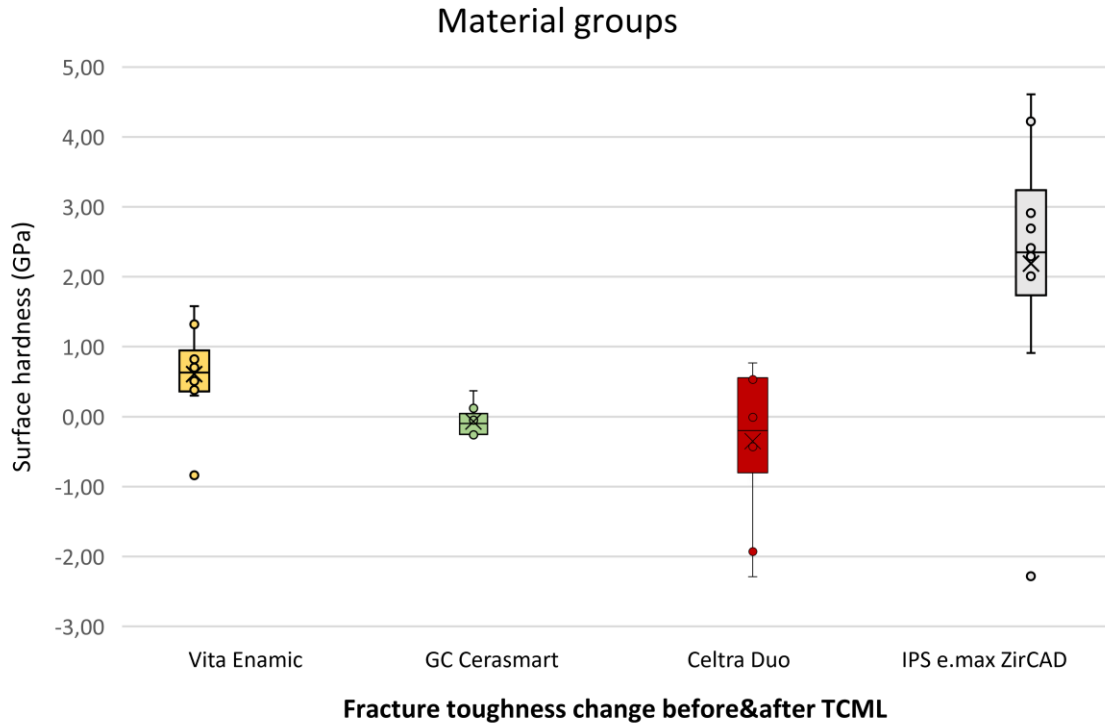


Figure 4.9. Box chart of fracture toughness change due to TCML in material groups

While the mean fracture toughness difference of materials was arranged as GC Cerasmart ($-0,07 \pm 0,20 \text{ MPa.m}^{1/2}$) < Celtra Duo ($-0,35 \pm 1,02 \text{ MPa.m}^{1/2}$) < Vita Enamic ($0,61 \pm 0,64 \text{ MPa.m}^{1/2}$) < IPS e.max ZirCAD ($2,00 \pm 2,11 \text{ MPa.m}^{1/2}$), the statistically significant differences were found as follows:

The mean fracture toughness change value of Vita Enamic was higher than GC Cerasmart ($p=0.049$).

The mean fracture toughness change value of IPS e.max ZirCAD was higher than Celtra Duo ($p=0.040$).

4.5.4. Fracture toughness change of each CAD/CAM restorative material group by differences in time due to TCML

For the data which were not normally distributed, Wilcoxon t test and for the normally distributed data, paired sample t test was used to evaluate differences in time by each group. ($\alpha=0.05$)

Table 4.16. Comparisons of fracture toughness values of every single CAD/CAM restorative material group by differences in time

Group		n	Mean	Std. Deviation	Median	Minimum	Maximum	P value
Vita Enamic	Before TCML	10	2.75	.67	2.86	.99	3.49	.047 ^{a*}
	After TCML	10	2.14	.48	2.14	1.39	3.19	
GC Cerasmart	Before TCML	10	1.17	.14	1.14	.99	1.39	.297 ^b
	After TCML	10	1.24	.16	1.27	1.01	1.43	
Celtra Duo	Before TCML	10	2.44	.40	2.37	1.82	3.09	.307 ^b
	After TCML	10	2.79	.75	2.67	1.84	4.11	
IPS e.max ZirCAD	Before TCML	10	13.67	1.25	13.52	11.26	15.25	.015 ^{b*}
	After TCML	10	11.66	1.19	11.37	10.51	13.81	

^aWilcoxon test ^bPaired samples t test * $p<0.05$ statistically significant

Vita Enamic and IPS e.max ZirCAD were the only groups showing change with differences in time, the results were statistically significant. ($p=0.047$ and $p=0.015$)

In Vita Enamic (2.75 ± 0.67 / 2.14 ± 0.48 MPa.m^{1/2}) and IPS e.max ZirCAD [13.52 MPa.m^{1/2} ($11.26-15.25$ MPa.m^{1/2}) / 11.37 MPa.m^{1/2} ($10.51-13.81$ MPa.m^{1/2})] groups the fracture toughness values before TCML were higher than after TCML. The fracture toughness of Vita Enamic and IPS e.max ZirCAD material groups had decreased due to TCML process.

4.6. Wear mechanism and surface analyses of CAD/CAM restorative material and cusp/material groups

The worn surfaces of cusps and material samples were analysed using scanning electron microscope to determine wear surfaces and wear patterns (**Figure 4.10-4.13**).

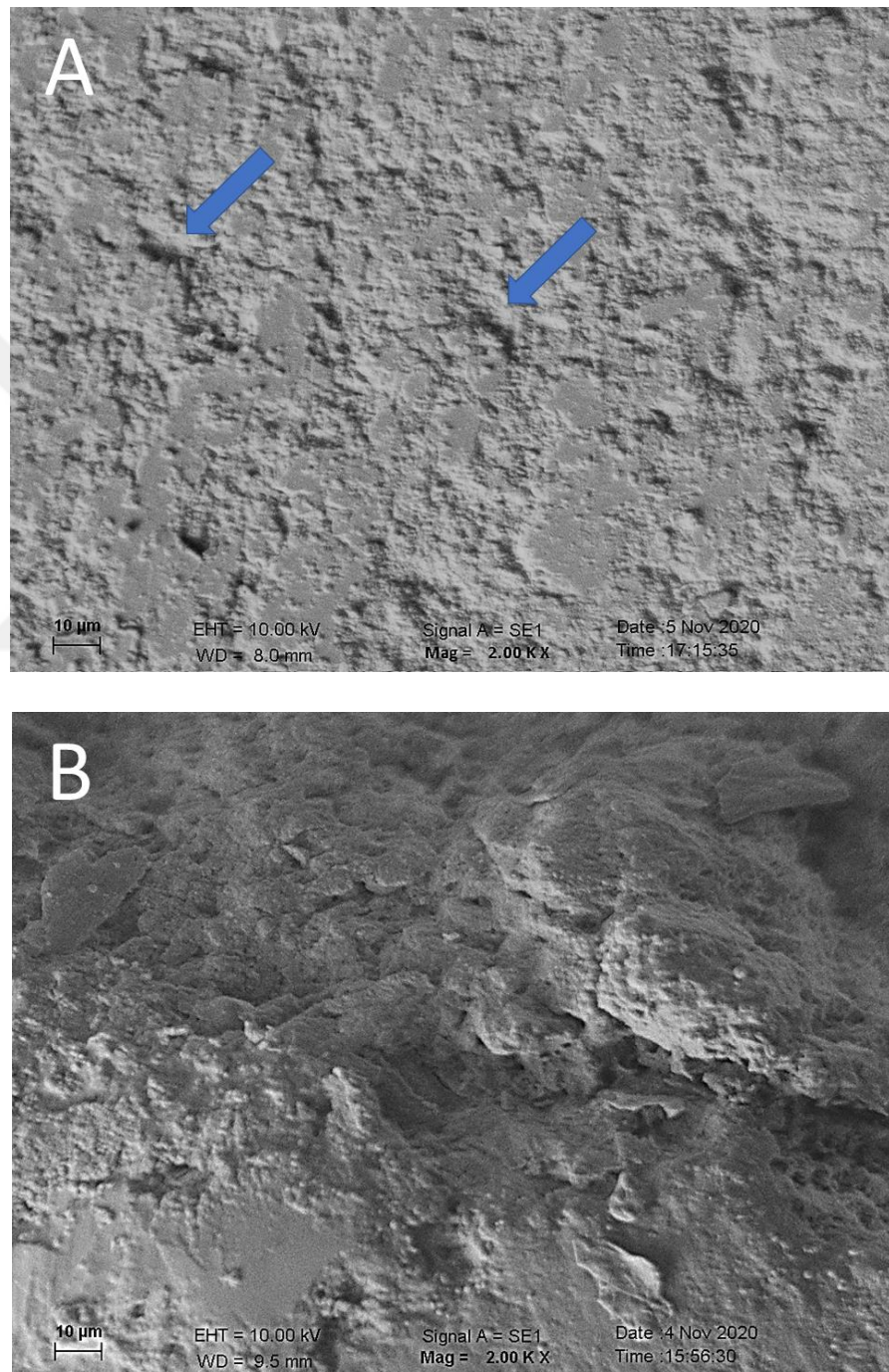


Figure 4.10. Scanning electron microscope images. A, Vita Enamic surface (original magnification $\times 2000$). B, Cusp/Vita Enamic surface (original magnification $\times 2000$)

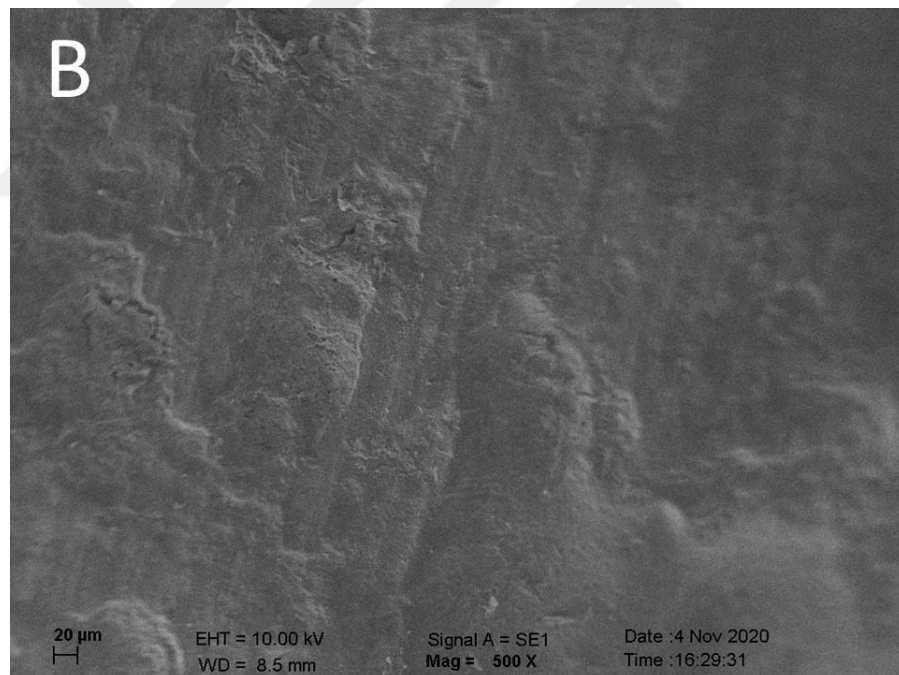
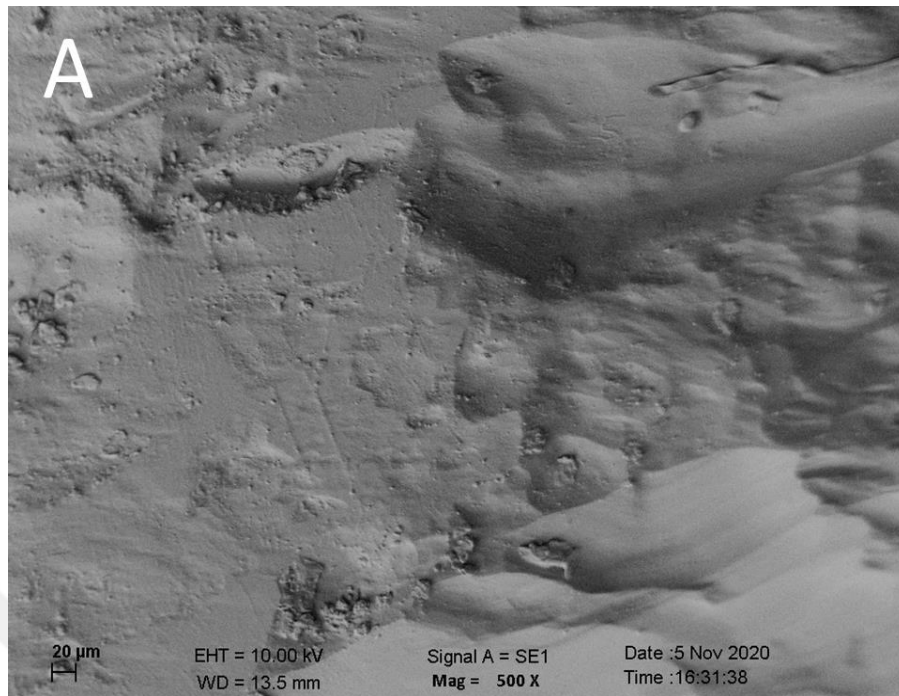


Figure 4.11. Scanning electron microscope images. A, GC Cerasmart surface (original magnification $\times 500$). B, Cusp/GC Cerasmart surface (original magnification $\times 500$)

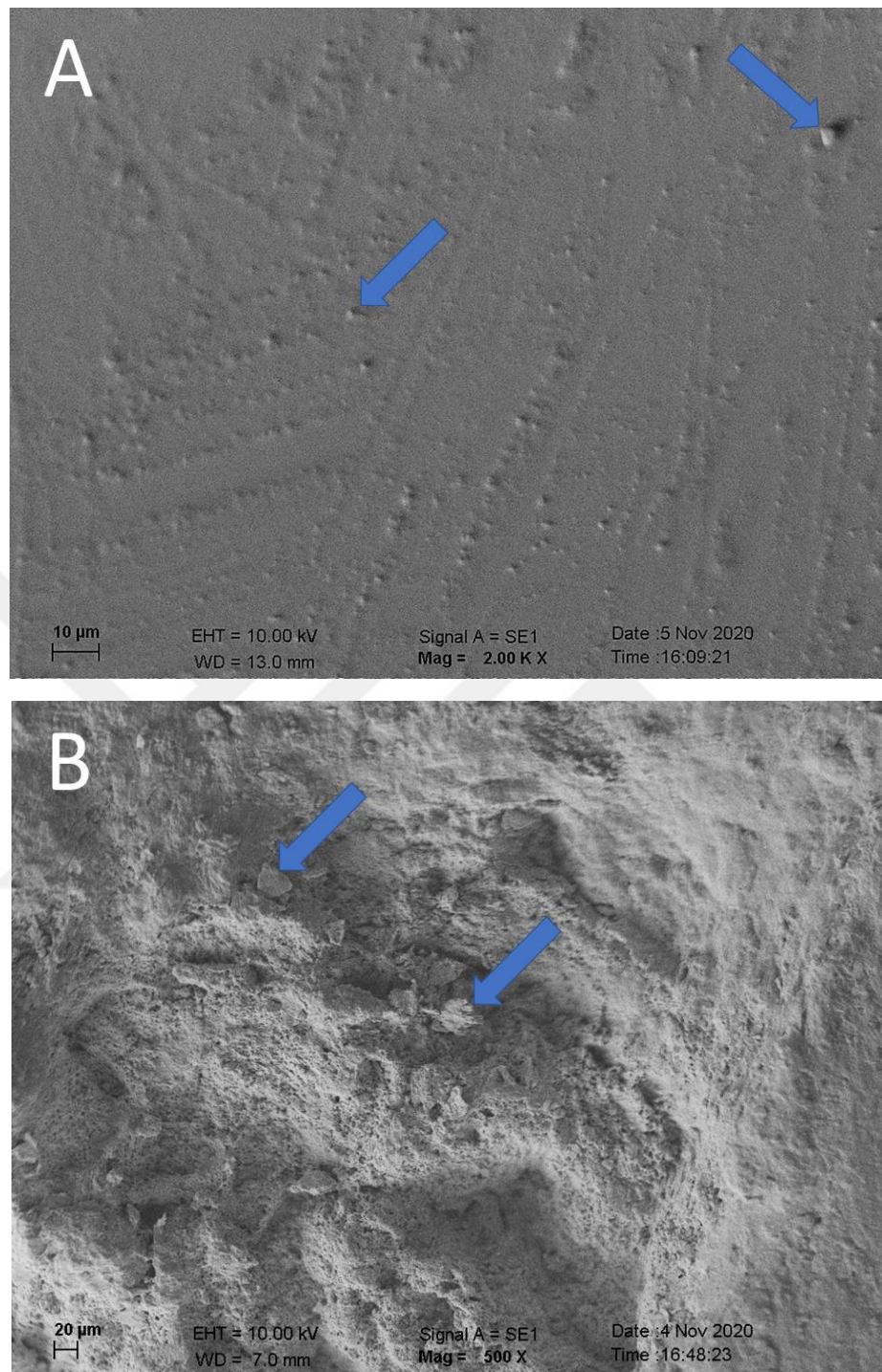


Figure 4.12. Scanning electron microscope images. A, Celtra Duo surface (original magnification $\times 2000$) B, Cusp/ Celtra Duo surface (original magnification $\times 500$)

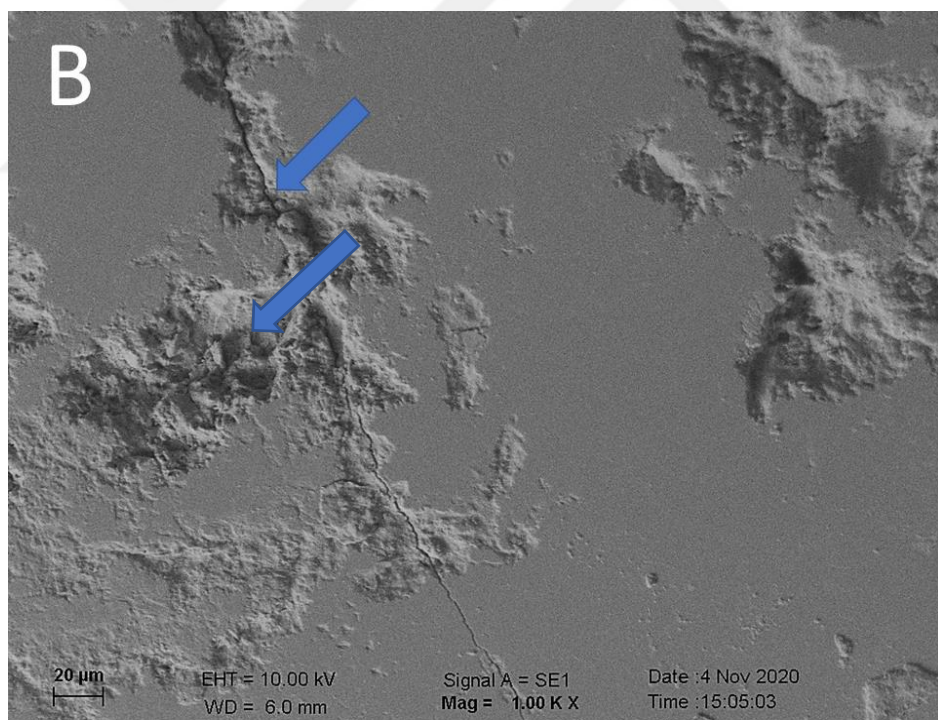
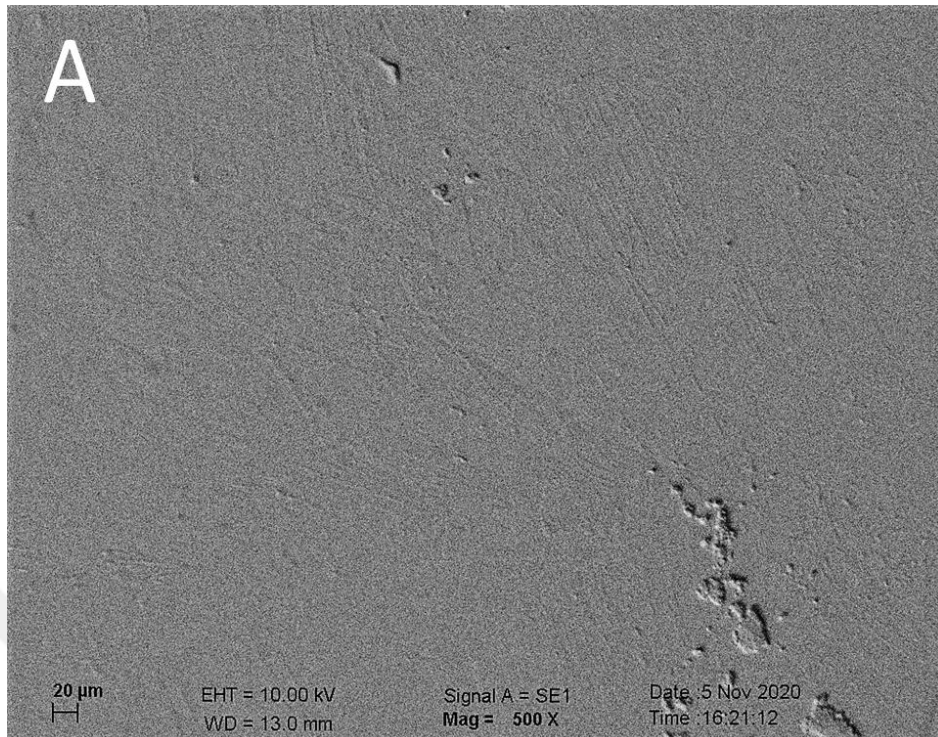


Figure 4.13. Scanning electron microscope images. A, IPS e.max ZirCAD surface (original magnification $\times 500$) B, Cusp/IPS e.max ZirCAD (original magnification $\times 1000$)

5. DISCUSSION

Regarding the mechanisms and controlling factors of wear, patient-related factors such as parafunctional habits, masseter muscle thickness, dentition status, age, gender, and material-related factors such as microstructural, physical, and mechanical properties have a critical importance.^{12–16}

Bruxism, a parafunctional activity, may contribute to excessive wear. Lambrechts et al.¹¹⁷ reported that the annual wear rate of the molar enamel in non-bruxists is very low as 20-40 μm compared to bruxist individuals. Xhonga,²²⁸ claimed that bruxist patients may experience wear rates as high as 220-243 μm . Gibbs et al.¹⁸⁶ compared the bite strength of some bruxist patients and reported that it was up to six times that of non-bruxists. Heintze et al.¹⁹ investigated the accelerator effect of high bite forces on tooth wear as a result of bruxism. Maximum bite force (MBF), tooth wear and bruxism were related. However, MBF was only assessed with force transducers placed between the patient's opposing teeth, usually on a single tooth, and maximum voluntary clenching (MVC) was measured with dynamometers. In addition to MBF, chewing force plays a significant role, especially in individuals with sleep bruxism (SB) due to muscle strengthening.^{19,20} While physiological chewing forces have a range of 10-120 N, parafunctional forces are approximately ten times greater at 200-800 N and can reach up to 1000 N.^{13,192}

Chewing simulators are most commonly used in-vitro three-body wear machines that better mimic the physiological conditions of chewing such as force, characteristics of chewing movement, and lubrication.^{10,197} Selection of loading force is an essential component of the wear testing method. Gibbs et al.¹⁸⁶ reported that, a load of 50 N can be considered the mean of the physiological occlusal forces in non-bruxist patients. According to Lutz et al.,²⁰⁰ higher forces/loads such as 75 N and 100 N, lead to higher wear rates in an in-vitro wear simulation. In this study tests were also performed using a chewing simulator with a load of 120 N to simulate the higher daytime chewing forces of patients and aim to achieve an evaluation of wear performance in a given time. 120 N was the maximum capacity of the chewing simulator and the upper limit of physiological forces. While planning a restoration, it is very important to choose the right material, especially for patients with high chewing forces. Therefore, it is necessary to evaluate the

effect of high chewing forces on wear characteristics of restorative materials and their enamel antagonists.

Enamel should be the best reference material to compare the results of a wear test. The type and shape of the enamel antagonist as well as the thickness of the cusp are considered to be possible wear influencing factors. Regarding the shape of the enamel antagonist, flat enamel specimens are not suitable for in vitro wear tests because they show high wear, which does not allow comparison of results with in-vivo situations. When the preference of the antagonist sample is enamel, extracted human teeth were usually used in studies. Most of these teeth were from human molars, as enamel thickness increases from incisors to third molars. The goal was to direct all of the wear that occurs within the enamel and not reach the underlying dentin. In terms of the type of antagonist, standardized enamel antagonists (in the same shape as the ceramic) or non-standardized enamel antagonists (usually palatal cusps of upper molars) can be used in in-vitro wear studies.¹⁹⁷ Therefore, this influenced the methodological design of the present study that the enamel antagonist was not used by a combination of flat-surface enamel. Human permanent, healthy third molars were used as antagonists against flat-surface material specimens to mimic the cusp-fossa relationship.

In terms of restorative materials, Heintze et al.¹⁹ reported that the vertical wear of flat-surface material specimens was significantly higher than that of crown specimens in most experimental set-ups indicating posterior restorations for molar teeth. As a possible explanation for this, they added that flat-surface specimens have a higher stress distribution than anatomically shaped specimens such as crowns or inlays, resulting in higher material wear for flat-specimens. However, this may vary depending on the different parameters and methods of in-vitro studies. For example, in an in-vitro wear study using flat-surface Vita Enamic specimens in a chewing simulator with 200.000 chewing cycles and 49 N of load, the material had a wear loss of $61.90 \pm 39.54 \mu\text{m}$.²²⁹ However, in another in-vitro wear study using Vita Enamic crowns, the wear volume loss was $135.58 \pm 24.07 \mu\text{m}$ at 1.200.000 chewing cycles and 50 N load.²³⁰ Although the applied loads are almost the same, the higher wear amount of the crown specimens in the second study might be related to the use of higher chewing cycles to mimic the long-term behaviour of the material. In this study, the volumetric wear (mm^3) of flat-surface materials under 1.200.000 chewing cycles and increased loads of 120 N was evaluated in three dimensions. In this way, the aim was to have 3D accurate results, to provide a

balanced strain distribution in the material, and to exclude possible rotation of the material or its antagonists in the supports of chewing simulator.

Krejci et al.¹⁵¹ concluded that the surface finish of the restorative material determines the rate of enamel wear. Many previous studies have reported that a ceramic with polished surface results in less enamel wear than a glazed one.^{143,151,156–158} It has been pointed out that a mere polish is acceptable after chairside adjustment.⁶² Heintze et al.¹⁹ claimed that in glazed ceramics, the surface under this glaze layer, which is not polished, may exhibit some surface roughness. The glaze layer has a thickness of only about 30–50 μm and the antagonist can easily wear it away. When the antagonist hits the rough surface of the ceramic layer, it can lead to increased amounts of enamel wear. Lower enamel wear is expected on polished material surfaces due to the lower friction coefficient. Since it is expected that the wear behaviour of the materials will be barely observed, it was not considered necessary to glaze the specimens. Moreover, the use of a glazing material is avoided from in order to eliminate the aforementioned enhancing effect on wear, since the aim of this study was to evaluate the effect of increased chewing forces. Moreover, it is not easy to achieve smooth surfaces with glaze as the methodology of this study involves flat-surface materials.

Regarding the completion of sample preparation, zirconia-reinforced lithium silicate (Celtra Duo) materials were subjected to a crystallization firing. In this firing process, zirconia grains act as crystallization nuclei and produce larger numbers of smaller crystallites (0.5-1 μm) instead of the fewer large crystallites (1.5 μm) present in conventional lithium disilicate ceramics that prevent crack propagation. The manufacturer of Celtra Duo claims that crystallization firing is not necessary, it can be polished and adhesively cemented immediately after grinding, gaining reduced working time compared to lithium disilicate and making it particularly suitable for the chairside fabrication. However, they also suggest that crystallization firing, although not mandatory, could be helpful in optimizing aesthetics and increasing the flexural strength of Celtra Duo.²³¹ In addition, a study in which the material was subjected to a firing cycle and then polished or just polished immediately after milling reported that a firing cycle can lead to favourable results in the abrasion resistance of zirconia ceramic balls similar to enamel and gold alloy. While the milled Celtra Duo showed a loss of $0.542 \pm 0.115 \text{ mm}^3$, the fired Celtra Duo showed a volume loss of $0.384 \pm 0.176 \text{ mm}^3$, which is close to enamel with $0.393 \pm 0.178 \text{ mm}^3$ and gold alloy with $0.331 \pm 0.138 \text{ mm}^3$ wear.¹³⁴ In a

study by Cook et al.,²³² polishing was shown to improve the strength after surface damage of glass-ceramics by removing the zone of deformation surrounding the surface defects. In accordance with the results of these studies and the recommendations of the manufacturers, samples from Celtra Duo were subjected to a subsequent firing process at 810 °C and then polished with silicon carbide sandpapers to grit sizes of 600, 800, and 1200, respectively.

In-vitro assessment of wear requires scanning of the sample surfaces, quantification of wear and statistical analysis of the wear measurements. Since the modern scanning equipment allows direct imaging of the sample surface, wear can now be identified three-dimensionally as volume loss rather than estimated one-dimensionally as depth or two-dimensionally as area.⁴¹ Herewith, 3D quantification of wear should specifically reflect volumetric loss.¹²⁹ Many previous studies have focused on the wear volume loss of materials after predefined wear cycles by simulating the chewing cycles of a human to estimate and evaluate wear resistance.^{134–136} 3D scanning is the preferred method for wear measurements because it is quantitative, accurate, provides storable 3D databases, and it is applicable in both clinical and the laboratory settings.²¹⁸ Therefore, in the present study, the wear volume loss of cusps and materials was estimated automatically using a 3D laser scanner. In addition to quantifying wear by evaluating the loss of substance, it is particularly important to qualify and describe the sample surface to investigate the surface properties that determine how future loss may progress. It is fundamental to visually image surfaces using microscopic techniques such as scanning electron microscopy, which is used in this study.²²¹

The results obtained showed that IPS e.max ZirCAD, a tetragonal polycrystalline zirconia, partially stabilized with 3% mol yttria (3Y-TZP) (88-95.5 % ZrO₂), had the lowest volumetric loss among the material groups (Vita Enamic, GC Cerasmart, and Celtra Duo). Vardhaman et al. confirmed that the composition and microstructure of first-generation zirconia (3Y-TZP) were introduced to optimize strength and toughness. The pull out of grains was the main mechanism of wear under mild wear conditions as no microcracks and fractures were identified in zirconia.²³³ Conrad et al.⁷¹ claimed that the yttria additions may have allowed to delay crack propagation in the crystal structure as the crack-free zirconia surface did not show fragmentation or chipping. The average grain size is an important parameter for the wear intensity of Y-TZP ceramics, which determines the surface topography and the tribological behaviour.^{160,234} He et al.²³⁵

reported that Y-TZP exhibits wear resistance at grain sizes of 0.7 μm or smaller by increasing the energy required to remove the grain from the matrix of the ceramic. The determined average grain size of the IPS e.max ZirCAD used in this study was 0.5 μm , with a homogenous distribution of crystal sizes and orientations. This polycrystalline structure could prevent the propagation of microcracks and provide low material loss. In an in-vitro wear study simulating 360.000 chewing cycles and 49 N of load, 5 Y-TZP flat-surface zirconia samples exhibited no measurable wear.¹⁰ In another in-vitro wear test, in which 3Y-TZP flat-surface zirconia samples were polished and subjected to 240.000 chewing cycles and 49 N load, showed $0.031 \pm 0.033 \text{ mm}^3$ wear volume loss.¹⁴⁹ In another study, the volume loss of crown-shaped 3 Y-TZP zirconia specimens under 100.000 chewing cycles and 50 N of load was $0.037 \pm 0.838 \text{ mm}^3$.²³⁶ In the present study, the wear volume loss of zirconia was lower at $0.024 \pm 0.012 \text{ mm}^3$. The microstructure of zirconia may have prevented substance loss despite the selected load of 120 N and 1.200.000 chewing cycles. Most studies have evaluated the wear of various restorative materials against enamel antagonists. Lohbauer and Reich did not find statistically significant differences between the enamel antagonist and zirconia restorations in terms of mean wear volume loss and maximum vertical loss.²³⁷ The results of a study of Mitov et al.,¹⁶⁰ clearly show the favourable wear behaviour of polished zirconia against natural human enamel. The YTZ ceramic showed extremely high wear resistance against enamel and no zirconia wear was detected after wear simulation. According to some in-vitro studies, zirconia causes less wear of enamel antagonist compared to enamel and metal-ceramics.^{136,238} Many studies have also reported that the wear of enamel opposed by zirconia is much lower than other traditional dental ceramics.^{135,149,239} Esquivel-Upshaw et al.²⁴⁰ have claimed that the wear of enamel antagonized by zirconia was lower than that of metal-ceramics and enamel after 6 months but higher after 12 months, with no statistically significant results. In accordance with the methodology of this study, the restorative materials were also opposed by enamel antagonists. Even though a 5-year chewing simulation was performed, the results obtained showed that cusp/IPS e.max ZirCAD exhibited a wear volume loss of 0.22 mm^3 ($0.15\text{-}0.49 \text{ mm}^3$) without statistical significance. However, it was possible to observe changes in the surface characteristics of cusp/IPS e.max ZirCAD. In SEM observations, cusp/IPS e.max ZirCAD showed a flattened surface on the contact area; however, it had obvious crack lines and showed clusters of small chipping areas of enamel pieces (**arrows in Figure 4.13 B**). While the zirconia appears to cause little volume loss of enamel, it may cause structural defects that

can lead to problems in the long-term use. These changes and long-term behavioural differences of enamel could occur due to the physical properties of zirconia, as its high surface hardness and fracture toughness can scratch out the enamel antagonist. Zirconia has tightly bound grains within its structure, which makes it more resistant to surface degradation; it also presents a difficulty in chairside adjustment and polishing in the clinical setting. In addition, IPS e.max ZirCAD exhibited a completely different wear mechanism than Vita Enamic, GC Cerasmart and Celtra Duo. It exhibited smooth scratches that extended along the entire length of the antagonist contact site and across the slide path of the antagonist tooth cusp, without any fractures or microcracks. After all this research, it is fair to say that it has unique wear characteristics that may be due to its intrinsic properties. Furthermore, with the increasing popularity, clinical use, and reinforced structures of CAD/CAM materials, it is considered necessary to conduct wear comparisons between these new generation materials and their enamel antagonists rather than comparing them with traditional dental ceramics.

Several material-related factors have been claimed to influence wear behaviour of the material, such as size and shape of grains, the presence of glassy, crystalline, and/or reinforcing crystalline phases and the composition of the ceramic.^{147–149} In general, with the exception of zirconia, materials contain grains bound by a relatively weak glassy or polymeric matrix. Deformation of these bonding phases is associated with interfacial microcracks that facilitate detachment and dislocation of the crystals. In lithium disilicate materials, the propagation of microcracks begins along weak crystal-glass interfaces, a weakness responsible for the machinability of this material and the resulting propagation of ejected debris due to wear.¹⁵⁰ However, zirconia-reinforced lithium silicate (ZLS) glass-ceramics have been reported to have a relatively homogeneous fine crystalline microstructure, unlike other lithium disilicate glass-ceramics with the characteristic needle-shaped and fine-grained crystals. According to this study, the homogeneous fine structure brings a positive effect on the flexural strength.⁷⁹ Also, in Celtra Duo the zirconia particles are incorporated to reinforce the structure of lithium silicate ceramic.⁷⁹ The submicron-sized zirconia grains in the glassy matrix improve the mechanical properties by increasing the flexural strength and fracture toughness compared to conventional lithium disilicate ceramics.⁷⁶ In- an in-vitro wear study, fired or polished Celtra Duo and enamel specimens with Y-TZP zirconia cusp antagonists were subjected to 120.000 chewing cycles and 49 N of load. While the fired and polished Celtra Duo

showed a wear depth of 0.278 ± 0.061 mm, the Y-TZP zirconia antagonist worn 0.005 ± 0.04 mm, and while the enamel showed a wear depth of 0.217 ± 0.095 mm, the Y-TZP zirconia antagonist worn 0.004 ± 0.003 mm.¹³⁴ Zirconia appears to exhibit a stable character with low wear values, but this time as an antagonist. Enamel opposed by zirconia cusps showed low wear loss than Celtra Duo against zirconia cusps. It can be concluded that if it is planned to use Celtra Duo for a restoration, it would be better to antagonize it with zirconia than with enamel. In another wear study with 400.000 chewing cycles and 20 N load, the fired Celtra Duo samples showed 0.220 ± 0.05 mm³, while the enamel antagonists showed 0.276 ± 0.007 mm³ wear volume loss.²⁴¹ In the present study, the fired Celtra Duo samples showed 0.69 ± 0.29 mm³ volume loss as the material that came after zirconia among the material groups. However, the results were not statistically significant as the volume loss value of Celtra Duo was close to Vita Enamic and GC Cerasmart, and far from IPS e.max ZirCAD. In contrast to these studies, it appears in the present study that Celtra Duo worn more than its enamel antagonist. However, no direct correlation between cusp and material groups of Celtra Duo could be established, as it leads to fractures in the tooth due to its abrasive effect and high forces. Thus, the material and cusp wear evaluated separately. Regarding the wear of cusp/Celtra Duo, it showed the highest wear volume loss 0.59 mm³ (0.50-1.63 mm³). According to some studies, the wear behaviour of enamel antagonist of ZLS-based ceramics did not differ from what was reported previously with the other lithium disilicate materials.^{148,242} Rosentritt et al.²⁴³ reported in their study that lithium silicate ceramics were more abrasive to opposing steatite compared to zirconia. This might be related to the wear mechanism of Celtra Duo and enamel. In the observations of SEM, Celtra Duo showed delamination of the surface as zirconia particles seemed to adhere half and half cling to the surface of the material **(arrows in Figure 4.12 A)**. Furthermore, as a probable result of the abrasive character and dislocation of ejected zirconia crystals, there were embedded zirconia particles in the worn surface of cusp/Celtra Duo, which were represented as accumulated nanometric irregular particles **(arrows in Figure 4.12 B)**. The higher values for wear volume loss of cusp/Celtra Duo could have been caused by the release of zirconia crystals from Celtra Duo due to wear. Composite materials such as ZLS-based and resin matrix ceramic materials (PICN), could have an abrasive character on enamel due to than zirconia with its uniform polycrystalline structure due to their microstructural loss during wear.

GC Cerasmart and Vita Enamic are included in the same category, resin-matrix hybrid ceramics. Leinfelder²⁴⁴ assumed that hybrid ceramics exhibit tooth-like wear and have similar deformation capacity. According to the findings of Mendonca et al.²⁴⁵, both nanoceramic and PICN groups showed lower elastic modulus and hardness values compared to lithium disilicate and ZLS-based ceramics. Moreover, the lower hardness of the hybrid materials allows them to prevent wear of the antagonist natural teeth. However, their structural differences lead to different wear values and different effects of the two resin matrix hybrid ceramic groups on enamel. By imaging with SEM, microcracks in Vita Enamic were visible on the entire surface of the sample. Closer examination of the wear cracks revealed residual spaces left over from breaking out pieces of polymer from the resin matrix (**arrows in Figure 4.10 A**). Also, when cusp/Vita Enamic is observed, a roughened and irregular surface with broken out pieces of enamel was visible. This could be caused by erupting polymer pieces of the material sample, which have an abrasive character. Aladag et al.²⁴⁶ reported that the most abrasive materials for enamel antagonist were resin-matrix ceramic groups such as Vita Enamic and GC Cerasmart rather than glass-matrix ceramic groups, and they have added that this could be the result of earlier abrasion of the polymer parts of these materials, while their ceramic components were not. The exposed ceramic network components could have stronger abrasive effect on enamel. It was expected that wear tracks of GC Cerasmart show similar wear patterns to Vita Enamic. However, closer inspection revealed large wear tracks in the area where the enamel antagonist encountered the surface of the specimen and slide against it due to the collapse of nanoceramic particles at the tip of the wear track that were pulled across the resin matrix. These accumulation areas could have consisted of enamel particles originating from antagonist tooth cusp. This is because the images from the cusp/GC Cerasmart showed the presence of flattened depressions running the length of the wear track with parallel fine scratches, which could be explained by tooth substance loss from the cusp. Most studies have evaluated the wear of these hybrid materials and their enamel antagonists. According to an in-vitro wear study using Vita Enamic samples in a chewing simulator with 400.000 chewing cycles and 50 N load, the material showed extremely lower values of volume loss ($0.25 \pm 0.15 \text{ mm}^3$) compared to the present study.⁴¹ In another in-vitro wear study with 360.000 chewing cycles and 49 N load, Vita Enamic samples showed $\sim 0.24 \text{ mm}^3$ and the cusp/Vita Enamic samples showed $\sim 0.09 \text{ mm}^3$ volume loss.¹⁰ Also, in a wear study with parameters of 400.000 chewing cycles and 20 N load, the Vita Enamic samples presented $0.21 \pm 0.06 \text{ mm}^3$ loss, while the enamel antagonist cusp/Vita

Enamic showed 0.28 ± 0.07 wear volume loss.²⁴¹ GC Cerasmart samples presented 0.18 ± 0.07 mm³ loss, while the enamel antagonist cusp/GC Cerasmart showed 0.056 ± 0.03 wear volume loss.²⁴¹ From these studies, generally it can be concluded that enamel antagonists of GC Cerasmart and Vita Enamic worn less than themselves, especially the resin nanoceramic, GC Cerasmart causes less opposing enamel wear than the polymer infiltrated ceramic (PICN). Also in the present study, cusp/Vita Enamic and cusp/GC Cerasmart worn less than their enamel antagonists. While the Vita Enamic showed 0.74 ± 0.28 mm³ wear volume loss, cusp/Vita Enamic showed 0.59 mm³ (0.50-1.63 mm³) wear; and while the GC Cerasmart showed 0.82 ± 0.52 mm³ wear, cusp/GC Cerasmart showed 0.17 mm³ (0.041-0.41 mm³) wear volume loss. However, no direct correlation between cusp and material groups could be established as some of the materials may have been fractured due to high loads and greater chewing cycles. Thus, material and cusp wear were evaluated separately. The only statistically significant result belonged to the cusp/GC Cerasmart between these resin-matrix ceramics and their antagonists. Since nanoceramic materials can absorb energy within the bonding phase, could be the reason for the lower wear volume loss of the cusp/GC Cerasmart.

In the case of enamel with a high degree of orientation of long hydroxyapatite rods near the enamel surface, the sliding of protein sheaths between these rods is more difficult as a dislodgment effect.¹²⁴ Zheng and Zou¹²⁵ studied enamel wear based on the observed topography of wear surfaces and reported that severe delamination was considered the dominant mechanism of two-body wear. In three-body wear due to increasing loads, significant scratches accompanied by small delamination pits were dominant. In addition, Zheng et al.⁵ reported that the enamel exhibited a surface damage by plastic deformation in the early stage of wear, and iridescent rings appeared around the impact side as a result of the accumulation and propagation of plastic deformation. In addition, the wear behaviour of enamel did not show subsurface cracks and exhibited metal-like properties to a certain extent. However, the surface characteristics of enamel against wear depend on various factors related to the antagonist material. In this study, cusp/Vita Enamic and cusp/Celtra Duo exhibited subsurface microcracks without exhibiting delamination. In addition, cusp/IPS e.max ZirCAD showed small chipping areas on a flattened surface, possibly a residual delamination. The wear mechanism cusp/IPS e.max ZirCAD showed remarkable small microcrack areas. The cusp/GC Cerasmart showed a total wear mechanism of delamination with parallel scratches on its surface, as no microcrack areas

were seen either. These results were consistent with the general wear mechanism of enamel due to three-body wear.

In the above-mentioned studies on the wear of the CAD/CAM restorative materials included in this study and their enamel antagonists, the wear parameters were approximately 200.000-400.000 chewing cycles and 49-50 N of loads. However, encountering with different wear values in this study might have been caused by the simulation of patient-related factors, such as high chewing forces with a load of 120 N and long-term use of restorations by simulating 1.200.000 chewing cycles, which corresponds to a clinical service of 5 years. According to Fontijn-Tekamp et al.²⁴⁷, higher loads than seen in routine chewing, such as 100-150 N, lead to the exploration of thresholds for failure modes of test materials that might occur at high occlusal loads. In addition, in this study Celtra Duo might have led to fractures of some cusp specimens, and cusp specimens might lead to fractures of the Vita Enamic and GC Cerasmart materials due to the simulation of high chewing forces. Considering the material-related factors, possible changes in the physical properties of restorative materials due to thermocycling, increased mechanical loading, and long-term chewing simulation might be the reason of these results.

In vitro studies performed in the laboratory cannot accurately simulate oral cavity conditions related to clinical environment, humidity, and stresses on teeth and restorations due to temperature fluctuations. In addition to evaluating the mechanical aging of various ceramic materials with the loads applied by chewing simulators in an in-vitro study, the evaluation of thermal aging is also important. Simulation of the oral cavity environment just by thermal aging of teeth and/or restorations is possible to some extent.²⁸ It is better to use various aging procedures together, including thermocycling and mechanical loading (TCML), to reproduce the intraoral conditions.²⁴⁸ These aging procedures, which affect wear generally in a material-dependent manner, could account for possible differences in physical properties of materials such as surface hardness and fracture toughness. Thermocycling (TC) may reduce the wear rates of some materials while increasing the wear rates of others, and for still others, wear is not affected by TC.²⁴⁹⁻²⁵¹ However, these studies have only examined the effects of TC on some specific materials. A few studies have examined the changes in mechanical properties and surface characteristics of recently introduced CAD/CAM restorative materials before and after TCML.^{10,41,248,252} In the present study the TCML process was performed to evaluate the

physical properties of CAD/CAM restorative materials, wear of themselves and enamel antagonists. Rosentritt et al.^{30,210} and Henriques et al.³⁴ reported that 1.200.000 chewing cycles and 50 N of mechanical loading, along with 6000 thermocycles, approximates 5 years of clinical service and provides an acceptable estimation of the survival rate. Regarding the thermocycling, most studies used their own procedures and showed only some consistency within the temperature parameter (5-55 °C) and a great deal of variability in the number of cycles and dwell time chosen.³⁸ There are still no reports found on the number of thermal cycles per unit time in-vivo. Furthermore, in this study the thermocycling process was conducted by the chewing simulator as it refers to a TCML study. With the increased chewing forces of 120 N accompanying 1.200.000 chewing cycles mimicking the 5-year clinic, 3189 thermal cycles with a dwell time of 2 min. could be applied as allowed by chewing simulator.

Conventionally, it was assumed that higher hardness leads to more wear of enamel antagonist.^{129,149} In the 1970s and 1980s, it was assumed that the surface hardness of the ceramic material determines the enamel wear.²⁵³ However, studies in the 1990s concluded that the surface hardness is not a predictor of enamel antagonist wear.^{152,254} In 2008, Heintze et al.¹⁹ also confirmed this uncorrelation between surface hardness and the wear of enamel antagonist in their study. It was obvious that the definition of a strict correlation between hardness and wear was inappropriate for brittle materials, as they wear by subsurface fractures and not by plastic deformation, unlike metals and enamel.¹² In contrast, Mörmann et al.²⁵⁵ claimed that the ceramic or polymer content could have influenced the wear properties as well as mechanical properties. In their TCML study various materials including CAD/CAM restorative materials and enamel antagonists were exposed to 1.200.000 chewing cycles, mechanical loading (ML) of 49 N load, and to TC with 5-55 °C temperature ranges with 3000 thermal cycles. Vita Enamic and Lava Ultimate (with a similar microstructure to GC Cerasmart) showed the lowest Martens hardness values among material groups, and the hardness of the Vita Enamic was not significantly higher than that of Lava Ultimate. They reported that the decrease in hardness could cause an increase in enamel wear, except zirconium dioxide ceramic. As zirconium dioxide ceramic was the hardest material in their study, it showed no material wear and low wear of enamel antagonist. In the present study, also the Vita Enamic and GC Cerasmart had close and lowest Vickers hardness values. According to results after TCML, GC Cerasmart ranked first with the lowest hardness value of 1.46 GPa. Vita

Enamic was the second material in the order with a hardness of 2.21 GPa. In contrast, these lower hardness values did not result in an increase of enamel wear, unlike the cusp/GC Cerasmart showed the lowest wear. However, in the study of Mörmann the Martens hardness was evaluated rather than Vickers hardness. Also, the materials and cusp antagonists were evaluated reciprocally not separately as in our study. In terms of restorative materials, Lawson et al.²⁴¹ observed that lithium disilicate (IPS e.max CAD) and the zirconia-reinforced lithium silicate materials (Celtra Duo) were generally stronger, stiffer, and harder than polymer-infiltrated ceramic (Vita Enamic) and resin nanoceramic (Lava Ultimate). The result of the current study is consistent with the study of Lawson et al. that Celtra Duo (6.34 GPa) was initially harder than GC Cerasmart (1.55 GPa) and after TCML Celtra Duo (6.26 GPa) was harder than both Vita Enamic (2.21 GPa) and GC Cerasmart (1.46 GPa), while Vita Enamic was intermediate with significant differences between all these materials. After TCML, the order of materials was from greater to lesser IPS e.max ZirCAD (12.55 GPa) > Celtra Duo (6.26 GPa) > Vita Enamic (2.21 GPa) > GC Cerasmart (1.46 GPa). IPS e.max ZirCAD was harder than all other material groups with a hardness of 12.55 GPa. Materials with ceramic network structures, such as Vita Enamic, have been reported to be less affected by aqueous conditions and thermocycling by some studies.^{107,256} In contrast, according to a TCML study, the polymer-based materials were more affected by thermocycling that may relate with the aqueous conditions which could have damaged the resin structure.²⁴⁸ In accordance, Niem et al.²⁵⁷ reported that most hybrid and polymer-based CAD/CAM materials, with few exceptions, were found to be highly susceptible to physical degradations after TC compared to ceramics. Also, in this study the PICN material, Vita Enamic showed a significant decrease of 1.41 GPa in hardness after aging by showing a greater hardness change than GC Cerasmart (0.10 GPa) and Celtra Duo (-0.01 GPa). In addition, Celtra Duo (-0.01 GPa) showed an extremely lower hardness change than IPS e.max ZirCAD (1.64 GPa). Since in the present study, mechanical loading was carried out simultaneously with thermocycling, the results were different than those studies by different aging process of the specimens. It was expected to overcome with the greatest change in GC Cerasmart in accordance with its microstructure. However, beside the thermal and mechanical aging, factors such as the grain size and shape, the type of stabilizing elements, the size and distribution of internal flaws, and the presence of preloads may influence the clinical failure of ceramic-based restorations.^{76,258}

Regarding the relation between surface hardness and wear of both materials and tooth cusps, despite the decrease in hardness of Vita Enamic and IPS e.max ZirCAD, there were no statistical results of cusp wear antagonized by these materials at the end of TCML. Although Vita Enamic (1.41 GPa) and IPS e.max ZirCAD (1.64 GPa) have a similar degree of hardness change due to TCML, IPS e.max ZirCAD was the hardest material, and hardness value of Vita Enamic was close to GC Cerasmart as very low. The wear of cusp/Vita Enamic and cusp/IPS e.max ZirCAD was not statistically significant as a correlation could not be established between the hardness of materials and the amount of material and cusp wear. However, cusp/Celtra Duo showed higher wear than cusp/GC Cerasmart, since their antagonist materials could not show significant changes in surface hardness. The results could be related to the wear behaviour of these materials due to their microstructure. Although several attempts have been made to relate the hardness of dental materials to their abrasiveness and wear resistance, recent studies have shown that other factors also influence the wear behaviour of a ceramic, such as microstructure, porosity, crystal size, surface roughness, and environmental factors.¹⁴⁵ The possible degradation of the surface by the change of the physical properties of the material may affect the wear in a positive or negative way. Several studies have found that enamel wear caused by ceramics is more related to fracture toughness than to hardness values.^{163,205,259}

The fracture toughness of the material represents a key role in preventing cracks. Zirconia is less susceptible to the microcrack mechanism because it has a higher fracture toughness than glass-ceramics.²⁵⁹ The microcrack formation is responsible for the surface degradation of ceramics and is considered the dominant wear. Due to the brittle nature of ceramic materials, toughness is an important parameter that should be considered to understand the wear behaviour of cusps. Under the same conditions of the wear process, the propagation of microcracks is likely to be more difficult due to the polycrystalline structure of zirconia.²⁶⁰ Wang et al.²⁶¹ stated that the high toughness and strength of zirconia imparts resistance to surface damage under load on material. Xu et al.,²⁶² reported that fracture toughness of occlusal enamel is in the range of $0.8 \text{ MPa.m}^{1/2}$. Moreover, if the fracture toughness of ceramic material is much higher than that of enamel, even more antagonist wear may occur. If the fracture toughness of enamel is accepted as $0.8 \text{ MPa.m}^{1/2}$, GC Cerasmart appears to have the closest toughness value at $1.14 \text{ MPa.m}^{1/2}$, and IPS e.max ZirCAD appears to have the farthest toughness value with $13.52 \text{ MPa.m}^{1/2}$. In a thermocycling study of Sonmez et al.²⁶³ the fracture toughness of resin-matrix

ceramic materials, Vita Enamic and Lava Ultimate (with a similar microstructure to GC Cerasmart), were significantly decreased after thermocycling. While the fracture toughness of Vita Enamic was changed from $1.23 \text{ MPa.m}^{1/2}$ to $1.02 \text{ MPa.m}^{1/2}$, the Lava Ultimate changed from $1.29 \text{ MPa.m}^{1/2}$ to $1.01 \text{ MPa.m}^{1/2}$. The values were close to each other and to enamel as being low. The present study showed that the toughness change of Vita Enamic from $2.86 \text{ MPa.m}^{1/2}$ to $2.14 \text{ MPa.m}^{1/2}$ was more than GC Cerasmart from $1.14 \text{ MPa.m}^{1/2}$ to $1.24 \text{ MPa.m}^{1/2}$. However, while the decrease in fracture toughness of Vita Enamic was statistically significant, the small increase in fracture toughness of GC Cerasmart was not. This might be caused by the addition of high mechanical loading via chewing simulator to thermocycling. After TCML, the order of materials was from greater to lesser IPS e.max ZirCAD ($11.66 \text{ MPa.m}^{1/2}$) > Celtra Duo ($2.79 \text{ MPa.m}^{1/2}$) > Vita Enamic ($2.14 \text{ MPa.m}^{1/2}$) > GC Cerasmart ($1.24 \text{ MPa.m}^{1/2}$). IPS e.max ZirCAD had highest fracture toughness among all other material groups. According to some studies, improving the reliability of a material in service can be achieved by increasing its fracture toughness.^{264,265} A study in which Y-TZP ceramics are exposed thermal and mechanical cycling, applied a cycle load of 200 N on zirconia materials, to reproduce the severe conditions of bruxism and to assess the performance of restorations. During loading, the material is subjected to highly localized stresses.²⁶⁶ The stresses lead not only to cracking but also to a change in crystal structure and transformation to an amorphous phase.²⁶⁷ This transformation also occurs in the presence of cyclic hydrothermal stresses, such as in water or water-based conditions.²⁶⁸ These stresses are often stimulated by thermal cycling with temperature changes. A study comparing the toughness of 3 Y-PSZ and 5 Y-PSZ ceramics, found the 3 Y-PSZ more durable when exposed to fatigue testing and thermocycling. This may be due to its ability to undergo transformation toughening.²⁶⁹ The introduction of surface defects (cracks) into the material results in a tetragonal to monoclinic transformation that effectively prevents further crack propagation and results in high fracture toughness of the stabilized zirconium.^{91,270} 5Y-PSZ was significantly less fracture resistant than 3Y-PSZ before and after fatigue testing with 1.200.000 cycles and 110 N load. Harada et al.²⁷¹, found that the hardness and fracture toughness of zirconia were not affected by thermal aging. This study used increased chewing forces of 120 N and long-term simulated chewing cycles of 1.200.000, in addition to thermal aging, which may produce different results. IPS e.max ZirCAD was the hardest and toughest material in this study, despite the statistically significant decrease in its hardness and fracture

toughness after TCML. As if the zirconia seems not a stable material, no crack formation could be observed on material surface, and it showed the lowest wear.

Regarding the relation between fracture toughness and wear of both materials and tooth cusps, the statistically significant lower wear of cusp/GC Cerasmart could be related to the lower toughness of GC Cerasmart near the enamel. Furthermore, since IPS e.max ZirCAD has the highest toughness, it should have resulted in higher cusp wear. According to some studies, enamel antagonized with zirconia suffers fatigue wear with subsurface microcracks due to the high toughness of zirconia.^{158,261} While the wear quantification results of cusp/IPS e.max ZirCAD showed less wear volume loss with no statistically significant results, it was possible to observe small chipping areas and microcracks on the enamel surface by wear qualification via microscopic evaluations. In addition, IPS e.max ZirCAD as the toughest material showed the lowest wear among material groups. Although GC Cerasmart and Celtra Duo showed an increase in fracture toughness ($-0.07 \text{ MPa.m}^{1/2}$ and $-0.35 \text{ MPa.m}^{1/2}$) with no significance after TCML, the statistically significant decrease in fracture toughness was observed in Vita Enamic and IPS e.max ZirCAD material groups ($0.61 \text{ MPa.m}^{1/2}$ and $2.00 \text{ MPa.m}^{1/2}$). Despite the decrease in fracture toughness of Vita Enamic and IPS e.max ZirCAD, there were no statistically significant results of their enamel antagonists. From the results of the present study, it appears that there is no clear correlation between physical parameters such as surface hardness and fracture toughness of the materials and the wear of themselves and of enamel antagonists.

Surface roughness is another important material-related factor among the factors of enamel wear opposed by restorative materials.²⁷² Turssi et al.²⁷³ stated that the surface roughness plays a significant role in the wear pattern of the restorative material or antagonist. According to Flury et al.²⁷⁴ high surface roughness can cause superficial cracks to develop and propagate, which can reduce the strength of a restoration.²⁷⁴ Heintze et al.¹⁹ claimed that the rough ceramic surface encountered during chewing simulation further accelerates the wear of the opposing enamel, and chipped pieces can act as an abrasive medium. Nevertheless, Kim and his colleagues¹³⁵ investigated the wear of enamel antagonist of zirconia and lithium disilicate in their study and concluded that the pressed e.max lithium disilicate ceramic resulted in a greater wear volume loss of opposing enamel than zirconia ceramic, even though it had lower initial Ra values compared to zirconia at the beginning of the experiment. Metzler et al.¹⁶³ reported that

although the roughness of the ceramic is important in the initial stages of wear, once the effect of roughness disappears as wear progresses, the internal properties of the ceramic begin to affect the wear rate. In the present study, it was found that the initial surface roughness values of Celtra Duo and GC Cerasmart were similarly low to those of the other material groups. Even though, they were able to provide statistically significant results of antagonist cusp wear with these lower Ra values. This suggests that the initial roughness is not a determining factor for tooth cusp wear, rather than the internal properties of material.¹⁰ When measuring surface roughness, the selection of 3D surface characterization methods is more reasonable as they could give more specific and solid descriptions about the evaluation of microgeometry and function-related properties for the material surfaces. In this study, a contact profilometer that performs 2D measurements on parallel surfaces was used. The initial roughness of all material samples was standardized by the established polishing protocol. However, after the wear test, the presence of indentation areas due to the chewing simulation meant that there were no flat surfaces left on the materials over which measurements could not have been made. Thus, the initial surface roughness values of the materials were measured to assess the effect of them on the wear of enamel antagonists. However, no direct correlation was found between the initial roughness values of the materials and the cusp wear.

With regard to the null hypotheses formulated at the beginning of the study,

- 1) “There was no difference between wear volume loss and wear mechanisms of the compared CAD/CAM restorative materials due to increased chewing forces” was rejected only for IPS e.max ZirCAD material group.
- 2) “There was no effect of different CAD/CAM restorative materials on the wear of enamel antagonist due to increased chewing forces” was rejected for Celtra Duo and GC Cerasmart material groups.
- 3) “There was no effect of thermocycling and mechanical loading (TCML) by increased chewing forces on the surface hardness and fracture toughness of CAD/CAM restorative materials” was rejected for Vita Enamic and IPS e.max ZirCAD material groups.
- 4) “There would be no effect of initial surface roughness of different materials on the wear of enamel antagonist” was accepted.

6. CONCLUSIONS

The conclusions of the present study are as follows:

1. Zirconia (IPS e.max ZirCAD) showed the lowest material loss (0.02 mm^3) due to wear among other three material groups (Vita Enamic, GC Cerasmart, and Celtra Duo) under increased chewing forces. The reason of this result is due to its uniform, polycrystalline structure which involves a different wear mechanism than other dental ceramics.
2. The enamel antagonist of zirconia (cusp/IPS e.max ZirCAD) did not show a statistically significant wear (0.22 mm^3), which could indicate zirconia as an enamel-friendly material, especially in patients with high chewing forces. However, zirconia causes microstructural defects of enamel antagonist with obvious crack lines and clusters of small chipping areas of enamel pieces. This can lead to problems in the long-term use. A thoroughly wear qualification is needed.
3. Composite materials such as ZLS-based ceramic (Celtra Duo), and resin-matrix hybrid ceramic (Vita Enamic) exhibit an abrasive character on enamel due to their microstructural loss against wear than zirconia with its uniform polycrystalline structure.
4. ZLS-based ceramic (Celtra Duo) causes more enamel antagonist wear (cusp/Celtra Duo: 0.59 mm^3) than resin nanoceramic (GC Cerasmart) (cusp/GC Cerasmart: 0.17 mm^3) due to the abrasive character of ejected zirconia crystals upon wear.
5. The force absorbing manner of nanoceramics within the bonding phase provide GC Cerasmart an enamel-friendly behaviour.
6. According to the observations made during this study, wear quantification by measuring the volume loss does not fully reflect wear characteristics, but also requires qualification of contact surfaces by visual microscopic evaluations such as scanning electron microscopy to assess wear mechanisms.
7. Increased chewing forces of 120 N and 5-year chewing cycles of 1.200.000 to simulate long-term behaviour of materials and their enamel antagonists lead to surface degradation, which affects wear mechanisms.

8. Thermocycling and mechanical loading (TCML) by increased chewing forces appears to be change the physical properties of materials. TCML decrease the surface hardness and fracture toughness of Vita Enamic (surface hardness decrease: 1.41 GPa, fracture toughness decrease: $0.61 \text{ MPa.m}^{1/2}$) and IPS e.max ZirCAD (surface hardness decrease: 1.64 GPa, fracture toughness decrease: $2.00 \text{ MPa.m}^{1/2}$).
9. Regarding the initial surface roughness, surface hardness and fracture toughness of materials, no direct correlation between these material-related factors and the wear of materials and enamel antagonists was found. The results appear to be related with the microstructural differences of these materials on their wear characteristics.
10. Clinicians expected to be considered the wear characteristics of restorative materials prior to antagonize them with natural tooth, and patient-related factors such as bite/chewing forces, parafunctional habits, occlusion and occlusal guidance to make case specific assessment about material selection and long-term clinical success.

Overall, considering the clinical implications of this study IPS e.max ZirCAD appears to be a suitable material choice for high chewing forces due to its wear characteristics. However, further studies are required to evaluate the effects of zirconia on enamel, rather than quantifying the wear volume loss, as the enamel did not show significant wear but showed a different wear mechanism with cracks and chipping areas on its surface by scanning electron microscopy. Since, the extremely high surface hardness and fracture toughness values of IPS e.max ZirCAD compared with enamel, change the microstructural properties and surface characteristics of enamel antagonist. GC Cerasmart also seems to be a choice with its low potential to wear off the enamel antagonist, as it has an enamel-friendly behaviour. However, it does not appear to be durable since the material has a higher wear potential and lower hardness and fracture toughness. Therefore, restorations should be protected by occlusal appliances when high chewing forces are applied. Even though this is an in-vitro study, the clinical implications of the results of this study can be derived.

7. REFERENCES

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8. APPENDICES

8.1. CURRICULUM VITAE

Personal Informations

Name	İzim	Surname	TÜRKER
Place of Birth		Date of Birth	
Nationality		TR ID Number	
E-mail		Phone number	

Education

Degree	Department	The name of the Institution Graduated from	Graduation year
Doctorate	Department of Prosthodontics	Yeditepe University Faculty of Dentistry	2021
University	Dentistry	Yeditepe University Faculty of Dentistry	2017
High school	-	Istek Belde High School	2012

All the grades must be listed if there is more than one (KPDS, ÜDS, TOEFL; EELTS vs),

Languages	Grades (#)
Turkish	Native
English	YÖKDİL (Score: 86.25)

Computer Skills

Program	Level
Word/Pages	Excellent
Powerpoint/Keynote	Excellent
Excel/Numbers	Good

*Excellent, good, average, or basic

Articles published in other journals:

Yoruk B, Turker I, Ozen E, et al. Diş Hekimliğinde Eğitime Dayalı Öğrenme Yaklaşımının Kullanılmasına İlişkin Bir Durum Çalışması. EDU7. 2017; 6(8): 54-68.

Proceedings presented in international scientific meetings and published in proceedings book:

<u>Türker İ.</u>, Kursoglu P, Aydın Aksu S, Ozen E, Baskak F. Evaluation of Masseter Muscle Stiffness, Transversal and Sagittal Diameter with Shear-wave Elastasonography- A Double-Blinded Study. EPA SEPES Congress: Digital Natives in Prosthodontics, Madrid, Spain, 2018.
Kursoglu P, <u>Özen E.</u>, Türker İ. Mobil Dişin Birleşik Lamina Splint Sistemi ile Tedavisi: Vaka Raporu. Marmara Üniversitesi Diş Hekimliği Sempozyumu, İstanbul, Türkiye, 2018.
Kursoglu P, <u>Türker İ.</u>, Özen E. Bruksizm ve Bruksizmi Etkileyen Farmakolojik Yaklaşımlar. Marmara Üniversitesi Diş Hekimliği Sempozyumu, İstanbul, Türkiye, 2018.
Kursoglu P, <u>Özen E.</u>, Türker İ. Geçici Restorasyon Gövde Dizaynı ile Çıkış Profili Şekillendirilmesi Vaka Serisi. 6. Uluslararası TPID Dicle Sempozyumu, Diyarbakır. 2018.
Kursoglu P, <u>Türker İ.</u>, Özen E. Baş-Boyun Ağrısı Şikayetiyle Başvuran Hastada Semptomatik Stylohyoid Ligament Kalsifikasyonu. 6. Uluslararası TPID Dicle Sempozyumu, Diyarbakır. 2018.
Kursoglu P, Özkut K, <u>Türker İ.</u>, Özen E. Oklüzyonun Yeniden Düzenlendiği Vakada Çenenin Ortopedik Stabil Pozisyonuna Ulaşmak İçin Stabilizasyon Splintinden Yararlanılması ve Oklüzal Kayıtların Alınması. EDAD 24. Uluslararası Estetik Diş Hekimliği Kongresi, Dijital Kongre. 2020.
<u>Kursoglu P.</u>, Özen E, Türker İ. Aşınmış Diş Vakasında Oklüzal Dikey Boyutun ve Estetiğin Yeniden Düzenlenmesi. EDAD 24. Uluslararası Estetik Diş Hekimliği Kongresi, Dijital Kongre. 2020.
Kursoglu P, Cabbar F, <u>Özen E.</u>, Türker İ. Bruksizmde Botoksun Rolü. EDAD 24. Uluslararası Estetik Diş Hekimliği Kongresi, Dijital Kongre. 2020.

8.2. ETHICAL COMMITTEE DECLARATION



Number : 37068608-6100-15- 1925

12th Augusts, 2020

Subject: The evaluation of Clinical Researches
Application File by CREC.

Dr. İzim Türker
Yeditepe University
Faculty of Dentistry
İSTANBUL

Yeditepe University, Faculty of Dentistry, Prof. Dr. Pınar KURSOĞLU, as a clinical researcher, the research project titled with “**Evaluation of Wear Characteristics of Different CAD – CAM Restorative Materials by Increased Chewing Forces via Chewing Simulator**” was evaluated by Yeditepe University Ethic Committee of Clinical Researches (CREC), at the meeting dated 12th Augusts, 2020 (CREC Application File with the (No: 1913) registered number).

As a result of the evaluation by the Ethic Committee of Clinical Researches, decided by majority vote that it was ethically and scientifically appropriate to conduct the study named above (**CREC Decision Number:1257**).