T.C
YEDITEPE UNIVERSITY
INSTITUTE OF HEALTH SCIENCES
DEPARTMENT OF PROSTHODONTICS

EFFECT OF VARIOUS SURFACE TREATMENTS AND LUTING CEMENTS ON SHEAR BOND STRENGTH BETWEEN PEEK AND INDIRECT COMPOSITE

PhD Thesis

Dt. Zeynep Çiğdem KILINÇ

İSTANBUL - 2022

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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.

20.07.2022

Zeynep Çiğdem KILINÇ

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LIST OF SYMBOLS AND ABBREVIATIONS

AB	Acid-base
AFM	Atomic Force Microscope
Al_2O_3	Aluminum Oxide
BioHPP	Ceramic reinforced High Performance Polymer
Bis-GMA	Bis-phenol-A di glycidyl methacrylate
CAD/CAM	Computer Aided Design Computer Aided Manufacture
CFR-PEEK	PEEK Coated with Carbon-Fiber
Er, Cr: YSSG	Erbium, chromium: yttrium
Er: YAG	Erbium: yttrium-garnet
FPD	Fixed Partial Denture
GFR-PEEK	Glass Fiber Reinforced PEEK
GPa	Giga Pascal
HA/PEEK	Hydroxyapatite Reinforced PEEK
H_2SO_4	Sulfuric Acid
IRC	Indirect Resin Composite
MPa	Mega Pascal
Nd:YAG	Neodymium yttrium-garnet
n-FA/PEEK	Nano-fluorapatite PEEK
PAEK	Poly aryl ether ketone
PEEK	Poly ether ether ketone
PEKK	Poly ether ketone ketone
RMGI	Resin-modified glass ionomer
SEM	Scanning Electron Microscopy
SBS	Shear Bond Strength
STM	Scanning Tunneling Microscope
TEM	Transmission Electron Microscopy
ZOE	Zinc oxide eugenol
ZONE	Zinc-oxide non eugenol
μm	Micrometer
°C	Degrees Celsius

ABSTRACT

Kılınç, Z.Ç. (2022). Effect of Various Surface Treatments and Luting Cements on Shear Bond Strength between PEEK and Indirect Composite. Yeditepe University, Institute of Health Sciences, Faculty of Dentistry, Department of Prosthodontics, PhD thesis, İstanbul.

The purpose of this *in vitro* study was to evaluate the effects of three different surface treatments, two luting cements and thermocycling on the shear bond strength between PEEK and indirect composite. A total of 240 disc-shaped PEEK specimens were randomly divided into four groups according to surface treatment methods: no-treatment (control), sandblasting with 110 μm particle Al_2O_3, 98% sulfuric acid etching, Er:YAG laser irradiation. The surface roughness was measured with Atomic Force Microscope. Each group was divided into two subgroups according to the luting cements: a temporary cement (TempBond NE) and a resin cement (RelyX U200). After the bonding between PEEK and an indirect composite material (Gradia) was completed, all subgroups were further divided into two subgroups according to whether thermocycling was applied or not (n=15). Shear bond strength was measured, and the failure modes evaluated. The data were statistically analyzed using a Three-Way ANOVA and Bonferroni adjustments for multiple comparisons. The sandblasting group demonstrated the highest surface roughness values (p=0.000). A higher level of shear bond strength was observed in the samples luted with RelyX U200 (6.871-25.134 MPa) compared to TempBond NE (1.958-6.772 MPa) (p=0.000). Thermocycling caused a decrease in shear bond strength in all groups (p=0.000). The most frequent failure mode was adhesive failure. The highest roughness on the PEEK surface was obtained by the sandblasting method. RelyX U200 cement provided superior bond strength compared to TempBond NE. The highest mean shear bond strengths were observed for sandblasted surfaces, followed by laser-irradiated, sulfuric acid etched, and untreated PEEK surfaces luted with RelyX U200. Thermocycling weakened the bonding to PEEK in all groups.

Keywords: PEEK, Sandblasting, Sulfuric acid, Er:YAG laser, shear bond strength.

ÖZET

Kılınç, Z.Ç. (2022). Çeşitli Yüzey İşlemlerinin ve Simanların PEEK ve Endirekt Kompozit Arasındaki Makaslama Bağlanma Dayanımı Üzerine Etkisi. Yeditepe Üniversitesi, Sağlık Bilimleri Enstitüsü, Diş Hekimliği Fakültesi, Protetik Diş Tedavisi Anabilim Dalı, Doktora tezi, İstanbul.

Bu *in vitro* çalışmanın amacı, üç farklı yüzey işleminin, iki simanın ve ısıl döngünün PEEK ve endirekt kompozit arasındaki makaslama bağlanma dayanımı üzerindeki etkilerini değerlendirmektir. Toplam 240 disk şeklindeki PEEK numunesi yüzey işlemi yöntemlerine göre rastlantısal olarak dört gruba ayrıldı: Yüzey işlemi uygulanmayan grup (control), 110 µm'lik Al_2O_3 ile kumlama uygulanan grup, %98'lik sülfürik asit uygulanan grup, Er:YAG lazer uygulanan grup. Yüzey pürüzlülüğü Atomik Kuvvet Mikroskobu ile ölçüldü. Her grup, kullanılan yapıştırma simanına göre iki alt gruba ayrıldı: geçici siman (TempBond NE) ve rezin siman (RelyX U200) ile simante edilen gruplar. PEEK ile bir endirekt kompozit materyal (Gradia) arasındaki simantasyon işlemi tamamlandıktan sonra, tüm alt gruplar ısıl döngü uygulanıp uygulanmadığına göre iki alt gruba ayrıldı (n=15). Makaslama bağlanma kuvveti ölçüldü ve başarısızlık tipleri değerlendirildi. Veriler, çoklu karşılaştırmalar için Üç Yönlü ANOVA ve Bonferroni ayarlamaları kullanılarak istatistiksel olarak analiz edildi. Kumlama grubu en yüksek yüzey pürüzlülük değerlerini göstermiştir (p=0,000). RelyX U200 ile yapıştırılan örneklerin makaslama bağlanma dayanımı (6.871-25.134 MPa) TempBond NE ile simante edilen örneklerinkinden (1.958-6.772 MPa) (p=0.000) daha yüksekti. Isıl döngü, tüm gruplarda makaslama bağlanma dayanımında azalmaya neden oldu (p=0.000). En sık görülen başarısızlık tipi adeziv ayrılma idi. PEEK yüzeyinde en fazla pürüzlülük kumlama yöntemi ile elde edildi. RelyX U200 rezin siman, TempBond NE'ye kıyasla üstün bağlanma dayanımı sağladı. En yüksek makaslama bağlanma dayanımı RelyX U200 ile yapıştırılmış, kumlanmış yüzeyler için gözlemlendi, bunu sırasıyla RelyX U200 ile yapıştırılmış lazer uygulanmış, sülfürik asitle dağlanmış ve işlem uygulanmamış PEEK yüzeyleri izledi. Isıl döngü, tüm gruplarda PEEK'e bağlanmayı zayıflattı.

Anahtar kelimeler: PEEK, Kumlama, Sülfürik asit, Er:YAG lazer, makaslama bağlanma dayanımı.

1.INTRODUCTION and PURPOSE

Implant-supported fixed partial dentures/crowns are frequently used as alternative means to conventional bridge restorations using natural teeth as abutments. After implant placement, an appropriate healing period is required for the osseointegration process until the definite prosthesis is made.

In order to maintain social interaction, patients need to feel confident both in terms of esthetics and function during the implant treatment period. Therefore, a provisional restoration is necessary to meet the functional and esthetic requirements of the patient during osseointegration. Provisional restorations may also serve as a reference for the design of definitive restoration (1). Furthermore, implant-supported fixed temporary restorations have numerous advantages, such as better tissue contours associated to emergence profile, development of an interdental/interimplant papillae, avoidance of an extra surgical procedure, fixation of the prosthesis, and customization during the healing process to create a more esthetic prosthesis (2).

Temporary implant abutments, either plastic (generally polymethyl methacrylate or polyetheretherketone) (3) or metal are used to fabricate a provisional restoration. This provisional restoration can either be cemented on a temporary abutment(s) or it can be integrated with a temporary abutment(s) by using a screw-retained prosthesis (4).

As temporary abutments, provisional solid titanium abutments are commonly employed. The grey color of the titanium is a significant drawback of these abutments. Also, the titanium abutment is not easy to prepare chair-side at the dentist office. Polyetheretherketone (PEEK) temporary abutments have been offered as an alternative to titanium abutments (5).

PEEK is a thermoplastic polymer with high performance that is appealing for use in dentistry due to its excellent thermomechanical characteristics, chemical stability, biological inertness, biocompatibility, and natural tooth-color. PEEK is chemically stable with virtually all chemical elements, and it has high strength, stiffness, toughness, and fatigue resistance (6, 7). Because of these advantages, PEEK is recently utilized in dentistry as useful material for implant retained overdentures, implant supported fixed prostheses, framework of removable partial dentures, framework for metal-free fixed prostheses, endocrowns, resin-bonded fixed prostheses, occlusal splints, dental implants, healing abutments and implant abutments (8).

Chair-side processing of PEEK abutments is simple, and their whitish color makes it easier to achieve an esthetic outcome (5, 9). Prefabricated PEEK temporary abutments facilitate the connection and adaption of temporary restorations, particularly during immediate loading protocols. When compared to temporary titanium abutments, these abutments are less expensive and may be effortlessly adjusted in the mouth to accommodate a transitional prosthesis (10). Unlike titanium abutments, this adjustment does not result in heat transfer to the peri-implant bone.

Despite its numerous advantages, PEEK has poor adhesion to resin-based materials due to its low surface energy and inert hydrophobic surface (11-13). That is, the major clinical drawbacks are challenges in generating a strong and durable adhesion between PEEK and dental materials (14). Previous studies examining the bonding ability between PEEK and composite resins indicated that the PEEK surface showed no or insufficient bonding without any additional surface treatment (12).

Various surface treatments are available to change the PEEK surface and improve the bond strength between PEEK and dental materials. These surface treatments include application of sandblasting (6, 7, 12, 14-26), silica coating (7, 14, 18, 31), sulfuric acid (6, 7, 11, 12, 17-20, 23, 26, 27), piranha acid (12, 22, 24, 26), different forms of plasma (19, 27-29) and laser (13-15, 22, 30).

Although there have been various studies in the dental literature on the improvement of PEEK surface to provide clinically long-term adhesion between PEEK and dental materials, there is not enough information yet about the potential and limitations of PEEK in bonding to dental materials (14). Since the luting procedure is one of the important issues in the clinical performance of fixed prostheses, current studies focus on improving of PEEK surface in order to enhance bonding to resins. Therefore, the aim of this study was to evaluate and compare the effects of three different surface treatments (sandblasting, sulfuric acid etching, Er:YAG laser application), two luting cements (Temp Bond NE: Zinc oxide non-eugenol cement, RelyX U200: resin cement) and also thermocycling on the bond strength between PEEK and indirect composite (Gradia). The null hypothesis was that surface treatments, luting cement types and thermocycling would have no influence on PEEK-indirect composite bond strength values.

2. GENERAL INFORMATION

2.1. Composite Resins

Composites have been available to the dentistry for many years. Composites are obtained by combining two or more different materials with different structures and qualities to form distinct phases. The purpose of this combination is to provide features that each of the parts that make up the composite cannot have alone (32, 33). Composite resins consist of three phases: organic phase, inorganic phase and interphase (34, 35).

Composite resins are classified according to inorganic filler particle sizes and percentages, polymerization methods and viscosity. The most widely used classification in composite resins is the classification made according to the size and amount of inorganic filler (36).

Classification of composites according to inorganic filler particle sizes and percentages:

- Conventional composite resins: Inorganic filler particle size 10-100 μm composites are called macrofill composites, and 1-10 μm composites are called midifill composites. Midifill and macrofill composites are called conventional composites.
- Small particled macro-filled composite resins: They are composite resins containing 70-80% by weight,1-5 μm sized filler particles.
- Microfilled composite resins: These composites contain 35-40% by weight of pre-polymerized silicon dioxide filler particles with a size of 0.02-0.4 μm.
- Hybrid composite resins: These composites contain 70-80% by weight of different filler particles in the size of 0.04 μm and 1-5 μm. Average particle size is generally 0.6 μm. Those with an average filler particle size of 1 μm and above are called hybrid, and those below 1 μm are called microhybrid. (34).

Dental restorative composite materials can be divided into two types which are direct and indirect resin composites (37).

2.1.1. Direct resin composites

The mechanical resistance of direct composite restorations is lower than indirect restorations. Non-ideal bonding with dentin, surface roughness, occlusal and proximal wear, postoperative sensitivity, loss of marginal adaptation, secondary caries can be counted as the disadvantages of the direct method (38).

2.1.2. Indirect resin composites

Prosthetic composites and laboratory composites are other names for indirect resin composites (IRC) (37). IRCs are used for onlays, overlays, inlays, short-span fixed denture prosthesis (FDP), as well as veneering material and repair material for a range of restorations (39).

The composition of IRC systems is similar to that of direct systems, with the difference being that alternative techniques of extra polymerization are used, allowing for greater radical conversion. Photo activation, pressure, heat, and a nitrogen atmosphere are examples of further polymerization methods (40).

There are two generations of indirect composites. The first generation was introduced in the 1980s. Its use has been abandoned because it does not provide clinical success due to reasons such as high wear resistance and low bending resistance. In order to overcome the disadvantages of first-generation indirect composites, second generation indirect composites were produced in the early 1990s. These are micro-hybrid composites containing 66% filler. They became more desirable due to its improved mechanical properties such as bending resistance and modulus of elasticity (38).

Despite having considerably inferior mechanical properties than ceramics, IRCs can support and enhance (rather than replace) ceramic restorations in some clinical situations, such as coronal restoration of dental implants. Because ceramics have a high modulus of elasticity and absorb minimal mastication energy, a significant amount of masticatory force is passed to the implant and periosseous structure, limiting the restoration's longevity. Polymers are the preferable materials in this case because they absorb a greater proportion of the occlusal stress. Stress-absorbing materials, such as IRCs, are recommended for individuals with weak periodontal tissues who require occlusal covering (41). Tsitrou *et al.* (42) reported that resin composites are less prone to marginal chipping than ceramics.

The clinical lifespan of indirect ceramic or indirect composite resin restorations is dependent on their adequate treatment and cementation. The kind of restorative material (ceramics/indirect resin composites) determines the cementation procedure; hence, their surface treatment should be undertaken in accordance with their specific compositions. Etching techniques, sandblasting, and silane coupling agents are the most common procedures with improved results (43).

According to studies, porcelain fused to metal restorations have a 10-year survival rate of 92%, a 15-year survival rate of 75%, and a 5-year survival rate of 93%. All ceramic fixed partial dentures have a five-year survival rate (44). To enhance esthetics, metal-free restorations such as ceramics and resins were introduced into dentistry. All-ceramic restorations are aesthetically pleasing, but they are hard and fragile (45).

Gradia composite is a micro-filled hybrid composite containing prepolymerized UDMA monomer and silica fillers that increase mechanical and physical qualities (46). The fracture strength of composite single crowns is determined by several parameters, including the component's modulus of elasticity, luting agent, thickness of the restoration, and mechanical characteristics of fiber, type of fiber and matrix, fiber and resin matrix adhesion, and composite type (47-49).

2.2. Luting Cements

Cementation is the process performed to close the gap between the tooth/abutment and the restoration. This procedure ensures that the restoration remains in the mouth for a long time by creating a connection and is considered the last stage of fixed restorative treatment. Cements are adhesive agents used in the cementation of fixed prostheses and provide temporary or permanent retention of restorations (50).

Depending on their physical qualities and the anticipated duration of the restoration, luting agents can be permanent or temporary (51).

2.2.1. Luting Cements for Definitive Luting

2.2.1.1. Zinc Phosphate Cements

Zinc phosphate is the oldest luting cement. It has been used successfully since 1800s. It is the traditional AB (acid-base) cement, delivered as a separate powder/liquid system, with the powder containing around 90% zinc oxide (ZnO) and the liquid containing ~67%

buffered phosphoric acid. Aluminum (1%–3%) in the liquid is required for the cement-forming process, while water (~33%) influences the reaction rate partially (52).

This cement is ideal for cementing cast and prefabricated posts, crowns, FPDs, metal inlays/onlays (51).

To delay the exothermic process and enable maximum amount of powder to be incorporated into the mix while keeping the viscosity in control, zinc phosphate should be combined on a cold, dry glass slab (52). Zinc phosphate cement has a reasonable working time. If mixing is done right, it has a good compressive strength which is one of the main advantages of this cement. Other benefits include adequate film thickness, usage in areas where the masticatory stress is high or when a long-span prosthesis is planned (51).

The disadvantages of zinc phosphate cements are low tensile strength, post-operative sensitivity due to low pH while setting, no chemical bonding, and the solubility in oral fluids (51).

2.2.1.2. Zinc Polycarboxylate Cements

Zinc polycarboxylate cements have been introduced in 1968. These cements are produced by the reaction of zinc oxide with poly carboxylic acid in water to prevent its dissolvement in oral fluids (53). Polycarboxylates are also supplied as powder–liquid compositions. The powder consists of sintered zinc oxide with fine particle sizes, minor amounts of magnesium oxide (1–5%), fumed silica and fluoride salts for the improvement of the mechanical strength and release of fluoride. The liquid is an aqueous solution of poly carboxylic acid, often a polyacrylic acid homopolymer or a copolymer of acrylic acid with itaconic or maleic acids. Polycarboxylate cements can create ionic bonds with enamel and dentine via the calcium ions found in hydroxyapatite minerals (54). Because of the rapid ascent in pH after mixing, it has high biocompatibility with dental pulp. Other advantages of zinc polycarboxylate cements are favorable tensile strength (8–12 MPa) and the sufficient water dissolution resistance (51).

The primary downsides of zinc polycarboxylate cement are, its non-resistance to acid dissolution, deformity under loading, critical manipulation and early rapid rise in film thickness which might interfere with proper seating of a casting (51).

2.2.1.3. Glass-ionomer cements

Wilson and Kent developed glass-ionomer (glass polyalkenoate) cement in 1969, and by the late 1990s, it had become the most widely used definitive luting agent on a global scale. The powder is typically a calcium aluminosilicate glass (other mixes substitute strontium or lanthanum for calcium) that contains fluoride to aid regulate cement formation and adjust properties. Dilute poly alkanoic acids, poly acrylic acid, maleic acid, itaconic acid, and other minor organic acids make up the liquid. Although the acid components are dried and blended with powdered glass, they are later mixed with water or a diluted tartaric acid. Its success has been attributed to its good flow properties, adherence to dental structure and base metals, cariostatic potential due to fluoride release (as well as fluoride recharge potential), high translucency, adequate strength, comparably cheap cost per unit dosage and its simplicity of mixing (52). It is most commonly used for luting metal and metal-ceramic restorations, but it may also be used with porcelain crowns (51).

The main drawbacks of glass-ionomer cement are sensitivity to moisture contamination and desiccation, its initial slow setting, insufficient wear resistance, and due to its lower modulus of elasticity than zinc phosphate, it is susceptible to elastic deformation in areas of strong masticatory stress (55).

2.2.1.4. Resin-modified Glass-ionomer Cements

The resin-modified glass ionomer (RMGI) is a hybrid material that consists of conventional glass ionomer cement and a small amount of light-curing resin. Therefore, it has properties of the two materials. They have superior characteristics compared to conventional glass ionomer materials. Good adhesion to the dental structure, esthetic, release of fluoride and easy manipulation and use are some of the advantages of resin modified glass ionomer cement (51).

RMGIs are indicated for luting crown and bridge prostheses, orthodontic brackets, zirconia, alumina-based ceramics, lithium disilicate pressed and milled (CAD/CAM) onlays and inlays. However, because of their hydrophilic nature, they exhibit increased water sorption that leads to hygroscopic expansion. As a result, they are not recommended for cementing low-strength all-ceramic crowns and veneers due to the risk of clinical fractures. It is used to bond metal or metal-porcelain crowns and FPDs to tooth, resin composite, amalgam, or glass ionomer core buildups (56).

Dehydration shrinkage is a drawback of RMGIs. Because of the glass-ionomer component, polymerization shrinkage has been documented as late as 3 months after maturity of the cement. As a result, stress fractures may occur at the exposed cement-tooth-restoration interface (56).

Due to free monomer, RMGIs may cause an allergic reaction. As a result, mixing should be done with care. Because removing the cement bulk is hard, this is considered a disadvantage (56).

2.2.1.5. Resin Cements

Today's resin cements are composed of a resin matrix of bis-GMA or urethane dimethacrylate and a filler of fine inorganic particles (20–80%) to achieve a thin film thickness (51).

Resin cements come in powder/liquid, encapsulated, and paste/paste forms. Resin cements are categorized into three varieties based on their polymerization method: chemical-cured, light-cured, and dual-cured (51).

Resin cements' benefits include exceptional compressive and tensile strengths (20–50 MPa) with minimal solubility, micromechanical adhesion to prepared enamel, dentin, alloys, and ceramic surfaces, its wide spectrum of shades and good translucency (51).

The disadvantages of resin cements are their diligent and critical manipulation technique, marginal leakage due to polymerization shrinkage, high film thickness, pulpal reactions when applied to vital dentin, no fluoride release or uptake, low elasticity modulus, and difficulty in removing hardened excess resin cement, especially from inaccessible areas, which precludes its use around subgingival margins. Furthermore, the application of eugenol based temporary cements inhibited the resin cement from completely polymerizing (51).

2.2.2. Provisional luting cements

Provisional cements come in a variety of compositions, including zinc oxide eugenol (ZOE), zinc-oxide non eugenol (ZONE), and resin-based cements (57).

2.2.2.1. Zinc Oxide Eugenol Cements

Dr. J. Foster Flagg developed zinc oxide eugenol cement in 1875 (58). It consists of zinc oxide, zinc stearate, zinc acetate and resin powder, liquid, eugenol, and oil. This cement was created by replacing the liquid in zinc oxychloride cement with creosote and then eugenol. Wessler created the first proprietary ZOE product, pulpol, in 1894. To enhance strength and minimize solubility, several additives such as alumina, silica, rosin, dicalcium phosphate, polymethylmethacrylate, polystyrene, and ortho-ethoxybenzoic acid have been mixed with ZOE cement (51).

Their primary application is in circumstances when tooth sensitivity is an issue. They are also used as short-term luting agents for provisional crowns and permanent partial dentures (51).

The main issue with eugenol-containing cements is that phenolic hydrogen interferes with the appropriate polymerization of resin composites, reducing their micro-hardness and color stability. Non-eugenol formulated cements are advised to be used as temporary luting cements (51).

2.2.2.2. Zinc Oxide Non-Eugenol Cements

Since eugenol is harmful when in direct contact with pulpal tissue, and the presence of residual eugenol impairs the adhesion of resin, ZONE cements were developed. While zinc oxide remains the primary powder component, several organic acids have been replaced for eugenol to make dental cements that are not adequate for definitive luting but satisfactory sealing and retain well-fitted temporary restorations (52).

2.2.2.3. Resin-Based Provisional Cements

The relatively recent development of resin temporary cements holds up the possibility of materials that do not produce ZOE challenges and have superior retentive properties (55). Flexure strength was related with retention for 3 resin-based temporary cements, and they were more retentive when compared against 5 other ZOE and ZONE cements, according to Lawson *et al*. (57).

2.3. Poly Ether Ether Ketone (PEEK)

It is a member of the poly aryl ether ketone (PAEK) polymer family, which is a new family of high temperature (over 300°) thermoplastic polymers. This polymer was first produced by ICI Advanced Materials in line with the recommendations of the British scientist in 1978 and was commercialized by the same company under the name of Victrex PEEK and was used for implant applications. When Victrex PEEK was first introduced, its main target was not medicine. In the 1980s, with the realization of the high potential of PEEK polymer, other manufacturers started polymer studies with these properties, and by the end of the 1990s, PEEK succeeded in enriching its application area as a high-performance thermoplastic material.

Poly ether ether ketone (PEEK) is a polymer consisting of chemically repeating ether and ketone functional groups and aromatic basic structure and molecular chain linked to each other (59).

When the polymer materials containing poly ether ether ketone are classified, they are simply included in the plastic class. To define PEEK more specifically, it can be classified as a linear homopolymer. The monomer parts that make up the polymer can be repeated and identical, in which case the homopolymer identification is used. Polymers containing two or more different monomers are called copolymers. A polymer can be linear or branched, this difference may vary depending on the synthesis of the polymer and the environmental conditions. The chain length, combination and shape of the molecules that make up the polymers are other factors that affect the heat resistance and bio-resistance of the molecule (60).

Recently, Poly ether ketone ketone (PEKK) has been developed and compared to PEEK polymer, PEKK has a compressive strength of more than 80%. PEKKTON, another member of the poly aryl ether ketone (PAEK) family, is a high-performance thermoplastic polymer that can be used in crown, bridge, and implant restorations with conventional or CAD/CAM production methods. PEKKTON polymer material has superior performance in many points compared to the materials used in the past in the dental industry. The advantages of this material are as follows: low weight, elastic modulus close to human bone, ability to absorb shock from the incoming force, no corrosion, not containing metal, not creating a foreign taste sensation in the patient's mouth, radiopacity and non-allergenic (61).

2.3.1. General Features of PEEK

PEEK polymer is a semicrystalline thermoplastic that has a fully aromatic linear structure. It consists of only carbon, hydrogen, and oxygen atoms. PEEK biomaterial, which is a pale and amber colored organic polymer, is an advanced high-performance plastic that is resistant to high temperatures. Semicrystalline aromatic polymers maintain their hardness even at high temperatures. This polymer, which has an exceptionally good resistance to heat and chemicals, is widely used in industrial applications, aerospace, automotive, electronics, medical equipment, chemical, petroleum, food and beverage industries due to its superior mechanical properties such as excellent biocompatibility, dissolution resistance and high fatigue resistance. It has been used in many fields, including PEEK material, with all these positive properties, has been made suitable for orthopedic and neurological surgery applications with various chemical modifications and commercial compositions available in the market (62). It has shown excellent results as an alternative to PEEK titanium, which has become widespread in orthopedic applications in medicine. The biggest feature of PEEK material used in orthopedics is that it has an elastic modulus of 3-4 GPa. This value is remarkably close to the elastic modulus of bone. In addition, with the addition of carbon fibers to PEEK, the elastic modulus can be increased up to 18 GPa. With its elastic modulus close to the bone, it absorbs the forces generated during chewing, keeps these forces away from the peri-implant region in the cervical area and prevents crystal bone resorption. PEEK polymer is also a rigid, tough material with high friction-wear resistance and is an important matrix material for thermoplastic applications (63, 64). Properties of PEEK are shown in Table 2.1.

Table 2.1. Properties of PEEK polymer

Properties of PEEK polymer	
Specific gravity	1.26 g/cm3
Initial modulus	3.6 GPa
Tensile strength	90-100 MPa
Moisture recovery	0.1%
Dielectric strength	190 kv/cm
Thermal conductivity	0.25w/m/°C

2.3.2. Chemical Structure of PEEK

PEEK is chemically composed of a repeating ketone and two ether groups. The resulting polyether ether ketone polymer is commercially produced by the polyetherification reaction. During the synthesis of PEEK polymer, the method of synthesis and the conditions in which it is carried out are of great importance. PEEK, consisting of aromatic basic structure and molecular chains, is a semicrystalline thermoplastic polymer. PEEK is chemically inert and has high chemical resistance due to the delocalization of high orbital electrons throughout the entire polymer chain. This high chemical resistance is due to the location of the ether and ketone groups at opposite ends of the aryl rings. Aryl rings join these groups at the chain's opposite ends. PEEK's stable chemical structure leads in greater orbital electron displacement across the macromolecule, making it resistant to chemical, thermal, and post-irradiation degradation. PEEK is known to be chemically resistant and to have an extremely low chemical absorption rate of 0.5 percent (65). It has been reported that PEEK polymer has solubility in water at a rate of 0.5% and is not chemically damaged in the presence of water for a long time, but water absorption reduces the crystalline percentage of PEEK (66). The reaction of 4.40-difluorobenzphenone with the disodium salt of hydroquinone in a polar solvent such as diphenyl sulphine at 300°C is the typical synthesis route for PEEK. Two different building blocks are used in the polycondensation reaction of the polymer of PEEK polymer, one of which is 4,4'-dichlorobenzphenone and the other is 4,4'-difluorobenzphenone. In the production of PEEK, 4,4'-difluorobenzophenone reacts with hydroquinone anions and performs the condensation process. 4,4'-difluorobenzophenone reacts with hydroquinone anions in high-boiling organic solvents such as N-cyclohexyl-2-pyrrolidone (67) (Figure 2.1).

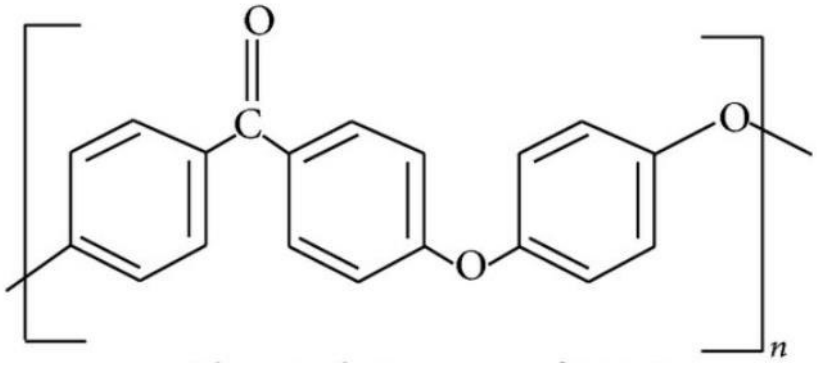

Figure 2.1. The chemical structural formula of polyetheretherketone (PEEK) (68).

PEEK material can be used in environments with an operating temperature of 260°C, due to its superior thermal degradation resistance and its melting temperature of 343°C. For example, while the degrees of thermal degradation leading to mass loss in commonly used polymers such as polyethylene and polypropylene are between 328°C and 335°C, this value range is between 575°C and 580°C in PEEK material (69).

2.3.3. Dental Usage of PEEK

PEEK has good physical, chemical and mechanical properties such as high biocompatibility, elastic modulus close to bone, high temperature resistance, resistance to dissolution and chemical agents, excellent electrical insulation, good abrasion and wear resistance. It has various usage areas such as temporary abutment, infrastructure material in fixed prosthetic restorations, healing caps, endocrowns, occlusal splints, infrastructure material in implant-supported hybrid prostheses, and major connector, clasp, and other components in removable partial dentures (70). It has been also used for the production of dental implants (61).

2.3.3.1. Use of PEEK as an Implant Material

The main material used in dental implants is titanium and its alloys. Titanium has a good physicochemical character, mechanical properties, biocompatibility, high resistance to corrosion and fatigue, but one of the major disadvantages of titanium material is that its modulus of elasticity (110 GPa) is significantly higher than that of the cortical bone elastic modulus (14 GPa). With excessive load on the bone, it causes implant fractures, bone resorption and results such as sufficient stress breaking effect (71). In addition, clinical problems are encountered with titanium materials. Although rare, there are many problems such as metal sensitivity and allergic reaction, wear of the implant surface and contamination with periimplantitis, dispersion of radiation, and the appearance of metallic color in aesthetic restorations (especially in the anterior region) in patients with thin gingival phenotype. Many researchers have developed materials to replace titanium dental implants, but the biggest disadvantage is that zirconia has a high elastic modulus and is broken down into small molecules originating from its own internal structure at low temperatures. Another material polymeric composition, PEEK, is a semicrystalline linear thermoplastic polymer developed in 1978. PEEK implant material is the material that can be applied as an abutment (72).

Because of PEEK's mechanical properties, elastic modulus (3-4 GPa), similar to human cortical bone, PEEK's good chemical and physical properties, stability at high temperatures, corrosion resistance and resistance to sterilization procedures it has been started to be used in medical implants. Its biocompatibility has also been proven in *in vitro* studies. Compared to titanium, PEEK materials allow the most optimal distribution of chewing forces and loads around the implant. The fact that its tooth-color, PEEK is more aesthetic compared to titanium. Other advantages are that it shows comparable properties with the response of soft and hard tissues to titanium and reduces stress (72, 73).

2.3.3.2. PEEK in Prosthodontics

As computer-aided design and computer-aided manufacturing (CAD/CAM) technology has advanced, a wider range of machinable materials suitable for dental prosthesis has become accessible. One of these materials is PEEK, a high-performance polymer with supreme physical, mechanical, and chemical characteristics. PEEK is a biocompatible material with strong mechanical qualities, high temperature resistance, chemical stability, good polishability, good wear resistance, and low plaque affinity. As a result, PEEK is now being utilized as a framework material in dentistry for metal-free fixed dental prostheses, removable dental prostheses, implant-supported fixed prostheses, endocrowns, implant-retained overdentures, and resin bonded fixed dental prostheses. It has also been employed in the fabrication of dental implants, implant abutments, temporary abutments, healing abutments, and occlusal splints. PEEK has a low modulus of elasticity (4 GPa) and is as elastic as bone when compared to rigid framework materials like metal alloys and zirconium oxide, providing cushioning and minimizing stresses transmitted to abutment teeth (74).

2.3.3.2.1 PEEK Implant Abutments

Materials used as implant abutments; titanium, gold, zirconium, and ceramic abutments are used (75). Despite the disadvantages of titanium and its alloys such as corrosion and sometimes hypersensitivity reactions, these materials, which are most frequently used in the production of implants and abutments, are considered the gold standard. However, in cases where the gingival phenotype is particularly thin in aesthetic areas, very satisfactory results cannot be obtained due to the reflection of metallic color from

the gingiva. Although gold material is less likely to be used due to its cost, zirconium abutments are more likely to break in the mouth due to their internal structure over time. There are few *in vitro* and *in vivo* studies on the use of alumina and zirconium ceramic abutments with zirconium being used exclusively with complete ceramic prostheses on a single tooth implant (76). PEEK is another material that may be utilized as a temporary abutment (77).

The fact that the elastic modulus of the PEEK surface (3-4 GPa) and the elastic modulus of the bone surface (1-30 GPa) are close, reduces the stress transmission of the loads to the jaw as a result of chewing forces and encourages bone regeneration. As a result, PEEK may be a viable alternative to titanium in the fabrication of temporary abutments. In addition, the microbial flora attachment of PEEK abutments is comparable to abutments made with titanium, zirconium and polymethylmethacrylate materials. Titanium reinforcement increases the resistance of PEEK abutments while increasing implant-abutment compatibility (78) according to Juvora (Juvora Ltd., Thornton Cleveleys, Lancashire, England).

2.3.3.2.2. The use of PEEK in removable partial prostheses

The partial dentures made of chromium-cobalt material are a suitable and predictable treatment option for the rehabilitation of partially edentulous patients. Many thermoplastic materials such as polyamide and acetal resins have emerged in clinical practice since metal clasps have aesthetic problems in the anterior region, heavy restoration due to metal weight, metallic taste and allergic reactions caused by metal alloys.

The acetal resins have more rigid infrastructures with retentive clasps, connectors and supporting elements compared to polyamides. It provides adequate tooth and tissue support against chewing forces. Polyamide materials have low elastic modulus and reduce the forces on teeth against aesthetic and rotational forces. The biggest and most important disadvantages are that Kennedy I and II removable partial prostheses made of polyamide material can cause occlusal embedding, they do not have a rigid infrastructure, and they are deficiencies in priming procedures (79).

Therefore, as an alternative to these materials, PEEK material has started to be used as a substructure material in removable partial dentures. Due to the color of the PEEK material, it is more aesthetic than metal substructures. PEEK material is also used as a clasp material in removable partial dentures. PEEK clasps showed lower retention compared to

that of chromium cobalt-based removable dentures (80), but the advantages of this material are its high polishing quality, low plaque affinity, and high wear resistance (81).With CAD/CAM systems, prostheses could be formed from PEEK (82). Since PEEK is a high-performance polymer, it can be used with acrylic teeth in removable dentures. In distal extension removable dentures, PEEK infrastructures are beneficial. Its elastic modulus reduces torque forces and reduces the stress on the tooth. As a result, a lighter prosthesis that provides comfort and satisfaction for the patient is obtained. This material, which is produced by scraping with the CAD/CAM system, is produced in a short time and can be recommended as a long-term restoration according to the results of previous studies due to the properties of PEEK material such as low water absorption and solubility (81).

2.3.3.2.3. The use of PEEK in fixed prostheses

PEEK can be used as a substructure in fixed prostheses, and PEEK substructures can be obtained by direct etching technique and restored with lost wax technique and superstructure with composite resin. Although the color of PEEK resembles tooth, it has, opaque color. Therefore, it should be used with composite resin to increase its aesthetic properties (72). Studies show that PEEK material can be used as coping under composite resin, since the mechanical properties of PEEK are like enamel and dentin. As a result, PEEK may offer an advantage over ceramic restorations. For CAD/CAM restorations, PEEK can be used instead of poly methyl methacrylate (73). Industrially manufactured PEEK blanks made with CAD/CAM had greater fracture strength than pressed granular PEEK or pressed pellet-shaped PEEK prosthesis in 3-unit fixed partial dentures (77). PEEK fixed prosthesis with CAD/CAM milling have a greater fracture resistance than lithium disilicate glass ceramics (950 N), alumina (815 N), and zirconia (981-1331 N) (83, 84). PEEK material is capable of withstanding compressive stresses of up to 1383 Newtons (plastic deformation starts from about 1,200 N) (55). Furthermore, PEEK has exceptional wear characteristics. Despite the low elastic modulus of PEEK, it has abrasion-wear resistance that competes with metal alloys. PEEK's good wear resistance, good mechanical properties and sufficient bonding with composite resin may be the long-term successful use of PEEK in fixed prostheses (78).

2.3.4. Types of PEEK

2.3.4.1. BioHPP PEEK

Dental material based on PEEK, trade name BioHPP (high performance polymer), was developed by Bredent (Senden, Germany). It is a high temperature thermoplastic polymer based on PEEK that was developed for use in dentistry. It has ceramic microparticles in it to improve the polishing qualities of restorations. This BioHPP polymer matrix contains ceramic fillers with a grain size of 0.3-0.5 μm that are equally dispersed and contribute for 20% of the overall content. Homogeneity is achieved in the macrostructure of polymers due to their micro dimensions. The high degree of polishability of the material also ensures color stability and less plaque retention over time. The fact that BioHPP is close to the elastic modulus (4 GPa) of human bone is especially important when bending forces occur in implant treatments, and chewing forces are transmitted as lightly as possible, thus reducing the risk of fracture (85).

BioHPP is particularly suitable for allergic patients because the polymer has a very low water solubility of <0.3 ig/mm (86). Restorations finished with BioHPP has a very low weight, which is considered extremely advantageous for patients. The research demonstrating good abrasion resistance for the BioHPP suggest that it might be used in dentistry as an alternative to chromium-cobalt alloys (Cr-Co) because it is lighter and does not generate corrosion. BioHPP structures may be produced utilizing both advanced CAD/CAM technology and traditional wax replacement methods. PEEK might also be used to fabricate dental implants as an alternative material. PEEK/BioHPP is an appealing material for dental implantology due to its mechanical qualities and great biocompatibility (85, 86). It is used as an infrastructure material for restorations due to its many advantages such as low weight, elastic modulus close to human bone, shock absorbing effect, no metallic components, high biocompatibility, low plaque retention, and non-corrosion (85). HPP polyether ether ketone consists of a single monomer, and this may be the reason of its low elastic modulus (74).

BioHPP can be utilized as the core material for removable partial dentures. However, due to a lack of research in this field, there is little evidence on the efficiency of clasps in BioHPP removable partial dentures. The use of ceramic nanoparticles considerably increases the strength of the substructure, which responds to the criteria of an elastic modulus that is suitable for permanent restorations. Its elastic modulus reaches 4000 MPa, which is similar

to bone. Furthermore, the breaking strength exceeds 150 MPa. Another use of BioHPP in Prosthetic Dentistry is the production of obturators in removable dentures. Because of its great biocompatibility and low density of 1.31 g/cm^3, BioHPP material is preferred. Other positive features are fracture-resistant, bone-like modulus of elasticity, easy polishability and processing (79).

Special laboratory composites are employed in the creation of BioHPP-based crown and bridge restorations. These polymer-coated indirect laboratory composites produce a comprehensive anatomical, functional, and aesthetic outcome. Full-contour BioHPP restorations are another possibility. BioHPP crowns are a great alternative for individuals who have parafunctional behaviors (for example, bruxism) since they do not corrode easily and can tolerate high chewing pressure without breaking. It has the potential to be utilized to build polymer endocrowns (88).

2.3.4.2. PEEK Coated with Carbon-Fiber (CFR-PEEK)

It is obtained by adding 30% short carbon-fiber (6-9 μm) to PEEK. CFR-PEEK is used for dental implants. PEEK has some advantages over titanium as an implant material in dentistry. It causes less allergic and hypersensitivity reactions. Studies on this matter show that titanium is an allergenic material. PEEK is radiolucent and causes less artifact on magnetic resonance imaging (MRI). PEEK has a more aesthetic appearance as it does not have a metallic gray color like titanium. Also, PEEK is a versatile base material that can be tailored for a specific purpose by changing its surface properties. The elastic modulus of PEEK is much lower compared to titanium, ceramic materials, and cortical bone. The elastic modulus of PEEK is much lower compared to titanium, ceramic materials, and cortical bone. PEEK with a greater elastic modulus is required for dental implant materials, particularly when used in the fabrication of abutments and superstructures. As a result, they reinforced PEEK using a variety of technologies, including carbon fiber reinforced PEEK (CFR-PEEK) and glass reinforced PEEK (GFR-PEEK). CFR-PEEK has an elastic modulus of 18 GPa, while GFR-PEEK has an elastic modulus of 12 GPa. With carbon fiber reinforced PEEK composites (CFR-PEEK) constructed with various fiber lengths and distributions, the elastic modulus of PEEK may be reached closer to the elastic modulus of cortical bone and titanium material. CFR-PEEK has attracted attention in medical implants due to its compatibility with modern imaging techniques, excellent physical and mechanical properties, and biocompatibility. A study compared the stress distribution of 30% carbon-fiber reinforced

PEEK and titanium using the finite element method. The findings of the study showed that less stress points occur at the bone-implant interface due to the reduction of elastic deformation in carbon-fiber reinforced implants (85). Another study compared the compression strength of CFR-PEEK, GFR-PEEK and titanium bars, and the highest compression strength value was obtained in titanium bars, while the lowest value was obtained in GFR-PEEK (87).

2.3.4.3. Glass Fiber Reinforced PEEK (GFR-PEEK)

It is made with 10% glass fiber reinforcement and radii ranging from a few m to 10 μm. It is mostly used as a substitute for dental implants. Glass fibers have a high elastic modulus, good thermal stability, a consistent coefficient of expansion, and an elastic modulus similar to bone. *In vitro* investigations have demonstrated that GFR-PEEK can offer a favorable environment to produce osteocalcin (87).

2.3.4.4. Hydroxyapatite Reinforced PEEK (HA/PEEK)

Particle size of hydroxyapatite is between 3-1000 μm and constitutes 30% in Hydroxyapatite Reinforced PEEK (HA/PEEK). Its elastic modulus is remarkably close to that of human cortical bone and supports the growth of osteoblasts (89).

2.3.4.5. Nano-TiO2 PEEK (N-TiO_2 /PEEK)

Titanium oxide has good biocompatibility, bioactive and hydrophilic. Nano-TiO2 PEEK composite significantly improves the biological activity of PEEK. It also supports the proliferation and adhesion of osteoblasts (90).

2.3.4.6. Nano-fluorapatite PEEK (n-FA/PEEK)

Fluoride ion from nano-fluorapatite affects the energy of metabolism and enzyme activity of bacteria, so because of this effect it can be stated that n-FA/PEEK has antibacterial effect. Fluoride ions, on the other hand, inhibit the activity of phagocyte and osteoclasts and thus stimulate new bone formation. (91).

2.3.5. Surface Treatment Methods of PEEK

Since PEEK has an inert surface, it bonds poorly to dental materials (12, 22). Because the clinical performance of FDP is mostly dependent on the luting procedure, scientific literature focuses on enhancing the PEEK surface for reacting with resins to provide bonding (12, 26, 86, 92).

Several research have been conducted to investigate the bond strength between resin and PEEK by applying various surface treatments such as silica coating (7, 18, 25), sandblasting (12, 21, 18, 27, 94), piranha etching (18, 22, 24, 95), sulfuric acid etching (6, 7, 11, 12, 17-20, 23, 26, 27), different forms of plasma (19,27-29) and laser etching (13-15, 22, 30, 96).

2.3.5.1. Sandblasting

It is one of the most often used methods for changing the surface energy of PEEK and increasing its hydrophilicity. Sandblasting is carried out with aluminum oxide (Al_2O_3) particles providing micromechanical retention. Sandblasting technique with Al_2O_3 particles is the most preferred method because of its oxidizing properties and because of the handicaps of using acid etching in the clinical setting. The sandblasting method is based on the principle that Al_2O_3 particles in diverse sizes up to 50-200 μm are sprayed on the PEEK surface from a certain distance and under pressure, creating a roughness on the surface and consequently forming retention grooves and providing retention. In studies on these subjects, sandblasting was applied with 50 μm or 110 μm Al_2O_3 particles at a pressure of 2.5 MPa, 3.5 MPa or 4 MPa from 10 mm distance for 10 seconds (12, 14, 17, 21, 94, 97).

In two studies comparing the surface roughness and contact angle values of the samples roughened with 50 and 100 μm Al_2O_3, Stawarczyk *et al.* (12, 21) stated that the surface roughness and contact angle values of the samples roughened with 100 μm Al_2O_3 were higher than those roughened with 50 μm Al_2O_3 sandblasting.

In the studies, it has been reported that the adhesion and surface roughness of PEEK increase with the sandblasting process applied on the PEEK surface with aluminum oxide particles, and a much stronger bond is obtained when bonding with the composite resin. This is based on the principle that the surface area increases with the increase in roughness on the PEEK surface after sandblasting, and the area to which the composite resin will be bonded increases, increasing the retention (94), Davies *et al.* (97) reported that PEEK achieved low

value in roughness and adhesion with the sandblasting method. Caglar I. *et al*. (14) evaluated the bond strength of PEEK samples roughened by sandblasting, laser and silica coating methods with composite resin and reported that the roughness values obtained in the sandblasted samples were significantly higher than the roughness values obtained in the other groups.

Ourahmoune *et al.* (98) discovered that sandblasting has a significant impact on the hydrophobic behavior of PEEK and its composites by modifying their morphology.

The effectiveness of the abrasive particles used for blasting depends on the hardness, shape, and size of the particles. As a rule, the particles to be used for sandblasting should be harder than the surface of the material to be applied. When abrasive particles hit a surface, they create surface defects. The size of this defect is proportional to the particle size. The shape of the particles is also important in the etching process. The most important function of sandblasting is to remove the contaminations on the surface and to increase the adhesion by increasing the surface area (24).

El-sherif *et al*. (99) summarized the advantages of aluminum oxide blasting as no specific equipment required and adequate retention.

2.3.5.2. Acid etching

With the acid etching method, pits are formed on the PEEK surface with various acids. It increases the surface polarity and thus increases the number of strong functional groups for binding due to breaking the aromatic ring. For this purpose, sulfuric acid and piranha acid are used in studies as well as various cauterizing solutions of some companies. Previous studies have suggested the application of 98% sulfuric acid for 1 minute to modify PEEK surfaces and increase the binding of PEEK. According to a study, etching with high concentration of sulfuric acid shows that the irregular differences on the surface of PEEK are formed due to the dissolution of the PEEK matrix by the sulfonation reactions. Sulfonation reaction is the displacement of the hydrogen atoms in the organic compound by sulfonic acid (11). In addition, scanning electron microscopes showed that the use of high concentrations of sulfuric acid resulted in more prominent pits and a porous surface at deeper depths. Thus, more resin-tags were obtained, and their binding increased (22)

Various roughening procedures were used to examine the bond strength between PEEK and composite. The bond strength of the samples etched with 98% sulfuric acid and

sandblasted samples were found to be remarkably high compared to piranha acid and samples that were not pretreated at all (22).

Piranha acid has also been widely used in the studies. Piranha solution is a powerful oxidizing agent that may eliminate organic residues and is composed of 98% sulfuric acid (H_2SO_4) and 30% hydrogen peroxide (H_2O_2). While sulfuric acid attacks the ether and carbonyl groups in functional groups, the atomic oxygen formed by the reaction of sulfuric acid and hydrogen peroxide in piranha acid directly reacts with the benzene ring of PEEK. With this reaction, PEEK increases the surface polarity and increases the number of functional groups that are much stronger for binding due to the breaking of its aromatic ring. Although some studies (12, 22, 26) have shown that the use of piranha acid alone does not affect the surface properties, it has been reported that the combination of piranha and sandblasting increases its bonding with composite resin.

This method is based on the principle of treating the metal surface with some acids to form pits on the surface and was developed as an alternative to electrolytic etching. Although acid-based gels create more superficial etching than the electrolytic technique, they have the advantages of being easy to use in clinical and re-etching when desired without the need for complex laboratory instruments. For this purpose, besides various cauterizing solutions produced by some companies, methanol, hydrochloric acid, nitric acid, sulfuric acid, ferrite chloride and their combinations are used (100).

2.3.5.3. Etching with Plasma

Plasma is a unique and reactive chemical environment that hosts several surface reactions. Plasma's high concentration of ionized and excited molecules can alter the surface characteristics of ordinarily inert materials such as ceramics and polymers. Unlike the risky application of aggressive acidic solutions mentioned above, chemical surface modification with plasma is a non-hazardous procedure. Plasma is the fourth state of matter. In this state, gases are ionized and electrons are separated from their atoms. This process is usually achieved by excitation of gases such as N_2, O_2, Ar and H_2 with electrons from radio frequency (rf), microwave or hot filament discharge. Surface modification with plasma has been widely used for more than 20 years to process polymeric materials. Plasma-generated charged particles cause certain effects on the treated surface, such as removal of organic residues, surface activation (101). Depending on the low penetration level (2-3 μm) plasma roughening preserves the excellent physical and chemical properties of the material, can

increase the strength, surface and coating properties, and biocompatibility of the materials by modifying the surface energies (102). Many studies have been conducted to enhance the adhesive properties of polymer surfaces with plasma, but only a few studies have reported improvement of the adhesive properties of plasma materials without the use of primers (103).

2.3.5.4. Laser Etching

There are many diverse types of lasers recommended for different dental treatments(103, 104):

- Argon Laser; advanced curing lights and bleaching.
- Carbon dioxide (CO2) Lasers; gingivectomy/gingivoplasty, periodontal curettage, second stage implant exposure, removal of oral mucosal lesions.
- Diode Lasers: gingivectomy/gingivoplasty, oral medicine uses, second stage implant exposure, periodontal curettage, removal of oral mucosal lesions.
- Neodymium yttrium-garnet (Nd: YAG) Lasers; gingivectomy/gingivoplasty, oral medicine uses, second stage implant exposure, periodontal curettage, removal of oral mucosal lesions.
- Erbium: yttrium-garnet (Er: YAG) Lasers; Gingivectomy/Gingivoplasty, second stage implant exposure, periodontal curettage, removal of hard tissues, removal of oral mucosal lesions.
- Erbium, chromium: yttrium (Er, Cr: YSSG) Lasers; Gingivectomy/Gingivoplasty, second stage implant exposure, periodontal curettage, removal of hard tissues, removal of oral mucosal lesions (103,104).

Laser surface modifications are a promising alternative to improve PEEK surface properties due to their high resolution, working speed, and lack of effect on general properties.

Several research have employed various forms of laser irradiation to improve the surface characteristics of PEEK (13-15, 30, 65, 96, 105-109). In the studies, researchers mostly used Er:YAG and Nd:YAG lasers to enhance surface properties. Because Er:YAG irradiation has a high optical penetration depth, it may be employed directly and efficiently in solid structures, which makes it a viable alternative to conventional methods of surface pretreatment of dental materials (110,111).

Laser surface treatments have been used effectively to increase the surface roughening and bond strength of Zr_2O_3, Al_2O_3 and polymeric materials. Riveiro *et al.* (30) reported that laser-irradiated PEEK surfaces show increased surface roughness compared to control group. Tsuka *et al.* (13) reported that it was the laser surface treatment greatly improved the mechanical adhesion, by creating pores on the PEEK surface and increasing the bond strength between PEEK and the resin based adhesive agent.

Jahandideh *et al.* (109) investigated the shear bond strength of PEEK to composite resin veneers after surface treatment with Er:YAG and CO_2 lasers. This study found that Er:YAG and CO_2 laser irradiations can improve the shear bond strength of PEEK to composite resin veneers, with the Er:YAG laser being more successful.

However, several studies about Er:YAG laser for surface modification of PEEK concluded that when this method alone is used, there is no difference in bonding to veneer composite resin (14, 15, 109).

2.3.5.5. Surface Treatments That Create Chemical Bonding

2.3.5.5.1. Application of Adhesive Agents

Primers containing functional monomers with high affinity for PEEK materials are used to provide a strong and continuous bond between PEEK material and composite resins. Especially the chemical content and solvents of adhesive systems are the most crucial factors in creating a strong adhesion between PEEK and composite resin. Monomers containing methyl methacrylate (MMA) have been shown to have a very high efficacy. It has been reported that high bond strength values are obtained after the application of methyl methacrylate-containing adhesive systems to the PEEK surface, after swelling the surfaces of PEEK and making it active.

The effect of adhesive primers with different monomer contents on the bond strength between PEEK and composite resin was investigated, and it was reported that the bond strength values were higher in sample groups where methacrylate-containing primers were applied, and these values were comparable to metal-ceramic restorations. Even in the evaluations made after aging, it has been reported that the samples applied with methyl methacrylate primers still show high values (112,113).

2.3.5.5.2. Tribochemical Silica Coating System (Rocatec)

Rocatec was first launched in Germany in 1989. This method, which was introduced to the market with a new type of bonding method between resin-metal-acrylic, is used to coat surfaces with silicate tribochemically. The purpose of tribochemistry is to create chemical and physicochemical changes in the material during the application of mechanical energy. Rocatec has types suitable for both laboratory and clinical use. The laboratory method is the rocket system, and two-stage sand application is made. Tribochemical silica sand is a modified form of alumina particles with silica and forms a silica layer on the ceramic surface when applied under pressure. The first stage, 110-μm sand is applied to the restoration with a pressure of 2.5 bar, then the second stage, the 110-μm size silicon oxide (SiO_2) sand called Rocatec-plus, is sprayed under the same pressure (2.5 bar) to form microscopic irregularities. Thus, mechanical retention is achieved (94). The effect of tribochemical silica coating on the bond can be explained by two mechanisms: sandblasting to create a surface to which the resin can be bonded micro-mechanically, and a chemical bond between the resin and PEEK by coating the PEEK surface with silica.

The method that can be used in the clinic is the Co-Jet system, another tribochemical silica coating method. It consists of silane and coating-abrasive. With this method, it is carried out by applying 30-110 μm Al_2O_3 particles modified with silicic acid for about 5-15 seconds under 2-3 bar pressure, and by embedding high-energy silica-coated aluminum oxides 15 μm into the PEEK surface. The connection between the PEEK surface and the silane layer is facilitated. While micromechanical retention is provided on the one hand, chemical retention is obtained with silane application on the other hand (25,94).

2.4. Bonding strength test methods in dentistry

In vitro tests have an important place in the evaluation of materials used in dentistry. There are 5 types of tests (115):

- Shear
- Tensile
- Push-out
- Micro shear
- Micro tensile

2.4.1. Shear test:

Shear bond strength (SBS) test has been one of the most preferred bond tests in dentistry. It is mostly used to evaluate the interface resistance of two bonded materials. In this test method, a force is applied to the sample placed on the fixed carrier by means of a tip and the load on which the sample is broken is determined. Various inserts are used when applying force, but it is often recommended to use blade-shaped inserts that apply a shearing force to the specimen from the surface (116, 117). Due to the design of the samples and the shape of the applied force, the forces affecting the interface are quite complex. The rupture of the specimen is not only caused by the shearing effect, but also by the tensile stress developed at the interface because of the bending effect (117).

2.4.2. Tensile Test

Force distribution in tensile tests is more homogeneous than in shear tests. This situation leads to much more stable results for the bonding test results. In tensile bond strength tests, it is critical that the bonded interface to be aligned perpendicular to the loading axis. If not adjusted, the force will cause bending of the samples. In addition, the test device must maintain the correct position between the tooth surface and the adhesive material. For these reasons, tensile tests require more technical precision than shear tests (118).

2.4.3. Push-out Test

This test is one of the tests used for measuring the bond strength. A hole in the appropriate size for the material to be worked on is made on the dentin prepared in the form of a round slice, and the material is placed in this hole with the adhesive system, and a force is applied to the material with a pointed tip whose ratio is less than 0.85 to the diameter of the material. The force at which the separation occurs is measured. This method mimics the clinical environment better than the shear and tensile test because in this method, the tested material is bonded to dentin with an adhesive system (119).

2.4.4. Micro Shear Test

The micro shear test was introduced to researchers in 2002. This is a test method in which the bond strength of samples placed on tooth tissue, or a material is measured with the help of a wire or a different mechanism. The surface area of the materials whose bond strength is measured is less than 1 mm^2. Calculation of the average value for a single tooth, more than one test sample can be obtained from a tooth, possibility to perform the test in the presence of irregular surfaces, the samples can be examined more easily under the electron microscope after the experiments are among the advantages of this method (120).

2.4.5. Micro Tensile Test

The value in MPa of the micro-tensile bond strength test method is higher than that found in tensile tests because the critical size of cracks is smaller at the micro-interface. However, another point to be considered when evaluating the bond strength between tissue and samples is as follows; Micro cracks that may occur when cutting samples with a diamond bur can concentrate in the area where the force is applied, resulting in a lower bond strength result (121).

2.5. Surface Roughness Measurement Methods

Various devices have been developed to evaluate the surface roughness. These are, profilometer, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic electron microscopy (AFM) (122-129).

2.5.1. Profilometer

It is a method that gives two-dimensional measurement results. In mechanical profilometer devices, the surface roughness values obtained by moving a diamond scanner tip on the sample surface are digitally calculated and recorded. While the diamond scanner tip travels on the sample surface at a certain speed, it records the vertical movements of the tip due to irregularities on the sample surface. Thus, numerical values related to the sample surface topography obtained as the arithmetic mean roughness of the surface (Ra value, in μm). Surface profilometry is the primary approach for assessing surface roughness, in which

a fine stylus is used to scan the topography in a single line of a preselected region. The disadvantages of this approach include that surface defects adjacent to the scanning line are not assessed and hence do not contribute to the total measured surface roughness, as well as its invasive nature, which may harm the surface while scanning it (125). Roughness values obtained with a profilometer provide a quantitative measure of surface irregularities; nonetheless, the Ra parameter is regarded as a poor predictor of surface roughness, even though it is commonly used to evaluate surface topography in dental materials (126- 130). In optical profilometer devices, there is no contact with the sample surface. Therefore, it can be used to evaluate the roughness of sensitive surfaces. Thus, the sample surface is not damaged. It works on the principle of reflection of the light directed to the surface. They are faster and have higher resolution than mechanical profilometers (119-121).

2.5.2. Scanning Electron Microscopy (SEM)

It is an optical method. The basic principle in SEM is scanning the sample surface with a primary electron beam. In the scanning process, the primary electron beam interacts with the electrons on the sample surface, allowing these electrons to scatter around. By detecting and collecting these scattered electrons by the sensors, information about the surface topography, surface components and surface structure is obtained. The higher the number of electrons detected by the detector, the brighter the image of the region, the less the image of the region is obtained. As a result, an image of the sample surface in gray tones is obtained. To obtain quality images in SEM analysis, sample surfaces such as ceramics should be coated with gold-palladium powder (122,131). Because it does not allow for the visualization of 3D surface texture, SEM has limits in terms of surface topography description (124).

2.5.3. Transmission Electron Microscopy (TEM)

This method is another optical method. While SEM is a method of collecting electrons scattered from the sample surface, TEM is a method that works with electrons collected by passing through the sample. TEM is a special material characterization device that uses imaging and diffraction techniques together, providing microstructural examination of the sample and determination of crystal structures. In TEM, precise and detailed images

of the atomic crystal structure are obtained without the need for coating ceramic samples (132).

2.5.4. Atomic Electron Microscopy (AFM)

This method is another optical method. In the AFM system, a needle consisting of a few atoms is moved in 3D on the surface to be examined by piezoelectric control elements. Distortions at the tip of the needle are detected and magnified by a laser light. Thus, images related to detailed surface topography are obtained at nanometer resolution (122). Figure 2.2 shows the AFM components.

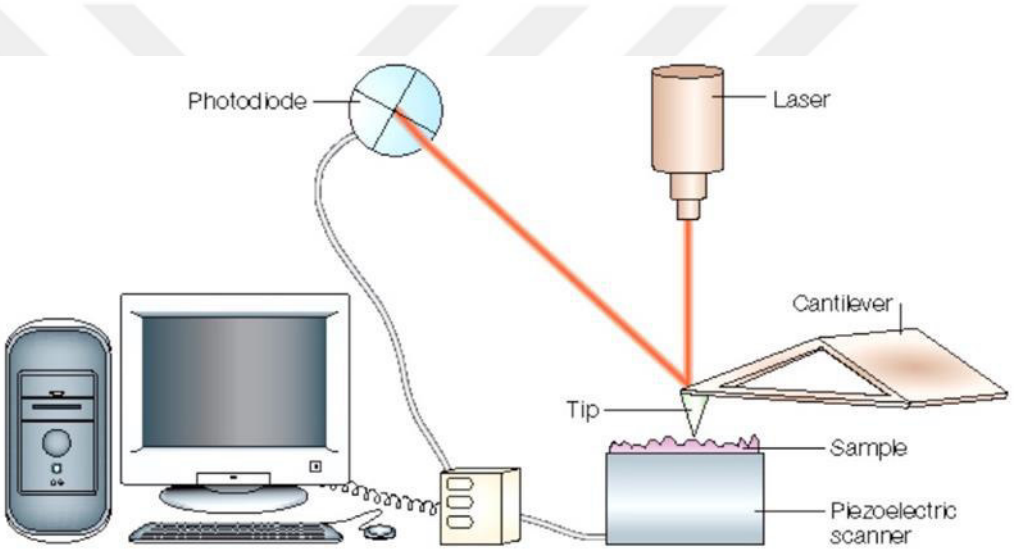

Figure 2.2. AFM components

Scanning Probe Microscopy (SPM) is an experimental method used to view the structure of a surface at the subatomic level. Another variant of SPM is Atomic Force Microscopy. Binnig, Quate, and Gerber introduced this microscope in 1986 as a combination of the Scanning Tunneling Microscope (STM) and the Contact Stylus Profilometer (133). As a result of their successful STM studies, researchers have developed a spring that can be sensitive to the forces between atoms. This spring part is the cantilever part of the AFM device, and its spring constant varies between 0.001 and 100 N/m. The cantilever or spring is 100 to 400 μ long and 0.5-5 μ thick. It is usually made of silicon or silicon nitride. AFM is based on the mapping of the atomic force field generated on the sample surface (122).

The use of AFM in the field of biomaterials has increased in recent years. Unlike optical microscopes, this type of microscope works by scanning the surface in three dimensions with the help of a needle instead of light. Unlike other microscopes, it is possible to examine in different liquid environments thanks to the liquid chamber of the AFM device. In addition to liquid, it could work in air and vacuum. The data obtained because of the interaction between the needle and surface atoms are used to measure the surface topography from Angstrom level to 100 μ. In addition to the surface topography, AFM analyzes the physical, chemical, and magnetic properties of the sample.

It can also be used to examine the structure, dynamics, and manipulation of biomaterials and to examine flat surfaces or interfaces. AFM has made possible to obtain high resolution images from the surfaces of varied materials and structures such as metals, polymers, ceramics, biomolecules, or cells (123). In AFM, the movement of the needle at the tip of the cantilever on the sample surface and the pushing and pulling forces of Newton (N) occurring between the tip and the surface are measured. With push and pull forces, a topographic image of the sample surface is obtained in computer environment.

AFM needs a constant and continuing atomic force. These atomic forces are used as feedback signals. It does not need electric current. This allows us to examine the surface of any object. Figure 2.3 shows the elements of AFM. The sharp tip is moved on the sample surface and the atomic forces between the tip and the sample are measured because of the movement of the balance bar. The beam from the laser source is reflected from the cantilever (balance bar) to the photodetector. The photodetector determines the position of the beam from the laser source and can measure variations of approximately 0.1 Angstrom. By measuring the difference between the signals of these two photodiodes, the displacement of the balance bar can be determined. The movement of the cantilever in the x, y and z directions is recorded and transferred to the computer, and the sample topography appears in three dimensions. The piezoelectric scanner can move in the x, y or z direction with a resolution of 0.5-127 μ on the sample surface of the balance bar. It is possible to obtain two- or three-dimensional surface images via the scanner. According to the contact between AFM, cantilever tip and sample, there are 3 different modes of operation: contact mode, non-contact mode and tapping mode (123, 134, 135).

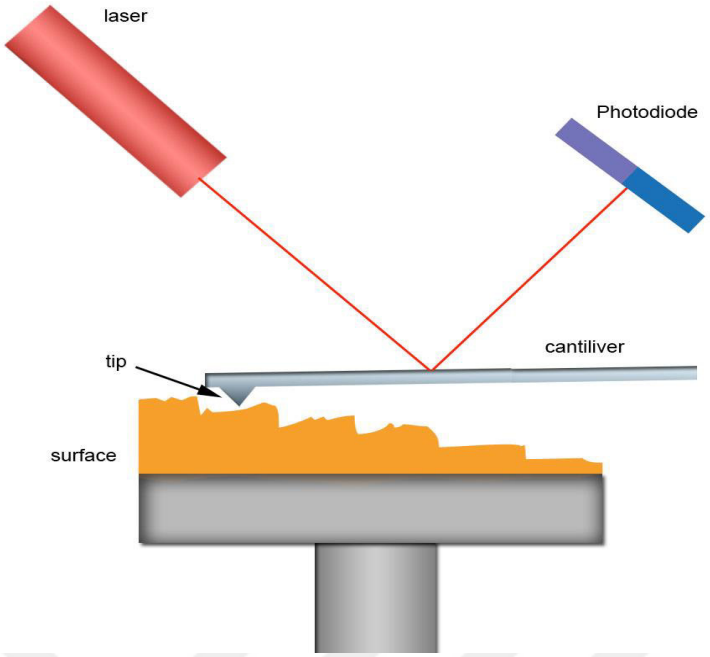

Figure 2.3. AFM elements (136)

AFM allows the surface properties of an object to be examined in detail. In addition to the surface topography, three dimensional images are obtained. Thus, intermolecular relations, hardness, reflection, morphological properties of the surface, surface interactions, electrical charge, magnetic, wear and corrosion properties, composition and amounts of molecules and elements that make up the sample, hydrophilic properties and melting point can be examined. Although the scanned area appears to be a small area to the naked eye, a very large area emerges at the nano-level. In the two-dimensional evaluation of surface roughness in *in vitro* conditions in dental studies, the arithmetic mean roughness of the surface (Ra value, in μm) or root mean square (RMS) of the average roughness value (Rq, in μm) is considered. A sample's amplitude parameters are defined by parameters that provide information about statistical average values, histogram height shape, and other extreme characteristics. The mean height obtained over the entire measured length/area is the average roughness (Ra). The term Ra is commonly used to define the roughness of machined surfaces. It can be used to identify general variances in overall profile height properties as well as to monitor an established production process. RMS roughness (Rq) is the square root of the surface height distribution and is regarded to be more sensitive than average roughness for substantial deviations from the mean line/plane. It is also used to

calculate the skew and kurtosis parameters. The finish of optical surfaces is described by RMS roughness (Rq) (137). Because surface topography is three-dimensional in nature, the parameters acquired from three-dimensional AFM examinations are more realistic. In comparison to the profilometer's 5 μm diamond stylus, the AFM has a 0.01 μm tip, allowing for more accurate measurement (124). The surface roughness parameters of AFM images are average roughness (Sa) and root mean square roughness (Sq). While the roughness of two-dimensional surfaces is evaluated using Ra and Rq values, the parameters for the roughness evaluation of three-dimensional surfaces are examined by looking at the mean roughness (Sa, in μm) and RMS deviation, that is, the square root roughness deviation (Sq, in μm) (134, 135, 138).

The AFM device's benefits include determining quantifiable values for examined parameters, eliminating the need for extra preparations, and producing topographical 3D images with excellent resolution (139). AFM is regarded as a promising tool for assessing the surface properties of dental materials (131, 140, 141).

3.MATERIALS and METHODS

This study was carried out in the Hard Tissue Laboratory at the Faculty of Dentistry, Yeditepe University, Turkey. The compositions and details of the materials used in this study are listed in Table 3.1.

Table 3.1. The compositions and details of the materials used in this study

Materials	Product Name	Manufacturer	Composition	Lot No.
PEEK	KETRON CLASSIX LSG PEEK white	Invibio Ltd., PEEK-CLASSIX, Germany	Polyether-ether ketone	-
Sulfuric acid	Sulfuric acid	-	98% Sulfuric acid	-
Aluminum oxide	Cobra	KOROX Renfert, Germany	110 µm pure Al_2O_3	15831012
Dental laser	VersaWave Er:YAG All-tissue Laser	HOYA ConBio, France	Er:YAG laser (10 Hz, 150mJ)	-
Zinc oxide/Non-Eugenol cement	TempBond NE	Kerr, CA, USA	Zinc Oxide, white mineral oil (petroleum), petrolatum	8586912
Resin cement (Self-adhesive Dual-cured)	RelyX U200 Automix	3M ESPE, Seefeld· Germany	Base: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst: Methacrylate monomers, Alkaline (basic)	609966
Indirect laboratory composite	Gradia Indirect	GC Corp, Tokyo, Japan	Alumino-silicate glass, Amorphous Precipitated Silica, Di-2-Methacryloyloxyethyl 2,2, 4-trimethylhexamethylene dicarbamate, Neopentylglycol dimethacrylate	1512031, 1510141

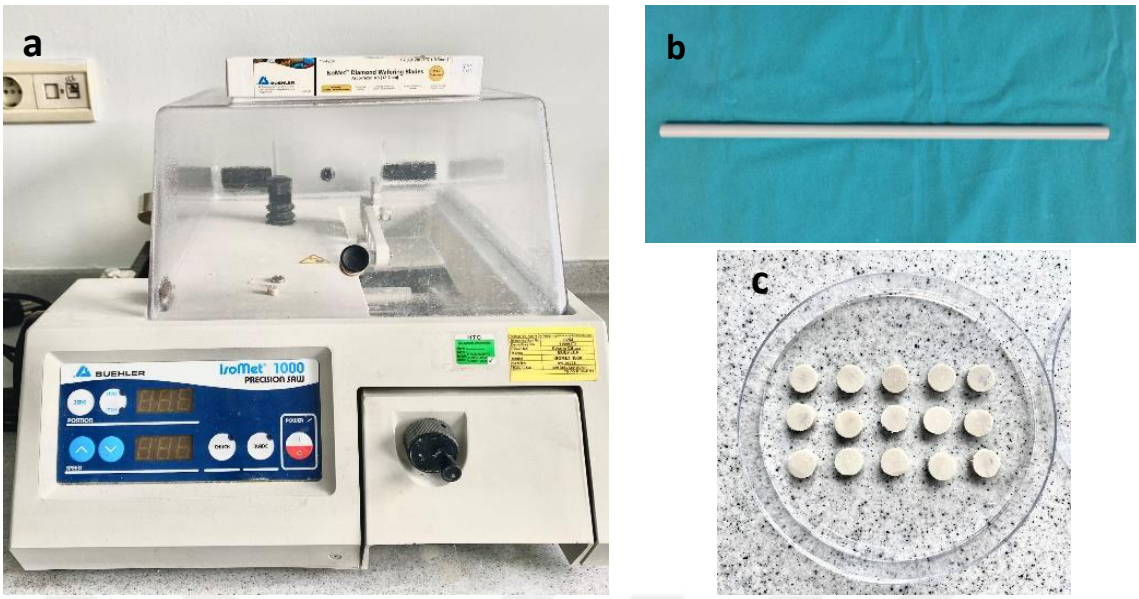

Figure 3.1.a. The cutting machine (Isomet ® 1000, Buehler, Germany), **b.** Prefabricated blank, **c.** Specimens after cutting

3.1. Preparation of PEEK specimens

Two hundred and fourty disc-shaped PEEK specimens (8 mm diameter; 3 mm thickness) were obtained by sectioning from a prefabricated PEEK blank (KETRON CLASSIX LCG PEEK, Invibio, Germany) with a cutting machine (Isomet ® 1000, Buehler, Germany). The cutting machine, a prefabricated blank and specimens after cutting are demonstrated in Figure 3.1.

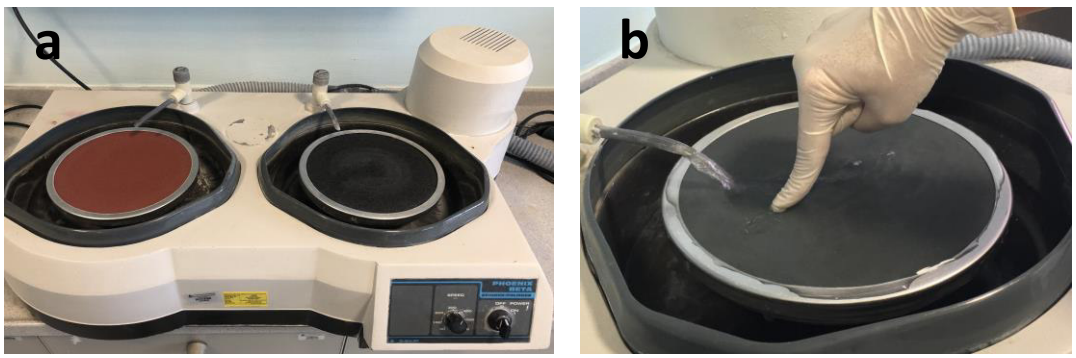

Figure 3.2.a. Polishing device (Phoenix Beta, Buehler, Germany), **b.** Polishing of PEEK specimens with water cooling.

Then, the bonding surface of each specimen was polished with the polishing device (PHOENIX BETA, BUEHLER, Germany) under water cooling with 600 grit silicon carbide abrasive papers (Figure 3.2). After polishing, the PEEK discs were cleaned in an ultrasonic water bath (Astra S, Tecno-Gaz, Parma, Italy) (Figure 3.3) for 10 minutes and air-dried.

Figure 3.3. Ultrasonic cleaning device.

3.2. Grouping of the PEEK specimens according to surface treatment

240 disc-shaped PEEK specimens were randomly grouped into four main groups according to surface treatment protocol (n= 60 per group) as follows:

- No surface treatment (control)
- Sandblasting (with 110 µm Al_2O_3)
- Acid etching (with 98% sulfuric acid)
- Laser irradiation (with Er:YAG Laser)

3.3. Surface treatments of PEEK specimens

3.3.1. No Surface treatment (Control group):

Non-treated PEEK group served as control. All specimens of this group were only rinsed with de-ionized water for 1 min. and air dried or 10 seconds (Figure 3.4).

Figure 3.4. Rinsing of specimens in de-ionized water

3.3.2. Sandblasting:

Surfaces of PEEK specimens in this group were sandblasted with 110 µm Al_2O_3 particle size for 10 seconds at a pressure of 2 bar and at distance of 10 mm between the tip of the sandblasting machine and the PEEK surface, and application angle of 45 degrees. All specimens were rinsed with de-ionized water for 1 min. and air dried for 10 seconds.

Figure 3.5 demonstrated the sandblasting machine and sandblasted specimens.

Figure 3.5. Sandblasting machine, and a group of the sandblasted specimens.

3.3.2. Acid Etching:

Surfaces of PEEK specimens in this group were etched with 98% sulfuric acid for 60 seconds (Figure 3.6). And then, all specimens were rinsed with de-ionized water for 1 min. and air drying was done for 10 seconds.

Figure 3.6. Etching of PEEK specimens with 98% Sulfuric acid.

3.3.4. Laser Irradiation:

Surfaces of PEEK specimens in this group were irradiated using Er:YAG laser (Er: YAG laser, Hoya ConBio, Fremont, USA) with a wavelength of 2.940 nm, a power setting of 150 mJ, and a10 Hz repetition rate at an average power output of 1.5 W in QSP mode (5 x 5 μs pulse duration) (Figure 3.7a) based on the method described by Caglar *et al.* (14). Laser energy was applied to the surface with a noncontact handpiece (H02-N, 0.9 mm spot size), and the laser beam was aligned perpendicular to the specimen surface at a distance of 10 m (Figure 3.7.b). Then, all specimens were rinsed with de-ionized water for 1 min. and air drying was done for 10 seconds.

Figure 3.7.a. Er:YAG laser device (Er: YAG laser, Hoya ConBio, Fremont, USA)
b. Er:YAG laser application on surface of the PEEK specimens

3.4. Surface Roughness Measurements Using Atomic Force Microscope

After surface treatments, a total of 60 PEEK discs that are randomly chosen from 4 main groups (n: 15) were examined using an Atomic force microscope (AFM) (QUESANT, INSTRUMENT COOPERATION, UNIVERSAL SPM, USA) (Figure 3.8).

Figure 3.8. Atomic Force Microscope (QUESANT, INSTRUMENT COOPERATION, UNIVERSAL SPM, USA)

The microscope was operated in the non-contact mode under dry condition. The specimens were placed below the cantilever of the AFM to obtain three-dimensional images of the surface (10 μm x 10 μm) and images were taken. AFM images reflect the surface in three dimensions. The numerical values obtained for the scanned surface are Root mean square (RMS, namely Rq), square root of mean roughness squares (Sq) and average roughness (Sa).

Surface roughness analysis was performed with Silicon (Si) tip AFM device (Quesant Instrument Corp., Agoura Hills, CA, USA) (Figure 3.8) in Boğaziçi University R&D Center. AFM images and roughness measurements obtained from the sample were acquired in tapping mode, and the rough spots detected in the sample were reflected on the images as height or small grains. Tapping mode is the most used imaging technique, and it allows us to see the surface morphology in 3D. The specimens were placed below the cantilever of the AFM to obtain three-dimensional images of the surface (10 μm x 10 μm) and images were taken. AFM images reflect the surface in three dimensions. The numerical values obtained from the scanned surface are Root mean square (RMS), square root of mean roughness (Sq) and average roughness (Sa).

3.5. Preparation of GC Gradia Indirect Composites

For the polymerization of the indirect composite resins (GC Gradia Indirect, GC Corporation, Tokyo, Japan) to be used in the present study, plexiglas molds (Figure 3.9.a) were prepared.

These Plexiglas molds allow light from all directions, and they are suitable for placing inside the light chambers of the light device. Each mold provides the polymerization of 8 samples simultaneously and from all directions. The prepared molds consist of 2 layers of plates. The bottom layer consists of plain untreated 5 mm thick plexiglas on which the Gradia composites can be stacked (Figure 3.9.b), and 5 mm thick plexiglas for the composites to be applied. All plates are attached to each other with neodymium magnets for ease of operation.

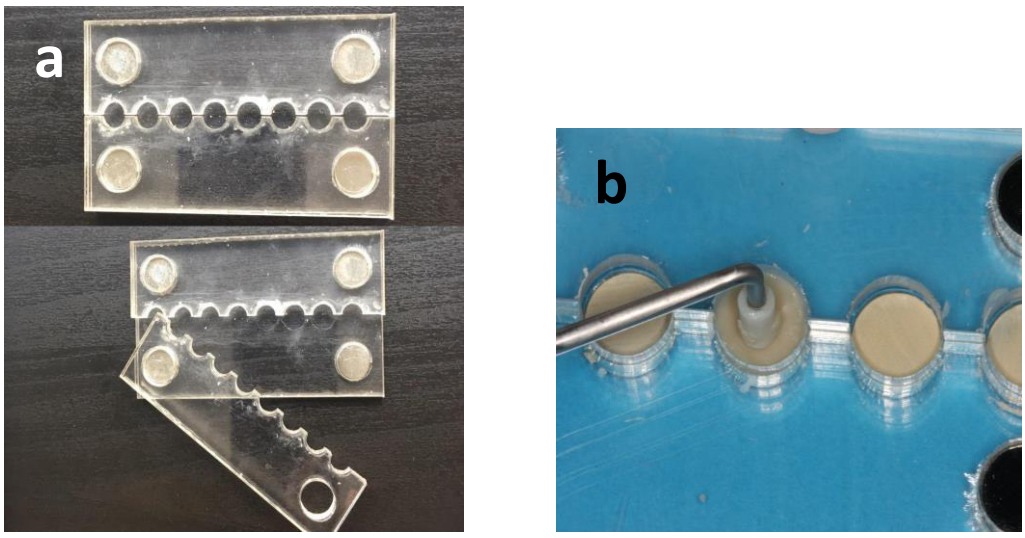

Figure 3.9.a. Plexiglas mold, and **b.** Application of Gradia indirect composite material in the plexiglas mold

The polymerization processes of indirect composite were carried out with a light device (GC Labolight Duo light, LV-III) (Figure 3.10).

Figure 3.10. GC Labolight Duo Light LV-III

After polymerization, the indirect composite specimens were removed from the plexiglas mold. Thus, Gradia indirect composite specimens with the dimensions of 5x5 mm were produced. Then, all specimens were rinsed with de-ionized water for 1 min. and air dried for 10 seconds.

Then, Gradia indirect composite is sandblasted with a pencil-tipped sandblaster (Microetcher ERC, Danville Eng., CA, USA) in order to increase the surface roughness and bond strength. Sandblasting was done with 110 μm Al_2O_3 particles under 0.5 MPa pressure for 15 seconds from a distance of 10 mm to the surface in accordance with manufacturer's recommendations.

After sandblasting, all composite discs were ultrasonically cleaned with distilled water for 10 minutes (Figure 3.11).

Figure 3.11. Cleaning of specimens in the ultrasonic bath

3.6. Bonding Procedure Between PEEK And Indirect Composite Specimens

After surface treatments, each main group was divided into two subgroups according to luting cements which are Zinc oxide non-eugenol cement (TempBond NE, Kerr, CA, USA) and self-adhesive resin cement (RelyX U200 Automix self-adhesive resin cement, 3M ESPE, Seefeld, Germany) (Figure 3.12).

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Figure 3.12. Luting cements used. **a.** TempBond NE (Kerr, CA, USA), **b.** RelyX U200 Automix self-adhesive resin cement (3M ESPE, Seefeld, Germany)

3.6.1. Bonding With Zinc Oxide Non-Eugenol Cement

TempBond NE (Kerr, CA, USA) was mixed on a paper pad following the manufacturer's instructions and placed between PEEK and Gradia® indirect laboratory composite. A 500 g weight was placed on top of the specimens for standardization during setting of the cement as seen in the Figure 3.13. Excess cement was removed using a microbrush. A standard time of six minutes was waited to allow for complete setting of the cement.

3.6.2. Bonding With Self-Adhesive Resin Cement

RelyX U200 Automix self-adhesive resin cement (3M ESPE, Seefeld, Germany) was applied on the bond surface of PEEK specimens according to the manufacturers' recommendations. Gradia discs were positioned and stabilized on the PEEK surface on which resin cement was placed. A 500 g weight was placed on top of the specimens for standardization during setting of the cement as seen in the Figure 3.13. Excess resin was removed with a microbrush. RelyX U200 was light polymerized with the light curing device (Kerr, Optilux, 21 Commerce Drive Danbury CT, USA) (Figure 3.14) from all sides for a total of 80 seconds. The distance of the light tip from the specimen was 5 mm.

Figure 3.13. 500 g weight placed

Figure 3.14. Light device (Optilux, Kerr)

3.7. Thermocycling of Test Specimens

Each subgroup was further divided into two additional subgroups, with and without thermocycling. Specimens in the thermocycling group (Figure 3.15.a) were subjected to a total of 5500 thermal cycle processes at 5° to 55°C (±2) to imitate the temperature changes in the mouth. The waiting time of the samples in the bath of the thermal cycler (Salubris Technica, Dentester, Turkey) (Figure 3.15.b) is 30 seconds and the transition time from one bath to the next is 2 seconds.

Figure 3.15.a. Subgroups to be subjected to thermocycling, and **b.** Bath of the thermal cycler

Table 3.2 shows classification of specimens in all test groups with respect to surface treatments, luting cements and whether thermocycling is applied or not. As a result, 16 test groups were organized in the present study (Table 3.3).

Table 3.2. Classification of specimens

Substrate	Surface treatment	Cements	Thermocycling	Indirect composite
PEEK	Control (No treatment) (n=60)	Zinc oxide non-eugenol cement (TempBond NE) (n=30)	TC + (n=15)	Gradia
			TC - (n=15)	
		Resin cements (RelyX U200 Automix) (n=30)	TC + (n=15)	
			TC - (n=15)	
PEEK	Sandblasting (with 110 µm Al_2O_3) (n=60)	Zinc oxide non-eugenol cement (TempBond NE) (n=30)	TC + (n=15)	Gradia
			TC - (n=15)	
		Resin cements (RelyX U200 Automix) (n=30)	TC + (n=15)	
			TC - (n=15)	
PEEK	Acid etching (with 98% sulfuric acid) (n=60)	Zinc oxide non-eugenol cement (TempBond NE) (n=30)	TC + (n=15)	Gradia
			TC - (n=15)	
		Resin cements (RelyX U200 Automix) (n=30)	TC + (n=15)	
			T C - (n=15)	
PEEK	Laser irradiation (Er:YAG Laser) (n=60)	Zinc oxide non-eugenol cement (TempBond NE) (n=30)	TC + (n=15)	Gradia
			TC - (n=15)	
		Resin cements (RelyX U200 Automix) (n=30)	TC + (n=15)	
			TC - (n=15)	

Table 3.3. Abbreviations of names of all test groups and applied processes.

Groups	Names of Groups	Procedures
Group 1	Group C.Z.T	No treatment + zinc oxide non-eugenol cement + thermocycling
Group 2	Group C.Z.nT	No treatment + zinc oxide non-eugenol cement + **no** thermocycling
Group 3	Group C.R.T	No treatment + resin cement + thermocycling
Group 4	Group C.R.nT	No treatment + resin cement + **no** thermocycling
Group 5	Group S.Z.T	Sandblasting + zinc oxide non eugenol cement + thermocycling
Group 6	Group S.Z.nT	Sandblasting + zinc oxide non eugenol cement + **no** thermocycling
Group 7	Group S.R.T	Sandblasting + resin cement + thermocycling
Group 8	Group S.R.nT	Sandblasting + resin cement + **no** thermocycling
Group 9	Group A.Z.T	Acid etching + zinc oxide non eugenol cement + thermocycling
Group 10	Group A.Z.nT	Acid etching + zinc oxide non eugenol cement + **no** thermocycling
Group 11	Group A.R.T	Acid etching + resin cement + thermocycling
Group 12	Group A.R.nT	Acid etching + resin cement + **no** thermocycling
Group 13	Group L.Z.T	Laser irradiation + zinc oxide non eugenol cement + thermocycling
Group 14	Group L.Z.nT	Laser irradiation + zinc oxide non eugenol cement + **no** thermocycling
Group 15	Group L.R.T	Laser irradiation + resin cement + thermocycling
Group 16	Group L.R.nT	Laser irradiation + resin cement + **no** thermocycling

3.8. Shear Bond Tests (SBS)

The cemented specimens (PEEK +cement+ Gradia) were embedded in self-curing acrylic resin (Meliodent, Bayer Dental Ltd., Newbury, UK) inside stainless steel rings 15 mm in diameter and 15mm in length (Figure 3.16).

Figure 3.16. Specimen embedded in self-curing acrylic resin

Shear bond strength was measured with the universal testing machine (INSTRON, 3345, Instron Corp., Norwood, MA, USA) (Figure 3.17). The specimens were attached to a custom jig of a universal testing machine and subjected to a shear force at a crosshead speed of 1mm/min until failure occurred (Figure 3.18). The setup for the shear bond test is illustrated in Figure 3.19.

Figure 3.17. Universal testing machine

The shear bond strength (SBS) values were calculated from this measurement and expressed in Mega-Pascals (MPa) using the following formula: Shear bond strength (MPa) = Load (N)/area (mm^2).

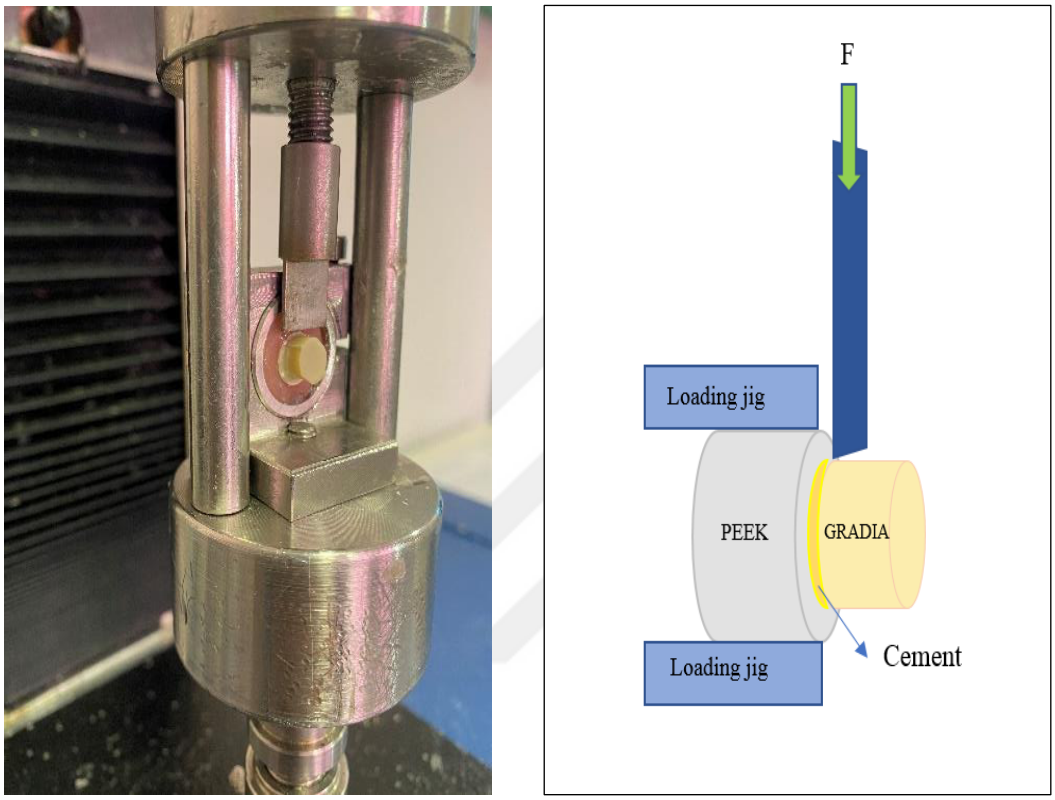


Figure 3.18. Illustration of the Universal Testing Machine with mounted specimens and schematic illustration of shear bond test setup.

3.9. Failure Mode Analysis with Stereomicroscope

After shear bond test, surfaces of all specimens were examined under a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) (Figure 3.19) at 40x magnification to evaluate the fracture pattern. Similar to the study of Gouveia et al. (142), failure modes were classified into one of three categories as follows:

1. **Adhesive failure:** If debonding occurred between PEEK and luting cement or resin cement and Gradia interface; when no cement remnants left on the PEEK surface or Gradia surface.

2. **Mixed failure:** When resin remnants were partially left on the PEEK's surface but also when part of the PEEK surface was exposed.

3. **Cohesive Failure:** When the fracture was occurred in the PEEK or in the Gradia.

As a result, the design of this study is summarized in Figure 3.20.

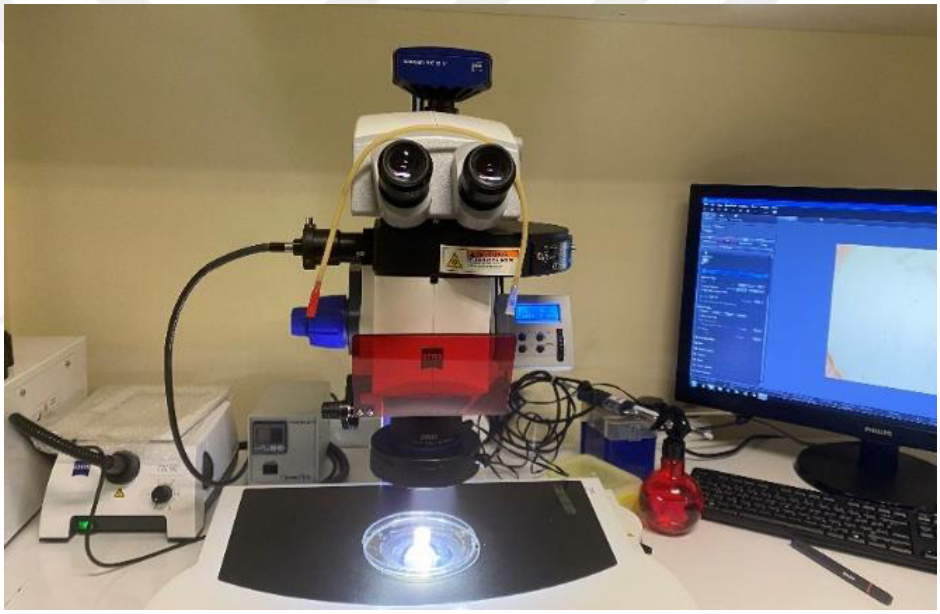


Figure 3.19. Stereomicroscope (SMZ 800, Nikon, Tokyo, Japan)

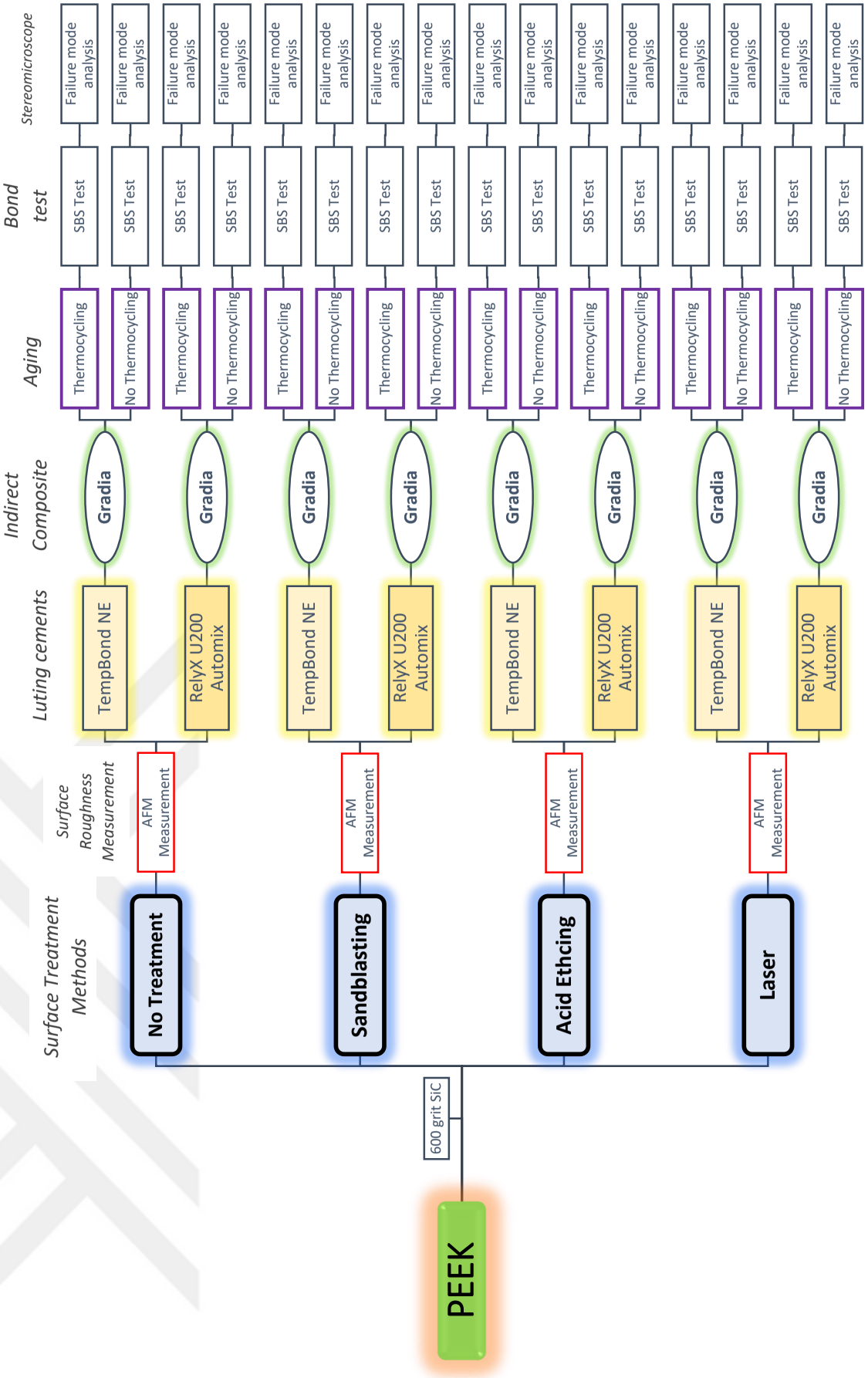


Figure 3.20. Overview of the study design.

3.10. Statistical analysis

Statistical analysis was calculated using the statistical package (IBM SPSS Statistics for Windows v26.0; IBM Corp).

While evaluating the study data, the normal distribution status of the data for each group was evaluated by using Kurtosis and Skewness statistics. The values for Kurtosis and Skewness between -2 and +2 are considered acceptable in order to prove normal univariate distribution (143). According to the Kurtosis and Skewness statistics, it was determined that the parameters were not normally distributed.

Data obtained from RMS Deviation and Mean Deviation tests were analyzed with Kruskal Wallis to examine the differences between four different surface roughening methods. Pairwise comparisons were completed with Dunn's Post Hoc test with Bonferroni adjustment.

Three-Way ANOVA test was used to examine main and interaction effects of roughening methods, cements and thermocycling factors on shear bond strength. Pairwise comparisons for all groups were completed with the EMMeans contrasts with sequential Bonferroni adjustments for multiple comparisons.

The statistical significance is set at 0.05 ($p<0.05$).

4. RESULTS

4.1. Results of surface roughness measurements

The means surface roughness values (Sa and Sq) and descriptive statistics of all surface treatment groups are presented in Table 4.1. The control group values (both Sq and Sa) showed the lowest surface roughness. The sandblasting group revealed the highest surface roughness values. When comparing the surface roughness values for specimens from all test groups, the ranking of both Sq and Sa values from high to low was **sandblasting>laser irradiation, acid etching, control** (Table 4.1) (Figure 4.1 and 4.2).

Table 4.1. Means, standard deviations and descriptive statistics of surface roughness values (µm) of test groups

		Descriptive Statistic				Kruskal Wallis		Pairwise Comparisons
Variable	**Roughening Method**	**N**	**Mean**	**SD**	**Mean Rank**	**K-W H**	**p**	**Dunn's Post Hoc**
RMS Deviation (Sq) (µm)	**Control(1)**	15	190.44	56.15	21.47	27.263	0.000	1-2[a] 2-3[a] 2-4[a]
	Sandblasting(2)	15	471.23	139.17	50.60			
	Sulfuric Acid(3)	15	206.11	125.89	23.03			
	Laser(4)	15	223.13	81.66	26.90			
Mean Deviation (Sa) (µm)	**Control(1)**	15	151.92	48.13	21.60	26.930	0.000	1-2[a] 2-3[a] 2-4[a]
	Sandblasting(2)	15	379.95	116.91	50.40			
	Sulfuric Acid(3)	15	163.52	103.01	22.57			
	Laser(4)	15	177.51	59.67	27.43			

Kruskal Wallis Test

a. Significance values have been adjusted by the Bonferroni correction for multiple tests.

*significant p value at 0.05 level.

As a result of the Kruskal Wallis test, significant differences in Sq and Sa values were observed among the experimental groups (p=0.001) (Table 4.1). As a result of the dual comparisons, it was seen that the Sq and Sa means of the **sandblasting group** was higher than the other 3 groups ($p<0.05$). No significant difference was detected among control, sulfuric acid and laser groups ($p>0.05$).

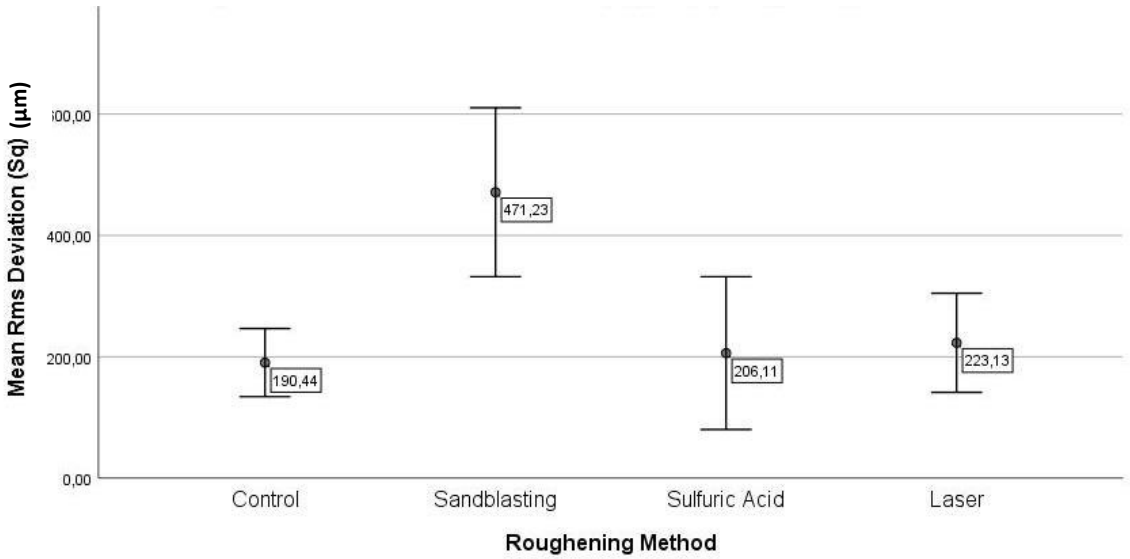


Figure 4.1. Bar graph of variation of RMS deviation (Sq) between different roughening

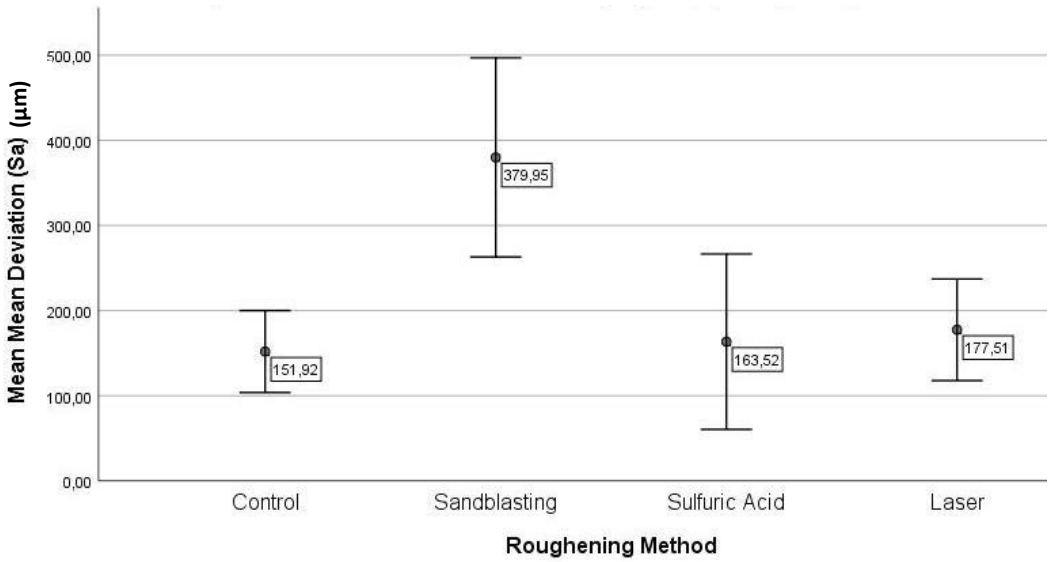


Figure 1.2. Bar graph of variation of mean deviation (Sa) between different roughening methods

4.2. Evaluation AFM images

Examples of three-dimensional Atomic Force Microscope images of PEEK surfaces after the different surface treatments are shown in Figure 4.3. The morphology of PEEK surfaces was affected by the various surface treatments as shown in Figures 4.3a-d.

In the control (no treatment) group, the PEEK surface showed relatively smooth and homogeneous surface, with irregular light striations (Fig 4.3.a). The sandblasted PEEK surface showed rougher surface with many convex precipitates compared with the untreated surface (Fig 4.3.b). The sulfuric acid etched PEEK surface showed uniform pits and pores, revealing rougher surfaces than did the control group (Fig 4.3.c). The laser irradiated PEEK surface showed regular grooves similar to that of the control group but slightly deeper (Fig 4.3.d).

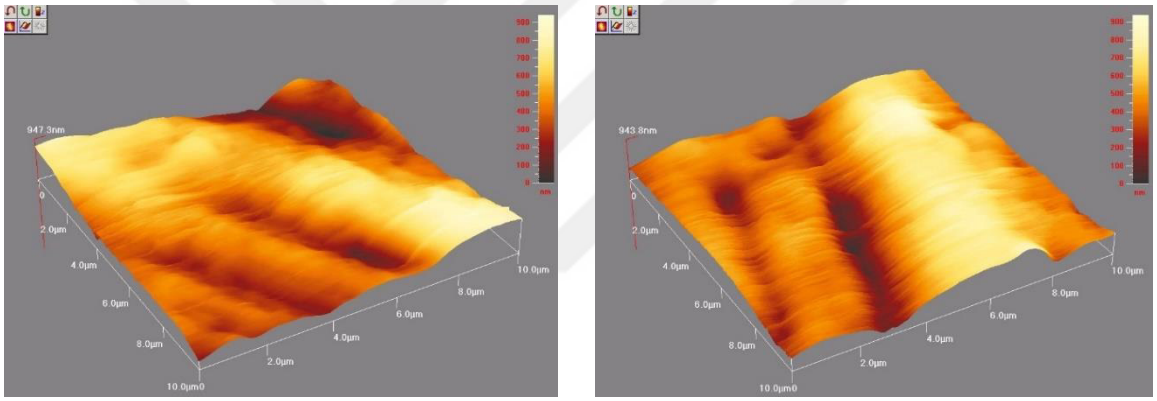


Figure 4.3.a. Two examples of AFM images from **control** group specimens.

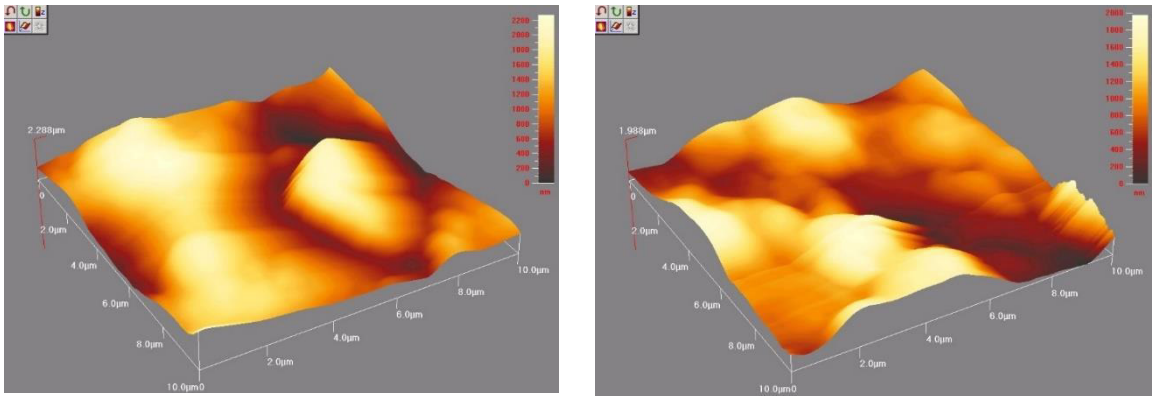


Figure 4.3.b. Two examples of AFM images from **sandblasting** group specimens.

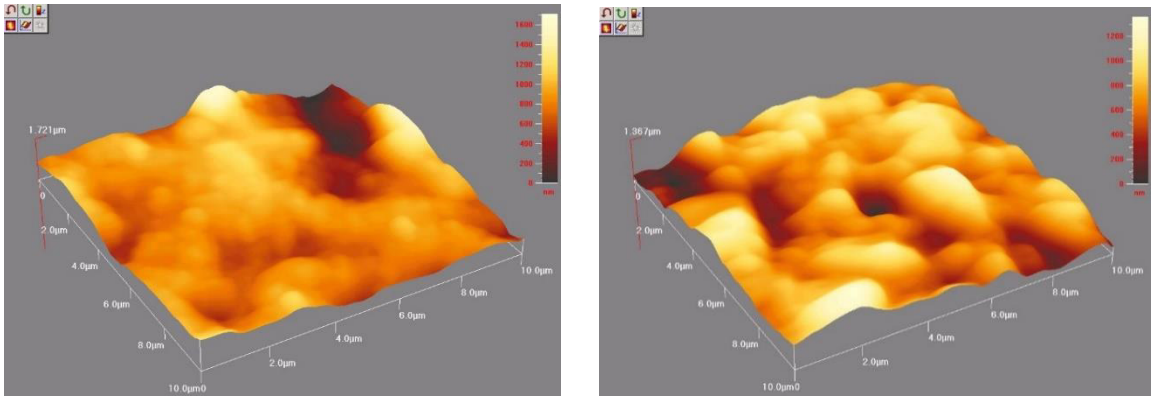


Figure 4.3.c. Two examples of AFM images from **sulfuric acid** group specimens.

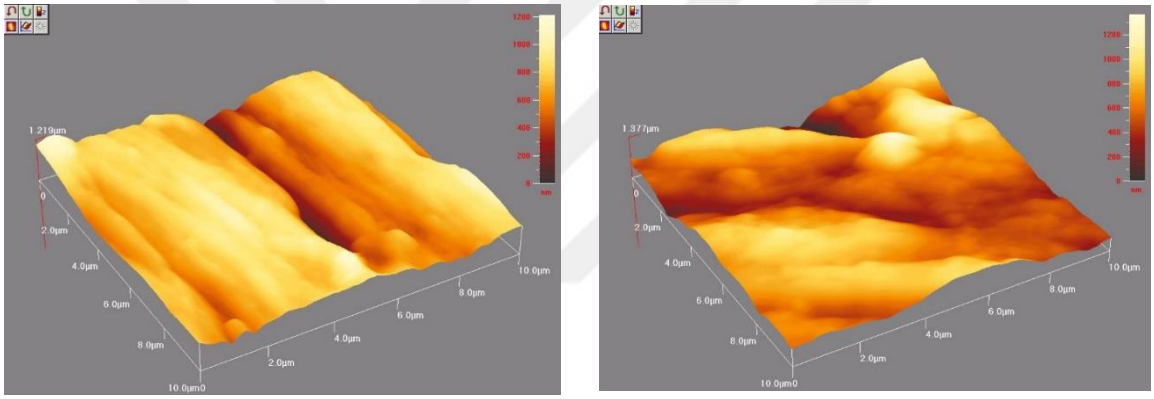


Figure 4.3.d. Two examples of AFM images from **laser** group specimens.

4.3. Results of shear bond strength (SBS) test

Mean SBS values of specimens with different surface treatments and luting cements are shown in Table 4.2 and Figures 4.4 and 4.5. The mean shear bond strengths values (MPa) ranged from 4.014 to 13.346 for the control group; and 1.958 to 25.134 for the sandblasting group; and 2.562 to 14.699 for the acid etching group; and 1.968 to 16.854 for the laser group.

Table 4.2. Means and standard deviations of SBS values (MPa) of specimens with different surface treatments and luting cements.

Surface treatments	TempBond NE + thermocycling (TempBond NE.T)	TempBond NE + no thermocycling (TempBond NE.nT)	RelyX U200 + thermocycling (RelyX. T)	RelyX U200 + no thermocycling (RelyX.nT)
Control	4.014 (±0.558)	6.772 (±0.946)	6.871 (±0.719)	13.346 (±1.948)
Sandblasting	1.958 (±0.345)	3.120 (±0.743)	17.488 (±2.073)	25.134 (±1.665)
Acid etching	2.562 (±0.315)	4.790 (±0.277)	9.405 (±0.837)	14.699 (±1.506)
Laser irradiation	1.968 (±0.544)	3.190 (±3.190)	11.727 (±1.724)	16.854 (±1.517)

For the control groups (untreated); the highest shear bond strength value (13.346 MPa) was obtained with RelyX cement in the samples that did not undergo thermocycling, according to Table 4.2. The lowest SBS value (4.014 MPa) was in the TempBond NE group that underwent thermocycle.

The ranking in terms of SBS value among the control groups was as follows: **RelyX. nT > RelyX. T > TempBond.nT > TempBond.T** (Table 4.2).

For the sandblasting groups; the highest shear bond strength value (25.134 MPa) was obtained with RelyX cement in the samples that did not undergo thermocycling, according to Table 4.2. The lowest SBS value (1.958 MPa) was in the TempBond group that underwent thermocycle.

The ranking in terms of SBS value among the sandblasting groups was as follows: **RelyX. nT > RelyX. T > TempBond.nT > TempBond.T** (Table 4.2).

For the acid etching groups; the highest shear bond strength value (14.699 MPa) was obtained with RelyX cement in the samples that did not undergo thermocycling,

according to Table 4.2. The lowest SBS value (2.562 MPa) was in the TempBond group that underwent thermocycle.

The ranking in terms of SBS value among the acid etching groups was as follows: **RelyX. nT > RelyX. T > TempBond.nT > TempBond.T** (Table 4.2).

For the laser irradiation groups; the highest shear bond strength value (16.854 MPa) was obtained with RelyX cement in the samples that did not undergo thermocycling, according to Table 4.2. The lowest SBS value (1.968 MPa) was in the TempBond group that underwent thermocycle.

The ranking in terms of SBS value among the laser irradiation groups was as follows: **RelyX. nT > RelyX. T > TempBond.nT > TempBond.T** (Table 4.2).

In summary; for all of the surface treatment groups (including control) the ranking in terms of SBS value was as follows:

RelyX. nT > RelyX. T > TempBond.nT > TempBond.T (Table 4.2).

When **TempBond NE cement** (both in groups with and without thermocycle) was used; the highest SBS value belongs to the control group. The order of SBS value in both TempBond NE groups was as follows: **Control > Acid etching > Laser irradiation > Sandblasting** (Table 4.2).

When **RelyX U200 resin cement** (both in groups with and without thermocycle) was used; the highest SBS value belongs to the sandblasting group. The order of SBS value in both RelyX U200 groups is as follows: **Sandblasting > Laser > Acid etching > Control** (Table 4.2).

In summary; as the surface roughness of PEEK increases, the bonding of Gradia luted with the temporary cement decreases; and as the surface roughness of PEEK increases, the bonding of Gradia luted with the resin cement increases.

When TempBond NE cement was used; the highest SBS value (6.77 μm) was in the control group, which did not undergo thermocycling; and the lowest SBS value (1.96 μm) was seen in the thermocycled sandblasting group.

When RelyX U200 resin cement was used; the highest SBS value (25.134 μm) was in the sandblasting group, which did not undergo thermocycling; and the lowest SBS value (6.87 μm) was seen in the thermocycled control group.

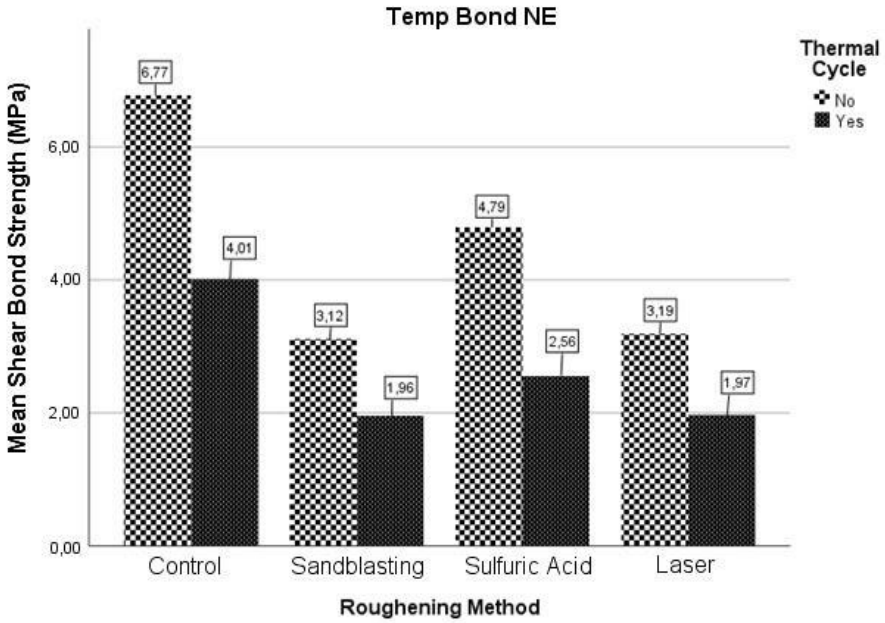


Figure 4.4. Means for shear bond strength (MPa) of groups luted with TempBond NE temporary cement.

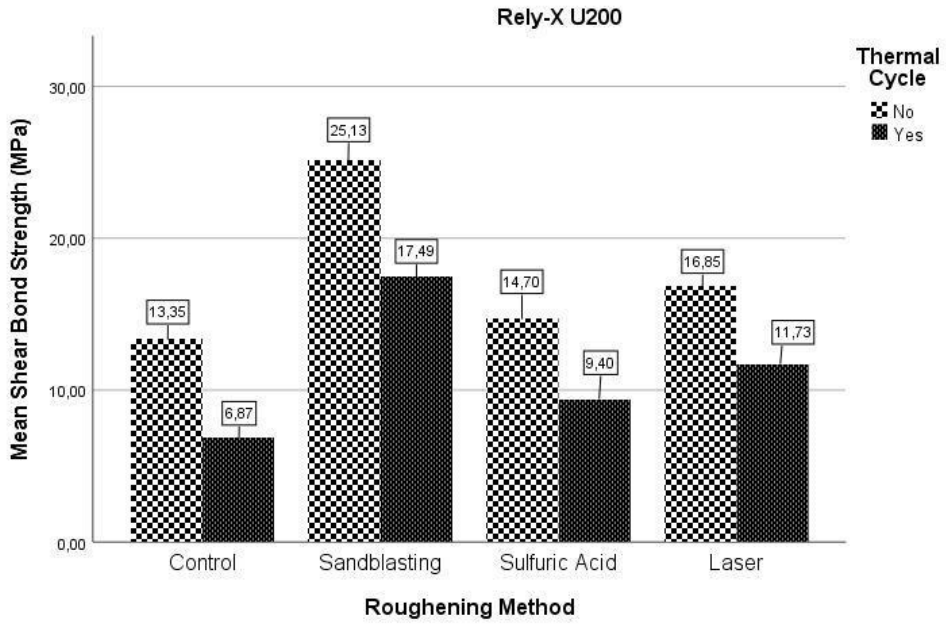


Figure 4.5. Means for shear bond strength (MPa) of groups luted with RelyX U200 resin cement.

4.3.1. Effects of thermal cycle, cement and roughening method factors on shear bond strength

According to results of Table 4.3, there is a significant difference between shear bond strength means of measurements by 4 surface treatment groups (F=167.742, p=0.000). This finding can be interpreted as shear bond strength changes between **roughening methods** without supervise any cement or thermal cycle effect.

Table 4.1. Statistical analyses of between-subjects effects

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	10708.655	15	713.910	510.632	.000*	.972
Intercept	19413.135	1	19413.135	13885.457	.000*	.984
Roughening Method	**703.557**	**3**	**234.519**	**167.742**	**.000***	**.692**
Cement	**7120.405**	**1**	**7120.405**	**5092.947**	**.000***	**.958**
Thermal Cycle	**954.869**	**1**	**954.869**	**682.980**	**.000***	**.753**
Roughening Method * Cement	**1608.654**	**3**	**536.218**	**383.536**	**.000***	**.837**
Roughening Method * Thermal Cycle	**19.233**	**3**	**6.411**	**4.585**	**.004***	**.058**
Cement * Thermal Cycle	**276.479**	**1**	**276.479**	**197.754**	**.000***	**.469**
Roughening Method * Cement * Thermal Cycle	**25.460**	**3**	**8.487**	**6.070**	**.001***	**.075**
Error	313.172	224	1.398			
Total	30434.962	240				
Corrected Total	11021.827	239				

*significant p value at the .05 level.

On the other hand, the difference between shear bond strength means of measurements by cement groups is found significant (F=5092.947, p=0.000). Accordingly, it was found that the shear bond strength values where 2 different materials were used

differed significantly, that is, the effects of being in **different cement groups** (RelyX U200, TempBond NE) on shear bond strength were found to be significant (Table 4.2).

Besides, the difference between shear bond strength means of **thermal cycle groups** is found significant ($F=682.980$, $p<0.05$). Accordingly, it was found that the shear bond strength of samples which applied thermal cycle differed significantly from not applied (Table 4.3).

Also, the difference between shear bond strength means by **roughening method*cement blocks** is found significant ($F=383{,}536$, $p=0.000$). Accordingly, it was found that the shear bond strength of the samples applied with 4 different surface treatments and 2 brands cements differed significantly in general, that is, the common effect of surface treatments and cement factors on bond strength were significant (Table 4.4).

The difference between shear bond strength means by **surface treatment*thermal cycle blocks** is found significant ($F=4.585$, $p<0.05$). Accordingly, it was found that the shear bond strength of the samples applied with 4 different surface treatments and thermal cycling differed significantly in general, that is, the common effect of surface treatments and thermal cycling on shear bond strength were significant (Table 4.5).

The difference between shear bond strength means by **cement*thermal cycle blocks** is found significant ($F=197.754$, $p=0.000$). Accordingly, it was found that the shear bond strength of the samples applied with 2 different cements and thermal cycling differed significantly in general, that is, the common effect of cement brand and thermal cycling on shear bond strength were significant (Table 4.6).

Lastly, it was found that the shear bond strength of samplings which **4 different surface treatment, 2 different cements and thermal cycling** applied showed a significant difference ($F= 6.070$, $p=0.001$), that is, common effect of these 3 factors on shear bond strength levels were significant (Table 4.7).

4.3.1.1. Effect of surface treatments on shear bond strength

Multiple comparisons between surface treatments methods for shear bond strength values are shown in the Table 4.4. The variation between groups is shown in Figure 4.6.

When the roughening methods (surface treatment) were compared, there was no statistically significant difference between the control and acid etching groups also between the laser and acid etching groups ($p>0.05$). Apart from this, significant changes were

observed in all pairwise comparisons ($p<0.05$). Accordingly, ranking from high to low of means of shear bond strength is **Sandblasting > Laser, Acid etching** groups (Table 4.4).

Table 8.4. Comparisons of shear bond strength (MPa) between roughening methods

(I) Roughening Method	(J) Roughening Method	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
					Lower Bound	Upper Bound
Control	Sandblasting	-4.174*	.216	.000	-4.749	-3.600
	Sulfuric Acid	-.113	.216	1.000	-.688	.461
	Laser	-.684*	.216	.010	-1.259	-.110
Sandblasting	Control	4.174*	.216	.000	3.600	4.749
	Sulfuric Acid	4.061*	.216	.000	3.486	4.636
	Laser	3.490*	.216	.000	2.915	4.065
Sulfuric Acid etching	Control	.113	.216	1.000	-.461	.688
	Sandblasting	-4.061*	.216	.000	-4.636	-3.486
	Laser	-.571	.216	.052	-1.146	.004
Laser irradiation	Control	.684*	.216	.010	.110	1.259
	Sandblasting	-3.490*	.216	.000	-4.065	-2.915
	Sulfuric Acid	.571	.216	.052	-.004	1.146

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

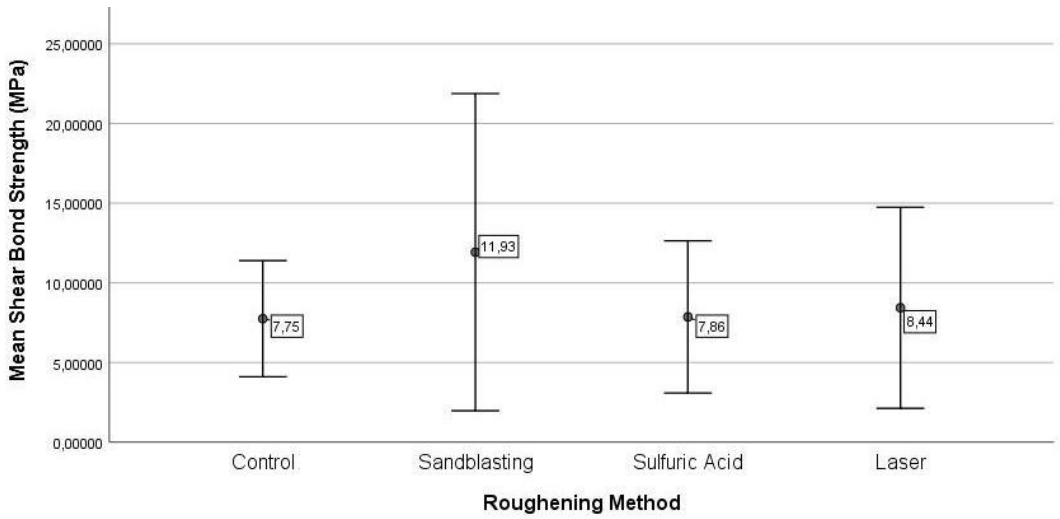


Figure 4.6. Bar graph of the variation of shear bond strength between different roughening methods.

When comparing roughening methods for "**RelyX U200**", there is a statistically significant difference between all methods (p=0.000). Accordingly, ranking from high to low of means of shear bond strength is **Sandblasting > Laser > Sulfuric Acid > Control Group** (Table 4.5).

Table 4.5. Comparisons of shear bond strength (MPa) between roughening methods for each cement.

Cement	(I) Roughening Method	(J) Roughening Method	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
RelyX U200	Control	Sandblasting	-11.202*	.305	.000	-12.015	-10.390
		Sulfuric Acid	-1.944*	.305	.000	-2.756	-1.131
		Laser	-4.182*	.305	.000	-4.995	-3.370
	Sandblasting	Control	11.202*	.305	.000	10.390	12.015
		Sulfuric Acid	9.259*	.305	.000	8.446	10.072
		Laser	7.020*	.305	.000	6.207	7.833
	Sulfuric Acid	Control	1.944*	.305	.000	1.131	2.756
		Sandblasting	-9.259*	.305	.000	-10.072	-8.446
		Laser	-2.239*	.305	.000	-3.051	-1.426
	Laser	Control	4.182*	.305	.000	3.370	4.995
		Sandblasting	-7.020*	.305	.000	-7.833	-6.207
		Sulfuric Acid	2.239*	.305	.000	1.426	3.051
Temp Bond NE	Control	Sandblasting	2.854*	.305	.000	2.041	3.667
		Sulfuric Acid	1.717*	.305	.000	.904	2.530
		Laser	2.814*	.305	.000	2.001	3.626
	Sandblasting	Control	-2.854*	.305	.000	-3.667	-2.041
		Sulfuric Acid	-1.137*	.305	.001	-1.950	-.324
		Laser	-.040	.305	1.000	-.853	.773
	Sulfuric Acid	Control	-1.717*	.305	.000	-2.530	-.904
		Sandblasting	1.137*	.305	.001	.324	1.950
		Laser	1.097*	.305	.002	.284	1.909
	Laser	Control	-2.814*	.305	.000	-3.626	-2.001
		Sandblasting	.040	.305	1.000	-.773	.853
		Sulfuric Acid	-1.097*	.305	.002	-1.909	-.284

Based on estimated marginal means

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

b. Adjustment for multiple comparisons: Bonferroni.

When comparing roughening methods for "**Temp Bond NE**", there is not a statistically significant difference between only sandblasting and laser methods (p>0.05).

Except this, accordingly, ranking from high to low, means of shear bond strength is **Control Group > Sulfuric Acid>Laser, Sandblasting** (Table 4.5).

Table 4.6. Comparisons of shear bond strength (MPa) between thermal cycle groups for each cement

Thermal Cycle	(I) Roughening Method	(J) Roughening Method	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
No	Control	Sandblasting	-4.068*	.305	.000	-4.880	-3.255
		Sulfuric Acid	.315	.305	1.000	-.498	1.127
		Laser	.037	.305	1.000	-.776	.850
	Sandblasting	Control	4.068*	.305	.000	3.255	4.880
		Sulfuric Acid	4.382*	.305	.000	3.570	5.195
		Laser	4.105*	.305	.000	3.292	4.917
	Sulfuric Acid	Control	-.315	.305	1.000	-1.127	.498
		Sandblasting	-4.382*	.305	.000	-5.195	-3.570
		Laser	-.278	.305	1.000	-1.090	.535
	Laser	Control	-.037	.305	1.000	-.850	.776
		Sandblasting	-4.105*	.305	.000	-4.917	-3.292
		Sulfuric Acid	.278	.305	1.000	-.535	1.090
Yes	Control	Sandblasting	-4.281*	.305	.000	-5.094	-3.468
		Sulfuric Acid	-.541	.305	.466	-1.354	.271
		Laser	-1.406*	.305	.000	-2.218	-.593
	Sandblasting	Control	4.281*	.305	.000	3.468	5.094
		Sulfuric Acid	3.740*	.305	.000	2.927	4.552
		Laser	2.875*	.305	.000	2.063	3.688
	Sulfuric Acid	Control	.541	.305	.466	-.271	1.354
		Sandblasting	-3.740*	.305	.000	-4.552	-2.927
		Laser	-.865*	.305	.030	-1.677	-.052
	Laser	Control	1.406*	.305	.000	.593	2.218
		Sandblasting	-2.875*	.305	.000	-3.688	-2.063
		Sulfuric Acid	.865*	.305	.030	.052	1.677

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When the samples **without thermal cycle** are compared according to the roughening method, there was no statistically significant difference between control, sulfuric acid and laser groups ($p>0.05$). But the mean of the sandblasting group is higher than the other 3 groups ($p<0.05$). According to that, ranking from high to low of means of shear bond strength is **Sandblasting > Sulfuric Acid, Laser, Control Group** (Table 4.6).

Thermocycled samples are compared according to the roughening method, there was no statistically significant difference between the control and sulfuric acid groups ($p>0.05$).

Apart from this, significant changes were observed in all pairwise comparisons ($p<0.05$). Accordingly, ranking from high to low of means of shear bond strength is **Sandblasting > Laser > Control, Sulfuric Acid** (Table 4.6).

4.3.1.2. Effect of luting cements on shear bond strength

When cement groups were compared, there was a statistically significant difference between RelyX U200 and Temp Bond NE ($p<0.05$). Accordingly, the mean shear bond strength of the specimens luted with RelyX U200 resin cement was 10.894 MPa higher than the mean of the specimens luted with Temp Bond NE. The variation between groups is shown in Figure 4.7.

Table 4.7. Comparison of shear bond strength (MPa) between cement groups.

(I) Cement	(J) Cement	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
					Lower Bound	Upper Bound
RelyX U200	**Temp Bond NE**	10.894*	.153	.000	10.593	11.195

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

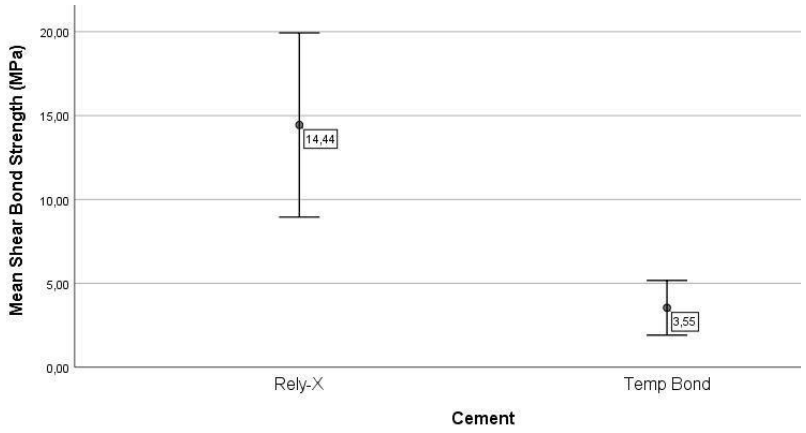


Figure 4.7. Bar graph of the variation of shear bond strength between different cements.

Table 4.8. Comparisons of shear bond strength (MPa) between cement groups for each roughening method groups

Roughening Method	(I) Cement	(J) Cement	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
Control	**RelyX U200**	**Temp Bond NE**	4.715*	.305	.000	4.114	5.317
Sandblasting	**RelyX U200**	**Temp Bond NE**	18.772*	.305	.000	18.170	19.373
Sulfuric Acid	**RelyX U200**	**Temp Bond NE**	8.376*	.305	.000	7.774	8.978
Laser	**RelyX U200**	**Temp Bond NE**	11.712*	.305	.000	11.110	12.313

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When comparing cement groups for **"Control Group",** there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 4.715 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.8).

When comparing cement groups for "**Sandblasting method",** there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 18.772 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.8).

When comparing cement groups for "**Sulfuric Acid method",** there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 8.376 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.8).

When comparing cement groups for "**Laser method",** there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 11.712 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.8).

Table 4.99. Comparisons of shear bond strength (MPa) between cement groups for each thermal cycle situation

Thermal Cycle	(I) Cement	(J) Cement	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
No	**RelyX U200**	**Temp Bond NE**	13.040*	.216	.000	12.615	13.466
Yes	**RelyX U200**	**Temp Bond NE**	8.747*	.216	.000	8.322	9.173

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When the samples without thermal cycle are compared according to the roughening method, there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 13.040 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.9).

When thermal cycle is applied samples are compared according to the roughening method, there was a statistically significant difference between RelyX U200 and Temp Bond NE (p=0.000). Accordingly, the mean shear bond strength of the samples applied with RelyX U200 cement was 8.747 MPa higher than the mean of the samples treated with Temp Bond NE (Table 4.9).

4.3.1.3. Effect of thermocycling on shear bond strength

The effect of thermal cycle on shear bond strength was statistically significant (p=0.000) (Table 4.9). According to this, the mean of the samples applied thermal cycling was 3.989 MPa lower than those of without.

Table 4.10. Comparison of shear bond strength (MPa) between thermal cycle groups

(I) Thermal Cycle	(J) Thermal Cycle	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
					Lower Bound	Upper Bound
No	Yes	3.989*	.153	.000	3.688	4.290

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

Table 4.11. Comparisons of shear bond strength (MPa) between thermal cycle groups for each roughening methods

Roughening Method	(I) Thermal Cycle	(J) Thermal Cycle	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
Control	No	Yes	4.617*	.305	.000	4.016	5.219
Sandblasting	No	Yes	4.404*	.305	.000	3.802	5.006
Sulfuric Acid	No	Yes	3.761*	.305	.000	3.160	4.363
Laser	No	Yes	3.174*	.305	.000	2.573	3.776

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When comparing Thermal Cycle groups for **"Control Group",** the effect of thermal cycle on shear bond strength was statistically significant (p=0.000). According to this, the mean of the samples applied thermal cycling was 4.617 MPa lower than those without (Table 4.11).

When comparing Thermal Cycle groups for "**Sandblasting",** there was a statistically significant difference between samples with and without thermal cycling (p=0.000). According to this, the mean of the samples applied thermal cycling was 4.404 MPa lower than those without (Table 4.11).

When comparing Thermal Cycle groups for "**Sulfuric Acid",** there was a statistically significant difference between samples with and without thermal cycling (p=0.000). According to this, the mean of the samples applied thermal cycling was 3.761 MPa lower than those without (Table 4.11).

When comparing Thermal Cycle groups for "**Laser",** there was a statistically significant difference between samples with and without thermal cycling (p=0.000). According to this, the mean of the samples applied thermal cycling was 3.174 MPa lower than those without (Table 4.11).

Table 4.12. Comparisons of shear bond strength between thermal cycle groups for each cement

Cement	(I) Thermal Cycle	(J) Thermal Cycle	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
						Lower Bound	Upper Bound
RelyX U200	**No**	**Yes**	6.136*	.216	.000	5.711	6.561
Temp Bond NE	**No**	**Yes**	1.843*	.216	.000	1.417	2.268

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When comparing Thermal Cycle groups for "**RelyX U200",** there was a statistically significant difference between samples with and without thermal cycling (p=0.000). According to this, the mean of the samples applied thermal cycling was 6.136 MPa lower than those without (Table 4.12).

When comparing Thermal Cycle groups for "**Temp Bond NE",** there was a statistically significant difference between samples with and without thermal cycling (p=0.000). According to this, the mean of the samples applied thermal cycling was 1.843 MPa lower than those without (Table 4.12).

Table 4.13. Comparisons of shear bond strength by thermal cycle condition for each roughening method and cement group

Roughening Method	Cement	(I) Thermal Cycle	(J) Thermal Cycle	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b]	
							Lower Bound	Upper Bound
Control	Temp Bond NE	No	Yes	2.759*	.432	.000	1.908	3.610
	RelyX U200	No	Yes	6.476*	.432	.000	5.625	7.327
Sandblasting	Temp Bond NE	No	Yes	1.162*	.432	.008	.311	2.013
	RelyX U200	No	Yes	7.646*	.432	.000	6.795	8.497
Sulfuric Acid	Temp Bond NE	No	Yes	2.228*	.432	.000	1.377	3.079
	RelyX U200	No	Yes	5.295*	.432	.000	4.444	6.146
Laser	Temp Bond NE	No	Yes	1.222*	.432	.005	.371	2.073
	RelyX U200	No	Yes	5.127*	.432	.000	4.276	5.978

Based on estimated marginal means
*. The mean difference is significant at the .05 level.
b. Adjustment for multiple comparisons: Bonferroni.

When comparing Thermal Cycle groups for "**Control Group & Temp Bond NE**", there was a statistically significant difference between samples with and without thermal cycling (p=0.000). Accordingly, the thermal cycle application reduces the shear bond strength mean by 2.759 MPa in the samples applied with Temp Bond NE brand cement and no roughening.

When comparing Thermal Cycle groups for "**Control Group & RelyX U200**", there was a statistically significant difference between samples with and without thermal cycling (p=0.000). Accordingly, the thermal cycle application reduces the shear bond strength mean by 6.476 MPa in the samples applied with Temp Bond NE brand cement and no roughening.

When comparing Thermal Cycle groups for "**Sandblasting & Temp Bond NE**", there was a statistically significant difference between samples with and without thermal cycling ($p<0.01$). Accordingly, the thermal cycle application reduces the shear bond strength mean by 1.162 MPa in the sandblasted samples cemented with Temp Bond NE.

When comparing Thermal Cycle groups for "**Sandblasting & RelyX U200**", there was a statistically significant difference between samples with and without thermal cycling ($p=0.000$). Accordingly, the thermal cycle reduces the shear bond strength mean by 7.646 MPa in the sandblasted samples cemented with Temp Bond NE.

When comparing Thermal Cycle groups for "**Sulfuric Acid & Temp Bond NE**", there was a statistically significant difference between samples with and without thermal cycling ($p=0.000$). Accordingly, the thermal cycling reduces the shear bond strength mean by 2.228 MPa in the samples applied with Temp Bond NE cement and etched with sulfuric acid.

When comparing Thermal Cycle groups for "**Sulfuric Acid & RelyX U200**", there was a statistically significant difference between samples with and without thermal cycling ($p=0.000$). Accordingly, the thermal cycling reduces the shear bond strength mean by 5.295 MPa in the samples applied with Temp Bond NE cement and etched with sulfuric acid.

When comparing Thermal Cycle groups for "**Laser & RelyX U200**", there was a statistically significant difference between samples with and without thermal cycling ($p<0.01$). Accordingly, the thermal cycle application reduces the shear bond strength mean by 1.222 MPa in the samples applied with Temp Bond NE cement and etched with sulfuric acid.

When comparing Thermal Cycle groups for "**Laser & RelyX U200**", there was a statistically significant difference between samples with and without thermal cycling ($p=0.000$). Accordingly, the thermal cycle application reduces the shear bond strength mean by 5.127 MPa in the samples applied with Temp Bond NE cement and etched with sulfuric acid.

In summary, thermocycling caused a statistically significant decrease in mean SBS value in all groups ($p<0.05$).

As a result of the statistical analyses, when the etching methods were compared, it was determined that the means of surface roughness values (both Sa and Sq) were higher in the samples where the sandblasting method was applied, compared to the other 3 groups (Control, Sulfuric Acid, Laser).

When the effects of roughening, cement and thermal cycle factors on shear bond strength measurements were examined, it was seen that all main and interaction effects of all these 3 factors were significant on shear bond strength. Therefore, **the null hypothesis that surface treatments, types of luting cement, and thermal cycling would have no effect on the bond strength values between PEEK and indirect composite was rejected.**

When the **main effect of roughening** was examined, it was determined that the samples showing the highest shear bond strength were in the sandblasting group. In addition, it was observed that the samples in the laser group, which had the second highest mean value after sandblasting, showed higher shear bond strength than the control group.

When these methods are analyzed separately according to the groups, the highest shear bond strength was observed in the sandblasting group in the samples using **RelyX U200** cement, followed by the laser, control and sulfuric acid groups in order from most to least (Sandblasting > Laser > Sulfuric Acid > Control Group).

This situation differed in the examples where **TempBond NE** cement was used. While the highest shear bond strength was in the control group, it was determined that the control and sulfuric acid groups had higher values than the sandblasting and laser groups (Control Group > Sulfuric Acid>Laser, Sandblasting).

When the **main effect of cement** factor on shear bond strength was analyzed, it was seen that RelyX U200 resin cement was at a higher bonding level than TempBond NE cement.

The results obtained did not differ when this comparison was made separately in the different roughening groups. In the samples that were not roughened and roughened with 3 different methods, a higher level of shear bond strength was observed in the samples applied with RelyX resin cement compared to TempBond NE.

When these brands are examined separately according to the thermal cycle factor, a higher level of shear bond strength was detected in the RelyX U200 group compared to TempBond NE, both in the samples with and without the thermal cycle.

When the **main effect of the thermal cycle** factor on shear bond strength was analyzed, it was found that the samples without thermocycling had higher shear bond strength than the thermocycled samples. In other words, the application of thermal cycling causes a decrease in shear bond strength. In addition, this decrease was observed in all groups when analyzed separately according to the roughening methods and cements too.

The application of thermal cycling causes a decrease in shear bond strength. In addition, this decrease was observed in all groups when analyzed separately according to the roughening method and cement brand too.

4.4. Failure mode analysis

After shear bond test, debonded surfaces of all specimens were examined under a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at 40x magnification to evaluate the fracture pattern. Similar to the study of Gouveia *et al.* (142), failure modes were classified into one of three categories as follows:

1. **Adhesive failure:** If debonding occurred between PEEK and luting cement or resin cement and Gradia interface, when no cement remnants left on the PEEK surface or Gradia surface.
2. **Mixed failure:** When resin remnants were partially left on the PEEK's surface but also when part of the PEEK surface was exposed.
3. **Cohesive Failure:** When the fracture was occurred in the PEEK or in the Gradia.

Failure mode distributions in all tested groups are shown in Table 4.14. Either adhesive or mixed failure was found in all tested groups (including control). Cohesive failure was not observed (Table 4.14). In other words, the cements were either completely separated or remained partial on the PEEK surface.

More adhesive debonding was observed in specimens luted with **RelyX U200 resin cement** in all surface treatment groups (including control). Similarly, adhesive failure generally occurred in most of the groups luted with **TempBond NE**; however, mixed failure was slightly more common in only sandblasted samples for TempBond NE (Table 4.14).

In the **control** groups (least roughness), luted with TempBond NE, adhesive debonding on the PEEK surface was mostly observed (86.7% (with TC) and 66.6% (no TC)). In the control groups luted with RelyX U200, 100% adhesive failure occurred on the PEEK surface (100% (with TC) and 100% (no TC)).

In the **sandblasting** groups that provided the most roughness, more mixed failure was seen for TempBond NE (100% (with TC) and 73.3% (no TC)), and more adhesive failure for RelyX U200 (100% (100 with TC) and 53.3% (no TC)).

Table 4.14. Distribution of failure modes according to all groups tested.

Surface Treatments	Names of Groups*	Failure Modes				n
		Adhesive		Cohesive	Mixed	
		Adhesive PEEK/Cement	Adhesive Cement/Gradia			
CONTROL	C.Z.T	13 (86.7%)	-	-	2 (13.3%)	15
	C.Z.nT	10 (66.6%)	-	-	5 (33.4%)	15
	C.R.T	15 (100%)	-	-	-	15
	C.R.nT	15 (100%)	-	-	-	15
SANDBLASTING	S.Z.T	-	-	-	15 (100%)	15
	S.Z.nT	4 (26.7%)	-	-	11 (73.3%)	15
	S.R.T	15 (100%)	-	-	-	15
	S.R.nT	7 (46.7%)	-	-	8 (53.3%)	15
SULFURIC ACID ETCHING	A.Z.T	3 (20%)	-	-	12 (80%)	15
	A.Z.nT	2 (13.3%)	-	-	13 (86.7%)	15
	A.R.T	9 (60%)	-	-	6 (40%)	15
	A.R.nT	11 (73.3%)	-	-	4 (26.7%)	15
LASER IRRADIATION	L.Z.T	12 (80%)	-	-	3 (20%)	15
	L.Z.nT	7 (46.7%)	-	-	8 (53.3%)	15
	L.R.T	14 (93.4%)	-	-	1 (6.6%)	15
	L.R.nT	13 (86.7%)	-	-	2 (13.3%)	15
Total		**150 (62.5%)**	**-**	**-**	**90 (37.5%)**	**240**

* Abbreviations of names of all test groups are shown in Table 3.3.

Figure 4.8 and Figure 4.9 show stereomicroscope images of debonded PEEK surfaces of randomly selected specimens from each test group.

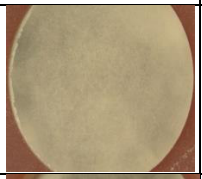
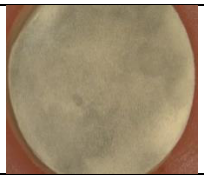
Groups*		Failure modes	
		Adhesive	Mixed
CONTROL	Group 1 C.Z.T		
	Group 2 C.Z.nT		
	Group 3 C.R.T		X
	Group 4 C.R.nT		X
SANDBLASTING	Group 5 S.Z.T	X	
	Group 6 S.Z.nT		
	Group 7 S.R.T		X
	Group 8 S.R.nT		
* Abbreviations of names of all test groups are shown in Table 3.3.			

Figure 4.8. Examples of stereomicroscope images of debonded PEEK surfaces of specimens from control and sandblasting groups.

Groups*		Failure modes	
		Adhesive	Mixed
SULFURIC ACID ETCHING	Group 9 A.Z.T		
	Group 10 A.Z.nT		
	Group 11 A.R.T		
	Group 12 A.R.nT		
LASER IRRADIATION	Group 13 L.Z.T		
	Group 14 L.Z.nT		
	Group 15 L.R.T		
	Group 16 L.R.nT		
* Abbreviations of names of all test groups are shown in Table 3.3.			

Figure 4.9. Examples of stereomicroscope images of debonded PEEK surfaces of specimens from acid etching and laser irradiation groups.

5. DISCUSSION and CONCLUSION

Nowadays aesthetic expectations of patients led dentists seek for new ways to make provisional crowns over implants especially in the anterior region. As titanium abutments are not good enough in color and zirconia abutments have high prices for provisionals, PEEK abutments began to attract attention. PEEK abutments are simple to prepare chairside, and their whitish color makes achieving a nice provisional aesthetic outcome easier (10). Furthermore, research have shown that PEEK implant abutments do not meet the biomechanical requirements to replace the definitive titanium abutment, although it is regarded an alternate material for temporary abutments (10, 105). However, due to its chemical composition and low surface energy, PEEK, an inert substance, has difficulty adhering to composite materials (105). Increasing the surface energy of PEEK and the bond strength with composite materials is a critical issue to be solved.

Different surface modification methods have been tried to overcome this problem. Many researchers recommended the use of sulfuric acid to change the surface properties of PEEK and increase the bond strength with the composite materials (7, 20, 23). In addition, studies have been conducted, in which piranha solution (12, 26), plasma application, sandblasting with Al_2O_3, tribochemical silica coating and various laser systems are used in surface modification of PEEK (12, 24, 26, 94, 98). In the studies, it has been reported that if no surface treatment is applied to the PEEK material, adhesion with the composite material will not be achieved or insufficient bond strength values will be obtained (20). Therefore, current study compared the effect of sulfuric acid etching, sandblasting with Al_2O_3 and Er:YAG laser application of PEEK surfaces on adhesion to composite material cementing with TempBond NE and RelyX U200.

There are many roughening methods on the PEEK surface, among which the most used methods are acid etching and sandblasting. The acids used in roughening the PEEK surface are sulfuric acid, hydrofluoric acid (27), and piranha acid (144). It has been reported that treatment with sulfuric acid supplies better adhesion compared to hydrofluoric acid, argon plasma and silica coating methods (85, 87, 145). Another acid used in PEEK surface modification methods is piranha solution (peroxymonosulfuric acid). The piranha solution consists of a mixture of 98% sulfuric acid (H_2SO_4) and 30% hydrogen peroxide (H_2O_2) in a 10:3 ratio. Piranha solution both increases microroughness and surface polarity by removing organic residues, thus increasing the number of strong functional groups to which the adhesive system can bind. However, since Piranha acid is a substance that is hard to work

with because of its explosive properties and its surface free energy (7), it was not preferred to be included in this study. In addition, Silthampitang *et al.* (22) reported that the bond strength of PEEK samples etched with Piranha acid was lower than that of the samples treated with Sulfuric acid.

Chaijareenont *et al.* (11) investigated the effects of different sulfuric acid concentrations on PEEK surface bonding to resin composite and found that the 90 and 98% sulfuric acid etching significantly achieved the highest shear bond strength. They suggested 90 and 98% sulfuric acid etching is the optimal concentration to improve adhesion between PEEK and the resin composite. In several studies (12, 20, 21, 23, 65, 77, 146) researchers etched PEEK specimens with sulfuric acid for 60 seconds and obtained durable bond strengths. In the current study the PEEK discs in the sulfuric acid etching group were also etched with 98% sulfuric acid for 60 seconds.

The simplest method for increasing surface roughness of PEEK is sandblasting with Al_2O_3. This method provides micro-mechanical adhesion between PEEK and composite resin materials (12, 18, 20, 21, 24, 25, 94, 97). Studies in which sandblasting with Al_2O_3 particles were investigated (16, 18, 20, 24, 25, 31), an increase in the adhesion of the PEEK surface and the bond strength between PEEK and composite was found. Stawarczyk *et al.* (31) roughened PEEK samples by blasting with different grain sizes (50 μm and 110 μm) and investigated the surface roughness and bond strength with composite resins. As a result of the study, they reported that blasting the samples with 110 μm Al_2O_3, compared to 50 μm Al_2O_3 created higher roughness values and lower contact angle on the surface and the bond strength was significantly increased. A lower contact angle value means that the wettability of the material increases, and it becomes hydrophilic (12, 16, 21). Rosentritt *et al.* (18) also reported that the roughness values and bond strength with the composite resin of the samples blasted with 110 μm Al_2O_3 were higher than the PEEK samples roughened by blasting with 50 μm Al_2O_3. Therefore, in the current study surface of the PEEK discs in the sandblasting group were also treated with 110 μm Al_2O_3 particles.

Laser irradiation is also used to improve PEEK surface roughness. Studies which examine the surface roughness of PEEK treated with laser irradiation reported that laser-irradiated PEEK surfaces show increased surface roughness compared to the untreated surfaces (13-15, 30, 65, 96, 105-109). Investigators mostly used Er:YAG and Nd:YAG lasers to improve the surface characteristics in the studies. Caglar *et al.* (14) compared the effect of sandblasting, silica coating and Er:YAG laser on the surface roughness of PEEK discs and reported that laser treated groups have increased surface roughness values than the

control group, but the difference was not statistically significant. Ates *et al.* (15) in 2018 compared Er:YAG laser and combination of Er:YAG laser with sandblasting and combination of Er:YAG laser with silica coating in terms of the effect of surface roughness of PEEK samples. They stated that pretreatment of PEEK surface with only Er:YAG laser is not effective for bonding, and they found significant differences in surface roughness values between control and combinations of laser groups. In both studies the investigators used 2.94 nm wavelength, 150mj power setting and an average power output of 1,5W. Ulgey *et al.* (96) compared the effects of Er:YAG, Nd:YAG and KTP lasers on the PEEK surfaces with the power settings of 3W. They reported that different laser types cause different topographies on the PEEK surfaces and by changing power values of lasers provide meaningful variation on the surfaces of the studied samples. In the current study, Er:YAG laser was used with the same parameters as Caglar *et al.* (14) and Ates *et al.* (15) to treat the surfaces of the PEEK samples in laser irradiation group.

To investigate the changes on the surface topography, roughness, and pattern of PEEK samples after surface treatments, researchers used Atomic Force Microscope (AFM), Scanning Electron Microscopy (SEM) and profilometer. Nayyer *et al.* (147) used both AFM and profilometer to assess the surface properties of various composites and compomer materials. They concluded that AFM showed higher precision compared to optical profilometer at a nanoscale level but both methods can be used in combination to perform a more precise roughness assessment. Porojan *et al.* (124) in 2020 evaluated the surface quality of thermoplastic dental appliances with profilometer, AFM and SEM. They concluded that roughness values obtained from profilometer allow a quantitative measure of the surface irregularities whereas parameters measured by AFM are more exact with the 0.01 μm tip, 3D in nature and more realistic (124).

Chaijareenont *et al.* (11) examined the surface topographies of PEEK samples etched with different concentration of sulfuric acid both with SEM and AFM. They reported that both SEM and AFM demonstrated a tendency of increased surface roughness and irregularities on the PEEK samples. Luo *et al.* (140) in 2001 investigated the surface of acid etched IPS Empress specimens both with AFM and SEM. They concluded that SEM provides a clear visual representation of the changes on the surface, while AFM supplies the numerical data to quantify those changes. In a study designed by Da Silva *et al.* (126) it was found that, since SEM does not observe 3D surface texture, it has limits in terms of surface topography description. AFM may also be employed for measurements, 3D assessments, and more comprehensive surface topography descriptions (126). The primary distinction

between AFM and profilometer is that AFM examines a specific area of the surface to determine surface properties, whereas profilometer uses a single line to determine surface characteristics. Scanning numerous lines in a row would provide a surface representation similar to that produced by AFM. This approach, however, would be time consuming and difficult to implement. Furthermore, the invasive nature of the profilometer may cause damage to the scanned surface and mislead the findings (125). As AFM gives numerical data and three-dimensional images of surface topographies, in the current study the surface topography and roughness values of the treated PEEK samples were examined with AFM.

PEEK material is mainly used as a temporary abutment in implant dentistry with fabrication of a provisional restoration. The potential and the limitation of bonding of PEEK to dental materials is the main concern of the studies about PEEK. The cements used and the bond strength between PEEK and the restoration material is an issue needs to be taken care of. Therefore, possible techniques for bonding PEEK to composite resin materials were assessed (7). Some researchers investigated the bond strength between PEEK and veneering resins, and some examined the bond strength between PEEK and the luting agents. Several studies tested the adhesion between PEEK and self-adhesive resin cement, used RelyX Unicem for luting (7, 12, 27, 94). Tsuka *et al.* (148) in 2017 and Kimura *et al.* (106) in 2022 bonded RelyX Ultimate resin cements on the PEEK samples. Uhrenbacher *et al.* (26) in 2014 used RelyX Unicem and Rocha *et al.* (149) in 2016 used RelyX ARC for luting procedure of PEEK specimens to human dentin surfaces. Previous studies (12, 16, 18, 20, 24) found that the bonding success between PEEK and resin is related to the content and solvents of the adhesive system. Adhesives that contain methyl-methacrylate monomers cause higher bond strength between PEEK and resin. For this reason, RelyX U200 was chosen as the adhesive cement in the current study as it has methacrylate monomers in its composition.

Some researchers (150-159) suggested the use of provisional cements to retain retrievability based on the assumption that provisional cements are less retentive than permanent cements. Therefore, Temp Bond NE is also used in the current study to compare the shear bond strength of a resin cement and a provisional cement, assuming that RelyX U200 will provide a better adhesion than TempBond NE.

Thermocycling is a method to simulate oral conditions for *in-vitro* aging of specimens prior to test bonding properties. This is a standardized and reproducible method for all tested specimens. Schimidlin *et al.* (7) reported that the lack of artificial aging by thermocycling or long-term water storage is a limitation of their study. In a study performed by Uhrenbaucher et al (26) PEEK samples were aged by thermal cycling for 5000 thermal cycles, and it was

concluded that thermal cycling plays an important role in long-term predictability. In another study, Gale *et al.* (160) stated that an artificial reference of 10000 thermal cycles indicates one service year. Since the current study is designed to use PEEK as a temporary abutment material the number of thermal cycles is considered according to pretend the 6-month period. Thus, to evaluate the effects of thermocycling process, half of all cemented specimens subjected to thermocycling in the current study as in Uhrenbaucher's (26) study.

Various bond strength tests have been used in studies investigating the bond strength between PEEK and different dental materials. Some researchers performed tensile bond strength test (12, 24, 25, 94, 154, 161), compression test (162), pull-out test (26, 163) whereas some used shear bond strength test (7, 15, 28, 29, 93, 108, 109, 113, 148, 149, 164, 165). Shear bond strength tests are commonly used by researchers because they are relatively simple compared to tensile bond strength tests. However, it is difficult to align the specimen in the testing machine without creating detrimental stress distribution while using the shear bond strength test (166, 167). Zhou *et al.* (27) reported that shear bond strength tests are more appropriate for evaluating adhesive capabilities of luting cements to composites. Advantages in shear tests include ease of specimen preparation and simple test protocol (168). In the current study the bond strength of PEEK specimens to RelyX U200 and TempBond NE is calculated with shear bond strength test because it can easily and rapidly reflect the clinical situation (27).

Shear bond test results are not the only criteria for the examination of the adhesion success. The type of failure occurred on the adhesion interface should also be evaluated. To examine the failure modes, researchers used optical microscopy (105), light microscopy (25, 164), SEM evaluations (20, 24, 93) and stereomicroscopy (7, 11, 22, 65, 148, 149). In the current study the surfaces of all PEEK specimens were examined under a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at 40x magnification to evaluate the fracture pattern. The failure modes were classified into one of three categories similar to the research of Gouveia *et al.* (142) in 2021.

According to the results of the current study, after surface treatments, sandblasted PEEK surfaces (Sq:471.23 μm and Sa: 379.95 μm) showed statistically higher surface roughness values than laser treated (Sq: 223.13 μm and Sa: 177.51 μm), the acid etched (Sq: 206.11 μm and Sa: 163.52 μm), and control groups (Sq: 190.44 μm and Sa: 151.92 μm). Researchers examining the bond strength of PEEK, mostly made pretreatments on the PEEK samples and investigated the surface topography and roughness. Schimidlin (7) and Silthampitang (22) investigated the surface of the samples with SEM and described the

effects of etching with %98 sulphuric acid and sandblasting. Schimidlin *et al.* (7) stated that the sulfuric acid etched PEEK resulted in a complex fiber network, while 50 μm sandblasting led to an irregular surface on PEEK but showed a more accentuated and dispersed surface pattern as compared to the 110μm pre-treatment according to SEM results. Silthampitang (22) stated that the samples treated by 98% sulfuric acid were characterized by a sponge-like porous fiber network and subsurface corrosion. The sandblasting group exhibited irregular, fissured surfaces with polygonal shaped alumina oxide embedded in PEEK surface observed with SEM. Parkar *et al.* (6) examined the treated PEEK surfaces with profilometer and found that the acid etched samples showed rougher surfaces than the sandblasted samples. Stawarczyk *et al.* (20) also evaluated the surface roughness values with profilometer and found that the sandblasted samples have higher roughness values than the acid etched and no pretreatment samples. Çulhaoğlu *et al.* (65) and Kimura *et al.* (106) also used profilometer for the measurement of the surface roughness values and both found that the laser treated groups have the highest surface roughness values continued with sandblasted, untreated, and acid etched samples respectively. Caglar *et al.* (14) used SEM for the topographical surface evaluations and profilometer for the measurements of the average roughness height values. They found that sandblasted surfaces have higher surface roughness values than the laser treated and untreated surfaces as in the current study. The surface roughness results of the current study are not consistent with the results of the studies designed by some of the researchers mentioned above. The reason for the disparities in the surface roughness results may be attributed to the differences in the study design, such as variation of laser type, changes in particle size of Al_2O_3 and the difference in the surface roughness assessment method used in the current study.

Studies showed that surface roughness of the materials influence the contact angle and the shear bond strength. The irregularities in the surface increases the mechanical retention and the bond strength by increasing the contact area compared to the smoother surfaces (169). According to the results of the current study, there is a direct correlation between the roughening rate and shear bond strength for RelyX U200 resin cement. As the surface roughness of PEEK increases, the bonding of Gradia luted with the resin cement increases. A study in which the researchers veneered composite to PEEK samples (20) found that the specimens air-abraded with coarser alumina particles yield higher roughness values consequently higher shear bond strength values. On contrary, they stated that the surface roughness of acid etched samples yield lower surface roughness values than the air abraded specimens but remarkably high contact angles which results in better wetting of the surface

and higher shear bond strength values than the air abraded specimens (20). Similar to the study of Stawarczyk *et al.* (20), in the study of Çulhaoğlu *et al.*, in which PEEK specimens were coated with Visio.link adhesive and veneered with a composite resin, and in another study designed by Kimura *et al.*, in which Visio.link was applied as the adhesive and 2 different resin cements were applied to the PEEK specimens, it was revealed that the highest shear bond strength in acid etched samples was seen in samples with the least surface roughness. They stated that this conflict is caused by the chemical alterations on the PEEK surface in consequence of acid etching (65, 106). In the current study, the highest surface roughness and shear bond strength values are measured in the sandblasted RelyX U200 groups. For the specimens cemented with RelyX U200, shear bond strength values decrease proportional to the decrease in the surface roughness values. The difference between the results of the current study and those of Stawarczyk (20), Kimura (106), and Çulhaoğlu (65) may be due to the difference in study designs. However, the current study indicated that the shear bond strength values of composite discs cemented with TempBond NE to PEEK specimens is lower than those cemented with RelyX U200 in all groups. This decrease in the TempBond NE groups may be attributed to the difference in the bonding properties as TempBond NE is a temporary luting cement. There was an inverse relationship between the surface roughness values and shear bond strength values in the samples cemented with TempBond NE in the current study. In other words, contrary to the expectations, the shear bond strength values of TempBond NE groups decreased while the surface roughness values increased. For samples cemented with Tempbond NE, maximum surface roughness values were measured on sandblasted PEEK specimens, while minimum shear bond strengths were calculated on sandblasted PEEK specimens. The possible reason for these results may be because of the high viscosity features of TempBond NE cement compared to RelyX U200 prevent the filling of the pits and micropores over the PEEK specimens with cement. Hence, it can be considered that penetration and micromechanical interlocking of cements through pits and pores is an important factor to achieve adhesion between PEEK and cements.

Researchers investigating the bond strength of pretreated PEEK specimens stated that the lack of artificial aging by thermocycling is a limitation of their studies (7, 20). The effect of thermocycling on the shear bond strength of resin cement to pretreated PEEK surfaces plays an important role in long term predictability (12, 14, 26). In the current study, half of both RelyX U200 and TempBond NE groups were underwent 5000 thermal cycles resulted in decreased shear bond strength values. This finding is similar to the findings of the studies designed by Kimura *et al.* (106), Bunz *et al.* (164) and Mayinger *et al.* (170) in which the

bond strength of the thermocycled samples was compared with that of the non-thermocycled samples. Thermal cycling may influence the bond strength. The reason in the decrease of the shear bond strength values after thermal cycling is thought to be caused because of the moisture, mechanical stress, and volume changes in the bonded areas by the heat changes (106).

According to Ateş *et al.*'s study (15) where they treated the PEEK surface with airborne particle abrasion, silica coating, Er:YAG laser etching, Er:YAG etching + airborne particle abrasion and Er:YAG etching + silica coating; the predominant failure type was adhesive. Only silica coated, Er:YAG etched + airborne particle abraded and Er:YAG etched + silica coated groups showed mixed failures. They observed no cohesive failures in tested specimens consistent with our study (15). In our study, control groups which has the least roughness; more adhesive failure and less mixed failure were observed in samples luted with TempBond NE. Namely, TempBond NE is usually not left on the PEEK surface at all. RelyX U200 resin cement groups, which are assumed to provide more bonding, showed 100% adhesive failure. This is probably because Gradia indirect composite has a similar structure to resin cement, and therefore resin cement bonds more strongly to Gradia than to PEEK.

In the sandblasting groups that provide the most roughness; TempBond NE groups have very little adhesive failure and more mixed failure; and adhesive failure is more common in RelyX U200 resin cement groups. Although TempBond NE seemed to adhere more to the PEEK surface, the shear bond strength value at debonding was lower than that of RelyX U200.

Based on the results of this study, if a temporary fixed prosthesis is to be cemented over PEEK implant abutments with TempBond NE cement, no surface treatment may be required as the shear bond strength is higher for untreated PEEK surfaces than for the surface treated groups. However, by conducting further studies, it can be investigated whether different modifications of the surface treatments used in our study or other types of treatments provide more bond strength than untreated surfaces.

If a temporary prosthesis is desired to be used in the mouth for a longer period of time, sandblasting the PEEK surface with 110 μm Al_2O_3 before the prosthetic restoration is cemented with RelyX U200 may be recommended.

Finally, it should be noted that this study has some limitations. These limitations are the *in vitro* nature of the study and the fact that the shear bond test does not fully represent

the mode of load application in a clinical situation. Although this study could not replicate all individual variations of the intraoral conditions, it may help in determining reliable bond formation between PEEK and indirect composite materials luted on PEEK with dental cements.

Within the limitations of this *in vitro* study, the conclusions can be summarized as follows:

1. Sandblasting (with 110 μm Al_2O_3 particles) resulted in the highest **surface roughness** values among the surface treatment methods applied to PEEK (p=0.000).
2. When comparing the surface roughness values for specimens from all test groups, the ranking of both surface roughness (Sa and Sq) values from high to low was: **sandblasting > laser irradiation, acid etching, control.**
3. Although acid etching and laser irradiation methods cause an increase in surface roughness of the PEEK compared to the control group, this increase is not statistically significant (p>0.05).
4. The specimens showing the highest **shear bond strength** value (6.871-25.134 MPa) in all surface treatment groups were the specimens that sandblasted, luted with RelyX U200 and non-thermocycled samples.
5. When TempBond NE cement (both in groups with and without thermocycle) was used; the highest shear bond strength value belongs to the control group. The ranking of SBS value in both TempBond NE groups is as follows: **Control > Acid etching > Laser irradiation > Sandblasting.**
6. When TempBond NE temporary cement was applied, the surface treatment methods did not increase the shear bond strength between PEEK and Gradia compared to control group.
7. When RelyX U200 resin cement (both in groups with and without thermocycle) was used; the highest shear bond strength value belongs to the sandblasting group. The ranking of SBS value in both RelyX U200 groups is as follows: **Sandblasting > Laser irradiation> Acid etching > Control.**
8. When RelyX U200 resin cement was applied, the surface treatment methods caused an increase in shear bond strength between PEEK and Gradia compared to control group.
9. There is a direct correlation with the roughening rate for the RelyX U200 resin cement, namely, as the surface roughness of PEEK increases, the bonding of Gradia luted with

the resin cement increases. However, for TempBond NE, there is an inverse relationship between the roughening rate and bond strength; that is, as the surface roughness of PEEK increases, the bonding of Gradia luted with the temporary cement decreases. Therefore, it can be concluded that roughness is not the only factor affecting bond strength.

10. For all surface treatment groups (including the untreated control group), a higher level of shear bond strength was observed in the samples luted with RelyXU200 resin cement compared to TempBond NE (p=0.000).
11. Thermocycling caused a decrease in shear bond strength value between PEEK and Gradia in all cement groups. Thermocycled groups showed significantly lower SBS values compared to non-thermocycled groups (p=0.000).
12. Either adhesive or mixed failure was observed in all tested groups (including control). The most frequent **failure mode** was adhesive failure. Cohesive failure was not observed. In other words, the cements were either completely separated or remained partial on the PEEK surface.
13. In control groups with the least roughness; more adhesive failure and less mixed failure were observed in samples luted with TempBond NE; and resin cement groups, which provide higher bonding value, showed 100% adhesive failure. This is probably due to the structural similarity between resin cement and thus the stronger adhesion of the resin cement to Gradia than PEEK.
14. In the sandblasting groups that provide the most roughness; TempBond NE groups have very little adhesive failure and more mixed failure; but adhesive failure is more common in RelyX U200 resin cement groups. Although TempBond NE seemed to adhere more to the PEEK surface, the shear bond strength value at debonding was lower than RelyX U200.
15. In order to increase the bonding strength between PEEK and composites, further studies can also be carried out using different types of luting cements, different concentration of sulfuric acid, different particle size of Al_2O_3, different types of dental lasers, combinations of surface treatment methods.

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7. CURRICULUM VITAE

Personal Information

Name	Zeynep Çiğdem	**Surname**	Kılınç

Education

Degree	Alan	The name of the Institution Graduated From	Graduation Year
PhD	Prosthodontics	Yeditepe University	2022
University	Yeditepe University	Faculty of Dentistry	2011
High School	FMV Özel Ayazağa Işık Lisesi	Scientific Section	2005

Language	Yabancı Dil Sınav Notu (#)
Turkish	Native Proficiency
English	Toefl 82

Computer Skills	Level
Microsoft Office: Word, Power Point, Excel	Good